UNIVERSIDAD MIGUEL HERNÁNDEZ DE ELCHE
ESCUELA POLITÉCNICA SUPERIOR DE ORIHUELA
Programa de Doctorado en Recursos y Tecnologías Agrarias, Agroambientales y Alimentarias

Valorization of Fruits and Leaves of *Ficus carica* L.

DOCTORAL THESIS

2025

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Director: Francisca Hernández García
Codirector: Marina Cano Lamadrid

Volverás a mi huerto y a mi higuera:
por los altos andamios de las flores
pajareará tu alma colmenera
de angelicales ceras y labores.

Miguel Hernández

The present Doctoral Thesis, titled "Valorization of the fruits and leaves of *Ficus carica* L." is presented in the format of a thesis by compendium, comprising the following seven publications:

- Teruel-Andreu, C., Andreu-Coll, L., López-Lluch, D., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2021). *Ficus carica* fruits, by-products and based products as potential sources of bioactive compounds: A review. *Agronomy, 11*(9). doi:http://doi.org/10.3390/agronomy11091834

- Teruel-Andreu, C., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2023). How does cultivar affect sugar profile, crude fiber, macro- and micronutrients, total phenolic content, and antioxidant activity on *Ficus carica* leaves? *Agronomy, 13*(1). doi:https://doi.org/10.3390/agronomy13010030

- Teruel-Andreu, C., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2023). Nutritional and functional compounds and antioxidant activity of edible and non-edible fruit part of brebas and figs (*Ficus carica* L.) among different varieties. *Scientia Horticulturae, 318, 112069*. doi: http://doi.org/10.1016/j.scienta.2023.112069

- Teruel-Andreu, C., Issa-Issa, H., Noguera-Artiaga, L., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2024). Volatile profile of breba and fig fruits (peel and pulp) from different *Ficus carica* L. varieties. *Scientia Horticulturae, 328*, 112892. doi:https://doi.org/10.1016/j.scienta.2024.112892

- Teruel-Andreu, C., Jiménez-Redondo, N., Muelas, R., Carbonell-Pedro, A. A., Hernández, F., Sendra, E., & Cano-Lamadrid, M. (2024). Techno-functional properties and enhanced consumer acceptance of whipped fermented milk with *Ficus carica* L. By-products. *Food Research International, 195*. doi: https://doi.org/10.1016/j.foodres.2024.114959

- Teruel-Andreu, C., Jiménez-Redondo, N., Muelas, R., Almansa, A., Hernández, F., Cano-Lamadrid, M., & Sendra, E. (2024). Flavonoids, microbial load and quality parameters changes during shelf-life of fermented milk enriched with pasteurized fig purée. *LWT, 211*. doi: https://doi.org/10.1016/j.lwt.2024.116918

- Teruel-Andreu, C., Cano-Lamadrid, M., Hernández, F., & Wojdyło, A. (2025). Bioactive compounds (LC-PDA-Qtof-ESI-MS and UPLC-PDA-FL) and in vitro inhibit α-amylase and α-glucosidase in leaves and fruit from different varieties of *Ficus carica* L. *Food Chemistry*, 465. doi: https://doi.org/10.1016/j.foodchem.2024.141977

Publication 1			

Teruel-Andreu, C., Andreu-Coll, L., López-Lluch, D., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2021). *Ficus carica* fruits, by-products and based products as potential sources of bioactive compounds: A review. ***Agronomy***, 11(9). doi:http://doi.org/10.3390/agronomy11091834

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Publication 3			

Teruel-Andreu, C., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2023). Nutritional and functional compounds and antioxidant activity of edible and non-edible fruit part of brebas and figs (*Ficus carica* L.) among different varieties. *Scientia Horticulturae*, 318, 112069. doi: http://doi.org/10.1016/j.scienta.2023.112069

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Publication 4			
Teruel-Andreu, C., Issa-Issa, H., Noguera-Artiaga, L., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2024). Volatile profile of breba and fig fruits (peel and pulp) from different *Ficus carica* L. varieties. Scientia Horticulturae, 328, 112892. doi:https://doi.org/10.1016/j.scienta.2024.112892			
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JCR® category		Horticulture	
Quartile 2023	Q1	**Rank**	6/38
Impact factor	3.9 (2023)	**5-year impact factor**	4.4

Publication 5			
Teruel-Andreu, C., Jiménez-Redondo, N., Muelas, R., Carbonell-Pedro, A. A., Hernández, F., Sendra, E., & Cano-Lamadrid, M. (2024). Techno-functional properties and enhanced consumer acceptance of whipped fermented milk with *Ficus carica* L. By-products. ***Food Research International***, 195. doi: https://doi.org/10.1016/j.foodres.2024.114959			
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Publication 6			
Teruel-Andreu, C., Jiménez-Redondo, N., Muelas, R., Almansa, A., Hernández, F., Cano-Lamadrid, M., & Sendra, E. (2024). Flavonoids, microbial load and quality parameters changes during shelf-life of fermented milk enriched with pasteurized fig purée. ***LWT***, 211. doi: https://doi.org/10.1016/j.lwt.2024.116918			
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JCR® category		Food Science and Technology	
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Impact factor	6.0 (2023)	**5-year impact factor**	6.0

Publication 7			
Teruel-Andreu, C., Cano-Lamadrid, M., Hernández, F., & Wojdyło, A. (2025). Bioactive compounds (LC-PDA-Qtof-ESI-MS and UPLC-PDA-FL) and in vitro inhibit α-amylase and α-glucosidase in leaves and fruit from different varieties of *Ficus carica* L. Food Chemistry, 465. doi: https://doi.org/10.1016/j.foodchem.2024.141977			
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Impact factor	8.5 (2023)	**5-year impact factor**	8.3

La Dra. Dña. Francisca Hernández García directora, y la Dra. Dña. Marina Cano Lamadrid, codirectora de la tesis doctoral titulada "Valorization of the fruits and leaves of *Ficus carica* L."

INFORMAN:

Que Dña. Candela Teruel Andreu ha realizado bajo nuestra supervisión el trabajo titulado "Valorization of the fruits and leaves of *Ficus carica* L." conforme a los términos y condiciones definidos en su Plan de Investigación y de acuerdo al Código de Buenas Prácticas de la Universidad Miguel Hernández de Elche, cumpliendo los objetivos previstos de forma satisfactoria para su defensa pública como tesis doctoral.

Lo que firmamos para los efectos oportunos, en Orihuela a 15 de abril de 2025.

Directora de la tesis	Codirectora de la tesis
Dra. Dña. Francisca Hernández García	Dra. Dña. Marina Cano Lamadrid

Dr. Dña. Juana Fernández López, Catedrática de Universidad y Coordinadora del Programa de Doctorado en Recursos y Tecnologías Agrarias, Agroambientales y Alimentarias (ReTos-AAA) de la Universidad Miguel Hernández de Elche (UMH),

CERTIFICA:

Que la Tesis Doctoral titulada **"Valorization of the fruits and leaves of *Ficus carica* L."** de la que es autora la graduada en máster universitario en tecnología y calidad agroalimentaria **Dña. Candela Teruel Andreu**, ha sido realizada bajo la dirección de la **Dra. Francisca Hernández García** y la codirección de la **Dra. Marina Cano Lamadrid**, actuando como tutora de la misma la Dra. Esther Sendra Nadal. Considero que la Tesis es conforme, en cuanto a forma y contenido, a los requerimientos del Programa de Doctorado ReTos-AAA, siendo por tanto apta para su exposición y defensa pública.

Y para que conste a los efectos oportunos firmo el presente certificado en Orihuela a 15 de abril de 2025.

Dra. Dña. Juana Fernández López
Coordinadora del Programa Doctorado ReTos-AAA

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A todos, gracias por estar siempre presentes.

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To all of you, thank you for always being there.

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DOCTORAL THESIS STRUCTURE

This Doctoral Thesis has been structured in accordance with the current internal regulation of the Miguel Hernández University of Elche using the opting for the Presentation of Doctoral Thesis by Compendium of Publications. Therefore, the structure includes:

• **Abstract/*Resumen*.** The most relevant results and conclusions are presented in this section (English and Spanish).

• **Introduction.** This section contains a brief bibliographic review of the botanical origin, taxonomy and economic importance of *Ficus carica* L.

• **Objectives.** The main objective and specific goals are presented in this part.

• **Material and Methods**. This part contains a description of plant material, methodology and statistical analyses used to reach the objectives of this research.

• **Publications:** The seven publications used to develop this Doctoral Thesis are listed below

Publications:

1- Teruel-Andreu, C., Andreu-Coll, L., López-Lluch, D., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2021). *Ficus carica* fruits, by-products and based products as potential sources of bioactive compounds: A review. ***Agronomy***, 11(9). doi:http://doi.org/10.3390/agronomy11091834

2- Teruel-Andreu, C., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2023). How does cultivar affect sugar profile, crude fiber, macro- and micronutrients, total phenolic content, and antioxidant activity on *Ficus carica* leaves? ***Agronomy***, 13(1). doi:https://doi.org/10.3390/agronomy13010030

3- Teruel-Andreu, C., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2023). Nutritional and functional compounds and antioxidant activity of edible and non-edible fruit part of brebas and figs (*Ficus carica* L.) among different varieties. ***Scientia Horticulturae***, 318, 112069. doi: http://doi.org/10.1016/j.scienta.2023.112069

4- Teruel-Andreu, C., Issa-Issa, H., Noguera-Artiaga, L., Sendra, E., Hernández, F., & Cano-Lamadrid, M. (2024). Volatile profile of breba and fig fruits (peel and pulp) from different *Ficus carica* L. varieties. ***Scientia Horticulturae***, 328, 112892. doi:https://doi.org/10.1016/j.scienta.2024.112892

5- Teruel-Andreu, C., Jiménez-Redondo, N., Muelas, R., Carbonell-Pedro, A. A., Hernández, F., Sendra, E., & Cano-Lamadrid, M. (2024). Techno-functional properties and enhanced consumer acceptance of whipped fermented milk with *Ficus carica* L. By-products. ***Food Research International***, 195. doi: https://doi.org/10.1016/j.foodres.2024.114959

6- Teruel-Andreu, C., Jiménez-Redondo, N., Muelas, R., Almansa, A., Hernández, F., Cano-Lamadrid, M., & Sendra, E. (2024). Flavonoids, microbial load and quality parameters changes during shelf-life of fermented milk enriched with pasteurized fig purée. ***LWT***, 211. doi: https://doi.org/10.1016/j.lwt.2024.116918

7- Teruel-Andreu, C., Cano-Lamadrid, M., Hernández, F., & Wojdyło, A. (2025). Bioactive compounds (LC-PDA-Qtof-ESI-MS and UPLC-PDA-FL) and in vitro inhibit α-amylase and α-glucosidase in leaves and fruit from different varieties of *Ficus carica* L. ***Food Chemistry***, 465. doi: https://doi.org/10.1016/j.foodchem.2024.141977

• **Results and Discussion**. This section gathers the key results and a brief discussion of each publication.

• **Conclusions/*Conclusiones*.** This section includes the main conclusions reached with this doctoral thesis and the future research lines (English and Spanish).

• **Future Research.** This section outlines the potential directions for future research.

• **References.** The present section contains all the literature used to write and justify this Doctoral Thesis following APA 7th edition.

02

ABSTRACT/ RESUMEN

Abstract

The fig tree (*Ficus carica* L.) is one of the oldest recorded fruit trees in terms of cultivation and domestication history. Native to the Middle East, its cultivation has expanded to Mediterranean and subtropical regions due to its remarkable adaptability to diverse climatic and edaphic conditions. Figs have been an integral part of the human diet since ancient times and are regarded as a symbol of health. Their therapeutic properties have been utilized in various traditional medicinal practices, such as Ayurveda, Unani, and Siddha.

Certain varieties are bifera, capable of producing two harvests per year: brebas and figss. The first harvest, known as breba, does not occur in all varieties and develops from buds formed in the previous year, often located at leaf axils. These buds remain dormant until the following spring. Various environmental factors, such as winter temperature and humidity, significantly influence brebas loss. Additionally, since breba begin developing before leaves emerge, there is direct competition between foliage and fruit for resources. Consequently, breba are valued for their exclusivity and limited production. In contrast, figs develop from buds at leaf axils of shoots grown in the current year and is harvested between July and September. The main differences between brebas and figs and stem from the distinct climatic conditions under which each develops.

The motivation for this doctoral thesis originates from my experience as a fig farmer. In Spain, fig cultivation is concentrated in various provinces, with Extremadura being notable for dried fig production and Alicante for breba production. In Alicante province, the most commonly cultivated variety is Colar. However, the economic viability of the second harvest (figs) is constrained by climatic conditions and high harvesting costs. As a result, between 50% and 60% of the total production remains unharvested and unmarketed. The underutilization of these fruits represents a substantial economic loss, given their high nutritional value and bioactive compound content, which could be harnessed across various industries, including food, cosmetics, and pharmaceuticals. It is crucial to highlight that the Food and Agriculture Organization of the United Nations (FAO) advocates for responsible production and consumption, with the objective of halving global per capita food waste. The overarching goal of this doctoral thesis is the physicochemical characterization of the most commercially relevant fig varieties in Spain, as well as the development and characterization of novel fig-based products.

To achieve this, the following specific objectives were established:

1. **Literature Review**: Analyzing the applications of figs, fig leaves, and other fig-derived by-products in various industries.
2. **Plant Material Characterization**: Determining the morphological, functional, and nutritional characterization of breba and figs, including their by-products (peel and leaves).
3. **Development and Optimization of Novel Products**: Creating and optimizing a fermented dairy product incorporating fig puree.

As a result of this research, seven articles were published in journals indexed in the Journal Citation Reports (JCR), addressing the following aspects:

- **Publication 1 (Agronomy)**: Literature review on the current state of research regarding figs and fig-derived by-products.
- **Publication 2 (Agronomy)**: Sugar profile, crude fiber content, macro- and micro-mineral content, total phenolic compounds, and antioxidant activity in fig tree leaves.
- **Publication 3 (Scientia Horticulturae)**: Sugar profile, crude fiber content, macro- and micro-mineral content, total phenolic compounds, and antioxidant activity in the peel and pulp of brebas and figs.
- **Publication 4 (Scientia Horticulturae)**: Volatile compound profile in the peel and pulp of brebas and figs.
- **Publication 5 (Food Research International)**: Techno-functional properties, volatile composition profile, and consumer acceptance of fermented milk enriched with pasteurized fig puree.
- **Publication 6 (LWT - Food Science and Technology)**: Flavonoid content, microbial load, and changes in quality parameters during the shelf life of fermented milk enriched with pasteurized fig puree.
- **Publication 7 (Food Chemistry)**: Carotenoids, chlorophylls, tocopherols, tocotrienols, amino acids, phenolic compounds, and in vitro biological activity of figs and fig tree leaves.

Through these studies and publications, knowledge regarding figs and their by-products (peel and leaves) has been significantly enhanced. Some key conclusions from this doctoral thesis include:

- Fig leaves represent a promising material for obtaining fiber, calcium, and bioactive compound rich extracts, providing an alternative source for incorporation into nutraceutical and food matrices.
- Fig leaves exhibit high concentrations of phenolic acids and flavonols, particularly derivatives of caffeic acid and apigenin, while figs contain exclusive anthocyanins, such as cyanidin-3,5-*O*-diglucoside.
- Clear differences in nutritional and functional properties, as well as antioxidant activity, were observed between the peel and pulp of brebas and figs and among different studied varieties. In general, the peel exhibited higher fiber, mineral, antioxidant, and phenolic content compared to the pulp.
- The most notable differences between breba and figs were found in fruit weight and sugar content. Figs are smaller in size compared to brebas. Additionally, both the peel and pulp of figs contained a higher total sugar concentration than brebas.
- The peel contains a higher quantity of volatile compounds responsible for the characteristic aroma of these fruits, with aldehydes such as hexanal, 2-hexenal, and benzaldehyde dominating the volatile profile of both brebas and figs.
- Incorporating 30%-40% fig puree into fermented milk improves texture, reduces syneresis, and enhances bioactive compounds such as quercetin-3-galactoside, thereby improving nutritional and functional qualities. Moreover, lactic acid bacteria stability and viability were maintained for 30 days of storage, making fig puree a valuable ingredient for functional fermented milks.

The information generated in this doctoral thesis contributes to a deeper understanding of the physicochemical properties of brebas, figs and fig tree leaves. Additionally, it provides solutions to current challenges facing fig cultivation in Alicante, which is essential for ensuring the continuity of this traditional crop, especially given the emerging challenges posed by climate change. Nonetheless, further research is necessary. Future studies should explore additional applications of figs and fig-derived by-products within the food, cosmetics, and pharmaceutical

industries to maximize the utilization of production. Additionally, advancing genetic improvement of fig varieties to enhance drought and high-temperature tolerance is critical to developing more efficient, resilient, and sustainable agri-food systems.

Resumen

La higuera es uno de los árboles frutales con una de las historias de cultivo y domesticación más antiguas registradas. Originaria de Oriente Medio, su cultivo se ha extendido a regiones mediterráneas y subtropicales gracias a su notable adaptabilidad a diversas condiciones climáticas y edáficas. Los higos han sido parte de la dieta humana desde tiempos remotos y se consideran un símbolo de salud. Sus propiedades terapéuticas han sido aprovechadas en distintas prácticas de medicina tradicional, como el Ayurveda, el Unani y el Siddha.

Algunas variedades son bíferas y pueden producir dos cosechas al año, brevas e higos. La primera cosecha, conocida como brevas no se produce en todas las variedades, se desarrolla a partir de las yemas del año anterior, a menudo de las axilas de las hojas. Estas yemas son higos latentes que no comienzan su desarrollo hasta la primavera siguiente. Diferentes factores ambientales como la temperatura invernal y la humedad pueden afectar especialmente a la pérdida de breva. Además, el hecho de que la breva empiece a desarrollarse antes que las hojas, provoca una competencia directa entre las hojas y los frutos por los recursos. Por lo que son apreciadas por su exclusividad y producción limitada. Mientras que la principal cosecha los higos, surge de las yemas en las axilas de las hojas de los brotes que han crecido durante el año actual y se recolectan entre julio y septiembre de ahí que las principales diferencias entre breba e higos se deban a las condiciones climáticas en las que se desarrolla cada uno.

La idea de esta tesis surge de mi experiencia como agricultora dedicada al cultivo de la higuera. En España, este cultivo se concentra en diversas provincias, destacándose Extremadura por la producción de higo seco y Alicante por la producción de brevas. En la provincia de Alicante, la variedad más cultivada es la Colar. Sin embargo, la viabilidad económica de la segunda cosecha (higos) se ve limitada por las condiciones climáticas, y los elevados costes de recolección. Como resultado, entre el 50 % y el 60 % de la producción queda sin recolectarse ni comercializarse.

La falta de aprovechamiento de estos frutos representa una pérdida económica significativa, ya que son una fuente valiosa de nutrientes y compuestos bioactivos que podrían ser utilizados en varias industrias, industria alimentaria, cosmética y farmacéutica. Es importante destacar que la Organización de las Naciones Unidas para la Alimentación y la Agricultura (FAO) promueve la producción y el consumo responsables, con el objetivo de reducir a la mitad el desperdicio mundial de alimentos per cápita.

El objetivo global de esta Tesis Doctoral es realizar una caracterización físico-química de las variedades comerciales más representativas de España, así como el desarrollo y caracterización de nuevos productos a base de frutos de higuera.

Por tanto, los objetivos marcados fueron:

1-**Revisión bibliográfica:** Analizar las aplicaciones de los higos, sus hojas y otros subproductos en la industria.

2- **Caracterización del material vegetal:** Determinar la caracterización morfológica, funcional y nutricional de los frutos de breva e higo, así como de sus subproductos (piel y hojas).

3- **Desarrollo y optimización de nuevos productos:** Crear y optimizar un producto lácteo fermentado a base de higo.

Como resultado de la investigación se publicaron 7 artículos en revistas del JCR en las que se abordaron los siguientes aspectos:

• **Publicación 1 (Agronomy):** Revisión bibliográfica sobre el estado actual de la investigación con frutos y subproductos de higueras.

• **Publicación 2 (Agronomy):** Perfil de azúcares, fibra cruda, contenido de macro y micro minerales, contenido total de compuestos fenólicos y actividad antioxidante en hojas de *Ficus carica*.

• **Publicación 3 (Scientia Horticulturae):** Perfil de azúcares, fibra cruda, contenido de macro y micro minerales., contenido total de compuestos fenólicos y actividad antioxidante en piel y pulpa de brevas e higos.

• **Publicación 4 (Scientia Horticulturae):** Perfil de compuestos volátiles en piel y pulpa de brevas e higos.

• **Publicación 5 (Food Research International):** Propiedades tecno-funcionales, perfil de composición volátil y aceptación por parte del consumidor de la leche fermentada enriquecida con puré de higo pasteurizado.

• **Publicación 6 (LWT - Food Science and Technology):** Flavonoides, carga microbiana y cambios en los parámetros de calidad durante la vida útil de la leche fermentada enriquecida con puré de higo pasteurizado.

• **Publicación 7 (Food Chemistry):** Carotenoides, clorofilas, tocoferoles, tocotrienoles, aminoácidos, compuestos fenólicos y actividad biológica *in vitro* de higos y hojas de higuera.

Fruto de estos estudios y de las correspondientes publicaciones, el conocimiento sobre los frutos y subproductos (piel y hojas) de la higuera ha mejorado. Algunas de las conclusiones principales que se han obtenido de esta tesis doctoral son:

- Las hojas de la higuera pueden ser un buen material para la obtención de extractos ricos en fibra, calcio y compuestos bioactivos proporcionar una fuente alternativa de estos compuestos para ser incorporados a otras matrices nutracéuticas y/o alimentarias.
- Las hojas presentan un alto contenido de ácidos fenólicos y flavonoles, en particular derivados del ácido cafeico y la apigenina, mientras que los higos contenían antocianinas exclusivas, como la cianidina-3,5-*O*-diglucósido.
- Se observaron cambios claros en los valores nutricionales y funcionales de los compuestos y la actividad antioxidante entre la piel y pulpa de brevas e higos y, entre las diferentes variedades estudiadas. En general, se encontraron valores más altos de los contenidos de fibra, minerales, actividad antioxidante y contenido fenólico en la piel que en la pulpa.
- Las diferencias más destacadas entre brevas e higos se han identificado en el peso del fruto y su contenido de azúcares. Los higos presentan un tamaño menor en comparación con las brevas. Asimismo, se ha determinado que tanto la piel como la pulpa de los higos contienen una mayor concentración total de azúcares en relación con las brevas.
- La piel contiene una mayor cantidad de compuestos volátiles responsables del aroma característico de estos frutos, con aldehídos como hexanal, 2-hexenal y benzaldehído dominando el perfil volátil tanto de las brebas como de los higos.

- Se comprobó que la adición de un 30%-40% de puré de higos a las leches fermentadas mejora la textura, reduce la sinéresis y potencia los compuestos bioactivos como la quercetina-3-galactosida, mejorando las cualidades nutricionales y funcionales. La estabilidad y la viabilidad de las bacterias del ácido láctico se mantuvieron durante 30 días de almacenamiento, lo que convierte al puré de higos en un ingrediente valioso para las formulaciones de yogur funcional.

La información generada en esta tesis doctoral contribuye a un mejor conocimiento de las propiedades fisicoquímicas de los frutos y hojas de la higuera. Además, proporciona soluciones a los desafíos actuales que enfrenta el cultivo de la higuera en Alicante, lo cual resulta fundamental para asegurar la supervivencia de este cultivo, tradicional en la región, especialmente ante los nuevos retos que impone el cambio climático. Sin embargo, aún se requiere de una mayor investigación. Es necesario, por un lado, continuar explorando las aplicaciones de los higos y sus subproductos en la industria alimentaria, cosmética y farmacéutica para garantizar el aprovechamiento total de su producción, y por otro, avanzar en la mejora genética de las distintas variedades para incrementar la tolerancia de la higuera al estrés hídrico y a las altas temperaturas. Para tratar de contribuir a desarrollar sistemas agroalimentarios más eficientes, resilientes y sostenibles.

03
INTRODUCTION

Origen, domestication, and evolution of the fig tree

The scientific name *Ficus carica* L. consists of two parts: the genus *Ficus*, derived from the Latin word for fig, referring to its enclosed flowers within the syconium (Zhang et al., 2019), and the species name *carica*, referencing Caria, a region in Asia Minor where edible figs were historically found (Ferguson et al., 2011). Fig domestication began in the early Neolithic, with archaeobotanical evidence from charred figs in the Jordan Valley dating back 11,200–11,400 years, preceding cereal domestication (Kislev et al., 2006).

Fig. 1 Schematic representation of the expansion of *Ficus carica* (Generated by ChatGPT)

Early trade routes shaped the modern distribution and genetic diversity of figs. The domestication and early migration of figs along these routes influenced their structure, as cultivation spread from southern Arabia to western Asia, including Mesopotamia, Anatolia, Transcaucasia, and Persia. Hybridization with wild figs and human selection, particularly in Transcaucasia, led to diverse varieties. Further expansion into Greece, Italy, Spain, Portugal, and Egypt increased cultivar diversity. Spanish missionaries introduced figs to the New World in the mid-16th century, reaching North America soon after (Aradhya et al., 2010). Around a thousand years ago, figs also spread to China, where research has since advanced in variety selection, cultivation techniques, processing, and medicinal applications (Lianju et al., 2003).

Taxonomy

The scientific nomenclature and taxonomy of fig trees is:

- Kingdom: Plantae
- Phylum: Streptophyta
- Class: Equisetopsida
- Subclass: Magnoliidae
- Order: Rosales
- Family: *Moraceae*
- Genus: *Ficus*
- Species: *Ficus carica*

The genus Ficus L. is divided into 17 sections, with *Ficus ca*rica L. classified under the Ficus section. This species includes two subspecies: (1) *Ficus car*ica L. subsp. *carica* (common fig), native to Afghanistan, Cyprus, Greece, Iran, Iraq, Crete, Lebanon-Syria, the North Caucasus, Palestine, Tajikistan, Transcaucasia, Turkey, and Turkmenistan; and (2) *Ficus carica* subsp. *rupestris* (Hausskn. ex Boiss.) Browicz, found in Afghanistan, Iran, Iraq, Lebanon-Syria, Pakistan, Turkey, and the Western Himalayas. Beyond its native range, the common fig and its numerous cultivars are widely cultivated in tropical and subtropical regions (Bandelj et al., 2023).

The characterization of fig germplasm has traditionally relied on morphological traits. One of the earliest comprehensive works on fig varieties was written by Condit (1955) monograph, based on over 30 years of study, in which he described more than 700 cultivars. However, the long history of domestication, extensive cultivar exchange, and global spread have led to significant ambiguity in fig cultivar nomenclature and identification. To standardize fig characterization, the International Plant Genetic Resources Institute (IPGRI) and the International Centre for Advanced Mediterranean Agronomic Studies (CIHEAM) released the *Descriptor List for Ficus carica L.* in 2003. This report identified key distinguishing traits, including leaf shape, fruit shape, ostiole diameter, fruit peel color, inner pulp color, and peel cracks (IPGRI & CIHEAM, 2003).

Morphology of the fig tree

Ficus carica L. is a medium-sized deciduous tree, with multiple spreading branches. The species is characterized by the presence of a milky white latex, primarily composed of ficin, a proteolytic enzyme, which is present in all parenchymatous tissues. The bark is smooth and silky, ranging in color from ash to silvery-grey, with irregular exfoliating flakes. The middle layer of the bark exhibits a light reddish-brown hue, while the inner tissue consists of pale yellowish to orange-brown granular layers (Al-Snafi, 2017). The root system of *F. carica* is is fasciculated, lacking a dominant taproot. In unirrigated soils, roots penetrate deeper in search of moisture, whereas in cultivated and irrigated conditions, they remain more superficial. The primary roots are thick, shallow, and spread widely, forming a network that extends up to twice the canopy's horizontal projection. About 80% of the fibrous, fragile roots are found at depths of 20–45 cm (Melgarejo, 1999).

Fig leaves are alternate, bright green leaves with a rough upper surface and a softer lower surface. Leaves are stipulated, petiolated, obovate to ovate in shape, with palmately lobed margins, a cordate base, and an acute to obtuse apex. They measure 10–20 cm in both length and width, typically featuring 3 to 5 lobes and a palmate venation pattern. The base is generally divided and heart-shaped. The upper surface is rough, while the underside has rigid, coarse hairs, contributing to its texture. The petioles range from 2 to 5 cm in length (Khadivi et al., 2018; Westwood & Romero, 1982).

The fig is not a true fruit but an infructescence called a syconium, which contains 200–300 flowers inside. After pollination, these flowers develop into small, hard fruits known as achenes. These achenes are highly resistant to digestion, facilitating seed dispersal by birds. The fleshy, sweet part of the fig is the enlarged floral receptacle. Figs grow continuously on the tree, emerging from leaf axils. Typically, only one axillary bud develops into a syconium, though sometimes both do. Syconia that mature in early summer (June) are called brebas and are more valuable in markets than the figs harvested from July to September. Though anatomically identical, brebas are usually larger due to more favourable growing conditions, whereas figs are more abundant (Melgarejo, 1999).

The fruit may be obovoid, turbinate, or pear-shaped, with a length ranging from 2–5 cm in diameter. Its color varies from yellowish-green to coppery, bronze, or dark purple, depending on the stage of ripening. The peel is thin and tender, while the fleshy wall can be whitish, pale yellow, amber, pinkish, rose-red, or purple, also

influenced by ripening. The juice is sweet when the fruit is ripe but gummy with latex when unripe. At peak maturity, the interior contains remnants of the flower structure, including a gritty texture commonly mistaken for seeds, which are actually unfertilized ovaries (Stover et al., 2007). Fig stalks and necks play a crucial role in harvesting ease and fruit damage prevention (IPGRI & CIHEAM, 2003). A larger ostiole increases susceptibility to spoilage and disease transmission and ostiole width varies among cultivars, with reported ranges from 0.00 to 10.8 mm in Iranian figs (Khadivi et al., 2018).

Fig flowers are extremely small and concealed within a specialized, fleshy, pear-shaped structure called the syconium. The flowers are embedded in the inner walls of the syconium and are not externally visible. Depending on the fig type, the syconium may contain only female flowers, only male flowers, or both. Male flowers become visible when the syconium matures and its ostiole bracts open, while female flowers remain hidden deeper inside. Some cultivated figs require pollen from wild figs (caprifigs) for fertilization, a process known as caprification, facilitated by the pollinating wasp *Blastophaga psenes* L., which transfers pollen from male flowers of wild figs into the syconia of cultivated varieties. However, most cultivated figs in certain regions develop fruit without pollination (Melgarejo, 1999).

Traditional varieties

Figs have been further grouped into 4 different types depending upon the sex of the flower and method of pollination employed. These types are commonly found in fig cultivation worldwide: *i*) Common Fig (Parthenocarpic Type): the most cultivate type of fig and includes many popular varieties; *ii*) the San Pedro type: the first crop (breba) that pollination is not needed and the second crop (the main crop: fig) in which pollination is required to achieve full maturation; *iii*) Smyrna type: production of fruit with viable seeds, pollination is required to set fruit; *iv*) The Caprifig type: the syconia (figs) contains pollen that will pollinate the Smyrna and San Pedro types (Ben et al., 2023).

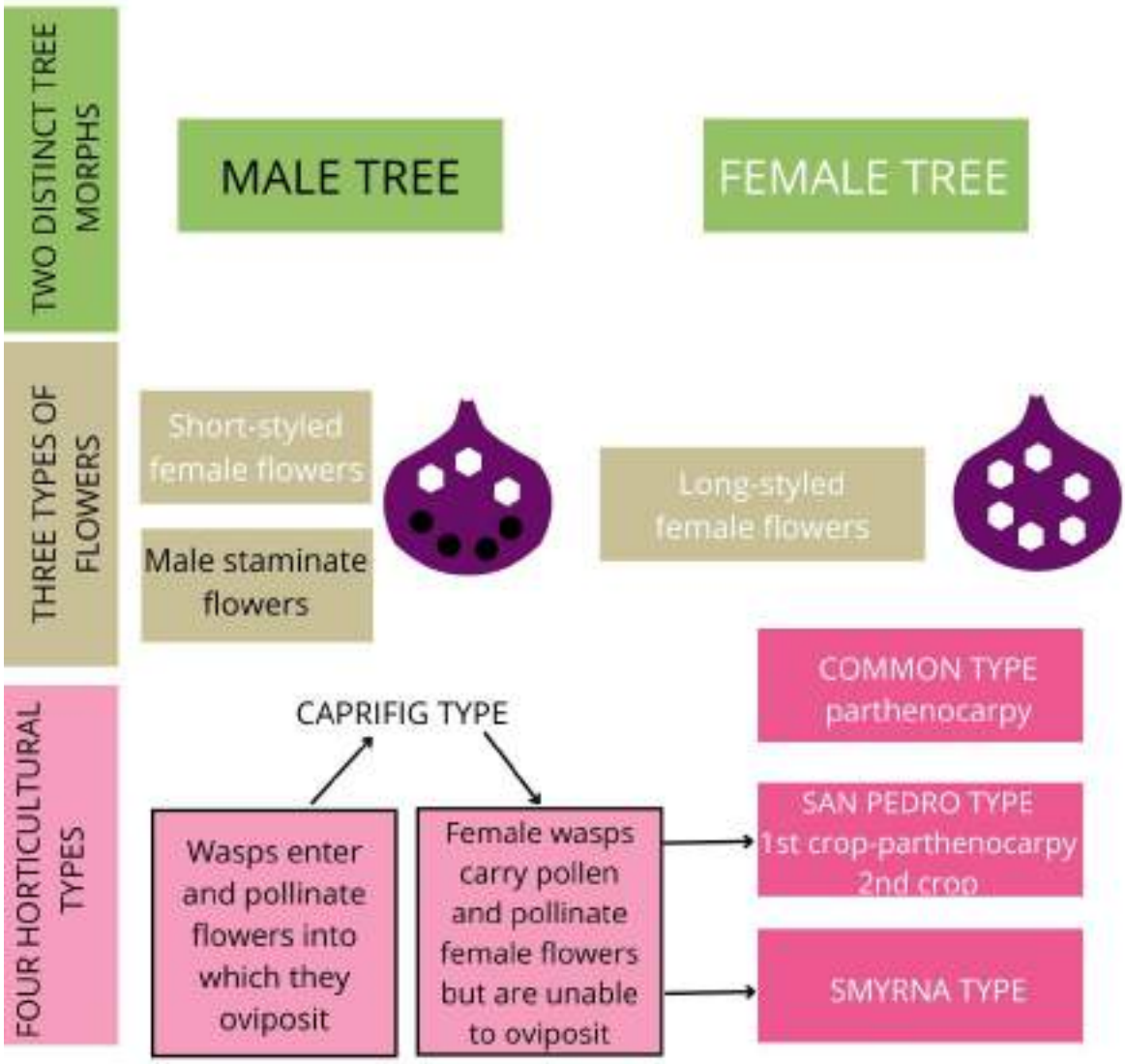

Fig. 2 Schematic representation of sex of the flower and method of pollination (*F. carica*) (Bandelj et al., 2023)

For this thesis, four parthenocarpic and biferous figs tree varieties have been analyzed (Melgarejo, 1999):

- San Antonio. A bifera variety of medium vigor and open growth habit. The breba is generally pyriform, although spherical fruits are relatively common. It has a medium-sized peduncle that detaches easily from the wood and an open ostiole. The peel is of medium thickness, dark green near the neck, gradually turning more purple towards the ostiole and the sun-exposed areas. It has prominent longitudinal ridges, also purple in color. The pulp is honey-colored with some pinkish tones, of medium texture, soft consistency, juicy, and has a sweet yet slightly acidic flavor. The achenes are large but do

not completely fill the receptacle. The average weight is 45 g. The fig harvest period extends from late July to late September, with the highest quantity and quality occurring in the early harvests. The average fig weight is 30 g. This variety is well adapted to rainfed cultivation and produces high-quality fruits, particularly brebas, during an early ripening period. However, it requires careful handling for fresh commercialization, which is its primary market.

- Colar. A black bifera variety that produces an excellent breba crop. The syconia are large, highly striped, rounded, and visually striking. The tree is highly productive, and the peduncle detaches easily with the fruit. This variety is the most widely cultivated, especially in southeastern Spain, due to its productivity, precocity, color, size, and excellent flavor. It accounts for over 95% of the fig tree cultivation area in this region. Breba harvesting begins in the second half of May, reaches excellent prices, and continues until late June or early July. This high-quality breba withstands transport well and has a very sweet flavor. Fig harvesting occurs from June to September, with fig production far exceeding that of brebas.
- Cuello Dama Negra. A bifera variety of medium to high vigor, with a semi-open growth habit, highly branched, and lower branches tending to be pendulous. The breba is obliquely pyriform, with a short peduncle that is difficult to detach from the wood, and a semi-open ostiole. The peel is dark purple and highly resistant. The pulp has a medium texture and garnet color. The breba ripening period occurs in late May. The fig has the same shape as the breba, with a medium to short peduncle and a semi-open ostiole. The peel is dark purple, almost black, thin but very resistant. When the fig ripens in conditions of high ambient humidity, it tends to develop many cracks, enhancing its visual appeal while maintaining peel integrity. The pulp is garnet-colored, with a fine texture and soft consistency, and the numerous achenes nearly fill the receptacle completely. The fig harvesting period extends from mid-August to late September, with peak production occurring in late August. This variety is well adapted to rainfed conditions and exhibits excellent handling and transport characteristics, making it highly suitable for direct fresh consumption.
- Superfig. A bifera variety of medium to high vigor and open growth habit. The syconia are large and pyriform, with a medium-sized peduncle that detaches easily from the wood and an open ostiole. The peel is greenish-

purple. The pulp has a medium texture and garnet color. The breba ripening period occurs in late May. The fig has the same shape as the breba, with a medium to short peduncle and an open ostiole. The fig harvesting period extends from June to September.

Cultivation conditions of the fig tree

Fig trees exhibit adaptability to various climates, their productivity and fruit quality are optimal in regions with warm and dry conditions. Environmental factors such as a decline in autumn temperatures, winter cold, and excessive rainfall during fruit maturation can negatively impact productivity (Hussain et al., 2021). The fig tree is highly suitable for cultivation under the ecological conditions of the Levante and Southeastern Spain. Due to low annual rainfall, both the soils and irrigation water in these regions often exhibit high salinity levels, regardless of areas specifically classified as salt flats. Under these conditions, certain crops such as the fig tree are among the best-adapted species, demonstrating resilience to the challenging edaphic environment while still allowing for the commercial production of high-quality fruit (Melgarejo, 1999).

Fig trees are most profitably cultivated in warm and temperate climates of the Northern Hemisphere, particularly between 35° and 40° latitude. Beyond 45°C, fruit fails to reach maturity and fruit quality begins to deteriorate beyond 39°C, while below 25°C, excessive heat disrupts the normal progression of vegetative phases. Fig trees can survive temperatures as low as −12.2°C, younger trees are more vulnerable and may suffer damage at frost levels between −5 and −10°C, with the optimal growth range being 15–21°C. High temperatures at the beginning of summer can cause premature fruit drop, leading to a phenomenon known as false ripening. This effect is exacerbated by insufficient irrigation and warm winds and typically occurs when temperatures reach 37.7°C (Ferguson et al., 2011).

Fig trees can thrive in various soil types, including light sand, rich loam, heavy clay, and limestone. However, the chosen soil should have adequate depth and proper drainage. Sandy soil is generally preferred due to its medium-dry texture and sufficient lime content. Fig trees adapt well to calcareous soils, tolerating pH levels between 8 and 8.5. Highly acidic soils are unsuitable for fig cultivation, so the soil pH should be maintained between 6 and 6.5. (Morton & Dowling, 1987). Fig trees exhibit high salinity tolerance, surpassed only by the date palm (*Phoenix dactylifera* L.), jujube (*Ziziphus vulgaris* L.), and prickly pear (*Opuntia ficus-indica*

L.). This characteristic makes fig cultivation particularly valuable in saline environments, where the profitable cultivation of many other fruit species is not feasible. Additionally, fig trees demonstrate exceptional tolerance to active limestone, comparable to that of the pomegranate (*Punica granatum* L.). Both species are successfully cultivated in certain regions of Alicante, where the active limestone content exceeds 22% without showing symptoms of chlorosis. (Melgarejo, 1999).

Fig trees thrive in regions with predominantly dry weather and light spring rains. Under these conditions, they produce the sweetest fruits. Excessive rainfall during early fruit development and ripening can result in excessively large, watery figs that are prone to rotting and difficult to dry. Additionally, the tree is highly susceptible to root rot. Fruit cracking is often caused by prolonged water deficiency followed by sudden rehydration due to heavy rainfall or excessive irrigation. However, prolonged extreme heat and dryness can lead to smaller leaves and reduced fruit yield, while extreme drought can result in complete crop failure and a significant reduction in both leaf size and number. An annual water supply of 600–700 mm is sufficient to achieve good productivity (Morton & Dowling, 1987).

In Southeastern Spain, fig cultivation is primarily focused on the production of first-crop fruit, known as brebas, which, like the second-crop fruit, figs, are mainly intended for fresh market commercialization. According to Flores (1990), fig trees grown under dryland conditions with a planting density of 9 × 9 m can yield approximately 8 t/ha of brebas or 15 t/ha of figs. Under irrigated conditions, with a planting density of 6 × 6 m, yields can reach 20 t/ha of brebas or 35 t/ha of figs. In the studied region, breba production is the most economically valuable, with nearly the entire harvest being marketable. In contrast, the second crop (figs) has a lower utilization rate (50–60%) (Melgarejo, 1999).

Global and Spanish production

According to the Food and Agriculture Organization (FAO) of the United Nations, global fig production has remained stable in recent years. In 2023, the total area under fig tree cultivation reached 299,768 hectares, with an estimated production of 1,304,849 tons. As illustrated in Figure 1, the global production of figs and the cultivated area of fig trees have shown fluctuations over the past nine years. In 2016, fig production reached its lowest point at 1,077,870 tons. However, production experienced a continuous increase until 2020. Over the last three years, global fig production has stabilized, with values ranging between 1,293,092 and

1,321,976 tons. Regarding the cultivated area, a decline was observed in 2017, reaching a minimum of 277,623 hectares. Since then, the cultivated area has increased gradually, stabilizing in the most recent year (FAOSTAT, 2023).

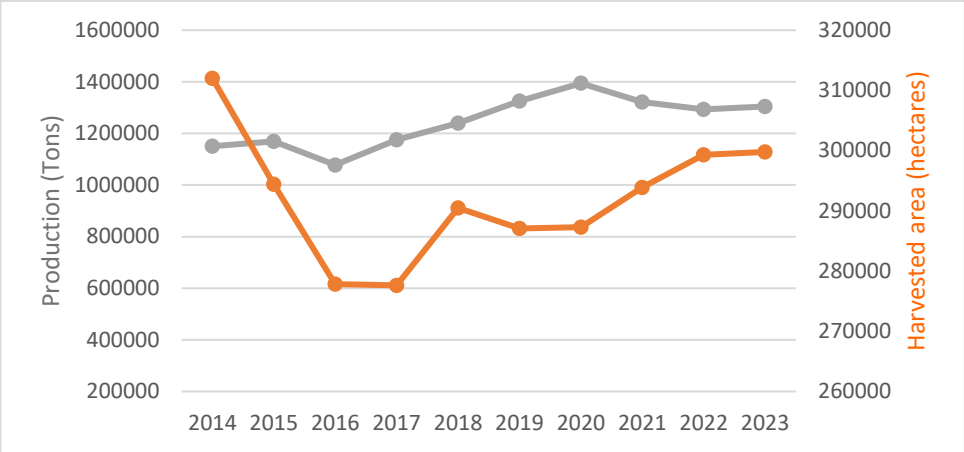

Fig. 3 Production and cultivated area of fig trees (*F. carica*) in the world (FAOSTAT, 2023)

Turkey remains the largest fig producer in the world, with an output of 356,000 tons in 2023, followed by Egypt with 193,058 tons and Morocco with 119,167 tons. The Mediterranean basin and the Near East continue to be of utmost importance for fig production (FAOSTAT, 2023).

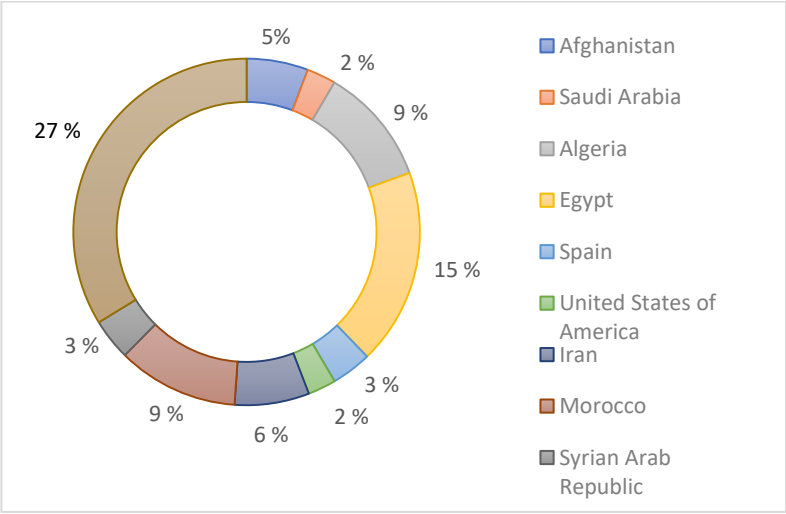

Fig. 4 Percentage of fig production by the top 10 Fig-Producing countries in the world (FAOSTAT, 2023).

In Europe, Spain is the primary fig producer, with a total production of 39,650 tons, accounting for 54% of total European production. It is followed by Italy with 13,030

tons (18% of European production) and Greece with 8,440 tons (11% of European production) (FAOSTAT, 2023).

Autonomous communities	Crop area			Production(t)
	Total (ha)	Non irrigated (ha)	Irrigated (ha)	
GALICIA	609	486	123	3075
P. DE ASTURIAS	–	–	–	45
CANTABRIA	–	–	–	–
PAÍS VASCO	1	1	–	170
NAVARRA	18	–	18	41
LA RIOJA	21	–	21	45
ARAGÓN	202	6	196	1633
CATALUÑA	556	10	546	5834
BALEARES	2218	2218	–	252
CASTILLA Y LEÓN	424	175	249	1850
MADRID	40	40	–	43
CASTILLA-LA MANCHA	863	770	93	2663
C. VALENCIANA	684	35	649	2932
R. DE MURCIA	135	36	99	819
EXTREMADURA	7034	5793	1241	37382
ANDALUCÍA	2643	2395	248	2595
CANARIAS	272	247	25	521
ESPAÑA	15720	12212	3508	59900

Fig. 5 Production and cultivated area of fig trees (*F. carica*) in the Spain (MAPA, 2021)

In Spain, the main fig-producing region is Extremadura, with a production of 37,382 tons, followed by Cataluña with 5,834 tons and Comunidad Valenciana with 2,932 tons. Within Comunidad Valenciana, the province of Alicante is the primary production area, concentrating 84% of fig output. The production in Alicante is mainly intended for fresh consumption, with breba figs of the Colar variety being predominant. In contrast, the production in Extremadura is primarily directed towards dried fig production (MAPA, 2021).

04
OBJECTIVES

The overall aim of this Doctoral Thesis was to perform a physico-chemical characterization of the most representative commercial fig varieties in Spain, as well as the development and characterization of new products based on fig fruits.

To reach the main purpose, the following specific objectives were established

- Objective 1: Know the applications of **figs**, **leaves** and other by-products in the **food** and **pharmaceutical industry** (Block I: Publication 1).

- Objective 2: Determine **morphological, functional, and nutritional characterization** of breba and fig fruits and by-products (Block II: Publications 2, 3, 4 and 7).

- Objective 3: Development and optimization of **fermented milk based on fig** (Block III: Publications 5 and 6).

05
MATERIALS AND METHODS

This section provides a brief description of the tools used to compile the bibliographic review (Publication 1). It also outlines the key characteristics of the experimental conditions, analytical methodologies, processing treatments, and statistical analyses employed in this thesis. Further details on the methodology and the materials used are available in the published manuscripts that form part of this thesis.

5.1 Block I (Publication 1)

Scientific Literature Review

A scoping review methodology was employed to synthesize evidence and assess the scope of 41 studies related to the topic. The review follows the PRISMA Extension for Scoping Reviews (PRISMA-ScR) guidelines (Page et al., 2021). A comprehensive literature search was conducted in August 2021 using Scopus and ScienceDirect databases, limited to articles published in English since 1990. Priority was given to studies published in journals indexed in the Journal Citation Reports.

5.2 Block II (Publications 2, 3, 4 and 7)

Plant material and experimental conditions

Leaves, brebas, and figs of *Ficus carica* were collected from four biferous varieties: San Antonio (SA), Colar (CA, CUMH), Cuello Dama Negra (CDN), and Superfig (SF). These samples were sourced from the experimental field of the Universidad Miguel Hernández de Elche (UMH) in the province of Alicante, Spain (02°03’50” E, 38°03’50” N). Additionally, the Colar variety was collected from two distinct localities: (i) at the aforementioned UMH experimental field (CUMH) and (ii) at a commercial plot in Albatera, Alicante, southern Spain (0°55’49” W, 38°13’17” N). The fig trees, aged 20 years, were trained to a vase-shaped system, planted at a density of 8 m × 5 m, and maintained with drip irrigation. Standard agricultural practices, including pruning, thinning, fertilization, and pest control treatments, were applied throughout the study. A selection of thirty brebas and figs were manually gathered at random upon reaching their optimal commercial maturity (brebas in May 2021 and figs in July 2021). Additionally, leaves were carefully hand-harvested from the trees and transported to the laboratory for processing. Within

the laboratory, the samples underwent freeze-drying and grinding, resulting in a uniform dry material suitable for further analysis.

Fig. 6 Breba Colar at different stages of maturity (CA - Left vs. CUMH - Right)

Morphological and physical characterization

For each variety, a total of 30 ripe brebas and figs were examined, obtained from three distinct trees, with 10 fruits selected per tree. The weight of the fruits was determined using a Sartorius digital bench scale (model AG204; Mettler Toledo, Barcelona, Spain) with a precision of 0.01 g. Their length and width were measured using an electronic digital caliper (model 500–197–20 150 mm; Mitutoyo Corp., Aurora, IL, USA) with an accuracy of 0.01 mm. Color evaluation was performed on the fruit peel at two opposing surfaces within the equatorial region. The CIELab color parameters were analyzed using a Minolta CR-300 Chroma Meter (Minolta Corp., Osaka, Japan), connected to a Minolta DP-301 data processor. A sample of 30 leaves per variety was analyzed, with 10 randomly selected leaves collected from each tree. Leaves were sampled from all orientations of the trees and from the midsection of shoots, ensuring only healthy and undamaged specimens were included. The parameters measured included leaf length (from the base of the petiole to the tip of the central lobe, expressed in cm), leaf width, petiole length, and the length of the central lobe.

Organic acids and sugars

A 0.2 g portion of the freeze-dried sample was blended with 5 mL of 50 mM phosphate buffer (pH 7.8) and subjected to centrifugation at 15,000 × g for 15 minutes at 4 °C using a Sigma 3–18 K centrifuge (Osterode am Harz, Germany).

Subsequently, 1 mL of the supernatant was filtered through a 0.45 µm Millipore filter (Billerica, MA, USA) prior to HPLC analysis. A Hewlett-Packard HPLC series 1100 system (Wilmington, DE, USA) was used for the identification and quantification of sugars and organic acids. Sugars detection was performed using a refractive index detector (RID; Hewlett-Packard, series 1100, G1362A) while the organic acid absorbance was read at 210 nm with a diode-array detector (DAD). All analyses were conducted in triplicate, and the results were expressed as g kg^{-1} dry weight (dw).

Total dietary fiber

The total dietary fiber content was analyzed according to the official methodology established by the Spanish Ministry of Agriculture, Fisheries, and Food, as detailed by (2021). The analysis was performed using an ANKOM200/220 fiber analyzer (ANKOM Technology, Macedon, NY, USA), and the results were reported as a percentage (%).

Mineral content

The mineral content of freeze-dried pulp and peel samples was determined by digestion using a microwave system (MARS ONE, 240/50 CEM). For each sample, 0.2 g was digested with 10 mL of concentrated nitric acid (65% w/v HNO_3), reaching 200 °C within 15 minutes and holding at this temperature for an additional 15 minutes. The processed samples were passed through quantitative filter paper, transferred into a volumetric flask, and diluted at ratios of 1:10, 1:20, and 1:60 for potassium using ultrapure deionized water (18 MΩ, Milli-Q® system; Millipore Corporation, Madrid, Spain). The mineralized samples were analyzed to determine the concentrations of macronutrients (Ca, Mg, and K) and micronutrients (Cu, Fe, Mn, and Zn) with an inductively coupled plasma mass spectrometer (ICP-MS), specifically the Shimadzu ICPS-2030 (Shimadzu Scientific Instruments, Inc., Columbia, MD, USA). The results were expressed as g/kg dw for macronutrients and mg/kg dw for micronutrients.

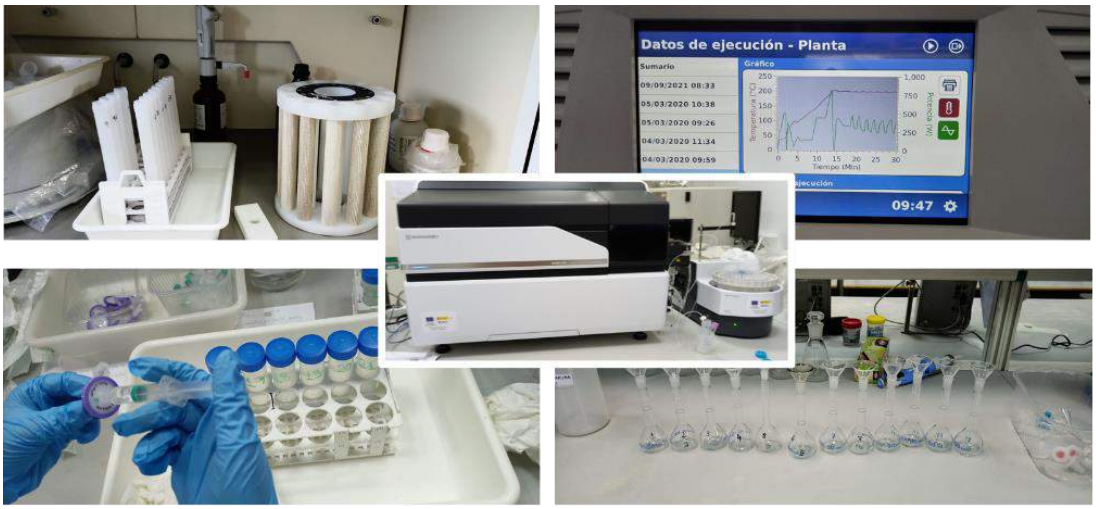

Fig. 8 Extraction and identification of the mineral compounds

Volatile compounds

Volatile compounds were extracted separately from fruit peel and pulp using headspace solid-phase microextraction (HS-SPME). A distinct extraction method was employed for the peel and pulp of each sample. For the peel, two grams (obtained using a peeler on frozen fruit) were placed into a hermetically sealed vial with a polypropylene cap and PTFE (polytetrafluoroethylene)/silicone septa, along with 1 g of NaCl. Regarding the pulp, eight grams were introduced into the vial alongside 2 mL of water and 1 g of NaCl. To facilitate compound absorption, a 50/30 µm DVB/CAR/PDMS fiber was utilized, with samples agitated at 500 rpm for 60 minutes at 40 °C using a Shimadzu AOC-6000 Plus autosampler. Analysis of volatile compounds was conducted via a Shimadzu GC2030 gas chromatograph fitted with an SLB-5 MS column (30 m × 0.25 mm × 0.25 µm) and linked to a Shimadzu TQ8040 NX mass spectrometer. Additionally, the relative intensity of each volatile compound was determined by calculating the ratio of its specific peak area to the sum of all identified peaks using the peak area normalization method in the chromatogram.

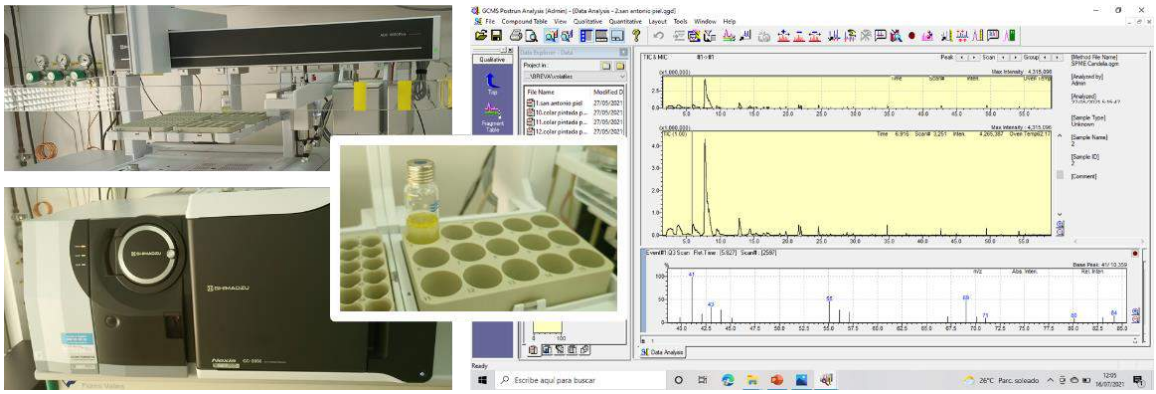

Fig. 9 Volatile compounds analysis

Amino acid

The content of free amino acid was measured according to the method described by Wojdyło et al. (2020). Analysis of amino acids was conducted using the ACQUITY UPLC system (Waters Corp., Milford, MA, USA) equipped with a photodiode array (PDA) detector coupled to a G2 Q/TOF micro-mass spectrometer system (MS) with an electrospray ionization (ESI) source. The PDA spectra of amino acids were measured at a wavelength of 260 nm and retention times and molecular masses were compared with certified reference standards for identification.

Carotenoids and Chlorophylls

Carotenoids and chlorophylls were extracted following the method described by Wojdyło et al. (2020). Analysis of carotenoids was performed using ultra performance liquid chromatography (Acquity UPLC System) with a binary solvent manager and a photodiode array (PDA) detector coupled to a G2 Q/TOF micro-mass spectrometer fitted with an electrospray ionization (ESI) source acting in positive ion mode (Waters Corp., Milford, MA, USA).

The characterization of single components was carried out via retention time and accurate molecular masses in positive ion mode, set to the base peak intensity (BPI) chromatograms. Retention times (Rt) and spectra (λ) were compared with those of pure standards. The detection wavelength for carotenoid compounds was 450 nm and for chlorophylls at 650 nm. Quantification was achieved by injection of solutions of known concentrations ranging from 0.05 to 0.5 mg/mL ($R^2 \leq 0.9998$). The results were expressed as mg per 100 g of dry matter (dm) for carotenoids and chlorophylls.

Tocols

The analysis of tocopherols and tocotrienols followed the methodology outlined by by Wojdyło et al. (2022) and was conducted using UPLC (Acquity UPLC Waters; Milford, MA, USA) equipped with a fluorescence detector (FL) and an Acquity UPLC BEH RP C18 column (1.7 μm, 2.1 mm × 100 mm, Waters Corp.; Milford, MA, USA). The separation process utilized the same Acquity UPLC BEH RP C18 column. Excitation and emission wavelengths were set at 290 nm and 330 nm, respectively. Identification and quantification were achieved through reference standards and

calibration curves. Calibration curves, spanning concentrations from 0.05 to 20 mg/mL ($R^2 \geq 0.998$), were established for α-, β-, γ-, and δ-tocopherols, as well as tocotrienols. The results were reported in mg per 100 g of dw.

Polyphenols and procyanidins

Polyphenols extraction and analysis was determined using the method proposed previously by Wojdyło et al. (2020). The sample (approximately 1 g) was extracted with 5 mL of a mixture containing methanol: H2O: ascorbic acid: acetic acid (30:68:1:1, v/v/w/v). For identification and quantitative analysis were performed using Acquity UPLC system (Waters Corp., Milford, MA, USA) equipped with a photodiode (PDA) and fluorescence (FL) detector with a binary solvent manager (Waters Corp., Milford, MA, USA). Polyphenolic compounds were monitored at the following wavelengths 320 nm (phenolic acids), 360 nm (flavonols), and 520 nm (anthocyanins). The content of polymeric procyanidins was analyzed using the phloroglucinol method (Wojdyło et al., 2020). All analysis was done in three repetitions and the results were expressed as mg per 100 g of dm.

Antioxidant capacity and total polyphenols content (TPC)

The antioxidant activities were tested as oxygen radical absorbance capacity (ORAC), ferric reducing ability of plasma (FRAP) and 2,2'-azino-bis (3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) antioxidant assays were determined as previously described by Ou et al. (2002), Benzie and Strain (1996), Re et al. (1999), respectively. The radical scavenging activity was evaluated using the DPPH radical method, as described by previous research (Brand-Williams et al., 1995), allowing a reaction time of 15 min. Total polyphenols content (TPC) was quantified using Folin–Ciocalteu reagent as described by (Singleton et al., 1999).

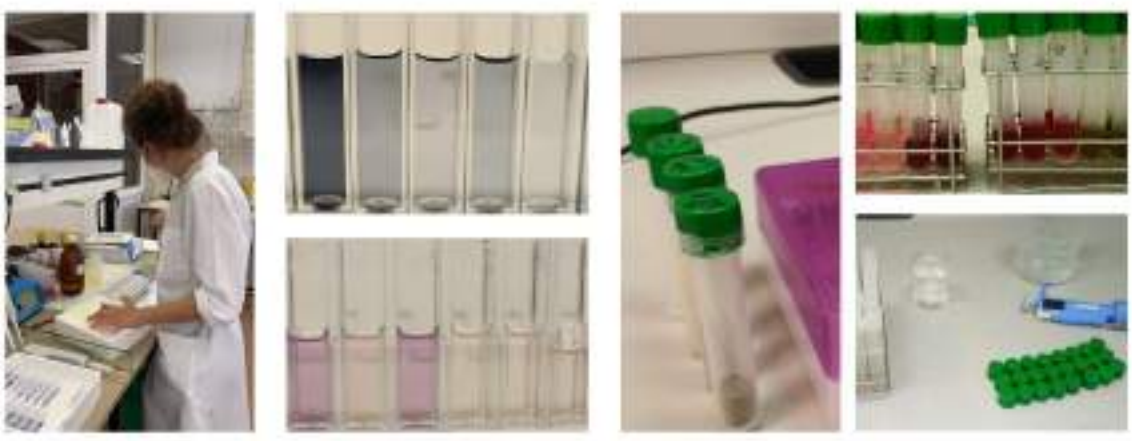

Fig. 10 Antioxidant capacity analysis

Biological activity

The α-amylase and α-glucosidase activity were determined as described by Tkacz et al. (2019). Briefly, the inhibition of α-amylase activity is based on a spectrophotometric measurement of the color change as a result of a reaction of iodine in potassium iodide with the remaining starch after enzymatic hydrolysis, and absorbance were measured at 600 nm. The analysis of α-glucosidase activity inhibitors was assessed based on the interaction of α-glucosidase with a β-d-glucosidase substrate producing a yellow solution upon cleavage. Absorbance were measured at a wavelength of 405 nm. Acarbose was used as a positive control.

5.3 Block III (Publications 5 and 6)

Pasteurized fig puree and fermented milk manufacture

For preparation of the pasteurized fig purée, Colar figs from commercial plot located in Albatera (Alicante, Spain, 2021 season) were harvested and frozen at −20 °C. Figs were disinfected with peracetic acid (200 mg/L, 15 °C, 10 min) and rinsed with water (15 °C, 5 min). Puree was made using a Thermomix® (crushing, then pasteurization at 100 °C for 20 min) and rapidly cooled to <4 °C. Mycotoxin analysis confirmed all levels were below EU regulatory limits.

Fermented milk was prepared using UHT whole cow's milk (3.6% fat, Hacendado, Spain) and a lyophilized lactic ferment containing *S. thermophilus, L. delbrueckii ssp. lactis, and L. delbrueckii ssp. bulgaricus* (CHOOZIT™ MY800, Rhodia Food-Danisco). The culture was hydrated in sterile peptone water (20 mL, 43 °C, 20 min) and dosed at 1000 µL per liter of milk. Milk was sterilized in Pyrex bottles, inoculated with the culture, shaken, and incubated at 43 °C until pH 4.6, then refrigerated for 24 h. Puree was added to yogurt after fermentation to preserve anthocyanins and enhance texture, with both stored at 4 °C for 24 hours before mixing. Samples were analyzed at 24 h (T0) and 30 days (T30).

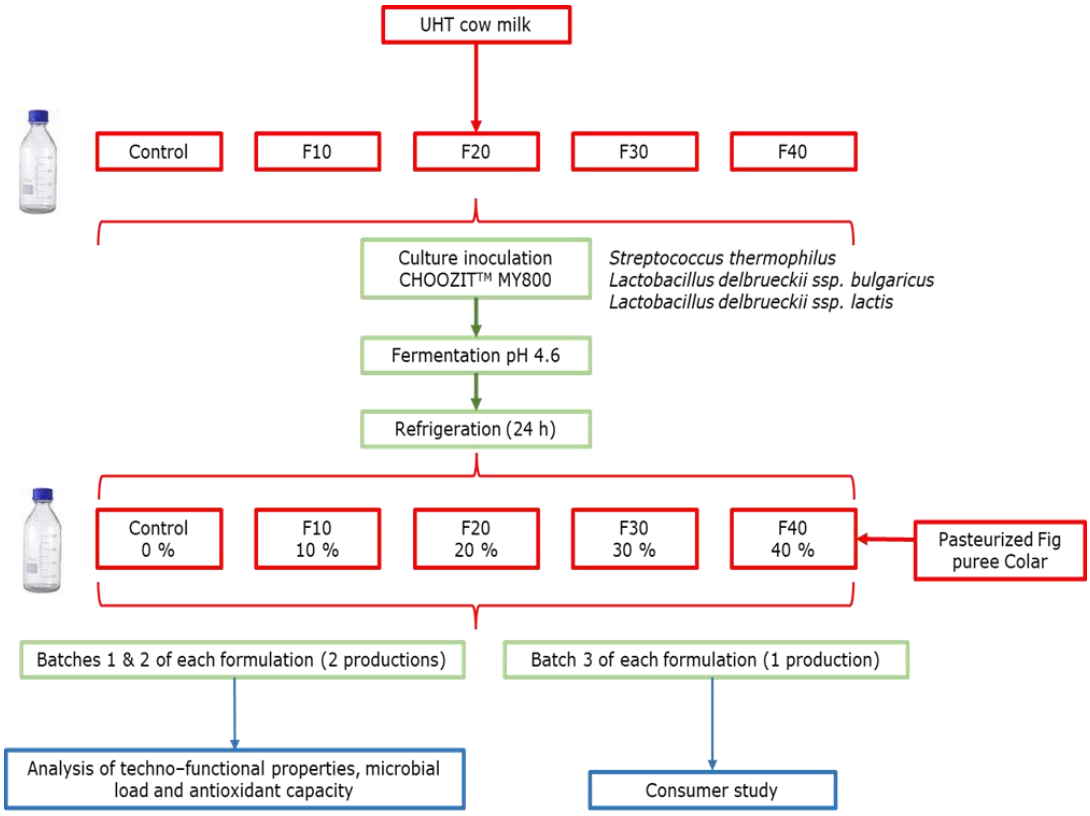

Fig. 11 representation of the experimental design with fermented milks and fig purée

Microbial load and techno-functional properties analysis

The microbial load was analyzed following the methodology described by Trigueros et al. (2012). MRS agar (Merck KGa, Sigma Aldrich, United States) was used to quantify *Lactobacilli* (LAB), incubated at 37 °C under microaerophilic conditions for 48 hours. For *Lactococci* (LAC), M17 agar (Merck KGa, Sigma Aldrich, United States) was utilized, with samples maintained at 30 °C under aerobic conditions for 48 hours. To identify molds and yeasts, Rose Bengal Agar (Merck KGa, Sigma Aldrich, United States) was applied, with incubation at 26 °C under aerobic conditions for 72 hours.

Fig. 11 Microbiological analysis: contamination observed on petri dish

The CIEL*a*b* color parameters of fermented milks were assessed using a Minolta CM-2002 spectrophotometer (Minolta Camera Co., Osaka, Japan), equipped with a CR-A70 liquid accessory (Minolta Camera Co., Osaka, Japan). Measurements were conducted under illuminant D65 with a 10° observer. Color evaluations took place at 12 ± 2 °C, with daily calibration performed using a Minolta white plate.

The pH was assessed across all batches to track the fermentation process and confirm attainment of the target value of 4.6. Furthermore, pH measurements were conducted on the fortified fermented milks following the addition of pasteurized fig puree at both time 0 and time 30. Readings were obtained using a pH meter (model pH/Ion 510, Eutech Instruments Pte Ltd., Singapore).

A texture penetration test was conducted utilizing a Texture Analyser TA-XT2 (Stable Micro Systems, Surrey, England) fitted with a 5 kg load cell. Constant-speed penetration testing was applied directly to cylindrical containers (4.5 cm in diameter and 4 cm in height), following the method outlined by Trigueros et al. (2012). A 10 mm diameter cylindrical ebonite probe (P-10) was inserted 15 mm into the samples at a rate of 1 mm/s. Each yogurt sample underwent three replicate measurements. Instrumental texture analysis was performed at 8 °C after the removal of spontaneous syneresis. Texture attributes (firmness, consistency, cohesiveness, and viscosity index) were analyzed using Exponent software.

Gel stability was evaluated through visual inspection following incubation to observe spontaneous syneresis and quantified by determining the volume of whey separated from the curd after centrifugation. Each formulation was tested with 3 replicates per batch, resulting in 6 replicates in total.

To extract organic acids and sugars from fermented milk, a 5 g sample is homogenized in 10 mL of ultrapure water with 0.1% phosphoric acid (H_3PO_4) and agitated at 13,500 rpm for 20 seconds using an IKA® ULTRA-TURRAX®. The mixture is then centrifuged at 15,000 rpm for 20 min at 4 °C, and the supernatant is filtered through a 0.45 µm membrane filter (Millipore Corporation, Bedford, USA). The analysis follows the same procedure previously applied to analyzing organic acids and sugars in fruits and leaves.

Flavonoids compounds

To extract flavonoids, 0.5 g of fresh sample was combined with 4 mL of a methanol/water/formic acid solution (80:19.9:0.1, v/v). The analysis of flavonoids was carried out in accordance with the method previously detailed by Uysal et al. (2023). Extracts were analyzed on a Shimadzu LC–MS/MS 8050 triple quadrupole mass spectrometer equipped with an electrospray ionization (ESI) as source operating in negative and positive modes and a Shimadzu High Performance Liquid Chromatography (HPLC) system (Shimadzu, Kyoto, Japan). The HPLC equipment was used with a photodiode array detector (PDA) SPD-M40 (Shimadzu, Kyoto, Japan). The identification process was conducted using authentic reference standards, with retention times and UV-visible spectra compared against values reported in the literature. Flavonoid compounds were monitored at specific wavelengths: cyanidin 3,5-diglucoside (611.10 nm), quercetin-3-galactoside (465.00 nm), and quercetin-3-glucoside (463.25 nm). Quantification was performed via external calibration using reference standards.

Sensory analysis

A total of 60 habitual consumers of yogurt and fermented milk, ranging in age from 18 to 70, were recruited at CIAGRO (UMH, Orihuela, Spain). Participants evaluated overall satisfaction using a 9-point hedonic scale and assessed attribute intensity through a Just About Right (JAR) scale. To familiarize them with the base milk matrix, they first tasted a control sample without fig puree. The consumer study was conducted in accordance with the ethical guidelines outlined in the Declaration of Helsinki and received approval from the UMH research ethics committee.

Statistical analysis

Statistical analyses were conducted using StatGraphics Plus version 5.0 (Manugistics, Inc., Rockville, MD, USA), XLSTAT (Addinsoft, Paris, France), and SPSS 24.0 (IBM SPSS Statistics, Chicago, IL, USA). Data analysis included one-way

and two-way analysis of variance (ANOVA) to evaluate statistical differences ($p < 0.05$) between varieties, locations, and sample types. Tukey multiple range test was applied for mean comparisons at a 95% confidence level.

Additionally, principal component analysis (PCA) was used to create regression maps, allowing the projection of samples based on significant variables. All analyses were conducted in triplicate, and results were expressed as mean values ($n = 3$) ± standard error (SE).

06
PUBLICATIONS

PUBLICATION 1:

FICUS CARICA FRUITS, BY-PRODUCTS AND BASED PRODUCTS AS POTENTIAL SOURCES OF BIOACTIVE COMPOUNDS: A REVIEW.

Teruel-Andreu, C.,

Andreu-Coll, L.,

López-Lluch, D.,

Sendra, E.,

Hernández, F.,

Cano-Lamadrid, M.

Agronomy, 11(9) (2021)

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Review

Ficus carica Fruits, By-Products and Based Products as Potential Sources of Bioactive Compounds: A Review

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Abstract: In this review, studies (n = 41) were searched in which the compounds and contents were determined for whole fig fruit, peel, leaves and pulp, the types of fig-based products were identified and their total phenols and antioxidant capacity as well as the potential uses of different extracts of fig parts were analyzed. There is a need to reduce the fruit's environmental impacts (zero waste), and bioactive compounds from fig fruits present a high added value as functional ingredients. Focusing on fig by-products (peel, seeds, no-optimal fruits and leaves), individual compounds and/or extracts can increase the functional, nutritional and techno-functional properties of food products such as additives. A high number of phenolic compounds was found in whole fruit (n = 19), peel (n = 26), pulp (n = 24) and leaves (n = 42). Quercetin-3-O-rutioside was reported as the major individual phenolic compound in whole figs, while cyanidin-3-rutinoside, epicatechin and caftaric acid were the highest compounds in peel, pulp and leaves, respectively. A potential strategy could be the development of novel additives and/or ingredients for food industry from fig by-products. Therefore, the use and valorization of the waste material produced during fig processing should be further investigated.

Keywords: figs; leaf; revalorization; pulp; added-value

Citation: Teruel-Andreu, C.; Andreu-Coll, L.; López-Lluch, D.; Sendra, E.; Hernández, F.; Cano-Lamadrid, M. *Ficus carica* Fruits, By-Products and Based Products as Potential Sources of Bioactive Compounds: A Review. *Agronomy* **2021**, *11*, 1834. https://doi.org/10.3390/agronomy11091834

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1. Introduction

Ficus carica L. (fig) is a species of the very large number of the genus *Ficus* belonging to the Moraceae famil, characterized by milky latex in all parenchymatous tissue, unisexual flowers, anatropous ovules and aggregated drupes or achenes [1]. Figs are infruitescences—the true fruits of the fig are located inside the fig or siconio, which are called achenes [2]. The fleshy and sweet part of the fig corresponds to the flower receptacles that, after fertilization, become swollen and fleshy [3]. It is worth mentioning that it is one of the oldest species domesticated by humanity [4]. In the Middle East and the Mediterranean region, the fig has been included in the diet since ancient times and is considered a symbol of health [5]. It has been suggested that the cultivation of the fig originated in the East Mediterranean region, which was later expanded into the West Mediterranean area [6].

According to the Food and Agriculture Organization (FAO) of the United Nations, the world production of fig fruit is stable. Worldwide, the area under cultivation of fig trees exceeds 289,818 ha, with an estimated production of 1,315,588 t [7]. Turkey is the biggest world producer, with 310,000 t in 2019, followed by Egypt, Morocco, Iran, Algeria and Spain. Therefore, of utmost importance for fig production is still the Mediterranean basin and the Near East [7]. Spain is the main source of figs in Europe (51,600 t), followed by Greece (19,730 t) and Italy (11,830 t) [7]. In Spain, the main producer is Extremadura (37,382 t), followed by Cataluña (5834 t) and the Comunidad Valenciana (2932 t) [8]. Because the fig tree is highly resistant to salinity and active calcium, it is quite suitable for marginal areas, such as southeastern Spain [9]. Taking production, yield and size of the cultivars cultivated

in Spain into account, "Banane" and "Brown Turkey" are the main cultivars [10]. On the other hand, other authors indicated that the most important cultivars are "Cuello Dama Blanco" and "Colar de Elche", because they exhibit the best organoleptic punctuation due to their higher content of sugars [11].

A high number of bioactive compounds have been found in the peel, flesh, leaves and whole fruits of figs, such as cyanidin, chlorogenic acid, rutin, luteolin and (+)-Catechin, among others [2,12,13]. Several authors have indicated that these compounds present potential health properties, such as antibacterial, hepatoprotective, antidiabetic, anti-inflammatory, antioxidant and anticancer activity [14–16]. Therefore, consumer demand for fig fruit and fig-based products has increased in the past decades [17]. It is essential to highlight that no health claim is yet authorized for "antioxidants", "anthocyanins" or "fig" by the European Food Safety Agency (EFSA). There is just one authorized claim (for polyphenols): hydroxytirosol and derivatives in olive oil. On the other hand, the high concentration of calcium (133 mg/100 g) [1] in fig fruits allows to mention the nutritional claim "rich in calcium", because its content is higher than 10% of the RDI (recommended dietary intake).

Fig products have been used in traditional medicine to treat many diseases, mainly in the dermatological field [18]. Abbasi et al. [19] studied the application of fig plant extracts and showed their effectiveness in relieving symptoms of atopic dermatitis, and can, hence, be used instead of cortisones. Moreover, another study reported the potential of fig plant extract to be used as a treatment of and prevention for skin warts and cervical cancer [20]. Ongoing research suggests anticancer effects of two components of fig leaf extract, bergapten and psoralen, which could be a good source for developing drugs to suppress the growth of cancer cells [18]. Additionally, other studies concluded that figs are a concentrated source of benzaldehyde [21]. There are studies that also show the potential of fig extracts to produce medicines for cardiovascular diseases, by the content in components such as flavone, rutine and quercetin [22]. Additionally, fig fruits as well as leaves have a high nutritional value and their high content of dietary fiber is widely known [22]. Constipation is a very common health problem, and laxative foods such as figs and their derivatives could be considered effective for this problem [23]. Similar results were also reported in a previous study, in which fig leaf extracts were used to help combat eating and lifestyle disorders [24]. Additionally, Ajmal et al. [25] recognized the efficacy of fig leaf extracts for reducing blood glucose levels.

On the other hand, there is a need to reduce the fig's environmental impacts (zero waste) and fruit-based products present a high added value as functional ingredients. Among fruit-based products, peels, seeds, no-optimal fruits and leaves, among others, can be found. Focusing on fig by-products, the peel and leaf extracts could increase the nutritional and pharmaceutical properties of food products such as additives [4]. Therefore, there is a great need to generate comprehensive information about the bioactive compounds of fig fruits, their derivatives/by-products (peel, leaves and oil) and fig-based products. How the processing (drying and preparation of jams) and storage have affected the phenolic composition of fig products will also be an objective of this review.

2. Scientific Literature Review

This review is organized as a research paper. A scoping review was used to synthesize the evidence and assess the scope of the 41 studies on the topic. This review was based on the PRISMA Extension (PRISMA-ScR) approach [26] for Scoping Reviews. A comprehensive literature search—Scopus and ScienceDirect—was performed in August 2021 and was limited to articles published in English since 1990 (Figure 1). Text words and controlled vocabulary for several concepts (*Ficus carica*, by-products, bioactive compounds, fig, peel, leaves and revalorization) within the titles, abstracts and keywords were used. The main focus has been given to studies published in journals included in the Journal Citation Reports. Only research papers that included the experimental design and data treatment were selected. The structure of the review allows a dissection of (i) which compounds

and their content in whole fig fruit, peel, leaves and pulp (n = 12); (ii) types of fig-based products and their total phenols and antioxidant capacity (n = 16); and (iii) uses of different extracts of fig parts (n = 13).

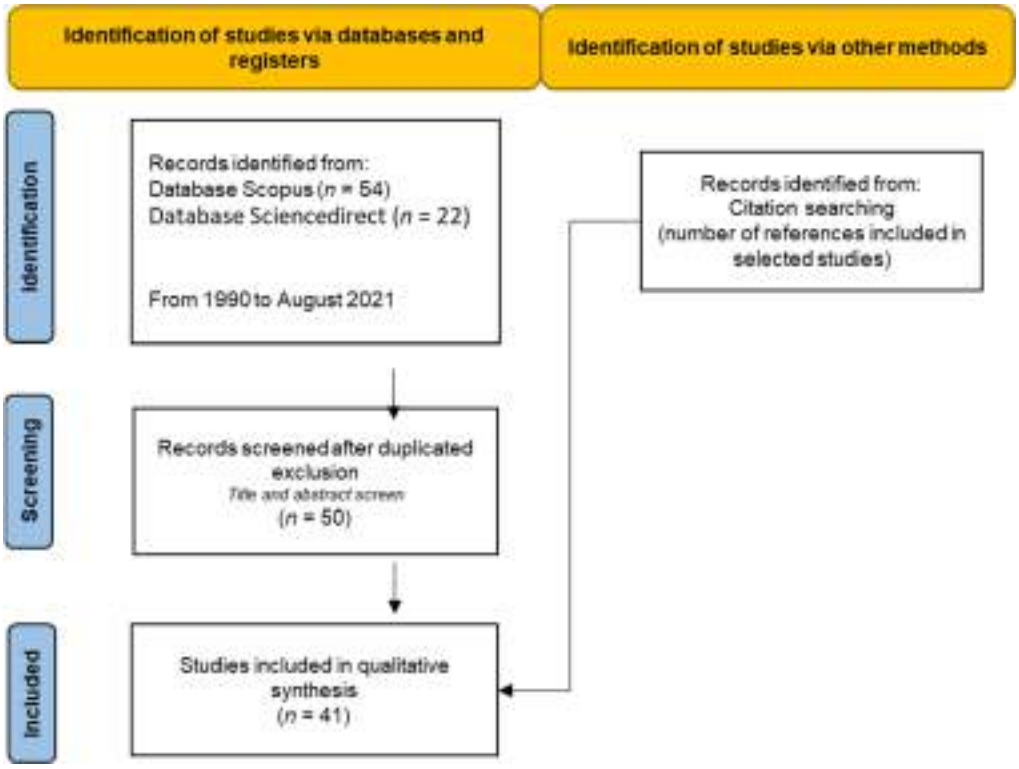

Figure 1. Flow diagram describing the study selection process of the scientific literature.

3. Bioactive Compounds in Different Fig Parts

Table 1 shows the individual phenolic compounds found in different part of fig parts: whole fruit (n = 19), peel (n = 21), pulp (n = 22) and leaves (n = 40). Identified compounds belong to different chemical families, such as phenolic acids (caffeic acid, chlorogenic acid, ferulic acid, coumaric acid, syringic acid, quinol and gallic acid) and flavonoids (catechin, kaempferol, quercitin and myricetin). The chemical structure of the main compounds found in fig fruits and derivatives are shown in recent studies related to chemical composition [14,16]. It is important to highlight that the results in the literature are not always reported in a unified manner, which makes it difficult to compare research findings. Data are expressed as dried matter/weight (dw) and fresh matter/weight (fw). Depending on the part of the fruit, maturity index, variety and region, the type of compounds varies. Quercetin-3-O-rutioside was reported as the major individual phenolic compound in whole figs (Table 1), followed by polymeric procyanidins, quercentin-3-glucoside, chlorogenic acid and cyanidin-3-0-rutinoside. As for the peel's bioactive compounds, cyanidin-3-rutinoside was the most abundant, followed by cyanidin-3,5-diglucoside, cyanidin-3-O-diglucoside, epitecatechin, catechin and quercetin-rutinoside. Epicatechin and cyanidin-3-rutinoside were the main compounds found in fig pulp, while caftaric acid, in the form of kaempferol 3-O-glucoside, was the main compound reported in fig leaves. On the other hand, Badgujar et al. [15] and Li et al. [14] reviewed the phytochemical composition of *Ficus carica* fruits and their derivatives. This study only indicated the profile of the bioactive compounds (isolation of phytosterols, anthocyanins, phenolic components and a few other classes of secondary metabolites), not the quantification. Therefore, these manuscripts were not added to Table 1. Most of these phytochemicals were found in latex, followed by leaves, fruit and root. Additionally, Li et al. [14] collected data of the phytochemical composition related to health properties, indicating that conventional and modern isolation and characterization techniques were used for the identification of about 126 chemical constituents, which were

divided into eight categories: hydroxybenzoic acids, hydroxycinnamic acids, flavonoids, coumarins, furanocoumarins, volatile constituents, triterpenoids and miscellaneous [14].

Table 1. Bioactive compounds and their content (minimum and maximum) in fig whole fruits, peel, pulp and leaves.

Plant Part	Compound	Minimun Value	Maximum Value	Unit	Reference
Whole figs					
	Catechin	0.2060	0.9570	(mg g^{-1} dw)	[12]
		0.0127	0.1670	(mg g^{-1} fw)	[6]
	Epicatechin	0.0900	0.4310	(mg g^{-1} dw)	[12]
		0.0058	0.3210	(mg g^{-1} fw)	[6]
	Polymeric procyanidins	0.5560	2.6800	(mg g^{-1} dw)	[12]
	Rutin	0.0489	0.2870	(mg g^{-1} fw)	[27]
		0.0089	0.2870	(mg g^{-1} fw)	[6]
	Cyanidin-3,5-*O*-diglucoside	0.0000	0.0190	(mg g^{-1} dw)	[12]
	Cyanidin-3-*O*-rutinoside	0.0040	1.1620	(mg g^{-1} dw)	[12]
	Pelargonidin-3-*O*-rutinoside	0.0000	0.0380	(mg g^{-1} dw)	[12]
	Chlorogenic acid	0.0880	1.2450	(mg g^{-1} dw)	[12]
		0.0105	0.0157	(mg g^{-1} fw)	[6]
	keampferol-3-glucoside	0.0013		(mg g^{-1} fw)	[6]
	keampferol-3-*O*-rutinoside	0.0060	0.3050	(mg g^{-1} dw)	[12]
	Quercetin 3-glucoside	0.0041	1.4020	(mg g^{-1} fw)	[6]
	Quercetin-3-*O*-rutioside	0.3990	3.2830	(mg g^{-1} dw)	[12]
	Quercetin-3-galactoside	0.0460	0.1420	(mg g^{-1} dw)	[12]
	Quercetin-3-*O*-malonyl-galactoside	0.0530	0.5800	(mg g^{-1} dw)	[12]
	Apigenin-*C*-hexoside-pentoside	0.0050	0.2430	(mg g^{-1} dw)	[12]
	Gallic acid	0.0010	0.0038	(mg g^{-1} fw)	[27]
		0.0030	0.0280	(mg g^{-1} fw)	[6]
	Syringic acid	0.0002	0.0010	(mg g^{-1} fw)	[27]
	Ellagic acid	0.0020		(mg g^{-1} fw)	[6]
	Syringic acid	0.0003	0.0008	(mg g^{-1} fw)	[6]
Peel					
	Catechin	0.0220	0.2060	(mg g^{-1})	[10]
		0.0009	0.0239	(mg g^{-1} dw)	[28]
	Epicatechin	0.0350	0.2570	(mg g^{-1})	[10]
		0.0027	0.0547	(mg g^{-1} dw)	[28]
	(epi)catechin-(4-8)-Cy 3-rutinoside	0.0004	0.0009	(mg g^{-1} fw)	[29]
	Carboxypyrano-Cy 3-rutinoside	0.0005	0.0013	(mg g^{-1} fw)	[29]
	Cyanidin-3,5-diglucoside	0.0020	0.0052	(mg g^{-1} fw)	[29]
		0.0008	0.4941	(mg g^{-1} dw)	[28]
	Cyanidin-3-malonylglycosyl-5-glucoside	0.0005	0.0014	(mg g^{-1} fw)	[29]
	Cyanidin-3-glucoside	0.0015	0.0154	(mg g^{-1} fw)	[29]
		0.1100	0.0060	(mg g^{-1} fw)	[2]
	Cyanidin-3-rutinoside dimer	0.0001	0.0083	(mg g^{-1})	[10]
		0.0004	0.0009	(mg g^{-1} fw)	[29]
	Cyanidin-3-malonylglucoside	0.0006	0.0035	(mg g^{-1} fw)	[29]
	Pg 3-rutinoside	0.0005	0.0035	(mg g^{-1} fw)	[29]
	Cyanidin-3-*O*-rutinoside	0.0079	0.1050	(mg g^{-1})	[10]
		0.0154	0.0783	(mg g^{-1} fw)	[29]
		0.2410	1.0890	(mg g^{-1} fw)	[2]
	Pelargonidin-3-*O*-rutinoside	0.0008	0.4787	(mg g^{-1} dw)	[28]
		0.0000	0.0107	(mg g^{-1})	[10]
		0.0042	0.0126	(mg g^{-1} dw)	[28]
	Chlorogenic acid	0.0005	0.0088	(mg g^{-1} dw)	[28]
		0.0200	0.0580	(mg g^{-1} fw)	[2]
	Luteolin-7- OGlucoside	0.0020	0.0260	(mg g^{-1})	[10]
		0.0019	0.0179	(mg g^{-1} dw)	[28]
	Luteolin 6C-hexose-8C-pentose	0.0010	0.0190	(mg g^{-1} fw)	[2]
	Kaempferol-rutinoside	0.0020	0.0070	(mg g^{-1} fw)	[2]

Table 1. *Cont.*

Plant Part	Compound	Minimun Value	Maximum Value	Unit	Reference
	Quercetin	0.0009	0.0595	(mg g^{-1} dw)	[28]
	Quercetine-acetilglucoside	0.0020	0.0170	(mg g^{-1} fw)	[2]
	Quercetin-rutinoside	0.0290	0.1580	(mg g^{-1} fw)	[2]
	Quercetine-glucoside	0.0020	0.0320	(mg g^{-1} fw)	[2]
	Ellagic acid	0.0150	0.0330	(mg g^{-1})	[10]
Pulp					
	Catechin	0.0140	0.0670	(mg g^{-1})	[10]
	Epicatechin	0.0140	0.1330	(mg g^{-1})	[10]
	(epi)catechin-(4-8)-Cy 3-glucoside	0.0000	0.0001	(mg g^{-1} fw)	[29]
	(epi)catechin-(4-8)-Cy 3-rutinoside	0.0000	0.0006	(mg g^{-1} fw)	[29]
	Cyanidin-3,5-diglucoside	0.0001	0.0006	(mg g^{-1} fw)	[29]
	Cyanidin-33-malonylglycosyl-5-glucoside	0.0000	0.0001	(mg g^{-1} fw)	[29]
	Cyanidin-33-glucoside	0.0005	0.0022	(mg g^{-1} fw)	[29]
	Cyanidin-33-rutinoside	0.0045	0.0102	(mg g^{-1} fw)	[29]
	Cyanidine-3-*O*Rutinoside	0.0008	0.0105	(mg g^{-1} dw)	[28]
	Cyanidin-33-malonylglucoside	0.0000	0.0001	(mg g^{-1} fw)	[29]
	Carboxypyrano-Cy 3-rutinoside	0.0000	0.0009	(mg g^{-1} fw)	[29]
	Pelargonidin-3-rutinoside	0.0000	0.0001	(mg g^{-1} fw)	[29]
	Pn 3-rutinoside	0.0000	0.0001	(mg g^{-1} fw)	[29]
	Chlorogenic acid	0.0010	0.0130	(mg g^{-1} fw)	[2]
		0.0010	0.0140	(mg g^{-1})	[10]
	Quercetinrutinoside	0.0040	0.0170	(mg g^{-1} fw)	[2]
	Cyanidin-3-rutinoside	0.0100	0.0950	(mg g^{-1} fw)	[2]
	Cyanidin-3-*O*-glucoside	0.0001	0.0083	(mg g^{-1})	[10]
	Cyanidin-3-*O*-rutinoside	0.0079	0.1050	(mg g^{-1})	[10]
	Pelargonidin-3-*O*-rutinoside	0.0001	0.0107	(mg g^{-1})	[10]
	Quercitin-3-*O*-rutinoside	0.0010	0.0190	(mg g^{-1})	[10]
	Quercitin-3-acetylglucoside	0.0010	0.0140	(mg g^{-1})	[10]
	Ellagic acid	0.0070	0.0140	(mg g^{-1})	[10]
Leaf					
	(+)-catechin	0.5200	0.7400	(mg g^{-1} dw)	[30]
	Caffeoylmalic acid	0.7900	5.9700	(mg g^{-1} dw)	[30]
		1.3860	7.4650	(mg g^{-1} dw)	[13]
	p-Coumaroyl derivative	0.3920	0.7130	(mg g^{-1} dw)	[13]
	p-Coumaroylquinic acid	0.3500	1.3710	(mg g^{-1} dw)	[13]
	p-Coumaroyl malic acid	0.3380	0.7740	(mg g^{-1} dw)	[13]
	Caffeic acid derivates	0.4240	0.5920	(mg g^{-1} dw)	[13]
	Caffeic acid	2.4800		(mg g^{-1} dw)	[31]
	Isoschaftoside	0.1420	0.9910	(mg g^{-1} dw)	[13]
	Schaftoside	0.0940	0.5180	(mg g^{-1} dw)	[13]
	Kampherol	0.8800		(mg g^{-1} dw)	[31]
	kaempferol 3-*O*-glucoside (astragalin)	12.4300	22.7000	(mg g^{-1} dw)	[30]
		0.0400	0.3890	(mg g^{-1} dw)	[13]
	Kaempferol derivative	0.0190	0.0600	(mg g^{-1} dw)	[13]
	Quercitin	13.4000		(mg g^{-1} dw)	[31]
	Quercetin derivative	0.0480	0.2110	(mg g^{-1} dw)	[13]
	Rutin (quercetin-3-*O*-rutinoside)	3.7200	7.5100	(mg g^{-1} dw)	[30]
		0.0097	0.6874	(mg g^{-1} fw)	[27]
		1.6480	8.2180	(mg g^{-1} dw)	[13]
	Quercetin 3-*O*-glucoside (isoquercetin)	5.3600	12.4500	(mg g^{-1} dw)	[30]
	Quercetin 3-*O*-malonyl-glucoside	0.1640	2.6210	(mg g^{-1} dw)	[13]
	Isoquercetin	0.0760	1.5460	(mg g^{-1} dw)	[13]
	Gallic acid		1.5000	(mg g^{-1} dw)	[31]
	Psolaren	0.3620	1.4920	(mg g^{-1} dw)	[13]
	Bergapten (5 methoxypsolaren)	0.4450	0.6270	(mg g^{-1} dw)	[13]
	Psolaric acid isobar	0.3440	0.4710	(mg g^{-1} dw)	[13]

Table 1. *Cont.*

Plant Part	Compound	Minimun Value	Maximum Value	Unit	Reference
	3-*O*-caffeoylquinic acid (chlorogenic acid)	0.0020		(mg g^{-1} dw)	[31]
		1.3100	3.5400	(mg g^{-1} dw)	[30]
		0.3400	0.5900	(mg g^{-1} dw)	[30]
	5-*O*-caffeoylquinic acid	0.4050	2.0610	(mg g^{-1} dw)	[13]
		0.4736	1.158.8	(mg g^{-1} dw)	[32]
	Ferulic acid	0.0320		(mg g^{-1} dw)	[31]
		11.9838		(mg g^{-1} dw)	[32]
	Pyrogallol	0.0060		(mg g^{-1} dw)	[31]
	Quinol	0.0110		(mg g^{-1} dw)	[31]
	p-Hydroxy benzoic acid	3.5000		(mg g^{-1} dw)	[31]
	Dihydroxybenzoic acid	1.1500	2.1500	(mg g^{-1} dw)	[30]
	Vanillic acid	0.0790		(mg g^{-1} dw)	[31]
	Syringic acid	0.0970		(mg g^{-1} dw)	[31]
	o-Coumaric acid	0.0110		(mg g^{-1} dw)	[31]
	p-Coumaric acid	0.0130		(mg g^{-1} dw)	[31]
	Benzoic acid	0.3200		(mg g^{-1} dw)	[31]
	Caftaric acid	40.2000		(mg g^{-1} dw)	[31]
	Ellagic acid	0.5240		(mg g^{-1} dw)	[31]
	Salicylic acid	0.0450		(mg g^{-1} dw)	[31]
	Myricetin	0.4140		(mg g^{-1} dw)	[31]
	Rosmarinic acid	0.2700		(mg g^{-1} dw)	[31]
	Ligstroside	0.1880		(mg g^{-1} dw)	[31]

4. Bioactive Content of Fig-Based Products and Their Antioxidant Activity

In general, fig fruits have mainly been consumed fresh and dried, but they have also traditionally been preserved and processed into jams [21]. Nowadays, consumer trends have changed and there is an increase in the range of other products based on figs [4]. Table 2 shows important information (type, cultivar and treatment) and bioactive compounds (total phenols, total flavonoids and total anthocyanins) as well as the antioxidant capacity of products based on fig fruits and their by-products. Additionally, the following lines include more information on the main reported fig-based products.

Table 2. Fig cultivar, total phenols, total flavonols and total anthocyanins, and the antioxidant capacity of the reported fig-based products.

Sample	Variety/Origin	Treatment	Total Phenols	Total Flavonoids	Total Anthocyanins	Antioxidative Capacity		References
Fig jam	Khudeiri	0 [a]	291.42 ± 44.9 (mg GAE kg^{-1})	nd	16.45 ± 1.2 (mg cya-3-glu kg^{-1})	DPPH	15.52 ± 0.5 (%)	[33]
Fig jam	Khudeiri	1 [a]	235.45 ± 2.6 (mg GAE kg^{-1})	nd	13.70 ± 1.0 (mg cya-3-glu kg^{-1})	DPPH	13.71 ± 0.3 (%)	[33]
Fig jam	Khudeiri	2 [a]	233.57 ± 0.5 (mg GAE kg^{-1})	nd	13.80 ± 1.1 (mg cya-3-glu kg^{-1})	DPPH	15.11 ± 2.0 (%)	[33]
Fig jam	Khudeiri	3 [a]	140.30 ± 5.3 (mg GAE kg^{-1})	nd	11.95 ± 1.7 (mg cya-3-glu kg^{-1})	DPPH	13.52 ± 0.4 (%)	[33]
Fig jam	Khudeiri	4 [a]	145.90 ± 13.2 (mg GAE kg^{-1})	nd	13.45 ± 0.1 (mg cya-3-glu kg^{-1})	DPPH	12.35 ± 0.5 (%)	[33]
Fig jam	Khudeiri	5 [a]	130.97 ± 2.6 (mg GAE kg^{-1})	nd	11.20 ± 0.6 (mg cya-3-glu kg^{-1})	DPPH	8.96 ± 2.1 (%)	[33]
Dry figs	"Bela petrovka"	Drying oven	530.2 mg (GAE kg^{-1} dw)	nd	nd		nd	[34]
Dry figs	Serbia		195.33 ± 1.07 (mg/100 g dm)	nd	nd	ABTS	0.388 ± 0.042 (mmol/100 g DM)	[35]
Dry figs	Turkey		19.2 (mg/100 g fw)	nd	nd		nd	[2]
Dry figs	Spain		19.1 (mg/100 g fw)	nd	nd		nd	[2]
Dry figs	Cuello dama		17.8 (mg/100 g fw)	nd	nd		nd	[2]
Dry figs	Saoudi douiret	Direct solar dryer	201.76 mg (GAE/100 g DM)	112.28 mg (QE/100g DM)	nd	DPPH	418.51 mg (TEAC/100 g DM)	[36]
Dry figs	Bayoudhi douiret	Direct solar dryer	73.74 mg (GAE/100 g DM)	57.96 mg (QE/100g DM)	nd	DPPH	131.55 mg (TEAC/100 g DM)	[36]
Dry figs	Mission	Freeze drying [b]	3.08 ± 0.4 (mg CEg^{-1})	nd	nd	DPPH	2.0 ± 0.3 (μM eq trolox g^{-1})	[37]
Dry figs	Mission	Drying 45 °C [b]	3.35 ± 0.2 (mg CEg^{-1})	nd	nd	DPPH	3.4 ± 0.3 (μM eq trolox g^{-1})	[37]
Dry figs	Mission	Drying 55 °C [b]	3.23 ± 0.3 (mg CEg^{-1})	nd	nd	DPPH	3.7 ± 0.2 (μM eq trolox g^{-1})	[37]
Dry figs	Mission	Drying 65 °C [b]	3.72 ± 0.2 (mg CEg^{-1})	nd	nd	DPPH	3.8 ± 0.3 (μM eq trolox g^{-1})	[37]
Dry figs	Mission	Freeze drying [c]	3.08 ± 0.4 (mg CEg^{-1})	nd	nd	DPPH	2.0 ± 0.3 (μM eq trolox g^{-1})	[37]
Dry figs	Mission	Drying 45 °C [c]	2.62 ± 0.2 (mg CEg^{-1})	nd	nd	DPPH	3.5 ± 0.3 (μM eq trolox g^{-1})	[37]
Dry figs	Mission	Drying 55 °C [c]	3.13 ± 0.3 (mg CEg^{-1})	nd	nd	DPPH	3.4 ± 0.3 (μM eq trolox g^{-1})	[37]
Dry figs	Mission	Drying 65 °C [c]	4.73 ± 0.7 (mg CEg^{-1})	nd	nd	DPPH	3.4 ± 0.7 (μM eq trolox g^{-1})	[37]
Fermented figs	Mission		4.77 (mg GAE/g of dm)	nd	nd	DPPH	0.53 (mg of GAE/g of dm)	[38]
Biscuit	Turkey	5% Fig seed [d]	145.28 ± 0.34 (mg GAE/100 g)	nd	nd	DPPH	10.36 ± 0.04 (%)	[39]
Biscuit	Turkey	10% Fig seed [d]	163.21 ± 0.16 (mg GAE/100 g)	nd	nd	DPPH	17.48 ± 0.09 (%)	[39]
Biscuit	Turkey	15% Fig seed [d]	76.84 ± 0.44 (mg GAE/100 g)	nd	nd	DPPH	25.36 ± 0.07 (%)	[39]

Table 2. *Cont.*

Sample	Variety/Origin	Treatment	Total Phenols	Total Flavonoids	Total Anthocyanins	Antioxidative Capacity		References
"Shir Anjir"	Iran	(13/0) [e]	No tested	No tested	No tested		No tested	[40]
"Shir Anjir"	Iran	(16.5/0) [e]	No tested	No tested	No tested		No tested	[40]
"Shir Anjir"	Iran	(20/0) [e]	No tested	No tested	No tested		No tested	[40]
"Shir Anjir"	Iran	(13/0.35) [e]	No tested	No tested	No tested		No tested	[40]
"Shir Anjir"	Iran	(16.5/0.35) [e]	No tested	No tested	No tested		No tested	[40]
"Shir Anjir"	Iran	(20/0.35) [e]	No tested	No tested	No tested		No tested	[40]
"Shir Anjir"	Iran	(13/0.7) [e]	No tested	No tested	No tested		No tested	[40]
"Shir Anjir"	Iran	(16.5/0.7) [e]	No tested	No tested	No tested		No tested	[40]
"Shir Anjir"	Iran	(20/0.7) [e]	No tested	No tested	No tested		No tested	[40]
Power fig	Iran	FP 707 [f]	No tested	No tested	No tested		No tested	[41]
Power fig	Iran	FP 505 [f]	No tested	No tested	No tested		No tested	[41]
Powerfig	Iran	FP 354 [f]	No tested	No tested	No tested		No tested	[41]
Powder figs	Cuello dama	Peel	4.78 ± 0.28 (mg GAE g^{-1})	17.61 ± 0.45 (mg RE g^{-1})	6.21 ± 0.28 (mg CGE g^{-1})	DPPH	9.50 ± 0.11 (%) [g]	[42]
Powder figs	Colar	Peel	5.76 ± 0.13 (mg GAE g^{-1})	19.12 ± 0.04 (mg RE g^{-1})	16.63 ± 0.85 (mg CGE g^{-1})	DPPH	21.48 ± 0.53 (%) [g]	[42]
Powder figs	Cuello dama	Pulp	2.67 ± 0.01 (mg GAE g^{-1})	13.51 ± 0.14 (mg RE g^{-1})	nd	DPPH	8.98 ± 0.08 (%) [g]	[42]
Powder figs	Colar	Pulp	1.92 ± 0.07 (mg GAE g^{-1})	9.24 ± 0.22 (mg RE g^{-1})	nd	DPPH	4.12 ± 0.10 (%) [g]	[42]
Smoothie	Colar	40% F + 60% Mo [h]	nd	nd	43.1 (mg/100 g fw)	ABTS	0.68 (mmol Trolox/100 g fw)	[43–45]
Smoothie	Colar	40% F + 60% W [h]	nd	nd	75 (mg/100 g fw)	ABTS	1.01 (mmol Trolox/100 g fw)	[43–45]
Smoothie	Colar	60% F + 40% Mo [h]	nd	nd	41.9 (mg/100 g fw)	ABTS	0.72 (mmol Trolox/100 g fw)	[43–45]
Smoothie	Colar	60% F + 40%W [h]	nd	nd	50.8 (mg/100 g fw)	ABTS	0.66 (mmol Trolox/100 g fw)	[43–45]
Wine	Brown turkey	HT-winess [i]	651 ± 12 (mg L^{-1})	126 ± 2 (mg L^{-1})	5.9 ± 0.5 (mg L^{-1})	DPPH	22.4 ± 0.9 (%) [j]	[46]
Wine	Brown turkey	Co-winess [i]	679 ± 9 (mg L^{-1})	135 ± 6 (mg L^{-1})	6.5 ± 0.3 (mg L^{-1})	DPPH	31.9 ± 1.3 (%) [j]	[46]
Wine	Brown turkey	HT-winedf [i]	705 ± 15 (mg L^{-1})	110 ± 7 (mg L^{-1})	3.0 ± 0.2 (mg L^{-1})	DPPH	25.2 ± 1.2 (%) [j]	[46]
Wine	Brown turkey	Co-winedf [i]	731 ± 9 (mg L^{-1})	116 ± 5 (mg L^{-1})	3.0 ± 0.1 (mg L^{-1})	DPPH	29.6 ± 0.8 (%) [j]	[46]
Wine	Hunan, China	WA:PF 1:7 [k]	725.58 ± 11.45 (mg L^{-1})	124.39 ± 3.36 (mg L^{-1})	148.94 ± 2.67 (mg L^{-1})	DPPH	88.21 ± 0.23 (%)	[47]
Wine	Hunan, China	PF:HU 3:1 [k]	682.67 ±16.13 (mg L^{-1})	180.7 ± 1.79 (mg L^{-1})	115.17 ± 4.96 (mg L^{-1})	DPPH	84.65 ± 0.54 (%)	[47]
Wine	Hunan, China	WA:HU 7:1 [k]	744.07 ± 9.81 (mg L^{-1})	143.58 ± 2.67 (mg L^{-1})	116.80 ± 1.35 (mg L^{-1})	DPPH	86.51 ± 0.42 (%)	[47]
Wine	Hunan, China	WA:PF:HU 3:1:3 [k]	765.20 ± 5.51 (mg L^{-1})	158.07 ± 0.71 (mg L^{-1})	142.37 ± 3.72 (mg L^{-1})	DPPH	88.65 ± 0.10 (%)	[47]
Wine	Hunan, China	Saccharomyces 1012 [k]	735.86 ± 8.15 (mg L^{-1})	115.74 ± 0.76 (mg L^{-1})	90.49 ± 0.70 (mg L^{-1})	DPPH	86.37 ± 0.34 (%)	[47]
Wine	Hunan, China	uninoculated [k]	538.35 ± 26.65 (mg L^{-1})	112.72 ± 1.84 (mg L^{-1})	52.50 ± 2.21 (mg L^{-1})	DPPH	6.62 ± 0.23 (%)	[47]

[a] Storage period (months), [b] ground figs, [c] half cut fig, [d] Wheat flour was replaced by fig seed powder at levels of 0%, 5%, 10% and 15%, [e] Dried fig (13%, 16.5% and 20%) and carboxymethylcellulose (CMC) (0%, 0.35% and 0.7%), [f] FP: Fig Powder; 707, 505 and 354: different particle size of fig powder based on micrometer-sized particles, [g] 10 mg/mL of sample, [h] Mo, Mollar de Elche pomegranate juice; W, Wonderful pomegranate juice; F: Fig purée, [i] Fig wine with/without prefermentation heating (HT-wineff/Co-wineff), and dried fig wine with or without prefermentation heating (HT-winedf/Co-winedf), [j] 50 mg/L of gallic acid, [k] Yeast strain/ Inoculation proportion/Abbreviations: HU, *Hanseniaspora uvarum*; PF, *Pichia fermentans*; WA, *Wickeramomyces anomala*.

The bioactive compound content and antioxidant activity strongly depend on the cultivar type in both fresh and processed fruits [48]. Khadhraou et al. [36] studied the main phenolic compounds, as well as the phenolic profiles and antioxidant activity, in nine sun-dried fig cultivars with different skin colors, originating from South-Eastern and Middle Eastern Tunisia [36]. For all evaluated parameters, a considerable variability with high significant differences was observed among the cultivars studied and the principal component analysis showed three groups of cultivars based on their similarity level. Dark cultivars contained the highest levels of flavonoids and phenolics and exhibited a high antioxidant capacity, while light-skinned cultivars contained the lowest levels. A recent study suggest that the preparation of fig jam preserves some bioactive compounds, especially carotenoids and phenolic compounds during storage [49]. On the other hand, Rababah et al. [33] studied the total phenolics and anthocyanins of fig jam after five months of storage and concluded that jam processing decreased the total phenolics (by 68.6%) and anthocyanins (by 60.2%). The minimum value to total phenolics and anthocyanins was 130.97 mg GAE kg^{-1} and 11.20 mg kg^{-1} of cyanidin-3-glucoside, respectively (Table 2).

As for dried figs, Slatnar et al. [34] showed results of total phenolics after a drying treatment. The drying process affected the degradation of phenolic compounds, the content of phenolic compounds being higher in fresh figs, followed by oven-dried figs and sun-dried figs. For example, Vallejo et al. [5] showed that around 15% of the total phenolics were lost in the drying processes in figs "Cuello Dama". Not only is the quality important, but safety is essential to be maintained. Mycotoxins have been found in quantities above the recommended limit in commercial samples of dry figs [50]. Therefore, a controlled drying process helps to reach a safety level. Alternatives to traditional sun drying is necessary for improving the protection of public health [51].

Nowadays, there is an increase on developing nutrient-rich value-added products by partially replacing its ingredients by others, such as underutilized fruits and added value by-products (pectins, colorants, emulsifier and antioxidants) from leaves and peels. As for fig by-products, Table 2 shows the reported inclusion alternatives; for example, fig powder as a colorant in the production of buns and muffins [52]. The authors also reported how the addition of fig seed powder to the formulation of a cookie improved its fiber content and also increased the total phenolic content and antioxidant activity [39]. Additionally, fig by-products' sweet extracts have been used for making traditional desserts without adding sugar, for example "Shir Anjir", an Iranian dessert [40]. Minimally processed fruits, such as smoothies, retain a large number of phytochemicals and they could in fact be considered a valid alternative to eating fresh fruits. Moreover, De Pilli et al. [53] found that the polyphenol content and antioxidant activity are strongly correlated in both fresh fruits and smoothies. In the same way, fig and pomegranate smoothies also showed a correlation between anthocyanins content and antioxidant capacity; smoothies with 60% of wonderful pomegranate juice showed a higher anthocyanin and antioxidant capacity (Table 2) [43–45]. Other studies have also suggested that fig leaf extracts presented a potential use as a source of natural antioxidant compounds [54]. Lu et al. [3] has noted that dried fig wines had lower contents of anthocyanins than fresh fig wines, which could be because of the thermal degradation of anthocyanins during the fig drying process. These wines also showed a lower antioxidant capacity.

Recent studies suggested that by-products/co-products obtained from peel and fig pulp showed potential properties to be used as ingredient in food products/additives (Tables 1–3). Table 3 shows the reported research about different raw fig by-product materials (different plant parts, peel, leaves and whole figs) and the extraction method used to obtain the desired ingredients/additives and their uses. For instance, peel extract could be used as a colorant due to its potential source of anthocyanin. Consequently, fig peel extract has great potential to be used as a natural food dye, where in addition to its ability to add natural purple colors, it also presents interesting antioxidant and antimicrobial activities. Table 3 also shows the extraction and uses of pectin from fig peel and pulp [55].

Table 3. Different potential uses of underutilized fruits and extracts of fig by-products.

Plant Part	Extract	Method	Uses	References
Peel	Lyophilized powdered	Extracted with 100 mL of acidified solvent 100% etanol	Natural purple colorants	[56]
	Lyophilized powdered	Heat-assisted extraction Microwave-assisted extraction Ultrasound-assisted extraction	Bioactive anthocyanin pigments	[57]
	Pectin	Hot-water extraction Ultrasound-assisted extraction Microwave-assisted extraction	The strong antioxidant and emulsification capacities	[58]
Leaves	Aqueous extract	Finely ground leaf powder suspended in 96 mL deionized water filtered by sterilized membrane filter, concentrated by using a rotary evaporator at 50 °C and followed by drying in an oven at 50 °C	Prolong the shelf life of pasteurized milk	[31]
	Powdered	Ethanol and chloroform were used as extracting solvents	Milk-clotting activity, which is most likely due to an enzyme component	[59]
	Fresh Leaf	Fig leaf extract, 96% ethanol. Using the maceration method	Antibacterial activity of fig leaf extracts	[60]
	Powdered	10 g of the finely divided leaf particles was dissolved in 200 mL of deionized water in a 500 mL flat bottom flask	Synthesis of eco-friendly and sustainable nanoparticles	[61]
	Fresh leaves and stems of the wild fig	Simple and chemical-free method (crushed and centrifuged).	Clotting ability in goat's fresh cheese production	[62]
	Powdered	Surfactant (PEG8000)-based microwave-assisted extraction method	Source of bioactive compounds	[63]
	Powdered	0.1 g of sample and 10 mL aqueous 50% acetone, centrifuged using Eppendorf centrifuge and filtered with a 0.22 µm PTFE syringe filter.	Source of bioactive compounds	[54]
Whole figs	Syrup	100 g of low-quality dried fig fruits were soaked in 500 mL distilled water, mixed and then centrifuged to remove solids.	Pullulan gum production from low-quality fig syrup using Aureobasidium pullulans	[64]
	Powdered	Samples (1 g) were mixed with ethanol (50 mL) and left macerating for 24 h; then, solutions were centrifuged (6800× *g*/20 min) and extraction was repeated three times.	Source of bioactive compounds	[65]
	Dry fig and stevia extract	Microwave-assisted extraction of stevia	Sugar replacement in ice cream	[66]

Regarding leaf extracts, El Dessouky Abdel-Aziz et al. [31] suggested that they can be used to extend the shelf life of pasteurized milk from 5 to 16 days without altering organoleptic properties. Moreover, other authors have reported that leaf extracts or fig powder can be a potential product for manufacturing functional foods [46] (Table 3). Fermentation is also known to promote the concentration of bioactive compounds of fruits and vegetables [55].

5. Conclusions

Although there has been an increase in research focused on the bioactive compounds of fig fruits and their by-products, more scientific evidence (combined with a unified way

of publishing data on bioactive compound content) is needed to establish the potential health properties. Future investigations should be focused on in vitro and in vivo studies to reveal their beneficial properties. There is scientific research about the potential use of underutilized fig fruit and figs by-products and its bioactive compounds as nutritional, functional and techno-functional properties. The use and valorization of the waste material (leaves, peel and pulp) produced during fig processing should be further investigated, since this could offer financial benefits to farmers and solve environmental issues by ensuring the sustainable management of these materials and, furthermore, bringing benefits to consumers' health and well-being. In addition, an economic estimation of the bioactive compounds of fig by-products could be essential to gain more knowledge and obtain added value. Although fig-based products and their uses were reported, such as smoothies, fig powders, colorants, fermented drinks and biscuits, among others, in the future, other products should be researched, for instance: fig coffee, dried figs using novel technologies and fermented milks based on fig by-products.

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PUBLICATION 2 (Open Access):

HOW DOES CULTIVAR AFFECT SUGAR PROFILE, CRUDE FIBER, MACRO- AND MICRONUTRIENTS, TOTAL PHENOLIC CONTENT, AND ANTIOXIDANT ACTIVITY ON *FICUS CARICA* LEAVES*?*

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How Does Cultivar Affect Sugar Profile, Crude Fiber, Macro- and Micronutrients, Total Phenolic Content, and Antioxidant Activity on *Ficus carica* Leaves?

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Abstract: The objective of this research was to evaluate the effect of the cultivar on the nutritional and functional parameters of *Ficus carica* leaves. This information will provide the basis for their potential use and future incorporation in other food matrices as food ingredients. Sucrose, glucose, and fructose were detected in all fig leaves, with mean values of 48.94, 66.74, and 43.70 g kg^{-1} dried weight (dw), respectively. The crude fiber range was between 6.53% and 22.67%, being an interesting source of fiber. The most abundant macronutrient was calcium (Ca), followed by potassium (K) and magnesium (Mg). All cultivars showed high concentrations of iron (Fe). *Ficus carica* leaves can be a good material for obtaining extracts rich in fiber and calcium and provide an alternative source of these compounds to be incorporated into other nutraceutical and/or food matrices.

Keywords: fig; bioactive compounds; functionality; mineral content

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1. Introduction

Today, there is an interest in searching for new sources of natural antioxidants due to the demand for healthier and "closer to zero waste" products [1]. Therefore, recent research has focused on the study of the nutritional composition and biological properties of different nonedible parts of plants, which can be called biomass waste. This biomass is rich in plant secondary metabolites, including bioactive compounds that play a key role in the defense against physiological and environmental stimulators, and the adaptation of plants to their environment. These compounds have extensive applicability in human health [2]. New sources of ingredients that are primary metabolites (sugars, fiber, etc.) are also of interest. All mentioned extracts/compounds may be used to increase the functional and techno-functional properties of new food matrices. They also can be of use in the pharmaceutical industry. According to Karim et al. (2012) [3], there are at least 14 groups of plant secondary metabolites with nutraceutical potential distributed in different anatomical parts of plants. For this reason, many authors are focusing their research on the study of leaves, for example, the leaves of *Citrullus colocynthis* [4], *Anredera cordifolia* leaves [2], *Arbutus unedo* leaves [1], *Mulberry leaves* [5], *Moringa olifeira* leaves [6,7], and other fruit tree leaves such as apple, pear, quince, apricot, peach, plum, sour cherry and sweet cherry [8].

The fig tree (*Ficus carica* L.; *F. carica*) is the most well-known *Ficus* species plant in the *Moraceae* family and is native to the Sub-Himalayan region and central India, although it is widely farmed around the world. *F. carica* is a species that has been widely farmed for its fruit and nutritional values [9]. The leaves are stipulated and petiolated with obovate, nearly orbiculate or ovate leaf blade, palmately lobed, cordate base, undulate or irregularly dentate margin, acute to obtuse apex, and scabrous-pubescent surfaces [10]. The therapeutic properties of *F. carica* have been used in traditional medicine practices such as Ayurveda,

Unani, and Siddha [11]. Fruits, roots, and leaves are used in traditional medicine to treat various conditions [12]. Recently, *F. carica* has been included in occidental Pharmacopoeias (i.e., Spanish Pharmacopoeia, British Pharmacopoeia) and therapeutic guides based on herbal medicines, such as the Physician's Desk Reference for Herbal Medicines (2000) [13]. Some recent research suggested the anticancer activities of *F. carica* leaf extracts [14] and another study found positive effects of the extract of *F. carica* leaves on liver cancer and colon cancer [9]. Also, another study showed that the ethanol extract of *Ficus carica* leaves promotes cancer cell death [15]. Other studies investigated and evaluated *F. carica* leaf extracts and found promising biological activities, such as hepatoprotective activity [10,11], the hypoglycemic effect [11], hypocholesterolemic activity of the decoction of leaves [11,16], hypolipidemic activity [10,11], strong antimicrobial activities [10,11], free radical scavenging activity [11], anti-HSV effect [10,11] and immunostimulant properties [11].

The modern pharmaceutical and food industry considers biomass waste as an almost infinite resource for functional product development [10]. For this reason, many authors are focusing their research on the analysis of *F. carica* leaves. A recent review compilated at least 40 bioactive compounds in *F. carica* leaves [17]. Some studies demonstrated that the phenol content in *F. carica* leaves is higher than that in either red wine or tea [18]. Some studies have reported the phenolic profile of fig leaves, which is composed of seven phenolic compounds, namely 3-CQA [3-*O*-caffeoylquinic acid], 5-CQA [5-*O*-caffeoylquinic acid], Q-3-Glu [quercetin 3-*O*-glucoside], Q-3-rut [quercetin 3-*O*-rutinoside], ferulic acid, psoralen, and bergapten. Other works have shown that in *F. carica* leaves, rutin, umbelliferone, and psoralen were the most abundant flavonoids, followed by coumarin and furanocoumarin compounds [18]. On the other hand, the presence of volatile compounds mainly distributed in *F. carica* leaves were alcohols, ketones, esters, sesquiterpenes, and norisoprenoid [13]. Therefore, due to the health-promoting potential of these compounds, the valorization of leaves with sustainable technologies to recover these high-value-added ingredients and their utilization in novel food formulation developments should be further investigated [19]. Until now, different potential uses of *F. carica* leaves have been reported and compilated in a recent review such as their use in nanoparticles, as antibacterial extracts, and as an additive for pasteurized milk for increasing shelf-life, among others [17].

For all the above-mentioned reasons, the objective of this research was to evaluate the effect of the cultivar on the nutritional and functional parameters of *Ficus carica* leaves. This information will provide the basis for their potential use as additives or extracts by the food and pharmaceutical industries. This is the first study that compares and characterizes the nutritional and functional parameters of the leaves of the *Ficus carica* of four dark varieties (the most relevant from the commercial point of view of southeastern Spain).

2. Materials and Methods

2.1. Vegetal Material

In this study, the leaves of four biferous varieties: San Antonio (SA), Colar ©, Cuello Dama Negra (CDN), and Superfig (SF) of *Ficus carica* were collected from the experimental field of the Universidad Miguel Hernández de Elche (UMH) in the province of Alicante Spain (02°03′50″ E, 38°03′50″ N). The Colar variety was collected in two zones: (i) in the above-mentioned coordinates (Colar UMH, CUMH) and, (ii) in a commercial plot in Albatera, Alicante, southern (CA) Spain (0°55′49″ W, 38°13′17″ N). The leaves were collected from 20-year-old trees. Fig trees were trained to a vase-shaped system and planted at a spacing of 8 m × 5 m. They were drip irrigated and were subjected to the standard farming practices (pruning, thinning, fertilization, and pest control treatments). Thirty leaves were collected one week after the fruit had been collected in May and July 2021, respectively. Leaves were moved to the laboratory for processing.

2.2. Leaf Characterization

Leaf characterization was done following The International Plant Genetic Resources Institute guidelines [20]. A sample size of 30 adult leaves per variety (10 random leaves per

tree) was characterized. All of them were taken from all tree orientations and middle parts of shoots, and only healthy and undamaged ones were selected. The parameters assessed were leaf length (from the base of the petiole to the tip of the central lobe, expressed in cm), leaf width, petiole length, and length of the central lobe (Figure 1).

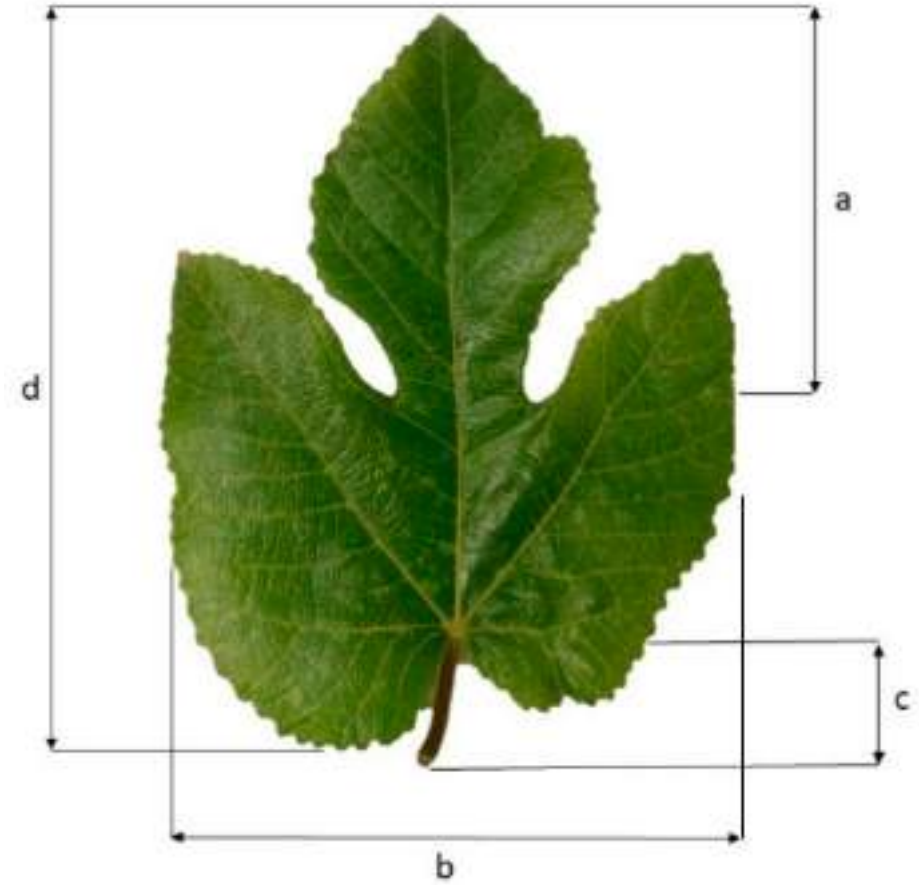

Figure 1. Representation of the measures considered to determine the size of the leaves, where "a" represents length of central lobe (cm); "b" leaf width (cm); "c" petiole length (cm); and "d" leaf length (cm).

2.3. Sugar Profile

Sugars were identified and quantified as previously described by Cano-Lamadrid et al. [21], with some modifications. Freeze-dried samples finely ground (0.2 g) were homogenized with 5 mL of phosphate buffer 50 mM (pH = 7.8) and centrifuged at 15,000× *g* for 15 min at 4 °C (Sigma 3–18 K; Osterode and Harz, Germany), Then, 1mL of supernatant was filtered through a 0.45 µm Millipore filter (Billerica, MA, USA) before HPLC analysis. Then, 10 µL were injected into a Hewlett-Packard high-performance liquid chromatography (HPLC) series 1100 (Hewlett-Packard, Wilmington, DE, USA). The elution system consisted of 0.1% phosphoric acid with a flow rate of 0.5 mL min^{-1}. The sugars were eluted through a Supelco column [SupelcogelTM C-610H column (30 cm × 7.8 mm)] coupled with a Supelguard column (5 cm × 4.6 mm, Supelco, Inc., Bellefonte, PA, USA) and detected with a refractive index detector (Hewlett-Packard, series 1100, G1362A, Wilmington, DE, USA). Analyses were run in triplicate, and results were expressed as g kg^{-1} dry weight (dw).

2.4. Crude Fiber, Macro- and MicroNutrient Content

Crude fiber content was determined according to methodology established by the Spanish Ministry of Agriculture, Fisheries, and Food as previously described by Sánchez et al. [22] using an ANKOM200/220 fiber analyzer (ANKOM Technology, Macedon, NY, USA). Each sample was tested in triplicate, and results were to be expressed as % dw. To determine the mineral content, 0.2 g of freeze-dried leaves were digested in a microwave (MARS

ONE, 240/50 CEM) reaching 200 °C in 15 min and held at this temperature for 15 min after the addition of 10 mL of concentrated, 65% (*w*/*v*), HNO_3. Later, samples were filtered with quantitative filter paper and transferred to a volumetric flask, and dilutions 1:10, 1:20, and 1:60 in the case of potassium were prepared using ultrapure deionised water, 18 MΩ (Milli-Q® system; Millipore Corporation, Madrid, Spain). Determination of macronutrients (Ca, Mg, and K) and micronutrients (Cu, Fe, Mn, and Zn) in previously mineralised samples was performed using an (ICPMS-2030, Shimadzu). Each sample was tested in triplicate, and results were to be expressed as g kg^{-1} dw for macroelements (Ca, Mg, K, and Na) and as mg kg^{-1} dw for microelements (Fe, Cu, Mn, and Zn).

2.5. Antioxidant Activity (AA) and Total Polyphenols Content (TPC)

Freeze-dried samples (leaves) (0.5 g) were blended with 10 mL of MeOH/water (80:20, *v*/*v*) + 1% HCl, sonicated at 20 °C for 15 min, and left at rest for 24 h at 4 °C. Later, the extract was again sonicated for 15 min and centrifuged at 15,000 rpm for 10 min. This extract was used to measure antioxidant activity by three methods (ABTS, DPPH, and FRAP methods) and total polyphenolic content (TPC). The radical scavenging activity was assessed using the DPPH radical (2,2-diphenyl-1-picrylhydrazyl) method, as described by Brand-Williams et al. (1999) [23], while the ABTS [2,2-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid)] radical cation and ferric reducing antioxidant power (FRAP) methods were measured as previously described by Re et al. (1995) [24], and Benzie et al., (1996) [25], respectively. All analyses were done using a UV-visible spectrophotometer (Helios Gamma model, UVG 1002E, UK). A calibration curve (3.5–5.0 mmol Trolox L^{-1}) with good linearity ($R^2 \geq 0.999$) was used for the quantification. Analyses were performed in triplicate and results were expressed as mM Trolox dw. Total polyphenols content (TPC) was quantified using Folin–Ciocalteu reagent as described by Singleton et al. (1965) [26]. Absorption was measured using a UV–visible spectrophotometer (Helios Gamma model UVG 1002E, Helios, Cambridge, UK). All determinations were performed in triplicate and results were expressed as grams of gallic acid equivalent (GAE) per kilogram of dw.

2.6. Statistical Analyses

Data were analyzed using StatGraphics Plus, version 5.0 (Manugis-tics, Inc., Rockville, MD, USA). A one-way analysis of variance (cultivar as factors) was performed and mean values were compared by Tukey's multiple range test. Also, a one-way analysis of variance (location as a factor) was performed, and mean values were compared by Tukey's multiple range test.

3. Results and Discussion

3.1. Leaf Characterization

Table 1 shows the morphometrics parameters of the studied *F. carica* leaves. As to size, significant differences were found among cultivars, being CA cultivar, presented the highest value of leaf length, leaf width, length of the central lobe, and petiole length (27.46, 21.78, 14.55, and 10.54 cm, respectively). Taking the values of the morphometrics parameters of the rest of the cultivars into account, the range of leaf length, leaf width, length of central lobe, and petiole length was 19.65–22.71 cm, 17.38–21.04 cm, 10.16–12.64 cm, and 8.16–8.98 cm, respectively. Our results agreed with other authors that the size depends on the cultivar. Abdelsalam et al. (2019) [27] obtained differences in the leaf sizes of several *F. carica* cultivars, the smallest leaf length and leaf width size was 'Komesrey-El-Hammam' (5.4 and 6 cm), and the greatest leaf length and width values were found for 'Abodey-Giza' (23.5 cm) and Black_Mission (23.0 cm), respectively. Almajali et al. (2012) [28] showed leaf length values of 'Kortomanee', 'Byadee', 'Kdaree', 'Ajlounee', and wild fig cultivars (25.78, 20.95, 18.80 cm, 15.3 cm, and, 14.6 cm, respectively). While, other authors [29] obtained a lower ranged value in the case of leaf lengths of 6.20–13.80 cm, leaf widths of 4.10–15.30 cm, and petiole lengths of 1.68–5.80 cm. For leaf length, the CA variety obtained higher values than those found in the literature, while for leaf width the data obtained in this study

are within the range of those found in the literature. These differences may be due to the different genotypes and to environmental factors. Many studies have shown that water stress reduces leaf area. On the other hand, when location factor was considered, significant differences were found for leaf morphological characteristics between CA and CUMH. The CA cultivar presented a larger leaf length, leaf width, length of the central lobe, and petiole length size than the CUMH cultivar. Another study [6] using mulberry leaves, collected in Orihuela, the same city as the UMH plot is located, where some fig trees used in this study are located, found that the mean values for leaf length and leaf width were lower than the mean values obtained in this study (1.66 and 2.02-fold lower), respectively. According to the results obtained by these authors and our results, cultivar, and crop location affect leaf size.

Table 1. Mean values of morphological characteristics of leaves of different varieties of *Ficus carica*.

Variety [a]	Size (cm)			
	Leaf Length	Leaf Width	Length of Central Lobe	Petiole Length
	ANOVA Test [b]			
	***	***	***	***
	Tukey's Multiple Range Test [c]			
SA	21.70 ± 0.35bc	21.04 ± 0.71ab	10.94 ± 0.30c	8.16 ± 0.29b
CA	27.46 ± 0.56aA	21.78 ± 0.39aA	14.55 ± 0.35aA	10.54 ± 0.39aA
CUMH	19.65 ± 0.52dB	17.38 ± 0.67cB	10.16 ± 0.26cB	8.64 ± 0.35bB
CDN	22.71 ± 0.42b	19.09 ± 0.27bc	12.64 ± 0.27b	8.98 ± 0.32b
SF	20.77 ± 0.36cd	18.60 ± 0.30c	10.52 ± 0.28c	8.71 ± 0.23b

[a] SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negra; SF: Superfig. [b] NS not significant at $p > 0.05$ and *** significant at $p < 0.001$, respectively. [c] Values (mean ± standard error; $n = 30$) followed by the same letter, within the same column, were not significantly different ($p > 0.05$), according to Tukey's least significant difference test. Lowercase letter shows significant differences among cultivars, and capital letter shows significant differences between location of the same cultivar.

3.2. Sugar Profile and Crude Fiber Content

Table 2 shows the sugars identified and quantified in leaves (fructose, glucose, and sucrose). Our results indicated that fructose was the major sugar in leaves (1.10 times higher than glucose and 1.84 times higher than sucrose) (calculations were made with the average of all the varieties). However, other authors reported that sucrose was the highest sugar found in *F. carica* leaves and that the total sugar concentration ranged from 10.7% (Fracasana cv.) to 20% (Kalamon cv.) [30]. Among cultivars, significant differences were found, being the highest value of sugars in the CA cultivar (159.38 g kg^{-1} dw). It should be clear that the same cultivar (Colar) between two locations (Albatera and UMH) showed differences, being attributed to the influence of agronomic factors such as differences in the irrigation of water (the UMH plot is irrigated with river water while the Albatera plot is irrigated with well water with a higher salt content), different location elevation (already mentioned in the plot description), and fertilization (no fertiliser was applied on the UMH plot while potash was used as fertilizer on the Albatera plot). The CA cultivar showed a 95.96, 14.77- and 3.55-times higher content than CUMH for sucrose, glucose, and fructose, respectively. It is essential to mention that the rest of the cultivars were in the same location with the same irrigation water and soil type, among other environmental conditions. Considering the values of total sugar content found in the *F. carica* leaves, these leaves could be used as natural sweeteners instead of other synthetic sweeteners.

As for crude fiber results, significant differences were observed between cultivars, being in the range between 6.53% and 22.67% for CUMH and CA, respectively. The author's hypothesis is the effect of water salinity on the content of pectin and fiber as mentioned above as the reason for the detected significant differences in the crude fiber content between both locations. These values are slightly lower than those reported by El Dessouky

Abdel-Aziz et al. [31]. The results showed that *F. carica* leaves can be a good source of fiber, especially if these results are compared with the crude fiber content of the fig fruit (2.2%) reported in another study (in this study Sultani fig trees' cultivar was used, the differences may be due to the cultivar) [32], being in agreement with results previously reported by Rusmadi et al. (2020) [33] for *F. carica* fruits of a different cultivar (0.88, 2.58, and 3.36%) [34]. It is important to mention that our results are expressed as crude fiber, this being necessary for future research to focus on total soluble and insoluble dietary fiber. The addition of fig leaf powders in other food matrices could be a good strategy to enhance fiber content to reach fiber contents suited to the mentions of 'high in fiber' or 'source of fiber'. In order to claim that a food is 'high in fiber' a minimum content of 6 g of fiber per 100 g of food is needed [35].

Table 2. Sugars profile [g Kg^{-1} dry weight (dw)] and crude fiber content (%) of different varieties of *Ficus carica* leaves.

Variety [a]	Sucrose	Glucose	Fructose	Crude Fiber
		ANOVA Test [b]		
	***	***	**	**
		Tukey´s Multiple Range Test [c]		
SA	0.47 ± 0.01b	4.47 ± 0.09b	12.43 ± 0.29b	16.75 ± 0.37a
CA	48.94 ± 5.74aA	66.74 ± 9.24aA	43.70 ± 10.02aA	22.67 ± 3.35aA
CUMH	0.51 ± 0.01bB	4.52 ± 0.03bB	12.32 ± 0.10bB	6.53 ± 0.35bB
CDN	0.52 ± 0.00b	4.61 ± 0.03b	12.53 ± 0.08b	19.3 ± 0.62a
SF	0.51 ± 0.01b	4.61 ± 0.04b	12.62 ± 0.11b	20.67 ± 0.1a

[a] SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negra; SF: Superfig. [b] NS not significant at $p > 0.05$; ** and *** significant at $p < 0.01$ and 0.001, respectively. [c] Values (mean ± standard error; $n = 30$) followed by the same letter, within the same column, were not significantly different ($p > 0.05$), according to Tukey's least significant difference test. Lowercase letter shows significant differences among cultivars, and capital letter shows significant differences between location of the same cultivar.

3.3. Mineral Content

Table 3 shows the mineral composition of the leaves of the five *F. carica* cultivars studied. Significant differences ($p < 0.05$) were found in the content of macro- and microminerals between all the varieties studied. The leaves of the SA cultivar obtained the highest contents for all the macro- and microelements except for Mn, which was the CA cultivar that showed the highest content. Leaves from all cultivars had a high content of calcium (Ca), ranging from 19.97 g kg^{-1} dw (CUMH) to 68.04 g kg^{-1} dw (SA), followed by potassium (K) ranging from 13.87 g kg^{-1} dw (CDN) to 18.63 g kg^{-1} dw (SA). A large variation was observed in the contents of magnesium (Mg) 2.57–8.46 g kg^{-1} dw and sodium (Na) 0.27–1.64 g kg^{-1} dw. Among the microminerals, the contents of copper (Cu) varied from 4.18–13.11 mg kg^{-1} dw; iron (Fe) varied from 201.07–342.21 mg kg^{-1} dw; manganese (Mn) varied from 33.07–66.56 mg kg^{-1} dw; and zinc (Zn) varied from 15.03–55.90 mg kg^{-1} dw. For macro- and microelement contents, taking the location factor into account, the only significant differences were found between CA and CUMH cultivars in Ca, Mg, Na, and Mn, being the CA cultivar, in which the highest content was found. There were few studies on the mineral content in *F. carica* leaves depending on the cultivar. Therefore, the results of this research could increase the knowledge of the effect of cultivar on the mineral content of leaves of the *Ficus carica* in the same soil conditions. Our results are in agreement with previous studies conducted by other researchers [31,36,37], in which calcium is found as the main mineral in the leaves of fig trees [31,36]. The values obtained by other authors were lower values than ours, being in the range of 13.98–15.70 g kg^{-1} dw. Calcium is a macroelement that adds a nutrient essential to the body's metabolism, and calcium deficiency is linked to osteoporosis [38]. The Codex Alimentarius, Guidelines for Use of Nutrition Claims states that solid foods must contain a calcium content of 15% of the nutrient reference value (NRV) of 800 mg of calcium to be labelled as a source of

calcium [39]. Therefore, the *Ficus carica* leaves supplementation of food matrices could be a good strategy to be able to use a nutritional claim. Regarding microelements, iron (Fe) showed a higher content of all microelements for leaves of the different cultivars tested. The differences found between the micronutrient contents in the different varieties may be due to the plants' tendency to accumulate greater amounts of micronutrients when there is a greater vegetative growth, therefore the lower the vegetative growth, the lower the concentration of elements [40].

Table 3. Macro (Ca, K, Mg, and Na; g Kg^{-1} dw) and microelements (Cu, Fe, Mn, and Zn; mg Kg^{-1} dw) of *Ficus carica* leaf.

	Macroelements				Microelements			
Variety [a]	**Ca**	**K**	**Mg**	**Na**	**Cu**	**Fe**	**Mn**	**Zn**
				ANOVA Test [b]				
	***	*	***	***	***	**	***	***
				Tukey´s Multiple Range Test [c]				
SA	68.04 ± 1.37a	18.63 ± 1.10a	8.46 ± 0.51a	1.64 ± 0.08a	13.11 ± 0.77a	342.21 ± 26.16a	60.13 ± 3.83a	55.90 ± 5.33a
CA	28.32 ± 0.30bA	17.24 ± 0.72abA	3.17 ± 0.07cA	0.32 ± 0.01bA	4.18 ± 0.29bA	226.35 ± 15.10bA	66.56 ± 3.76aA	15.23 ± 1.22bA
CUMH	19.97 ± 1.06cB	15.03 ± 0.72bA	2.57 ± 0.09dB	0.27 ± 0.00cB	4.73 ± 0.04bA	201.07 ± 11.25bA	33.07 ± 1.36bB	18.73 ± 1.63bA
CDN	23.87 ± 0.46bc	13.87 ± 0.18c	4.58 ± 0.08b	0.40 ± 0.03b	4.44 ± 0.68b	225.37 ± 3.13b	37.91 ± 0.37b	15.03 ± 2.58b
SF	26.72 ± 0.12b	18.41 ± 3.12a	3.03 ± 0.16c	0.43 ± 0.02b	5.80 ± 0.07b	275.21 ± 9.21ab	58.56 ± 1.68a	22.29 ± 0.42b

[a] SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negra; SF: Superfig. [b] NS not significant at $p > 0.05$; *, **, and ***, significant at $p < 0.05$, 0.01, and 0.001, respectively. [c] Values (mean ± standard error; $n = 30$) followed by the same letter, within the same column, were not significantly different ($p > 0.05$), according to Tukey's least significant difference. Lowercase letter shows significant differences among cultivars, and capital letter shows significant differences between location of the same cultivar.

3.4. Antioxidant Capacity and Total Phenolic Content

Table 4 shows the antioxidant capacity by three method assays and the total phenolic content by a Folin assay. The radical-scavenging activity by the ABTS assay of fig leaves' cultivars revealed the highest antioxidant activity was shown by cultivar SF (52.43 mM Trolox dw) followed by fig cultivars CDN (52.07 mM Trolox dw), SA (44.91 mM Trolox dw), CUMH (42.46 mM Trolox dw), and CA (33.81 mM Trolox dw). On the other hand, significant differences were found only between the CA and CUMH cultivars for the ABTS method. The DPPH assay of fig leaves' cultivars showed the highest activity to fig cultivar SF (72.45 mM Trolox dw), followed by fig cultivars CUMH (70.14 mM Trolox dw), CA (68.84 mM Trolox dw), CDN (59.27 mM Trolox dw), and SA (52.54 mM Trolox dw respectively). Regarding FRAP, the order of cultivars from highest to lowest was CDN (124.79 mM Trolox dw) > SF (115.66 mM Trolox dw) > CUMH (67.15 mM Trolox dw) > CA (60.70 mM Trolox dw) > SA (56.09 mM Trolox dw). It can be observed that the highest content of the antioxidant activity, measured by the three methods (sum of ABTS, DPPH, and FRAP methods) was for the SF cultivar, followed by CUMH, while the lowest antioxidant activity was for the SA cultivar. As to TPC, the highest TPC for cultivar CUMH (18.86 g GAE kg^{-1} dw), followed by SA (18.62 g GAE kg^{-1} dw), CDN (18.06 g GAE kg^{-1} dw), SF (17.83 g GAE kg^{-1} dw), and CA (16.64 g GAE kg^{-1} dw). The main phenolic compounds found in the leaves of the *Ficus carica* were compiled previously by Teruel-Andreu et al. (2021) [17], being phenolic acids (caffeoylmalic acid, 3-*O*-caffeoylquinic acid, 5-*O*-caffeoylquinic acid) and flavonols (quercentin and kaempherol derivatives), among others. Caftaric acid was the highest value reported in *Ficus carica* leaves. Although more studies about the individual phenolics that were found in the different Ficus carica leaves, the antioxidant activity can be correlated with these mentioned compounds. Mahmoudi et al. [41] also found that the total phenolics content varies depending on the varieties, detecting higher total phenolics content for the biferous "Dhokkar" variety followed by the uniferous "Hamra" (46.074 mg GAE g^{-1} dw and 42.889 mg GAE g^{-1} dw, respectively). Several studies have shown that the content of total phenols in fig leaves is influenced by the type of solvent used for extraction. Authors included in previous studies comparisons of different solvents since there are few scientific manuscripts related to this topic, specifically with the same solvent used. Thus, Ghazi et al. (2016) [36] indicated that TPC, DPPH, and FRAP values

were higher in the methanolic extract of *Ficus carica* leaf (412.37 mg GAE 100 g^{-1}, 63.29 and 131.39 mmol Fe^{2+} 100 g^{-1}, respectively), in comparison with the total phenolic content of water and methanol extracts. However. Gillani et al. (2012) [42] showed that the TPC of the water extract was highest as compared to the TPC of the methanol extract. Accordingly, extractions for antioxidant capacity and total phenolic content analysis in this study have been made with methanol extracts. On the other hand, other authors [43] studied the content of bioactive compounds with antioxidant capacity in the leaves of different fruit trees and found a high variation with results of α-tocopherol equivalents in a range of 74.14 (µg g^{-1} dw) in sweet cherry to 194.22 (µg g^{-1} dw) in apricot. They also found differences in the content of bioactive compounds with an antioxidant capacity between the leaves collected at two weeks after blooming and leaves collected at two weeks after fruits had been collected. Leaves collected two weeks after blooming had the highest contents of bioactive compounds with antioxidant activity. Thus, in the present study, the leaves were collected at the same time to see the effect of the cultivar only.

Table 4. Antioxidant activity (ABTS, DPPH, and FRAP; mM Trolox dw) and total polyphenol content (TPC; g GAE kg^{-1} dw) of *Ficus carica* leaves.

Variety [a]	ABTS	DPPH	FRAP	TPC
		ANOVA Test [b]		
	***	***	***	*
		Tukey´s Multiple Range Test [c]		
SA	44.91 ± 0.26b	52.54 ± 1.48b	56.09 ± 2.58c	18.62 ± 0.64a
CA	33.81 ± 1.08cB	68.84 ± 0.41aA	60.70 ± 1.70bcA	16.64 ± 0.57bA
CUMH	42.46 ± 1.03bA	70.14 ± 2.33aA	67.15 ± 1.98bA	18.86 ± 1.05aA
CDN	52.07 ± 0.11a	59.27 ± 1.67b	124.79 ± 1.14a	18.06 ± 0.22a
SF	52.43 ± 0.53a	72.45 ± 1.05a	115.66 ± 1.44a	17.83 ± 0.15ab

[a] SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negra; SF: Superfig. [b] NS not significant at $p > 0.05$; *and *** significant at $p < 0.05$ and 0.001, respectively. [c] Values (mean ± standard error; $n = 30$) followed by the same letter, within the same column, were not significantly different ($p > 0.05$), according to Tukey's least significant difference test. Lowercase letter shows significant differences among cultivars, and capital letter shows significant differences between location of the same cultivar.

3.5. PCA Analysis

For a better understanding of the relationships among the sixteen statistically significant variables studied for varieties of *F. carica* leaves, a PCA was carried out (linear dimensionality reduction method for processing of multivariate data) (Figure 2). This statistical test was run for all varieties studied. Figure 2 shows the first two components of the correspondence analyses PCA plot, which explained 80.51% of the variability in the data. The PCA explained analytical variables in two axes, F1 46.80% and F2 33.71%. The results showed three groups can be differentiated based on the position of the samples along the F1-axis, as can be seen (Figure 2). The first group included the CA cultivar which was linked with all the variables related to the sugar content (sucrose, glucose, and fructose). A second group consisted of the SA cultivar which was related to all mineral content variables. Finally, the rest of the cultivars CUMH, CDN, and SF appear grouped together with antioxidant capacity variables.

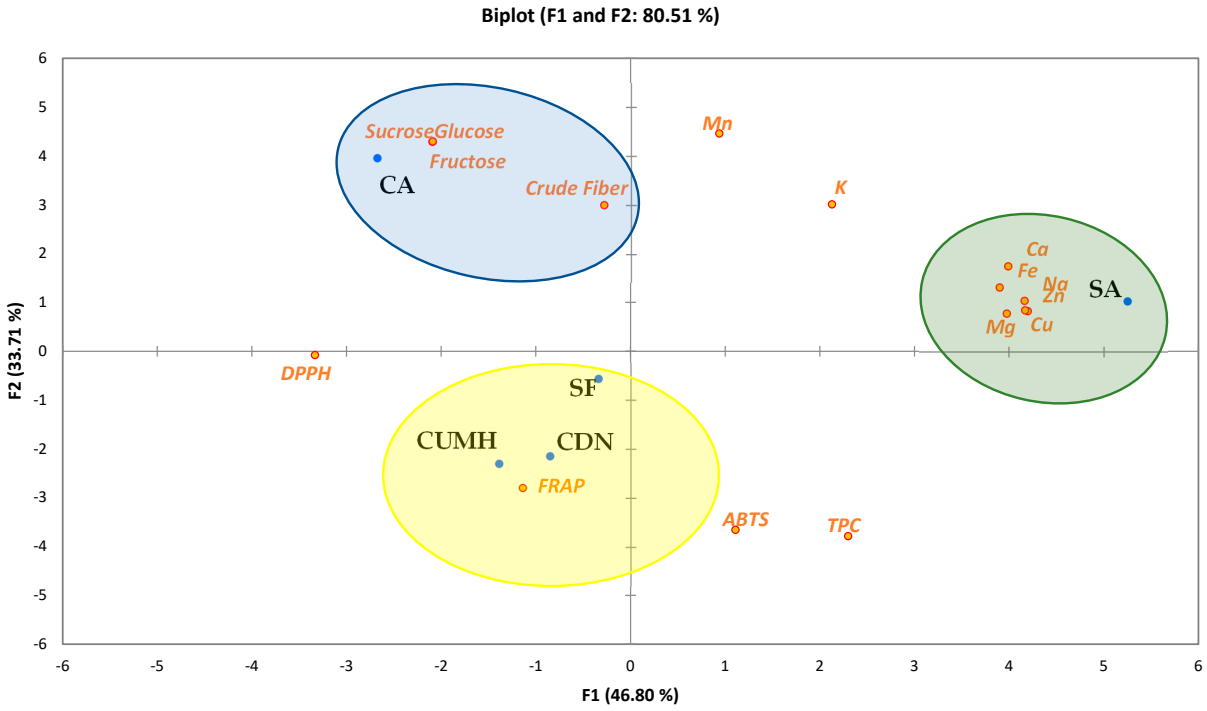

Figure 2. Differences between cultivars for all variables analysed in this study (PCA). Legend: ● Study varible ● Cultivar.

4. Conclusions

It is essential to highlight that this study is the first to study morphometric parameters, sugar profile, crude fiber, mineral concentration, and antioxidant activity in fig tree leaves grown in Southeastern Spain. Among the analyzed leaf cultivars, significant differences were found in all studied parameters. Also, both locations of the Colar cultivar affected the studied parameters. In conclusion, it could be said that Colar de Albatera showed the highest sugar content, being a potential sweetener. San Antonio cultivar presented the highest content of macro and mineral elements, being a suitable raw material to enrich another food matrix. Superfig and Cuello de Dama Negra cultivars presented the highest antioxidant capacity (ABTS and DPPH for Superfig; and FRAP for Cuello de Dama Negra). Future research should be carried out to know the specific bioactive compound which presents antioxidant capacity. Due to their functional compounds and properties, fig tree leaves could be used by the food industry for their health benefits and for pharmaceutical purposes. In addition, their use could offer benefits to farmers ensuring the sustainable management of this waste. For future research, the incorporation of this material and/or its extracts on food matrices would be of great interest.

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NUTRITIONAL AND FUNCTIONAL COMPOUNDS AND ANTIOXIDANT ACTIVITY OF EDIBLE AND NON-EDIBLE FRUIT PART OF BREBAS AND FIGS (*FICUS CARICA L.*)

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Nutritional and functional compounds and antioxidant activity of edible and non-edible fruit part of brebas and figs (Ficus carica L.) among different varieties

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ABSTRACT

Background: The objective of this work was to evaluate the effect of variety on the key nutritional and functional compounds (organic acids and sugars, dietary fiber, macro- and micro- elements, antioxidant activity and total phenols) and antioxidant activity on edible and non-edible fruit part (pulp and peel, respectively) from breba and fig fruits.

Results: Significant differences among variety were found in most of the parameters studied. Among pulps, the highest total sugar content and% dietary fiber was found in Colar de Albatera fig one. Taking the macro-elements content among peels into account, the greatest value was found in the San Antonio breba peel. On the other hand, both Cuello de Dama Negra breba and figs peel showed the highest values of ABTS, DPPH, FRAP and TPC. Principal component analysis (PCA) easily shows that sugars and organic acids, dietary fiber, mineral content, antioxidant activity, and total phenolic content are more correlated with the peel than with the pulp. Sugars content was higher in figs than in breba, while the percentage of total dietary fiber was higher in breba than in fig.

Conclusions: Finally, clear changes in nutritional and functional compounds and antioxidant activity values of edible and non-edible fruit parts were observed in this study among the different studied varieties. Also, higher values of key components were found in non-edible than edible fruit part. A potential strategy could be the development of novel additives/ingredients for food industry from non-edible fruit part or unharvested edible fig fruits since it can be catalogued such as fig by-products.

1. Introduction

The common fig (Ficus carica L.) tree belongs to the Eusyce section of the Moraceae. It is an unusual tree because it can produce multiple crops of fruits each year. "Biferous" varieties are those that are harvested twice a year, while the "uniferous" ones produce a single harvest (Núñez-Gómez et al., 2021). The breba crop, which is not produced in all varieties, is borne laterally on the growth of the previous season from buds produced in leaf axils. These buds are dormant figs that do not begin their development until the following spring and environmental factors such as winter temperature, photoperiod, and humidity affect specially the loss of breba (Flaishman et al., 2007).

According to the Food and Agriculture Organization (FAO) of the United Nations, the area under cultivation of fig trees exceeds 281,522 ha, with an estimated production of 1264,943 t (FAO Food and Agriculture Organization. FAOSTAT, 2022). In Spain, 14,599 ha and 51,598 tons were the total area and total production of figs/brebas in 2019. Related to fig/breba production, *Extremadura* is the highest producer (28,749), followed by *Cataluña* (5978) and *Comunidad Valenciana* (3502) (Ministerio de Agricultura, 2019) .

Recently the fresh breba and figs trade has gained economic importance because consumers are increasing demand of fig. But, due to its perishability, brebas and figs are not suitable for long term storage, so in order to avoid wasting the mature fruits there is a long tradition in Mediterranean countries of producing fig-based product such as dried figs, jams, syrups and fig spirits, therefore this fruit represents an

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important source of income, and those products offer a good way to reutilise and market poor-quality fig fruits (Rodríguez-Solana et al., 2018). It is important to highlight that the sustainable management of resources throughout production, industrialization, and consumption is one of the main concerns to be addressed to improve the world by 2030.

Nowadays, consumer trends have changed to a healthier and more sustainable consumption, and the industry and research are focusing on developing novel products, attracting the public and achieving higher consumer acceptance. It is worth mentioning that some categorized non-edible fruit part and/or by-products are rich in bioactive compounds and compounds with techno-functional properties. Previous research related to fig fruit observed that the fig peel contains bioactive compounds such as anthocyanins at higher levels than the edible fruit part (Backes et al., 2018). Various phytoconstituents such as amino acids, phytosterols, anthocyanins, organic acid, hydrocarbons, aliphatic alcohols, volatile components, fatty acids, phenolic components, etc. have been previously isolated from different parts of *Ficus carica* (Farooq et al., 2019). Although there is evidence related to the concentration of bioactive compounds differences between pulp and peel, more reports are needed (L Hssaini et al., 2021), for example taking the variety into account. Also, it is important to highlight that other author compared juices of *F. carica* peels, pulps and total fruits of different green varieties and the functionality of the juices influenced not only by the variety, but also by the fruit part. They concluded that it is necessary to increase the knowledge about dark figs, especially fig peel, which is a by-product, to be utilized a useful source of natural food preservative from a nutritional related point of view and other fig parts as a potential new source of natural antioxidants for food and pharmaceutical industries (Harzallah et al., 2016). Taking all above mentioned into account, the aim of this research was to know the effect of variety on the key quality parameters of peel and pulp from breba and fig fruits: i) physical-chemical characterization (weight, size, color), and ii) chemical (organic acids and sugars, fiber, minerals, antioxidant activity and total phenols).

2. Materials and methods

2.1. Plant material

In this study, brebas, and figs of five biferous varieties: San Antonio (SA), Colar de Albatera (CA), Colar UMH (CUMH), Cuello Dama Negra (CDN), and Superfig (SF) of *Ficus carica* were collected in the experimental field of Universidad Miguel Hernández de Elche (UMH) in the province of Alicante Spain (02° 03′ 50′' E, 38° 03′ 50′' N). Also, Colar variety was collected in two zones: i) in the above-mentioned coordinates (Colar UMH) and ii) in a commercial plot in Albatera, Alicante, southern Spain (Alicante, Spain; 0° 55′ 49′' W, 38° 13′ 17′' N). Fig trees were trained to a vase-shaped system and planted at a spacing of 8 $m \times 5$ m. They were drip irrigated and were subjected to the standard farming practices (pruning, thinning, fertilization, and pest control treatments). Thirty undamaged brebas (first crops) and figs (second crops) were randomly hand-harvested at commercial maturity stage (above 15°Brix and 9°Brix for brebas and fig, respectively) in May and July 2021, respectively.

2.2. Physicochemical parameters of breba and fig fruits

For each variety, a total of 30 fruits were evaluated from three different trees (10 fruits per tree). The parameters assessed in brebas, and fig fruits were whole weight, size (length and width), color, total soluble solids, pH and total titratable acidity. Fruit weight was accounted by a Sartorius digital bench scale (model AG204 scale; Mettler Toledo, Barcelona, Spain) 0.01 g accuracy. Fruit length and width were measured with an electronic digital calliper (model 500–197–20 150 mm; Mitutoyo Corp., Aurora, IL, USA) 0.01 mm accuracy. color measurement was made in the peel of the fruit on two opposite faces at the equatorial zone. The CIELab parameters were measured using a Minolta CR-300 Chroma Meter (Minolta Corp., Osaka, Japan) coupled to a Minolta DP-301 data processor. After the determination of the physical parameters, a portion of the plant material was squeezed to get the juice to analyze pH, total soluble solids (TSS), titratable acidity (TA), while other portion of fruits and peel were immediately frozen in liquid nitrogen and later freeze-dried in an Alpha 2–4 freeze drier (Alpha 2–4; Christ, Osterode am Harz, Germany) for 24 h at a pressure reduction of 0.220 mbar. Total soluble solids (TSS) were measured with a digital Atago refractometer (model N-20; Atago, Bellevue, Wash., U.S.A.) at 20 °C with values being expressed as°Brix. The titratable acidity (TA) and pH was determined by acid-base potentiometer (877 Titrino plus, Metrohm ion analyses CH9101, Herisau, Switzerland), using 0.1 N NaOH up to pH 8.1, values were expressed as g of citric acid per L^{-1}. Finally, the maturity index (MI) was calculated as the ratio between TSS/TA.

2.3. Organic acid and sugar profile

0.2 g of freeze-dried sample was homogenized with 5 mL of phosphate buffer 50 mM (pH = 7.8) and centrifuged at 15,000 x *g* for 15 min at 4 °C (Sigma 3–18 K; Osterode and Harz, Germany), Then, 1 mL of supernatant was filtered through a 0.45 μm Millipore filter (Billerica, MA, USA) before HPLC analysis. 10 μL was injected into a Hewlett-Packard high-performance liquid chromatography (HPLC) series 1100 (Hewlett-Packard, Wilmington, DE, USA). The elution system consisted of 0.1% phosphoric acid with a flow rate of 0.5 mL min $^{-1}$. The organic acids were eluted through a Supelco column [SupelcogelTM C-610H column (30 cm × 7.8 mm)] and a Supelguard column (5 cm × 4.6 mm, Supelco, Inc., Bellefonte, PA, USA) and detected by absorbance at 210 nm. For sugar determinations, the same HPLC, elution system, flow rate and columns were used. Sugars were detected with a refractive index detector (Hewlett-Packard, series 1100, G1362A, Wilmington Del., USA). Sugars and organic acids were determined in triplicate and results were expressed as g Kg^{-1} dry weight (dw).

2.4. Total dietary fiber

Total dietary fiber content was determined following the official methodology established by the Spanish Ministry of Agriculture, Fisheries and Food as previously described by Lipan et al. (Lipan et al., 2021) using an ANKOM200/220 fiber analyzer (ANKOM Technology, Macedon, NY, USA). Results were expressed as%.

2.5. Macro- and micro-mineral content

For determined the mineral content 0.2 g of sample of pulp and peel freeze dried, were digested in a microwave (MARS ONE, 240/50 CEM) reaching 200 °C in 15 min and holding at this temperature for 15 min after the addition 10 mL of concentrated, 65% (w/v), HNO_3. Later, samples were filtered with quantitative filter paper and transferred to volumetric flask and dilutions 1:10, 1:20 and 1:60 in the case of potassium were prepared using ultrapure deionised water, 18 MΩ (Milli-Q® system; Millipore Corporation, Madrid, Spain). Determination of macronutrients (Ca, Mg, and K) and micro-nutrients (Cu, Fe, Mn, and Zn) in previously mineralised samples was performed using an inductively coupled plasma mass spectrometer (ICP-MS), Shimadzu ICPS-2030 (Shimadzu Scientific Instrument, Inc., Columbia, MD, USA). Results were expressed as g kg^{-1} dry weight (dw) and mg kg^{-1} dw for macro-elements and micro-elements, respectively.

2.6. Antioxidant activity (AA) and total polyphenols content (TPC)

Freeze-dried samples (0.5 g) were mixed with 10 mL of MeOH/water (80:20, v/v) + 1% HCl, sonicated at 20 °C for 15 min and left for 24 h at 4 °C. Then the extract was again sonicated for 15 min, and centrifuged at 15,000 rpm for 10 min. The radical scavenging activity was evaluated

using the DPPH$^{\bullet}$ radical method, as previously described (Brand-Williams et al., 1995), while the ABTS$^{\bullet+}$ radical cation and ferric reducing antioxidant power (FRAP) methods were measured as previously described (Re et al., 1999; Benzie and Strain, 1999). Analyses were run in triplicate and results were expressed as mM Trolox dw. Total polyphenols content (TPC) was quantified using Folin–Ciocalteu reagent as previously described (Singleton et al., 1999). Results were expressed as grams of gallic acid equivalent (GAE) per kilogram of dw.

2.7. Statistical analyses

Analyses were run in triplicate and results were expressed as the mean value ($n = 3$) ± SE (standard error). Data was analysed using StatGraphics Plus, version 5.0 (Manugis-tics, Inc., Rockville, MD, USA). A one-way analysis of variance (variety as factor) was performed and means values were compared by Tukey's multiple range test. Principal component analysis (PCA regression map) was conducted to project the samples for brebas and for figs depending on studied variables with significant differences.

3. Results and discussion

3.1. Morphological characteristics

Table 1 shows weight, size and external color coordinates of Breba and fig fruits. Related to these parameters, significant differences were found among varieties.

As to Breba fruits, it can be said that SF Breba fruit was the heaviest, followed by CUMH. CA, SA and CDN presented similar values. The results of Breba fruits weight obtained in this study was in accordance with previous study (Núñez-Gómez et al., 2021).

Regarding with fig fruits, similar tendency was observed, being SF the heaviest, followed by CUMH and CA.

Breba fruits in all varieties were by far higher than fig fruits (1.5, 1.7, 2.2, 1.6 and 1.9-fold higher in SA, CA, CUMH, CDN and SF, respectively). Pulp percentage, the edible part of the fruit, ranged from 84.5 to 96.2, and from 88.7 to 93.8 in Breba and Fig fruit, respectively. It means that 4–15% of the Breba/fig fruit can be described as by-products, other authors indicated that peels could represent approximately 27% of the total weight of the fig fruit, generally discarded as industrial waste (Ayuso et al., 2022). Previous study indicated that the leaves and peels represent the main by-products resulting from fig production and are rich in bioactive molecules with potential nutritional value, including organic acids, phenolic acids, triterpenoids, flavonoids, tocopherols, and fatty acids, among others (Ayuso et al., 2022). It can be observed that statistical differences values were found in size parameters among varieties, being the ratio length/width since 1.3 to 1.8 and since 1.1 to 1.4 in Breba and Fig fruit, respectively. Breba fruits were more elongated while the figs had a more rounded shape.

The external color of the brebas and fig fruits showed variability among the varieties studied ($p > 0.05$). The luminosity coordinate (L*) was high and positive both for the external color of the brebas (values between 26.54 and 34.83). The order from the highest to the lowest value of a* was as follows: SF=CUMH > CA > CDN = SA. As to b* coordinate and C* in breba samples, the highest values were found in CA variety followed by SF >CUMH>CDN>SA. Values are in accordance with previous studies (Núñez-Gómez et al., 2021).

As to figs, the luminosity coordinate (L*) was high for the external color of the figs (values between 27.01 and 34.12). For the external color of figs, the order from the highest to the lowest value of a* was as follows: SF=CUMH>SA>CA=CDN. On the other hand, while the highest b* coordinate and C* values in fig samples were detected in CUMH, the lowest values of b* coordinate and C* were found in CDN variety.

3.2. Organic acid, sugar profile and total dietary fiber

Table 2 shows organic acid and sugar profile of pulp and peel of breba and fig fruit. As to breba fruits, no significant differences were detected in pulp and peel breba of malic acid among varieties (ranged between 910.62 and 2009.90 mg 100 g^{-1} fw). In agreement with other authors, citric acid was no detected in peel breba fruit (Oliveira et al., 2009), while it was detected in pulp Breba fruit, showing significant differences among varieties (216.52 and 756.25 mg 100 g^{-1} fw for CDN and CA, respectively).

As to fig, significant differences on the citric acid content among varieties were detected in pulp, while no differences were observed in fig peel.

Citric acid in pulp fig was lower 1.38 times than breba pulp, except CDN, being 2-fold higher in breba than fig. Related to malic acid, pulp of fig of CA and CDN varieties presented higher values than breba 1.12 and 1.64 times, respectively. While the fig peel of all varieties presented a total of 1.18 times lower than Breba. The organic acids detected in the

Table 1
Morphological characteristics and external color coordinates of Breba and Fig fruits from different varieties of *Ficus carica*.

Breba Variety[a]	Weight whole fruit (g)	Pulp (%)	Size Length (mm)	width (mm)	External color coordinates L*	a*	b*	c*
ANOVA test [b]	***	***	***	***	***	***	***	***
Tukey's Multiple Range test [c]								
SA	69.30±2.04c	84.49±0.89c	67.77±0.86d	51.73±0.73b	26.54±0.46b	4.61±0.47c	2.13±0.48b	5.27±0.62b
CA	83.41±2.55b	88.11±0.63b	87.45±1.66bc	47.45±0.56c	33.23±1.15a	6.88±0.67b	10.04±1.58a	13.37±1.38a
CUMH	115.75±3.55a	96.17±0.16a	91.64±1.18ab	57.91±1.02a	32.83±0.62a	9.82±0.50a	7.82±0.72a	13.13±0.48a
CDN	69.17±2.92c	95.79±0.20a	83.38±1.56c	46.38±0.62c	28.01±0.27b	6.42±0.31bc	2.58±0.32b	7.04±0.38b
SF	120.35±4.00a	94.76±0.33a	93.04±1.09a	59.94±0.81a	34.83±0.78a	10.40±0.55a	10.00±0.88a	15.23±0.51a
Fig								
ANOVA test [b]	***	***	***	***	***	***	***	***
Tukey's Multiple Range test [c]								
SA	46.10±1.47bc	92.26±0.47ab	48.56±0.83c	44.10±0.57b	28.76±0.36b	9.70±0.41b	3.11±0.24b	10.27±0.40b
CA	48.48±0.82bc	93.79±0.29a	60.48±0.91a	43.69±1.24b	27.01±0.25b	3.74±0.25c	0.24±0.15c	3.83±0.25c
CUMH	52.86±2.73b	89.38±0.68c	60.76±1.25a	45.50±0.95b	34.12±0.79a	15.65±0.61a	8.79±0.91a	18.54±0.68a
CDN	44.22±1.36c	91.54±0.55b	52.94±0.83b	40.01±0.49c	29.27±0.35b	3.48±0.29c	−0.15±0.21c	3.70±0.27c
SF	64.62±2.44a	88.66±0.49c	59.32±1.37a	52.32±1.07a	33.67±0.89a	15.31±0.58a	8.59±0.87a	18.00±0.74a

[a] SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negra; SF: Superfig.
[b] NS not significant at $p > 0.05$; *, **, and ***, significant at $p < 0.05$, 0.01, and 0.001, respectively.
[c] Values (mean ± standard error; $n = 25$) followed by the same letter, within the same column, were not significantly different ($p > 0.05$), according to Tukey's least significant difference test.

Table 2
Organic acids and sugars profile [g Kg^{-1} dry weight (dw)] and total dietary fiber (%) of different varieties of Breba (pulp and peel), and Fig (pulp and peel).

Breba Fruit						
Variety[a]	**Citric acid**	**Malic acid**	**Sucrose**	**Glucose**	**Fructose**	**Total dietary fiber**
ANOVA test[b]						
Pulp	***	NS	nd	***	***	***
Peel	nd[d]	NS	nd	NS	NS	*
Tukey's Multiple Range test[c]						
Pulp						
SA	14.76±0.61b	30.61±0.90	nd	4.74±0.01b	12.82±0.04b	8.74±0.27bc
CA	18.48±0.40a	26.20±0.62	nd	353.71±8.44a	354.47±8.79a	11.57±0.27a
CUMH	15.34±1.28ab	28.26±0.85	nd	336.75±9.02a	320.46±4.51a	8.47±0.57c
CDN	5.93±0.75c	24.94±6.11	nd	307.01±7.02a	305.41±1.86a	5.95±0.26d
SF	12.09±0.63b	30.14±1.19	nd	328.06±6.38a	308.87±6.61a	10.57±0.31ab
Peel						
SA	nd	29.14±0.72	nd	4.72±0.01	12.75±0.03	7.64±0.39c
CA	nd	28.11±0.45	nd	4.73±0.01	12.79±0.04	8.04±0.15bc
CUMH	nd	28.22±0.34	nd	4.66±0.02	12.65±0.06	13.77±1.72a
CDN	nd	32.00±0.71	nd	4.68±0.03	12.72±0.06	13.10±1.02ab
SF	nd	30.11±0.83	nd	4.67±0.02	12.65±0.06	12.04±0.35abc
Fig Fruit						
Variety[a]	Citric acid	Malic acid	Sucrose	Glucose	Fructose	Total dietary fiber
ANOVA test[b]						
Pulp	***	***	***	*	*	***
Peel	NS	NS	***	***	***	***
Tukey's Multiple Range test[c]						
Pulp						
SA	9.38±1.12b	21.35±2.42c	24.15±2.88bc	292.32±39.00b	285.34±7.73b	5.92±0.44b
CA	7.96±0.09b	29.47±0.45b	32.84±0.10a	379.33±6.03a	363.97±4.48ab	8.93±0.11a
CUMH	9.94±0.11b	26.21±0.09bc	20.81±0.37cd	384.37±3.61a	367.22±3.83a	7.11±0.20b
CDN	12.43±0.25a	40.90±0.45a	31.33±2.13ab	358.63±2.82ab	343.10±1.63ab	4.31±0.27c
SF	8.71±0.19b	25.61±0.32bc	15.57±0.35d	371.30±4.85ab	351.97±5.15ab	7.07±0.12b
Peel						
SA	10.88±0.27	23.61±0.87	11.32±1.25b	333.92±9.17a	326.12±9.27a	6.97±0.43ab
CA	10.34±1.39	26.61±0.66	26.17±5.05a	295.24±5.15b	284.49±5.46bc	4.76±0.34bc
CUMH	10.93±0.09	24.31±0.16	7.44±0.73b	336.09±3.14a	317.99±3.76ab	3.51±0.17bc
CDN	15.17±3.90	26.69±3.77	7.05±0.30b	288.99±6.90b	273.01±6.06c	10.45±1.21a
SF	9.29±0.25	24.19±0.79	6.60±0.67b	357.72±11.82a	333.95±12.40a	2.77±0.48c

[a] SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negra; SF: Superfig.
[b] NS not significant at $p > 0.05$; *, **, and ***, significant at $p < 0.05$, 0.01, and 0.001, respectively.
[c] Values (mean ± standard error; $n = 3$) followed by the same letter, within the same column, were not significantly different ($p > 0.05$), according to Tukey's least significant difference test.
[d] nd: no detected.

peel and pulp of brebas and figs were citric acid and malic acid. The main acid detected in all the samples analysed was malic acid agreeing to previous results (Núñez-Gómez et al., 2021). Other study indicated the highest organic acid was citric acid in both peel and pulp parts (827 and 259 mg 100 g^{-1} fw, respectively), followed by succinic acid (317 and 484 mg 100 g^{-1} fw, respectively) and malic acid (165 and 91 mg 100 g^{-1} fw, respectively) (Palmeira et al., 2019). The highest total content of organic acids was higher in the pulp than in the peel of the fruits analysed and the highest value was found in the pulp of the CDN variety (2642.52 mg 100 g^{-1} fw).

Glucose and fructose were detected in all parts of the fruits analyzed as previous studies indicated (these sugars were the major sugars found in several Spanish fig varieties whole fruits) (Wojdyło et al., 2016), while sucrose was only found in figs in a range of values from 15.57 to 32.84 g Kg^{-1} dw and pulp from 6.60 to 26.1 g Kg^{-1} dw. Previous studies indicated that sucrose was detect in low amounts in different Spanish varieties (Wojdyło et al., 2016).

For glucose and fructose in breba significant differences were only found in the pulp, showing the lowest values in the variety SA (4.74 g Kg^{-1} dw) and (12.82 g Kg^{-1} dw) for glucose and fructose respectively.

Besides, fig fruits showed significant differences between varieties for sucrose, glucose and fructose in peel and pulp. For sucrose CA variety showed the higher content for pulp (32.84 g Kg^{-1} dw) and peel (26.17 g Kg^{-1} dw). All varieties showed higher contents of glucose and fructose in pulp than in peel, except the SA variety that showed a content 1.14 times higher in peel than pulp for glucose and fructose. Finally, the highest total sugar content was found for fig pulp of the CA (776.14 g Kg^{-1} dw) variety.

Currently, dietary fiber is a hot topic in the formulation of healthy food products and refers to nutrients in the diet that are not digested by gastrointestinal enzymes but still fulfill an important role. On the other hand, crude fiber refers to one type of dietary fiber (cellulose, pentosans, lignin, and other components), the type that remains as a residue after food receives a standardized laboratory treatment (Buttriss and Stokes, 2008). Data of crude fiber, total dietary fiber, in our samples are shown in Table 2.

It can be observed that the peel breba presented a content 1.21 times higher than pulp, although variety effect was more statistically significant evidence. The highest value was observed in CA (11.57%) followed by SF (10.57%) in Breba pulp, while the highest values in Breba peel was identified in CUMH (13.77%), CDN (13.10%) and SF (12.04%).

In the case of fig fruits, the highest total dietary fiber in pulp was detected in CA (8.93%), followed by CUMH (7.11%) and SF (7.07%); while the highest value in peel was CDN (10.45%) followed by SA (6.97%).

It is important to highlight that the content of sucrose, glucose and fructose was higher in figs than in breba, both for pulp and peel in all the varieties studied. SF fig peel presented 76.60-fold and 61.75-fold more glucose and fructose values than SF breba peel. For total dietary fiber, it is essential to mention that CUMH breba peel presented 3.92-fold more value than CUMH fig peel and SF breba peel presented 4.34-fold more value than SF fig peel. The Regulation on Nutrition and Health Claims (European Commission 2007) allows claims to be made with respect to the fiber content of food if fiber levels exceed 3 g per 100 g (source of

fiber). The content of total dietary fiber in our samples is between 20 and 50% of the minimum content of fiber. It can be a good strategy the incorporation of pulp and/or peel of *Ficus carica* fruits in other food matrix to fortify and reach 3 g/100 g to be able to use the nutritional claim (Buttriss and Stokes, 2008).

3.3. Macro- and micro-elements

Mineral contents are involved in many biochemical processes in plants and are known to be affected by genotype, environmental conditions, use of fertilizers, and the nutritional status of the plant (Pereira et al., 2017). K, Ca, Mg, Fe, P, S, and N are the major elements detected in plants mainly accumulated during fruit growing and ripening (Lipan et al., 2020). Potassium has an important function in the regulation of osmotic pressure and cellular turgor; therefore, a deficiency of this element has a negative effect on metabolism of the plant (Garza-Alonso et al., 2019). Table 3 shows the macro- (Ca, K, Mg and Na; g Kg^{-1} dw) and micro- (Cu, Fe, Mn and Zn; mg Kg^{-1} dw) elements content of breba (pulp and peel), and fig (pulp and peel) while Fig. 1 and Fig. 2 shows the sum of micro- and macro-elements, respectively.

Firstly, the results of the breba will be discussed. As to K, SA variety presented the highest value among breba pulps (16.52 g kg^{-1} dw), this result was higher than those found in the research literature [21,24,25]. Also, the maximum value among breba peels was detected in the above-described variety (6.3 g kg^{-1} dw). However, the micro elements did not follow the same trend between pulp and peel in each of the samples analysed. For breba pulp, the descending order of the results were (mean values of the all varieties): Zn (29.78 mg kg^{-1} dw)>Fe (18.48 mg kg^{-1} dw)>Cu (8.13 mg kg^{-1} dw)>Mn (5.07 mg kg^{-1} dw), while in breba peel the descending order of detection was (mean all variety): Fe (41.98 mg kg^{-1} dw)>Zn (12.18 mg kg^{-1} dw)>Mn (6.84 mg kg^{-1} dw)>Cu (5.98 mg kg^{-1} dw). SA (54.65 mg kg^{-1} dw) and CA (53.32 mg kg^{-1} dw) cv. breba pulp showed higher values for the element Zn than the rest of the varieties, being these results higher than those found in the literature (Pereira et al., 2017; lo et al., 2020; GARZA-ALONSO et al., 2020). CA cv breba peel (17.09 mg kg^{-1} dw) obtained Fe in lower content than the rest of the varieties, although it is within the range of values found in the literature (lo et al., 2020; GARZA-ALONSO et al., 2020) (12.7 g kg^{-1} dw to 36.82 mg kg^{-1} dw). On the other hand, Cu and Mn were the micro-elements found in lower contents, ranged 15.09–3.34 and 8.47–2.28 mg kg^{-1} dw, respectively. Thus, the values obtained for Cu were higher than those found in the literature [21,24,25], however, in the case of Mn, the values found in the literature are higher (>13.3 mg kg^{-1} dw) (Lipan et al., 2020) than those obtained in our varieties. Furthermore, if the results obtained in pulp and peel for macro and microelements are compared, the results showed the same tendency, always showing higher values in peel than in pulp. To analyze the relationship between pulp and peel in breba fruits, the Ca content was 3.2, 3.8 and 3.0 times higher in peel than in the pulp in CUMH, CDN and SF, respectively. For sodium, CUMH and SF varieties showed 2.2- and 2.9-times higher values in peel than in the pulp. Regarding microelements for the element Fe, values between 2.2 and 3.5 times higher in the peel than in the pulp were found, except for CA that was 1.3 times lower in the peel than in the pulp. In addition, the Mn content was 2.0 and 2.2-fold higher in the peel than in the pulp in CUMH and CDN *cv*.

Figs, in relation to their K content, are considered a K-rich food, as

Table 3
Macro (Ca, K, Mg and Na; g Kg^{-1} dw) and micro-elements (Cu, Fe, Mn and Zn; mg Kg^{-1} dw) of Breba (pulp and peel), and Fig (pulp and peel).

Breba Fruit								
Variety[a]	Ca	K	Mg	Na	Cu	Fe	Mn	Zn
ANOVA test[b]								
Pulp	***	***	***	***	***	NS	***	***
Peel	***	*	***	***	***	***	**	**
Tukey's Multiple Range test[c]								
Pulp								
SA	4.92±0.11a	16.52±0.77a	1.54±0.01a	1.10±0.01a	12.06±0.73b	20.68±0.99	6.89±0.00b	54.65±1.50a
CA	4.76±0.12a	10.87±0.97b	1.17±0.07b	1.08±0.05a	15.09±0.85a	21.93±2.12	8.04±0.31a	53.32±0.79a
CUMH	1.34±0.13b	9.97±0.15bc	0.68±0.01c	0.10±0.01b	4.61±0.12c	17.71± 0.04	3.52±0.07cd	14.00±1.18b
CDN	1.05±0.12b	6.60±0.07c	0.44±0.01c	0.15±0.00b	4.11±0.15c	14.95±2.45	2.62±0.05d	14.75±1.46b
SF	1.40±0.07b	9.20±0.57bc	0.66±0.07c	0.08±0.01b	4.79±0.20c	17.13±1.64	4.29±0.20c	12.18±0.69b
SA	6.30±0.12a	15.52±0.02a	1.69±0.06a	1.15±0.01a	10.46±0.49a	44.59±0.73a	8.47±0.00a	28.65±3.72a
CA	5.57±0.06a	7.41±1.57b	0.42±0.09c	0.74±0.07b	3.62±0.37c	17.09±3.21b	4.99±0.07c	1.86±0.18c
CUMH	4.26±0.12b	10.20±0.21ab	0.70±0.01bc	0.22±0.02c	5.10±0.01bc	46.06±1.77a	7.15±0.23ab	13.91±1.34b
CDN	3.94±0.15b	9.84±0.33ab	0.73±0.04b	0.21±0.00c	5.02±0.37bc	52.26±2.07a	5.71±0.56bc	6.99±0.06bc
SF	4.21±0.20b	12.04±1.64ab	0.66±0.03bc	0.23±0.01c	5.71±0.16b	49.88±0.13a	7.90±0.53a	9.49±0.62bc
Fig Fruit								
Variety[a]	Ca	K	Mg	Na	Cu	Fe	Mn	Zn
ANOVA test[b]								
Pulp	*	*	***	NS	*	NS	*	NS
Peel	**	**	***	***	*	*	***	*
Tukey's Multiple Range test[c]								
Pulp								
SA	2.15±0.15a	8.54±0.31a	0.8 ± 0.01a	0.12±0.01a	3.72±0.04a	14.19±0.67a	2.93±0.26ab	7.99±0.07a
CA	1.65±0.13ab	7.20±0.55ab	0.52±0.02c	0.15±0.01a	3.34± 0.4a	13.42±2.24a	3.10±0.05a	10.31±1.16a
CUMH	1.44±0.04b	6.29±0.02b	0.60±0.01bc	0.15±0.00a	3.34±0.69a	14.55±2.66a	2.28±0.02b	7.80±0.92a
CDN	1.53±0.08b	6.50±0.07b	0.73±0.03a	0.14±0.01a	5.27±0.26 a	20.27±1.89a	2.72±0.17ab	10.84±0.2a
SF	1.55±0.04b	5.84±0.07b	0.62±0.02b	0.16±0.02a	4.79±0.08 a	9.93±0.73a	2.40±0.05ab	7.72±1.14a
Peel								
SA	2.48±0.22b	5.06±0.08b	0.58±0.02b	0.43±0.01a	5.41±0.58ab	15.19±0.45ab	2.95±0.05b	16.32±2.81a
CA	3.67±0.06a	4.94±0.10b	0.42±0.00c	0.44±0.00a	7.07±0.26a	10.94±1.22b	2.71±0.01b	17.15±0.55a
CUMH	2.59±0.04b	7.71±0.08a	0.76±0.00a	0.19±0.00c	4.56±0.02b	18.39±0.04a	3.98±0.02a	5.79±1.11b
CDN	3.29±0.03a	7.72±0.61a	0.77±0.02a	0.17±0.00c	6.34±0.04ab	17.25±0.63ab	3.02±0.08b	12.69±1.61ab
SF	2.42±0.13b	6.47±0.27ab	0.64±0.04b	0.28±0.03b	5.29±0.42ab	19.42±2.14a	4.25±0.28a	10.82±1.98ab

[d]nd: no detected.

[a] SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negra; SF: Superfig.

[b] NS not significant at $p > 0.05$; *, **, and ***, significant at $p < 0.05$, 0.01, and 0.001, respectively.

[c] Values (mean ± standard error; $n = 3$) followed by the same letter, within the same column, were not significantly different ($p > 0.05$), according to Tukey's least significant difference test.

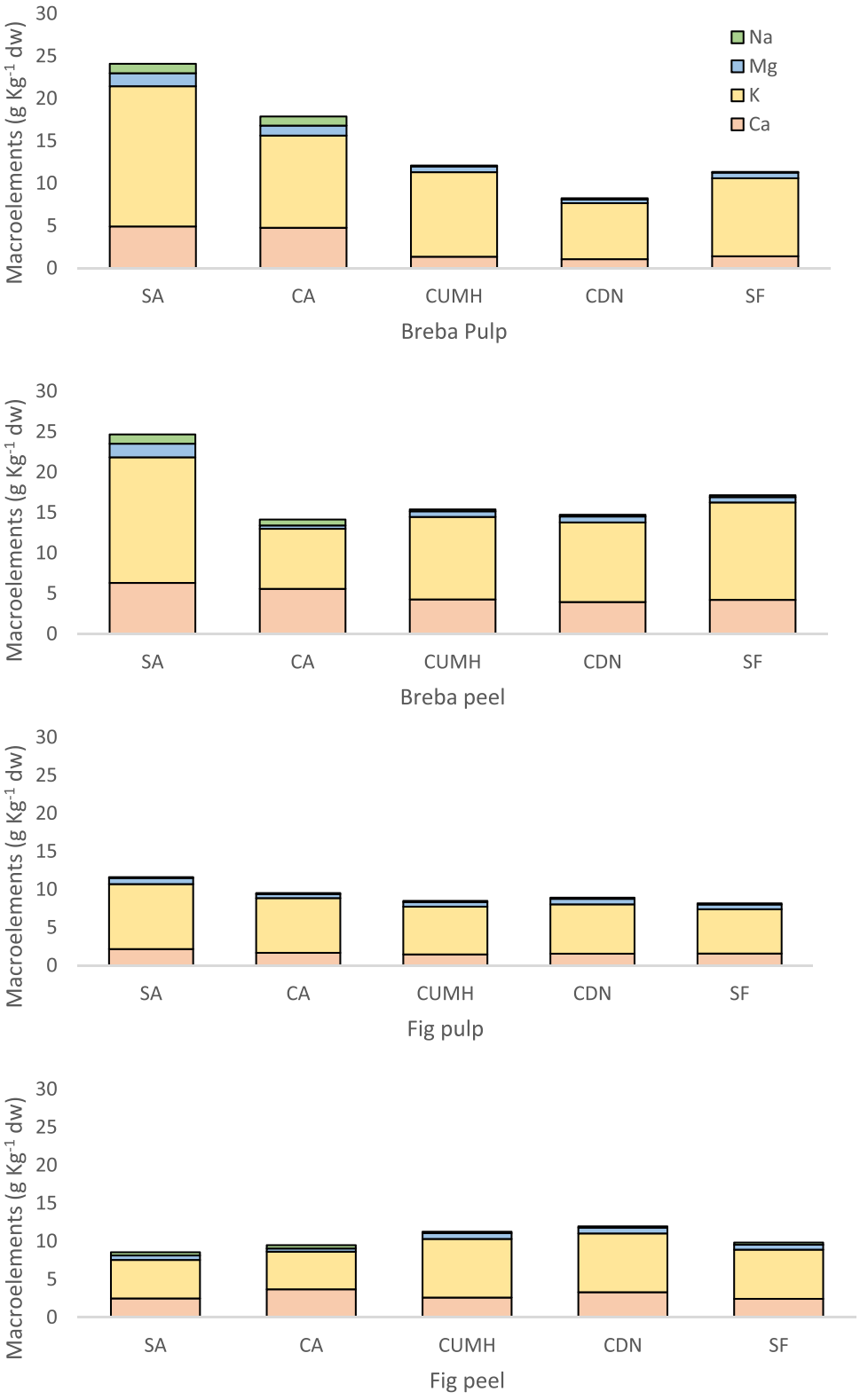

Fig. 1. Sum of the macro-elements (Ca, K, Mg and Na; g Kg^{-1} dw) of Breba (pulp and peel), Fig (pulp and peel).

their content is above the minimum threshold (600 mg/100 g) established in Regulation (EU) No. 1169/2011 of the European Parliament and of the Council (Lipan et al., 2020) . On the other hand, the highest values of Mg and Na were detected in the peel of cv SA (1.69 and 1.15 g kg^{-1} dw), respectively. These values are within the range of those reported in the literature (0.07 to 2.02 g kg^{-1} dw) (GARZA-ALONSO et al., 2020; Sadia et al., 2014). The tendency was the same in both figs peel and pulp as follows (mean value of all variety peel-mean values of all variety pulp): Fe (16.24–14.47 mg kg^{-1} dw)>Zn (12.55–8.93 mg kg^{-1} dw)>Cu (5.73–4.09 mg kg^{-1} dw)>Mn (3.38–2.69 mg kg^{-1} dw). As observed, fig fruits showed a Ca content 2.2- fold higher in peel than pulp for CA and CDN varieties. For Na, SA and CA varieties showed 3.6-

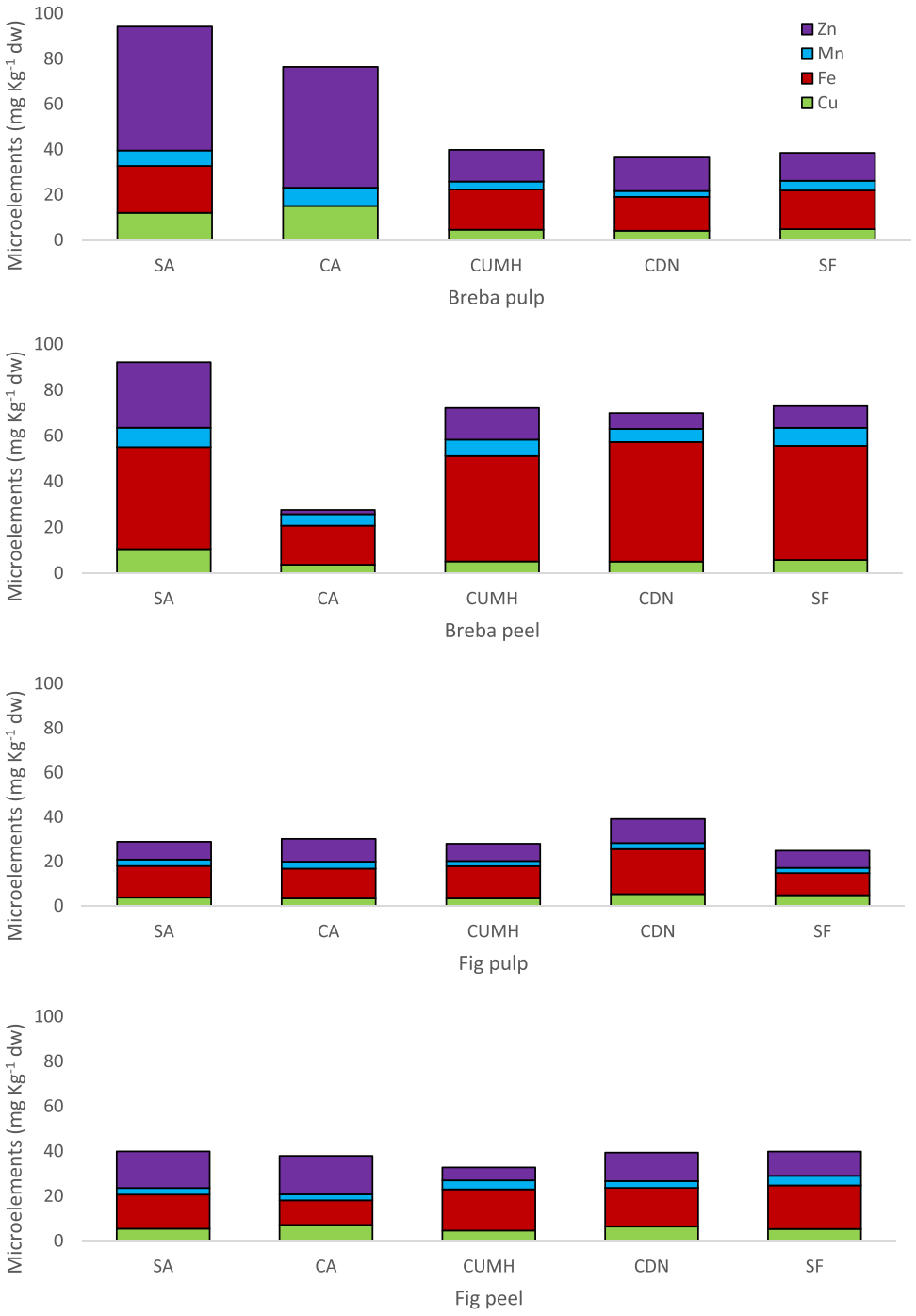

Fig. 2. Sum of the micro-elements (Cu, Fe, Mn and Zn; mg Kg^{-1} dw) of Breba (pulp and peel), Fig (pulp and peel).

and 2.9-times higher values in peel than pulp. In respect of microelements, Cu showed values 2.1 times higher in peel than pulp for CA variety. In addition, Fe and Zn element, both showed values 2.0 times higher in peel than pulp for SF and SA varieties. Finally, Commission Regulation (EC) No, 1881/,2006, does not set explicit maximum limits for any elements in figs or dried figs (Io et al., 2020).

When comparing breba and fig, it is important to note that SA and CA breba pulp presented values 2.29 and 2.88 higher in calcium than fig pulp. These same varieties SA and CA also presented higher values for breba pulp than for fig pulp in Na (9.17 and 7.20-fold more respectively), Cu (3.24 and 4.52-fold more respectively) and Zn (6.84 and 5.17-fold more respectively). Regarding the differences between breba and fig for peel, SA variety presented higher values in breba peel than in fig peel for all macro and micro elements, these values were higher 2.54 fold (Ca), 3.07 fold (K), 2.91 fold (Mg), 2.67 fold (Na), 1.93 fold (Cu), 2.94 fold (Fe), 2.87 fold (Mn) and 1.76 fold (Zn).

3.4. Antioxidant activity and total phenolic content

Fig. 3 and Table 1S (Supplementary material) shows the effect of variety on the ABTS, DPPH, FRAP and TPC values in breba pulp and peel, and in fig pulp and peel.

As to Breba pulp, the tendency of the effect of variety depends on the antioxidant activity assay: i) the highest value of ABTS was found in CUMH, while the lowest value was found in SA; ii) the highest DPPH value was obtained in SA while the lowest value was detected in SF, and,

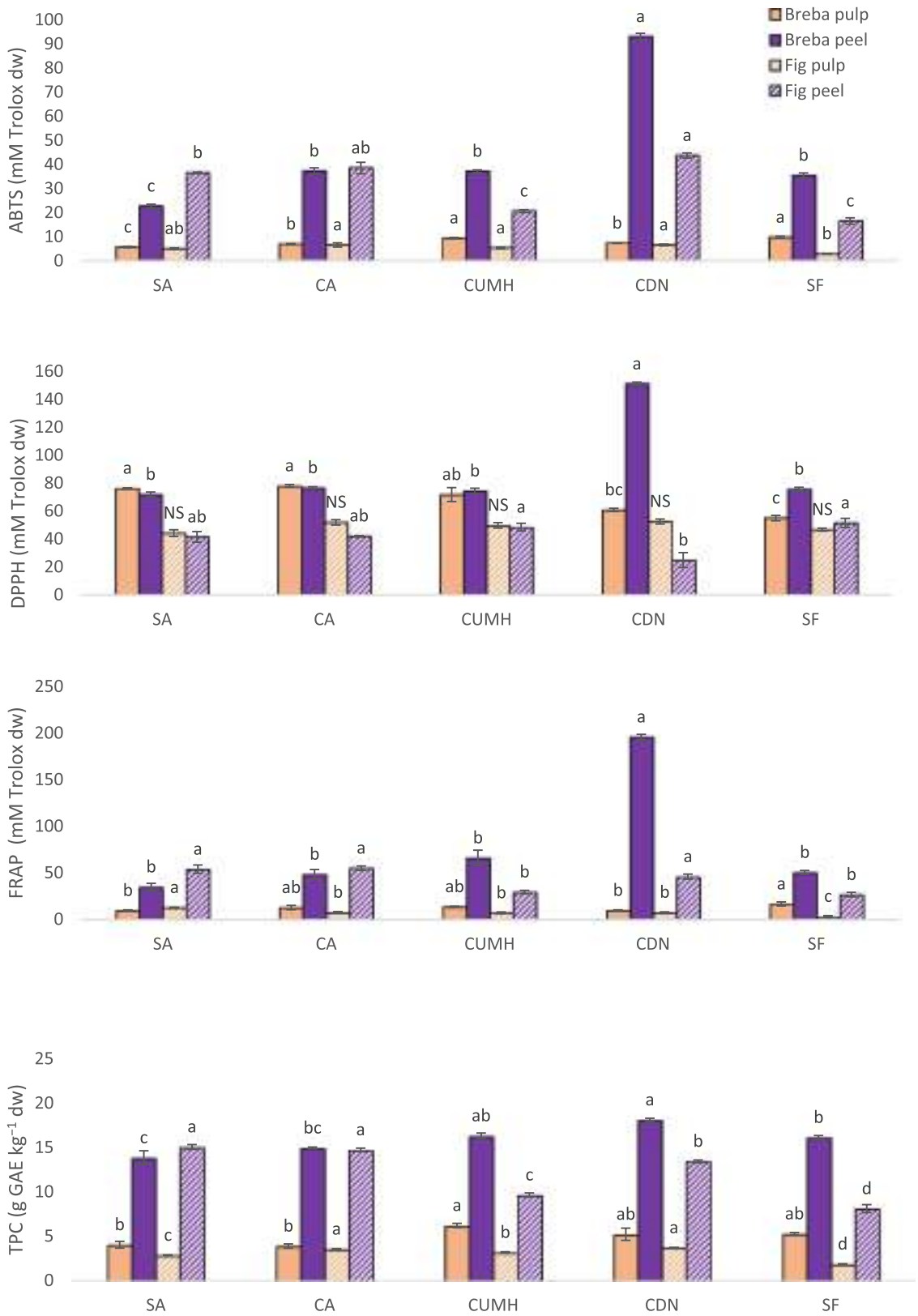

Fig. 3. Antioxidant activity (ABTS, DPPH and FRAP; mM Trolox dw) and total polyphenol content (TPC; g GAE kg^{-1} dw) of Breba (pulp and peel), and Fig (pulp and peel).

[a] SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negra; SF: Superfig. [b] NS not significant at $p > 0.05$; [c] Columns with different letter, within the same color, were significantly different ($p > 0.05$), according to Tukey's least significant difference test.

iii) the highest value of FRAP assay was observed in SF, and the lowest in SA and CDN. For TPC, CUMH pulp presented the highest values, being the lowest value the same variety but in other location (CA). On the other hand, it is important to highlight that the highest values of ABTS, DPPH, FRAP and TPC in breba peel was found in CDN variety, whilst he lowest ABTS, DPPH, FRAP and TPC values in peel was found in SA varieties. It can be appreciated in Fig. 1 and Table 1S (Supplementary material) that ABTS, DPPH, FRAP and TPC Breba peel values were between 3- and 13-fold, 1- and 2.5-fold, 3- and 21-fold, and 2.6- and 3-fold higher than pulp, respectively.

As to fig pulp, the tendency of the effect of variety depends on the antioxidant activity assay as occurs in breba pulp: i) the highest value of ABTS was found in CA and CDN, but the lowest value was found in SF, ii) the highest DPPH values was obtained in CDN while the lowest values was detected in SA, and, iii) the highest FRAP values was obtained in SA while the lowest value was detected in SF. For TPC, CA pulp presented the highest value, being SF variety the lowest. Regarding with fig peel, the highest values of ABTS, DPPH, FRAP and TPC were found in CDN, SF, CA and SA, respectively; whilst the lowest ones were found in SA, CDN, SF and SF, respectively. As to the differences between peel and pulp in fig fruits, ABTS, DPPH, FRAP and TPC peel values were between 4- and 7-fold, 0.5- and 1-fold, 4- and 9-fold, and 3- and 5.6-fold higher than pulp, respectively (Fig. 1 and Table 1S). A recent study compared 16 local and 9 introduced varieties, all them located in station on the National Institute for Agricultural Research of Meknes (INRA) in the northern Morocco and found big difference between the content total phenols and antioxidant activity of different varieties [27]. Showing a range of values for TPC, DPPH and ABTS since 389.05 to 2860.48 (mg GAE 100 g^{-1} dw), since 5.27 to 333.99 (mMol Trolox g^{-1} dw) and since 0.07 to 527.25 (mMol Trolox g^{-1} dw) in fig peel. In the case of fig pulp the values were found to range since 205.71 to 1186.6 (mg GAE 100 g^{-1} dw), since 19.54 to 42.63 (mMol Trolox g^{-1} dw and since 15.85 to 40.83 (mMol Trolox g^{-1} dw) (L Hssaini et al., 2021) . Del Caro et al., 2008 (del and A, 2008) also detected higher values of polyphenolic content in the peel than in the pulp of two Italian fig varieties analysed, only detecting a slight anthocyanin content in the pulp, and reported significant differences in polyphenolic content between black and green fig varieties, with higher results in polyphenolic content for black-skinned figs. Previous study about the characterization of peel of four Argelian fig varieties (Bakor Noir, Bouankik, Azenjer, and Tazegaght) revealed the extraction of large quantities of flavonoids (up to 50.4 mg g^{-1} of peels), mainly flavonols (up to 81% of total flavonoids). These compounds from fig peel extracts could be considered as multifunctional bioactive ingredients to be used in food industry and pharmaceutical formulations (Meziant et al., 2021). These results are in accordance with previous studies in which *Ficus carica* peel has been described as a source of bioactive compounds with antioxidant activity as anthocyanin pigments, with potential uses in various industrial fields, such as food, pharmaceutical, and cosmetic (Backes et al., 2018).

Although more studies are needed to obtain a correlation between antioxidant activity and individual bioactive compounds in our samples, previous studies indicated that each antioxidant activity assays are correlated with different bioactive compounds. For example, a significant positive correlation was observed between ORAC assay and keampferol-3-O-rutinoside, apigenin-3-O-hexoside-pentoside and total anthocyanins (Wojdyło et al., 2016). On the other hand, a recent study about the comparison of the content level of phytochemicals on juices of *F. carica* peels, pulps and total fruits indicated that not only results were influenced by the variety, but also varies significantly from one fruit part to the other (Harzallah et al., 2016).

Finally, it is important to highlight that SF Breba pulp presented 3-fold, 5.4-fold and 2.9-fold more ABTS, FRAP and TPC values than SF fig pulp. As to peel, it is essential to mention that CDN Breba peel presented 6.1-fold, 4.3-fold more DPPH and FRAP values than CDN Fig peel.

3.5. Principal component analysis (PCA)

For an easy visualization of the relationships among all variables, two PCA were run for all samples of Breba and fig samples, including only significantly different variables: sugar and organic acid profile, crude fiber, mineral content, antioxidant activity, and total phenolic content. Fig. 4 shows the two principal components which explained 73.3 and 65.3% of the samples`variation in Breba samples (Fig. 4, above) and figs (Fig. 4, below) samples, respectively.

As observed in Fig. 4 (above), Breba peel samples were grouped together and were characterized by higher antioxidant activity (DDPH, ABTS and FRAP assays), total phenolic content, crude fiber, micro-minerals (Fe) and macro-minerals (K, Mn and Ca). On the contrary, pulp samples in Breba were grouped separately and far from the first group with a higher fructose and glucose and citric acid.

The tendency in fig samples can be observed in Fig. 4 (below). Fig peel samples were mainly grouped together, except same varieties as CUMH and SF, and characterized by high content in Ca, Cu, Zn, Na, antioxidant activity by ABTS and FRAP assays, and total phenolic content. On the other hand, fig pulp samples were grouped together and were characterized by glucose, fructose, sucrose, malic acid, and antioxidant activity by DPPH assay. This tendency is according with the ANOVA test between Pulp and Peel included in Tables 2, 3 and 1S.

Antioxidant activity of fig seemed to be mostly related to the peel part and not the pulp part. The evaluation of antioxidant activity using cell-based methods and antibacterial potentials of peel and pulp of a Portuguese green fig fruit variety were previously published, confirming that the fig peel is superior to the corresponding pulp as it relates to nutritional and phenolic profiles as well as biological activities, this suggest the urgency in valorising and exploiting this usually discarded agro-industrial by-product (Palmeira et al., 2019). The peel extracts could increase the nutritional properties of food products (Teruel-Andreu et al., 2021). which can be used as value-added ingredients or pharmaceutical and so improve the economic performance, besides promote a circular economy and new product adapt with current consumer preferences towards sustainability and healthy food (Ayuso et al., 2022).

4. Conclusion

Globally, data presented here showed that variety affected the nutritional and functional components in both edible and non-edible Ficus carica L. fruit parts (organic acids, sugars, total dietary fiber, macro- and micro- elements, total polyphenolic content, and antioxidant activity). On the other hand, differences have also been observed between edible and non-edible fruit part: pulp was richer in sugar content while peel showed higher values of the rest of the studied parameters. Considering these results, the different parts of the fruits not only does it have a high content of phenolic compounds, but it can also be a potential material by his sweetener, dietary fiber, and mineral source. All these findings can help to select interesting varieties from the nutritional and commercial point of view improving consumer health and the economy of farmers. Further research is needed to know the profile of volatile compounds and individual phenolic compounds.

CRediT authorship contribution statement

Candela Teruel-Andreu: Conceptualization, Methodology, Writing – original draft, Software. **Esther Sendra:** Conceptualization, Writing – review & editing. **Francisca Hernández:** Conceptualization, Visualization, Investigation, Supervision. **Marina Cano-Lamadrid:** Conceptualization, Visualization, Writing – review & editing, Investigation, Supervision.

Fig. 4. Principal Component Analysis (PCA) of breba (above) and fig samples (below): micro and macro-elements, antioxidant activity, total phenolic content, sugar and organic acid (73.7 and 65.34%, respectively).

Declaration of Competing Interest

The authors declare no conflict of interest.

Data availability

Data will be made available on request.

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Supplementary materials

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VOLATILE PROFILE OF BREBA AND FIG FRUITS (PEEL AND PULP) FROM DIFFERENT *FICUS CARICA* L. VARIETIES.

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Volatile profile of breba and fig fruits (peel and pulp) from different *Ficus carica* L. varieties

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ABSTRACT

Background: It is currently estimated that around 50 % of fig production in Spain is not marketed and it is wasted, increasing the quantity of food loss. It is necessary to highlight that this is the first study comparing peels and pulps of breba and figs fruits to help improve the knowledge of volatile profile in four different Spanish varieties. The aim of this study was to investigate the volatile composition by HS-SPME of breba and figs (peel and pulp) of different varieties selected for their commercial relevance in Spain.
Results: In this study, 35 compounds have been detected in the different parts of breba and figs fruits. It can be said that the data presented here showed that variety affected the volatile profile in both edible (pulp) and non-edible (peel) *Ficus carica* L. fruit parts in both brebas and fig fruits, being Colar de Albatera which presented higher content in key volatile compounds. On the other hand, differences have also been observed between pulp and peel fruit part in each fruit: peel was richer in key volatile compounds than pulp, especially in Colar de Albatera variety.
Conclusion: Apart from the high content of phenolic compounds and nutritive properties of the edible and non-edible part of brebas and figs, specially Colar variety, it can be concluded that this material can also increase the olfactory sensory attributes.

1. Introduction

The fig tree (*Ficus carica* L.) is the most important *Ficus* species plant in the Moraceae family and is native to the Sub-Himalayan region and central India, although its cultivation is currently widespread in the Mediterranean area and the Near East due to its mild winters and hot and dry summers (Teruel-Andreu et al., 2023a). Therefore, the main figs/brebas -producing countries in the world are Turkey with 320,000 t in 2021, followed by Egypt, Morocco, Algeria, Iran, and Spain. In Europe, Spain is the major producer of figs/brebas (60,190 t), followed by Italy (12,760 t) (FAOSTAT, 2021).

However, the *Ficus carica* crop in Spain has been mainly grown in marginal areas traditionally cultivated under restrictive conditions (Lipan et al., 2020), but those under irrigation provide high-quality fruit for the fresh market and exports. Fig culture is oriented towards producing both breba and fig crops, using parthenocarpic and biferous cultivars (Melgarejo et al., 2007). Biferous varieties produce two crops – brebas and figs. The first crop Breba (dormant figs that develop from the previous year's growth and begin their development in the following spring) and the second crops Figs (develops on the stems of the current season). These varieties are characterized by the first crop being grown from the flowers of the previous year, this fruit is known as breba and it ripens at the beginning of the summer, whereas the second crop produces the figs, that emerge on the stems of the current season, and the fruit is harvested between mid-July and September, hence the main differences between breba and figs are due to the climatic conditions in which each develops (Palassarou et al., 2017) (Melgarejo et al., 2007; Núñez-Gómez et al., 2021; Palassarou et al., 2017).

Consumers are not only looking for the appearance of fruit (size, color, texture, etc.), but are also looking for internal quality (flavor, volatile compounds, functional compounds, etc.) (Sánchez-Bravo et al., 2022). Aroma present in fresh and processed fruit is affected by a

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complex group of chemical substances, such as aldehydes, alcohols, ketones, esters, lactones, and terpenes, which play an important role in the sensory quality (Villalobos et al., 2018). The volatile compound profile present in fresh fig can be used to identify each variety because it is considered to be unique and has a great influence on flavor and quality of the aroma and therefore on consumer acceptance (Pereira et al., 2020). Besides genotype other components that can influent aroma are geographical origin due to diversity in climatological conditions, maturity degree, agronomic techniques, and post-harvest treatment (Palassarou et al., 2017).

Volatile compounds belong to several chemical families, mainly aldehydes, terpenes, esters, alcohols, acids, and ketones contributing to the aroma of fresh figs (Pereira et al., 2020; Russo et al., 2017). In addition, other studies suggests that terpenes are the main volatile compound that influences the aroma of figs (Gozlekci et al., 2011). Similarly, Sertkaya et al. (2021) reported that the terpenes followed by esters and alcohols, were the most dominant aroma compounds in fig samples. Other compounds associated with fig aroma include 2-furancarboxaldehyde, 5-hydroxymethyl-2-furancarboxylic acid, benzaldehyde, furfural and phenol, 2,6-bis(1,1-dimethylethyl)−4-methyl phenol (Villalobos et al., 2018).

Currently, to determine the direct relationships between the odor or taste of a sample and the responsible volatile compounds, it is possible to compare sensory analysis, using GCMS– to detect volatiles and find associations or using GC olfactometry ports -MS to detect and identify the responsible compounds (Sánchez-Bravo et al., 2022). For the determination of volatile compounds with gas chromatography analysis with a mass detector (GC–MS) the most widely used extraction and pre-concentration of volatile compounds technique is solid-phase microextraction of headspace (HS-SPME) that does not produce alterations in the volatile compounds due to temperature or solvent effect (Oliveira et al., 2010).

Previous studies have reported the presence of volatile compounds in *F. carica* L. as mentioned above, but no-published data related to the volatile profile comparison research between pulp and peel in breba and/or fig fruits from *F. carica* L. was found. Therefore, the aim of this work was to determine the volatile profile of breba (pulp and peel) and fig (pulp and peel) of four varieties of *F. carica* L., one of them grown in two different localities. This information can will be used to select of the most suitable varieties, and can contribute valuable insights to the field of functional foods and potentially contribute to the development of new, health-promoting fig/ fig peel-based products.. It is important to highlight that this is the first work comparing breba and figs and their different parts (pulp and peel) of *F. carica* L. Spanish varieties.

2. Materials and methods

2.1. Plant material and sample processing

The fruit of 4 varieties of *F. carica* were used for this study. The *F. carica* variety "San Antonio" (SA), *F. carica* variety "Colar" (CA, CUMH), *F. carica* variety "Cuello Dama Negro" (CDN) and *F. carica* variety "Superfig" (SF) varieties were harvested at the experimental field station of Universidad Miguel Hernández de Elche (UMH) (Alicante, Spain; 02° 03′ 50″ W, 38° 03′ 50″ N, and 25 masl), while the "Colar" variety was harvested both at the experimental field station of University (CUMH) and at the local producers in the Albatera area (CA) (Alicante, Spain; 0° 55′ 49″ W, 38° 13′ 17″ N). The study was conducted in the year 2021 and fruits were harvested in two different periods. (i) June: for the breba crop, which is the first crop of figs in the season, (ii) August: For the main fig crop. The average number of fruits collected per tree was five. Mature fruits were randomly collected from four trees of each variety for both brebas and figs. All the harvested materials were immediately frozen at a temperature of −20 °C and stored until they were ready for analysis. Previous study (Teruel-Andreu et al., 2023b) about the nutritional and functional characterization of the same material has recently published.

2.2. Extraction procedure of volatile aroma compounds

Different extraction system was used to each sample peel and pulp. In the case of the first, two grams of peel (obtained using a peeler on frozen fruit) was added to a hermetic vial with polypropylene cap and PTFE (polytetrafluoroethylene)/silicone septa, together with 1 g NaCl. As to pulp samples, eight grams of pulp was added to vial with 2 mL of water and 1 g of NaCl.

The extraction of the volatile compounds of samples of peel and pulp was carried out by headspace solid-phase microextraction (HS_SPME) method. A fiber of 50/30 mm DVB/CAR/PDMS (Divinylbenzene/Carboxen/Polydimethylsiloxane) of 1 cm of length was used to absorb the compounds along the extraction. Samples were exposed for 60 min at 40 °C, with constant agitation (500 rpm) by using a Shimadzu AOC-6000 Plus autosampler (Shimadzu Corporation, Kyoto, Japan).

2.3. Chromatographic analyses

Volatile compounds were determined as previously described by Oliveira et al. (2010) using a chromatograph Shimadzu GC2030 (Shimadzu Scientific Instruments, Inc., Columbia, MD, USA) for isolation and identification of the volatile compounds. The gas chromatograph was equipped with an SLB-5 MS column of 30 m x 0.25 mm x 0.25 μm (length, diameter, and film thickness, respectively) (Teknokroma, Barcelona, Spain). For the identification of compounds, the chromatograph was coupled with a Shimadzu TQ8040 NX mass spectrometer detector. The parameters of the mass spectrometer were: (i) mass range 40–350 *m/z*, (ii) scan speed 3333 amu/s, (iii) event time of 0.100 s, and (iv) electronic impact of 70 eV Helium was used as gas carrier at a column flow of 1 mL min^{-1} in a splitless mode, purge flow of 6 mL min^{-1}, and a total column flow of 17.0 mL min^{-1}. The temperature of the interface was 280 °C, the ion source was 230 °C, and the injector was 220 °C. The desorption time of the sample in the injection port was 3 min. The oven program was the following: (i) initial temperature of 40 °C, and holded 1 min, and (ii), ramp of 2 °C min^{-1} up to 220 °C, and holded for 30 min.

The volatile compounds were identified using 3 methods: (i) retention indexes (RI) that were calculated with a commercial alkane standard mixture (C8–24) (Sigma-Aldrich, Steinheim, Germany), (ii) GC–MS retention time of the chemical pure compounds, and (iii) comparison of the compound mass spectrum with those of databases (NIST, 2023). In addition, the relative intensity of each volatile compound has been calculated as the ratio between the area of the specific molecule and the sum of the areas of all identified peaks (peak area normalization method) in the chromatogram. Compounds with spectral similarity >90 % and with a deviation of less than 10 units of linear retention similarity were considered as correctly identified.

2.4. Statistical analyses

Descriptive statistical analysis was done to check the normality and homogeneity of the variance. Once completed, a one-way analysis of variance (ANOVA) was performed to determine whether there were statistical differences ($p < 0.05$) between cultivars, and two-way analysis of variance was performed to determine whether there were statistical differences ($p < 0.05$) between brebas and figs. Tukey's multiple range test were performed for the analysis of the results. The XLSTAT Premium (2016.02.27444 version, Addinsoft: New York, NY, USA) was used to perform statistically significant differences, with a significant level $p < 0.05$.

Table 1
Aromatic compounds found in *F. carica fruits* pulp using headspace solid phase microextraction (HS-SPME).

Code	Volatile Compounds	Pulp	Peel	Chemical Family	[¥]RT (min)	Kovats index (KI)[¶] Exp	Lit	Descriptors
V1	Hexanal	Yes	Yes	Aldehyde	6.263	803	803	Fresh, cut grass [a]
V2	2-Hexenal	Yes	Yes	Aldehyde	8.396	849	850	Almond, apple, green, sweet, vegetable [a]
V3	Heptanal	Yes	No	Aldehyde	10.769	900	900	Oily, fruity, woody, fatty, nutty [a]
V4	2,4-Hexadienal	No	Yes	Aldehyde	11.238	908	909	Floral, citrus, green [a]
V5	Methyl hexanoate	No	Yes	Ester	12.010	921	919	Cheese, fatty, sour [a]
V6	Benzaldehyde	Yes	Yes	Aldehyde	14.061	954	955	Almond, anise, balsam, cherry, floral [a]
V7	1-Octen-3-ol	Yes	Yes	Alcohol	15.488	977	977	Cheese, creamy, earthy, herbaceous [a]
V8	2,3-Octanedione	Yes	No	Ketone	15.790	982	983	Herbal, earthy, fatty [b]
V9	2,2,4,6,6-Pentamethylheptane	Yes	No	Alkane	16.067	987	985	—
V10	2,4-Heptadienal	Yes	Yes	Aldehyde	16.474	993	993	Cinnamon, hazelnut, fatty [a]
V11	Octanal	Yes	Yes	Aldehyde	16.948	1001	1001	Honey, fruity, fatty, citrus [a]
V12	2-Octenal	Yes	Yes	Aldehyde	20.647	1054	1056	Spicy, herbaceous, green [a]
V13	3,5-Octadien-2-one	Yes	No	Ketone	21.436	1065	1068	Fruity, fatty, mushroom [a]
V14	2-Nonanone	Yes	No	Ketone	23.050	1088	1091	Herbaceous, floral, fruity, [a]
V15	Linalool	Yes	Yes	Terpenoid	23.640	1097	1097	Lemon, floral, citrus [a]
V16	Nonanal	Yes	Yes	Aldehyde	23.966	1102	1102	Apple, coconut, grape, lemon, vegetable [a]
V17	Phenylethyl alcohol	No	Yes	Alcohol	24.186	1105	1109	Honey, rose [a]
V18	Methyl octanoate	No	Yes	Ester	25.331	1121	1120	Cheese, oily [a]
V19	2,6-Nonadienal	No	Yes	Aldehyde	27.226	1147	1148	Vegetable, green [a]
V20	Benzenepropanal	No	Yes	Aldehyde	27.675	1153	1160	Floral, Green, Fresh, Powerful [b]
V21	Pinocarveol	No	Yes	Terpenoid	27.809	1155	1147	Herbal,woody, pine, balsam [b]
V22	2-Nonenal	Yes	No	Aldehyde	27.822	1155	1159	Waxy, fatty [a]
V23	Decanal	Yes	Yes	Aldehyde	31.133	1201	1201	Floral, citrus, sweet [a]
V24	2,4-Nonadienal	Yes	No	Aldehyde	31.664	1209	1208	Melon, fatty, floral, vegetable [a]
V25	2-Phenethyl acetate	No	Yes	Ester	34.308	1246	1250	Sweet honey, floral, balsamic [b]
V26	2-Decenal	Yes	Yes	Aldehyde	34.941	1255	1255	Oily, orange, floral, citrus, green, meaty [a]
V27	3-Phenyl 2-propenal	Yes	No	Aldehyde	35.324	1261	1260	Balsam, hyacinth, floral, sweet [a]
V28	Undecanal	No	Yes	Aldehyde	38.045	1299	1301	Orange, fatty, rose, waxy [a]
V29	Copaene	Yes	Yes	Terpene	42.368	1364	1366	—
V30	Caryophyllene	Yes	Yes	Terpene	45.073	1405	1405	Spicy, woody [a]
V31	Aromandendrene	No	Yes	Terpene	47.535	1443	1447	—
V32	*β*-Ionone	No	Yes	Ketone	48.824	1463	1470	Woody [a]
V33	1-Dodecanol	Yes	No	Alcohol	48.904	1464	1466	Coconut, honey, fatty, earthy, soapy, waxy [a]
V34	1-Tetradecanol	No	Yes	Alcohol	60.831	1669	1670	Fatty waxy, dairy creamy, fishy, fruity [b]
V35	Methyl hexadecanoate	No	Yes	Ester	73.788	1921	1921	Floral, waxy [a]

[¥] RT = retention time,.
[¶] KI (Exp.) = experimental Kovats index, (Lit.) = literature Kovats index,.
[a] SAFC (SAFC, 2012),.
[b] TGSC (TGSC, 2023).

3. Results and discussion

3.1. Volatile composition in pulp and peel of breba and figs

The volatile compounds were determined using HS-SPME standard method combined with GC for the isolation, identified and their relative abundance determined. Thirty-five volatile compounds were identified in the pulp and peel of the Ficus carica L. (Table 1).

The volatile compounds identified in the pulp (Table 2) of fruits studied were classified as aldehydes (n = 13), alcohols (2), alkanes (1), terpenes (4), terpenoids (1) and ketones (3). While the volatile compounds identify in the peel (Table 4) were classified as aldehydes (13), esters (4), alcohols (3), terpenes (2), terpenoids (2) and ketones (1). This trend was in agreement with those reported by others authors (Lachtar et al., 2022; Yao et al., 2021; Zidi et al., 2021).

For the pulp samples in the breba of the different varieties, hexanal was the one that obtained the highest percentage in the CA variety with 57.86 %, while for the CUMH and SF varieties it was benzaldehyde with a percentage of 43.45 and 34.38 % respectively. Previous works indicated that hexanal and benzaldehyde were the main compounds detected in figs (Gibernau et al., 1997; Lachtar et al., 2022; Pereira et al., 2020).

For alcohols only, significant differences have been found in the 1-octen-3-ol compound, also in this case the CA variety had the highest percentage (1.99 %). In a study dried fig cultivar of the major fig-producing geographical regions in Greece were analysed (Palassarou et al., 2017) and detected this compound in some varieties such as "Tsapelosika" and "Vassiliko" in values 1.33 % and 0.36 %, respectively.

Regarding the alkanes, the 2,2,4,6,6-pentamethyl heptane compound was found with a percentage range of 2.91–24.19 % between all the varieties. As in the terpene group, two compounds were detected copaene and caryophyllene but no significant differences were found between the varieties for any of the compounds.

The only one identified terpenoid was Linalool in this study. This compound also was found in figs of others varieties as "Azegzaw", although it was not the only one terpenoid found but it was the most abundant (Zidi et al., 2021). In ketone group, the CA variety obtained the highest percentage for the three compounds 2,3-octanedione (1.42 %), 2,3-octanedione (5.20 %) and 2-nonanone (1.69 %). 2-nonanone. Moreover, increase of some of these compounds such as 2-nonanone were detected in oven-dried "Dottato" figs (Palassarou et al., 2017; Russo et al., 2017).

For fig pulp, the compound that had the highest percentage was hexanal which belongs to the aldehyde group and CA (64.69 %) variety was the variety that obtained the highest percentage. Benzaldehyde is also a relevant compound in fig pulp other authors (Zidi et al., 2021) argue that hexanal and benzaldehyde were the most abundant aldehydes in "Taamriwthe" and "Azegzaw" figs. Benzaldehyde, showed a range of percentages between the different varieties studied (3.68–43.20 %), other authors agree with this and confirm results of 7.13 % (average value of several fig varieties) (Pereira et al., 2020). In the alcohol group, significant differences were found for the compounds 1-octen-3-ol and 1-dodecanol, CUMH (2.34 %) and SA (1.88 %) were showed the highest percentage respectively, this compound was also detected in low

Table 2
Volatile compound (% of volatile profile) of breba and fig pulp as affected by cultivar.

	Breba pulp (%)						Fig pulp (%)						Breba*Fig pulp (%)		
Volatile compound	ANOVA	SA	CA	CUMH	CDN	SF	ANOVA	SA	CA	CUMH	CDN	SF	ANOVA	Breba	Fig
Aldehyde															
Hexanal	***	26.14 b	57.86 a	4.61 c	10.22 bc	4.34 c	***	2.70 c	64.69 a	31.46 b	3.96 c	2.86 c	NS	20.63 a	21.13 a
2-Hexenal	NS	6.23 a	6.41 a	3.69 a	7.35 a	4.47 a	NS	5.01 a	2.06 a	1.90 a	3.38 a	3.63 a	**	5.63 a	3.20 b
Heptanal	NS	0.79 a	2.09 a	0.21 a	5.71 a	7.77 a	NS	0.40 a	0.53 a	0.48 a	0.20 a	0.54 a	*	3.31 a	0.43 b
Benzaldehyde	**	19.43 bc	3.17 d	43.45 a	9.05 cd	34.38 ab	**	20.76 ab	3.68 b	33.82 a	43.20 a	23.81 ab	NS	21.90 a	25.05 a
2,4-Heptadienal	**	2.01 a	1.68 ab	0.42 bc	0.20 c	0.13 c	NS	0.09 a	0.87 a	0.25 a	0.27 a	1.01 a	NS	0.89 a	0.50 a
Octanal	*	2.64 ab	1.46 b	2.88 ab	3.87 a	2.89 ab	NS	2.73 a	1.55 a	2.10 a	1.84 a	4.15 a	NS	2.75 a	2.47 a
2-Octenal	**	3.19 b	5.12 a	1.18 c	1.56 bc	0.67 c	NS	0.78 a	2.75 a	4.31 a	0.91 a	0.44 a	NS	2.34 a	1.84 a
Nonanal	**	8.64 b	3.57 c	10.54 b	17.71 a	8.72 b	NS	11.45 a	5.22 a	4.20 a	8.26 a	26.11 a	NS	9.84 a	11.05 a
2-Nonenal	*	0.92 ab	0.62 b	1.84 a	0.92 ab	1.17 ab	*	1.11 a	0.49 b	1.01 ab	0.83 ab	1.09 a	NS	1.10 a	0.91 a
Decanal	NS	2.90 a	0.77 a	2.28 a	2.35 a	1.76 a	NS	3.13 a	0.75 a	1.33 a	1.87 a	1.96 a	NS	2.01 a	1.81 a
2,4-Nonadienal	**	1.36 a	1.55 a	0.69 b	0.66 b	0.45 b	NS	0.82 a	1.12 a	1.39 a	0.31 a	0.47 a	NS	0.95 a	0.82 a
2-Decenal	NS	0.64 a	0.57 a	0.59 a	0.78 a	0.37 a	NS	0.53 a	0.43 a	0.66 a	0.40 a	0.51 a	NS	0.59 a	0.51 a
3-Phenyl 2-propenal	*	1.17 bc	0.49 c	1.62 abc	2.91 a	2.28 ab	NS	2.04 a	1.04 a	0.54 a	1.45 a	1.28 a	NS	1.69 a	1.27 a
Alcohol															
1-Octen-3-ol	*	1.59 a	2.00 a	0.65 a	1.04 a	0.39 a	*	0.65 b	1.11 ab	2.34 a	0.54 b	0.63 b	NS	1.13 a	1.05 a
1-Dodecanol	NS	0.90 a	0.48 a	1.46 a	1.39 a	0.93 a	*	1.88 a	0.51 b	0.57 b	0.85 b	0.94 ab	NS	1.03 a	0.95 a
Alkane															
2,2,4,6,6-Pentamethylheptane	***	5.12 bc	2.92 c	9.16 b	24.20 a	7.10 bc	NS	12.51 a	8.27 a	2.06 a	23.07 a	20.63 a	NS	9.70 a	13.31 a
Terpene															
Copaene	NS	0.18 a	0.39 a	1.85 a	3.07 a	2.19 a	NS	2.12 a	0.99 a	0.35 a	1.03 a	1.51 a	NS	1.54 a	1.20 a
Caryophyllene	NS	0.12 a	0.22 a	2.10 a	3.49 a	2.15 a	NS	2.14 a	0.86 a	0.18 a	2.10 a	2.27 a	NS	1.62 a	1.51 a
Terpenoid															
Linalool	***	12.24 b	0.33 c	9.77 b	1.89 c	17.21 a	**	27.98 a	0.39 b	7.19 b	4.04 b	5.47 b	NS	8.29 a	9.01 a
Ketone															
2,3-Octanedione	**	1.35 ab	1.42 a	0.83 bc	0.70 c	0.44 c	*	0.79 ab	0.88 ab	2.10 a	0.51 b	0.52 b	NS	0.95 a	0.96 a
3,5-Octadien-2-one	***	1.82 b	5.20 a	0.11 c	0.35 c	0.11 c	NS	0.17 a	1.51 a	1.68 a	0.26 a	0.10 a	NS	1.52 a	0.75 a
2-Nonanone	*	0.59 ab	1.69 a	0.09 b	0.60 ab	0.10 b	**	0.23 b	0.31 b	0.10 b	0.73 a	0.08 b	NS	0.61 a	0.29 a

NS: not significant at $p > 0.05$, ** and ***: significant at $p < 0.01$ and 0.001, respectively. Values followed by different letters, within the same column, were significantly different ($p < 0.05$). SA – San Antonio; CA – Colar Albatera; CUMH – Colar UMH; CDN – Cuello Dama negro; SF – Superfig.

Table 3
Volatile compound (% of volatile profile) of breba and fig peel as affected by cultivar.

	Breba peel (%)						Fig peel (%)						Breba*Fig peel (%)		
Code	ANOVA	SA	CA	CUMH	CDN	SF	ANOVA	SA	CA	CUMH	CDN	SF	ANOVA	Breba	Fig
Aldehyde															
Hexanal	***	7.60 b	52.72 a	5.23 b	4.62 b	8.89 b	***	2.09 c	28.08 a	9.57 b	4.56 c	2.91 c	NS	15.81 a	9.44 a
2-Hexenal	NS	36.78 a	11.86 a	51.36 a	54.86 a	45.68 a	***	37.16 a	14.36 bc	6.24 c	37.55 a	25.24 ab	**	40.11 a	24.11 b
2,4-Hexadienal	NS	0.27 a	0.44 a	0.13 a	0.48 a	0.04 a	**	0.30 ab	0.33 a	0.15 ab	0.13 ab	0.09 b	NS	0.27 a	0.20 a
Benzaldehyde	NS	40.59 a	9.72 a	37.74 a	32.72 a	33.29 a	***	47.12 ab	28.74 b	62.48 a	48.91 a	65.63 a	***	30.81 b	50.58 a
2,4-Heptadienal	***	3.27 ab	3.74 a	0.51 c	0.28 c	1.59 bc	***	0.36 b	1.33 a	1.82 a	0.46 b	0.53 b	NS	1.88 a	0.90 a
Octanal	**	0.66 ab	1.44 a	0.50 b	0.34 b	0.80 ab	***	0.59 b	1.39 a	1.15 a	0.35 b	0.29 b	NS	0.75 a	0.75 a
2-Octenal	***	0.90 b	5.42 a	0.39 b	0.48 b	0.82 b	***	0.56 b	2.89 a	1.87 a	0.32 b	0.24 b	NS	1.60 a	1.17 a
Nonanal	***	1.89 b	4.05 a	1.14 b	1.60 b	2.18 b	***	1.38 b	9.39 a	2.71 b	1.72 b	1.20 b	NS	2.17 a	3.28 a
2,6-Nonadienal	***	0.36 d	2.06 a	0.55 cd	1.02 bc	1.30 b	NS	1.03 a	1.08 a	0.60 a	1.10 a	0.39 a	NS	1.06 a	0.84 a
Benzenepropanal	***	0.16 a	0.01 b	0.01 b	0.05 b	0.03 b	***	0.15 b	0.70 a	0.11 b	0.18 b	0.11 b	**	0.05 b	0.25 a
Decanal	***	1.40 ab	1.98 a	0.37 c	0.63 c	0.84 bc	***	0.48 b	2.47 a	0.57 b	0.44 b	0.27 b	NS	1.04 a	0.85 a
2-Decenal	NS	0.10 a	0.24 a	0.12 a	0.02 a	0.03 a	NS	0.38 a	0.29 a	0.10 a	0.08 a	0.06 a	NS	0.10 a	0.18 a
Undecanal	***	0.13 ab	0.21 a	0.02 b	0.03 b	0.06 b	***	0.16 b	0.28 a	0.31 a	0.05 c	0.09 c	**	0.09 b	0.18 a
Ester														0.00	0.00
Methyl hexanoate	**	0.19 ab	0.37 a	0.06 b	0.08 b	0.10 b	***	0.03 b	0.47 a	0.42 a	0.02 b	0.02 b	NS	0.16 a	0.19 a
Methyl octanoate	***	0.17 a	0.18 a	0.07 b	0.02 c	0.01 c	***	0.09 b	0.29 a	0.32 a	0.06 b	0.05 b	NS	0.09 a	0.16 a
2-Phenethyl acetate	NS	0.01 a	0.02 a	0.14 a	0.21 a	0.57 a	NS	0.11 a	0.08 a	0.29 a	0.06 a	0.05 a	NS	0.19 a	0.12 a
Methyl hexadecanoate	***	0.10 b	0.42 a	0.01 c	0.01 c	0.01 c	***	0.01 b	0.10 a	0.13 a	0.01 b	0.01 b	NS	0.11 a	0.05 a
Alcohol														0.00	0.00
1-Octen-3-ol	***	0.61 b	2.46 a	0.36 b	0.35 b	0.84 b	***	0.67 c	3.38 a	2.07 b	0.52 c	0.33 c	NS	0.92 a	1.39 a
Phenylethyl Alcohol	**	0.24 b	0.19 b	0.52 ab	0.64 ab	1.03 a	**	0.63 a	0.41 ab	0.47 ab	0.25 b	0.11 b	NS	0.53 a	0.37 a
1-Tetradecanol	**	0.01 a	0.00 ab	0.00 b	0.00 b	0.00 ab	***	0.10 ab	0.11 a	0.04 bc	0.03 c	0.03 c	***	0.00 b	0.06 a
Terpene														0.00	0.00
Copaene	***	0.14 b	0.52 a	0.08 b	0.17 b	0.29 b	***	0.36 b	0.47 b	0.26 b	0.84 a	0.24 b	NS	0.24 a	0.43 a
Aromandendrene	NS	0.02 a	0.02 a	0.01 a	0.02 a	0.01 a	***	0.06 c	0.36 a	0.04 c	0.12 b	0.03 c	**	0.02 b	0.12 a
Terpenoid														0.00	0.00
Linalool	***	3.79 a	0.65 c	0.52 c	1.00 bc	1.26 b	***	5.46 ab	1.79 b	7.63 a	1.49 b	1.91 b	**	1.44 b	3.65 a
Pinocarveol	**	0.18 ab	0.48 a	0.09 b	0.20 ab	0.14 ab	***	0.31 bc	0.67 a	0.22 bc	0.42 ab	0.06 c	NS	0.22 a	0.34 a
Ketone														0.00	0.00
β-Ionone	***	0.45 ab	0.82 a	0.07 b	0.17 b	0.22 b	***	0.43 ab	0.58 a	0.45 ab	0.32 bc	0.13 c	NS	0.34 a	0.38 a

NS: not significant at $p > 0.05$, ** and ***: significant at $p < 0.01$ and 0.001, respectively. Values followed by different letters, within the same column, were significantly different ($p < 0.05$). SA – San Antonio; CA – Colar Albatera; CUMH – Colar UMH; CDN – Cuello Dama negro; SF – Superfig.

concentration (1.09 mg kg^{-1}) in other study with dried figs "Dottato" cv. (Russo et al., 2017). For alkane group only one compound (2,2,4,6, 6-pentamethylheptane) was detected and no significant differences were found between varieties. Copaene and caryophyllene were the two compounds found belonging to the group terpene and no significant differences were found for these compounds. On the other hand, Oliveira et al. (2010) found this compound only in pulp, while in this study it has been detected in peel and pulp (the amount found in pulp is 5.75 times higher than in peel). The linalool compound is a terpenoid for which significant differences have been found between the varieties studied. For the SA (27.98 %) variety, this compound was detected in a higher percentage than for the rest of the varieties. In the ketone group, no differences were found for compound 3,5-octadien-2-one, but for compounds 2,3-octanedione and 2-nonanone significant differences were found, being the CUMH (2.10 %) and CDN (0.73 %) varieties obtaining the highest percentage, respectively, for each compound. Finally, considering breba and fig as factors, we only found significant differences between breba and fig for compounds 2-hexenal and heptanal. Breba had the highest percentages in these compounds.

The aldehyde group represents the highest percentage of the total compounds found in the breba peel (Table 3). The main compound of this group was 2-hexenal, followed by benzaldehyde and hexanal. No significant differences were found for 2-hexenal and benzaldehyde among the varieties. Hexanal, was found in a ranged 4.62–52.72 % between varieties for CDN and CA, respectively. The percentage of hexanal detected in this study was similar to that reported by Pereira et al. (2020). Its percentage detected in fresh figs was 1.76 %. Besides, Villalobos et al. (2018) mentioned that 2-hexenal and hexanal are key compounds to the volatile aroma profile in figs. The CA variety also obtained the highest percentages for the main compounds of each one of each of the families, ester (methyl hexadecanoate 0.42 %), alcohol (1-octen-3-ol 2.46 %), terpene (copaene 0.52 %) and ketone (β-ionone 0.82 %). On the other hand, the SA variety showed a linalool percentage 5.82, 7.34, 3.78 and 3.01 times higher than the varieties CA, CUMH, CDN y SF respectively. The mean of all the varieties in peel breba for linalool 1.44 %. Previous work reported that the percentage for linalool

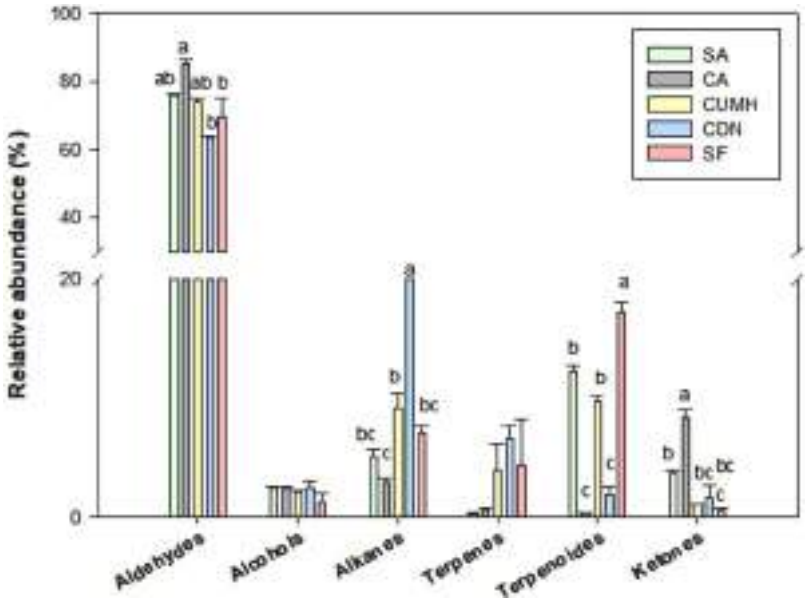

Fig. 1. Group of volatile compounds of breba pulp. SA – San Antonio; CA – Colar Albatera; CUMH – Colar UMH; CDN – Cuello Dama negro; SF – Superfig.

in fresh figs collected in national germplasm bank of the fig tree (Badajoz, Spain). Its percentage detected was 0.89 % (Pereira et al., 2020).

The aldehyde group was the one that obtained the highest representation with respect to all the compounds detected in the peel of figs. The main compound of this group was benzaldehyde, which showed the highest percentage for the SF variety (65.63 %), followed by the 2-hexenal compound with the highest percentages for the SA (37.55 %) and CDN (37.16 %) varieties, on the other hand for the compound hexanal it was the CA variety that obtained the highest percentage (28.08 %). In the group of alcohols, the two compounds with the highest percentage of the total were 1-octen-3-ol and phenylethyl alcohol, the varieties CA (3.38 %) and SA (0.63 %) respectively showed the highest percentage. For the ester group, the CA variety showed the highest percentage in compounds methyl hexanoate, while the CUMH variety showed the highest percentage for compounds methyl octanoate, 2-phenethyl acetate, and methyl hexadecanoate. Therefore, a trend is observed in the "colar" variety due to the volatile compounds of the ester group with respect to the other varieties studied. Three compounds belonging to the alcohol group were detected. The main compound of the alcohols was 1-octen-3-ol, for this compound the CA variety obtained a percentage 5.02, 1.63, 6.53 and 10.33 times higher than the SA, CUMH, CDN and SF varieties, respectively. For the terpene group, significant differences were found in the two compounds. Coapene showed a percentage range of 0.24–0.84 for SF and CDN respectively and aromandendrene showed a range of percentages of 0.03–0.36 for SF and CA, respectively. For the terpenoid group, the linalool compound was the main one, the CA variety was the one that showed the highest percentage 1.40, 4.26, 5.14 and 4 times higher than the SA, CUMH, CDN and SF variety. The last group is ketone, only β-ionone compound was detected and also the CA variety showed the highest percentage (0.58 %).

Using breba and fig as factors, only significant differences were found in the compounds 2-hexenal, benzaldehyde, benzenepropanal, undecanal, 1-tetradecanol, aromandendrene and linalool. Except for compound 2-hexenal, which showed a higher percentage in breba than in Fig. 1.66 times higher, for the rest of the compounds figs that obtained a higher percentage.

For breba, 12 volatile compounds were detected both in peel and pulp of the analyzed fruits. 2-Hexenal and 2,4-heptadienal compounds were detected in the highest percentages in the peel than in the pulp in all the varieties studied. However, octanal and 2-decenal it was the opposite. These compounds were detected in a higher percentage in pulp than in the breba peel. The CA variety showed higher percentages in peel than pulp for compounds benzaldehyde, decanal and linalool with percentages 3.06, 2.58 and 1.96 times higher respectively. For figs 2-hexenal and benzaldehyde compounds were detected in the highest percentages in the peel than in the pulp in all the varieties studied, however for octanal, 2-decenal and copaene it was the opposite, these compounds were detected in a higher percentage in pulp than in the breba peel. CA variety showed higher percentages in peel than pulp for the decanal, 1-octen-3-ol and linalool compounds with percentages 3.31, 3.05 and 4.60 times higher, respectively. Previous works (Gozlekci et al., 2011)indicated that the content of aldehydes were 4–9 times higher in pulps rather than in peel of figs varieties of Turkey ("Bursa Siyahi", "Karabakunya", "Sari Lop" and "Sultan Selim"). On the contrary, in this study the% of aldehydes detected in the peel was 1.30 times higher than for the pulp in breba and 1.29 higher in fig. On the other hand, Oliveira et al. (2010) found for "Borrasota" Tradicional and "Preta Tradicional" varieties aldehyde content was 1.48 and 1.17 times higher in peel than pulp . But for "Verbera preta" the aldehyde content in pulp was 2.87 times higher than in peel.

The data in peel and pulp of breba and figs aroma compounds is limited, several works reported that aldehydes, alcohols and ketones were the main volatile compounds contributor's aroma of figs (Russo et al., 2017) being aldehydes the most important chemical family to the of this fruits (Gozlekci et al., 2011). These volatiles compound founded in fruits of *F. carica* are related with different aromatic descriptors including high fruity, green notes and a moderate sweet and floral aroma, as well as a slight note of fatty aroma (Zidi et al., 2021).

Aldehyde represented (73.62 %) of total composition in breba pulp, the main percentage was for CA variety (85.36 %). The alcohol represented (1.32–2.49 %) of total composition and no significant differences were found in the total percentage of alcohols between the varieties studied. While for alkane the range of percentages was from (24.19 %) for the CDN variety to (2.91 %) for the CA variety. However, for terpenes no significant differences were found between varieties but for terpenoids SF variety showed the higher percentage (17.21 %). Finally, CA variety showed the highest percentage of ketone (8.31 %). According to Lachtar et al. (2022) who analyzed volatile compounds in peel and pulp of dryed fig affected by two drying methods (open sun drying and drying in a greenhouse) and different varities ("Bither Abiadh", "Bouhouli" and "Bidhi"). Its volatile profile was dominated by aldehydes (24.12–54.61

Table 4
Differences in volatile compounds (%) between the pulp of figs and figs of different varieties.

Pulp (%) Code	ANOVA	SA	CA	CUMH	CDN	SF	FSA	FCA	FCUMH	FCDN	FSF
Hexanal	***	2.70 d	64.69 a	31.46 b	3.96 d	2.86 d	26.14 bc	57.86 a	4.61 d	10.22 cd	4.34 d
2-Hexenal	NS	5.01 a	2.06 a	1.90 a	3.38 a	3.63 a	6.23 a	6.41 a	3.69 a	7.35 a	4.47 a
Heptanal	NS	0.40 a	0.53 a	0.48 a	0.20 a	0.54 a	0.79 a	2.09 a	0.21 a	5.71 a	7.77 a
Benzaldehyde	***	20.76 bcd	3.68 cd	33.82 ab	43.20 a	23.81 abc	19.43 bcd	3.17 d	43.45 a	9.05 cd	34.38 ab
2,4-Heptadienal	**	0.09 b	0.87 ab	0.25 b	0.27 b	1.01 ab	2.01 a	1.68 ab	0.42 ab	0.20 b	0.13 b
Octanal	*	2.73 a	1.55 a	2.10 a	1.84 a	4.15 a	2.64 a	1.46 a	2.88 a	3.87 a	2.89 a
2-Octenal	**	0.78 bc	2.75 abc	4.31 ab	0.91 bc	0.44 c	3.19 abc	5.12 a	1.18 bc	1.56 abc	0.67 bc
Nonanal	**	11.45 ab	5.22 b	4.20 b	8.26 b	26.11 a	8.64 b	3.57 b	10.54 ab	17.71 ab	8.72 b
2-Nonenal	**	1.11 ab	0.49 b	1.01 ab	0.83 b	1.09 ab	0.92 b	0.62 b	1.84 a	0.92 b	1.17 ab
Decanal	NS	3.13 a	0.75 a	1.33 a	1.87 a	1.96 a	2.90 a	0.77 a	2.28 a	2.35 a	1.76 a
2,4-Nonadienal	NS	0.82 a	1.12 a	1.39 a	0.31 a	0.47 a	1.36 a	1.55 a	0.69 a	0.66 a	0.45 a
2-Decenal	NS	0.53 a	0.43 a	0.66 a	0.40 a	0.51 a	0.64 a	0.57 a	0.59 a	0.78 a	0.37 a
3-Phenyl 2-propenal	**	2.04 abc	1.04 bc	0.54 c	1.45 abc	1.28 abc	1.17 bc	0.49 c	1.62 abc	2.91 a	2.28 ab
1-Octen-3-ol	**	0.65 bc	1.11 abc	2.34 a	0.54 bc	0.63 bc	1.59 abc	2.00 ab	0.65 bc	1.04 abc	0.39 c
1-Dodecanol	*	1.88 a	0.51 b	0.57 b	0.85 ab	0.94 ab	0.90 ab	0.48 b	1.46 ab	1.39 ab	0.93 ab
2,2,4,6,6-Pentamethylheptane	**	12.51 abcd	8.27 bcd	2.06 d	23.07 ab	20.63 abc	5.12 cd	2.92 d	9.16 abcd	24.20 a	7.10 cd
Copaene	NS	2.12 a	0.99 a	0.35 a	1.03 a	1.51 a	0.18 a	0.39 a	1.85 a	3.07 a	2.19 a
Caryophyllene	NS	2.14 a	0.86 a	0.18 a	2.10 a	2.27 a	0.12 a	0.22 a	2.10 a	3.49 a	2.15 a
Linalool	***	27.98 a	0.39 d	7.19 bcd	4.04 cd	5.47 cd	12.24 bc	0.33 d	9.77 bcd	1.89 cd	17.21 ab
2,3-Octanedione	**	0.79 b	0.88 b	2.10 a	0.51 b	0.52 b	1.35 ab	1.42 ab	0.83 b	0.70 b	0.44 b
3,5-Octadien-2-one	***	0.17 b	1.51 b	1.68 b	0.26 b	0.10 b	1.82 b	5.20 a	0.11 b	0.35 b	0.11 b
2-Nonanone	**	0.23 b	0.31 b	0.10 b	0.73 ab	0.08 b	0.59 b	1.69 a	0.09 b	0.60 b	0.10 b

NS: not significant at $p > 0.05$, ** and ***: significant at $p < 0.01$ and 0.001, respectively. Values followed by different letters, within the same column, were significantly different ($p < 0.05$). SA –Breba San Antonio; CA – Breba Colar Albatera; CUMH – Breba Colar UMH; CDN – Breba Cuello Dama negro; SF – Breba Superfig; FSA – Fig San Antonio; FCA – Fig Colar Albatera; FCUMH – Fig Colar UMH; FCDN – Fig Cuello Dama negro; FSF – Fig Superfig.

%) and also detected alcohols (6.06–13.37 %) and ketone (2.46–3.42 %).

Aldehydes (95.74 %) represent the highest percentage of the composition of the breba peel, the two main varieties by their percentage of aldehyde are CUMH (98.07 %) and CDN (97.13 %). On the other hand, CA variety showed the highest percentages for esters (1.00 %), alcohols (2.65 %), terpenes (0.53 %) and ketones (0.82 %) content. However, for the terpenoids content, the highest percentage was obtained by SA (3.97 %). In recent years, some authors have reported that aldehydes were the most abundant volatile compounds in figs (Gozlekci et al., 2011; Lachtar et al., 2022; Pereira et al., 2020; Zidi et al., 2021). Moreover, the concentration of aldehydes was influenced by variety (Pereira et al., 2020).

For fig pulp, the families with the highest percentage of total compounds in order from highest to lowest were aldehydes (70.98 %), alkanes (13.31 %), terpenoids (9.01 %), terpenes (2.71 %), alcohols (2.00 %) and ketones (1.99 %). Significant differences between varieties have been found for aldehyde and terpenoids only. CA (85.18 %) and CUMH

Table 5
Differences in volatile compounds (%) between the peel of figs and figs of different varieties.

Peel (%) Code	ANOVA	SA	CA	CUMH	CDN	SF	FSA	FCA	FCUMH	FCDN	FSF
Hexanal	***	7.60 c	52.72 a	5.23 c	4.62 c	8.89 c	2.09 c	28.08 b	9.57 c	4.56 c	2.91 c
2-Hexenal	***	36.78 abc	11.86 bc	51.36 a	54.86 a	45.68 ab	37.16 abc	14.36 bc	6.24 c	37.55 abc	25.24 abc
2,4-Hexadienal	**	0.27 abc	0.44 ab	0.13 abc	0.48 a	0.04 c	0.30 abc	0.33 abc	0.15 abc	0.13 abc	0.09 bc
Benzaldehyde	***	40.59 ab	9.72 b	37.74 ab	32.72 ab	33.29 ab	47.12 ab	28.74 ab	62.48 a	48.91 ab	65.63 a
2,4-Heptadienal	***	3.27 a	3.74 a	0.51 cd	0.28 d	1.59 bc	0.36 cd	1.33 bcd	1.82 b	0.46 cd	0.53 cd
Octanal	***	0.66 bc	1.44 a	0.50 bc	0.34 c	0.80 abc	0.59 bc	1.39 a	1.15 ab	0.35 c	0.29 c
2-Octenal	***	0.90 bc	5.42 a	0.39 c	0.48 c	0.82 bc	0.56 c	2.89 b	1.87 bc	0.32 c	0.24 c
Nonanal	***	1.89 bc	4.05 b	1.14 c	1.60 bc	2.18 bc	1.38 bc	9.39 a	2.71 bc	1.72 bc	1.20 c
2,6-Nonadienal	***	0.36 d	2.06 a	0.55 cd	1.02 bcd	1.30 b	1.03 bcd	1.08 bcd	0.60 bcd	1.10 bc	0.39 cd
Benzenepropanal	***	0.16 b	0.01 e	0.01 e	0.05 cde	0.03 de	0.15 b	0.70 a	0.11 bcd	0.18 b	0.11 bc
Decanal	***	1.40 bc	1.98 ab	0.37 d	0.63 cd	0.84 cd	0.48 d	2.47 a	0.57 cd	0.44 d	0.27 d
2-Decenal	NS	0.10 a	0.24 a	0.12 a	0.02 a	0.03 a	0.38 a	0.29 a	0.10 a	0.08 a	0.06 a
Undecanal	***	0.13 cde	0.21 bc	0.02 f	0.03 f	0.06 ef	0.16 cd	0.28 ab	0.31 a	0.05 ef	0.09 def
Methyl hexanoate	***	0.19 b	0.37 a	0.06 b	0.08 b	0.10 b	0.03 b	0.47 a	0.42 a	0.02 b	0.02 b
Methyl octanoate	***	0.17 bc	0.18 b	0.07 d	0.02 d	0.01 d	0.09 cd	0.29 a	0.32 a	0.06 d	0.05 d
2-Phenethyl acetate	**	0.01 b	0.02 b	0.14 ab	0.21 ab	0.57 a	0.11 b	0.08 b	0.29 ab	0.06 b	0.05 b
Methyl hexadecanoate	***	0.10 b	0.42 a	0.01 c	0.01 c	0.01 c	0.01 c	0.10 b	0.13 b	0.01 c	0.01 c
1-Octen-3-ol	***	0.61 c	2.46 b	0.36 c	0.35 c	0.84 c	0.67 c	3.38 a	2.07 b	0.52 c	0.33 c
Phenylethyl Alcohol	***	0.24 b	0.19 b	0.52 ab	0.64 ab	1.03 a	0.63 ab	0.41 b	0.47 b	0.25 b	0.11 b
1-Tetradecanol	***	0.01 b	0.00 b	0.00 b	0.00 b	0.00 b	0.10 a	0.11 a	0.04 b	0.03 b	0.03 b
Copaene	***	0.14 de	0.52 b	0.08 e	0.17 de	0.29 bcde	0.36 bcd	0.47 bc	0.26 bcde	0.84 a	0.24 cde
Aromandendrene	***	0.02 d	0.02 d	0.01 d	0.02 d	0.01 d	0.06 c	0.36 a	0.04 cd	0.12 b	0.03 cd
Linalool	***	3.79 bc	0.65 cd	0.52 d	1.00 cd	1.26 cd	5.46 ab	1.79 cd	7.63 a	1.49 cd	1.91 cd
Pinocarveol	***	0.18 bcd	0.48 ab	0.09 d	0.20 bcd	0.14 cd	0.31 bcd	0.67 a	0.22 bcd	0.42 abc	0.06 d
β-Ionone	***	0.45 bcd	0.82 a	0.07 e	0.17 cde	0.22 cde	0.43 bcd	0.58 ab	0.45 bc	0.32 bcde	0.13 de

NS: not significant at $p > 0.05$, ** and ***: significant at $p < 0.01$ and 0.001, respectively. Values followed by different letters, within the same column, were significantly different ($p < 0.05$). SA –Breba San Antonio; CA – Breba Colar Albatera; CUMH – Breba Colar UMH; CDN – Breba Cuello Dama negro; SF – Breba Superfig; FSA – Fig San Antonio; FCA – Fig Colar Albatera; FCUMH – Fig Colar UMH; FCDN – Fig Cuello Dama negro; FSF – Fig Superfig.

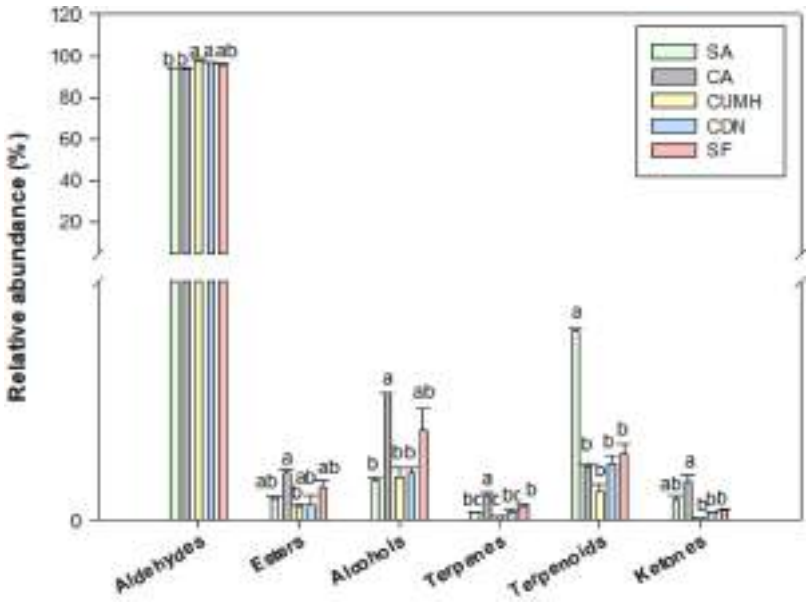

Fig. 2. Group of volatile compounds of breba peel. SA – San Antonio; CA – Colar Albatera; CUMH – Colar UMH; CDN – Cuello Dama negro; SF – Superfig.

(83.45 %) showed the highest percentage of aldehyde while SA variety (27.98 %) showed the highest percentage of terpenoids. Similar chemical classes were reported by Russo et al. (2017) for Italian oven dried figs: Aldehydes, furans, ketones, alcohols, terpenes, and esters in descending order of concentration.

The three main families of compounds in breba peel, in order of relevance, were aldehyde (92.72 %), terpenoids (3.99 %), and alcohols (1.8 3 %). SF (97.04 %) was the varieties with the highest percentage of aldehydes. For terpenoids, the CUMH variety (7.84 %) showed the highest percentage. Finally, for alcohols CA variety (3.89 %) showed the highest percentage. This same variety also obtained the highest percentages for the ketones (0.58 %). Moreover, aldehydes, terpenes and alcohols were also the main family compounds detected by (Andreu-Coll et al., 2020) in prickly pear fruit pulp from Spanish varieties. These results suggest that not only does the variety of brebas and figs influence the volatile compounds, but there are also significant differences between the edible (pulp) and non-edible (peels) parts of the fruit. These authors (Del Caro and Piga, 2008; Harzallah et al., 2016; Hssaini et al., 2021) indicated differences in contain concentrations of nutrients and bioactive compounds between different parts of the fruit of brebas and figs.

3.2. Comparation of volatile compounds between breba and figs of different varieties

For the volatile compounds detected in the pulp (Table 4), significant differences have been found between breba and figs of the different cultivars studied in 15 of the 22 volatile compounds detected. In general, for most of the volatile compounds detected in the pulp, the highest percentages have been found in figs. Fig CDN variety obtained the highest percentages for six compounds 2-hexenal (7.35 %), 2-decenal (0.78 %), 3-phenyl 2-propenal (2.91 %), 2,2,4,6,6-pentamethylheptane (24.19 %), copaene (3.07 %) and caryophyllene (3.48 %) but for hexanal compound, breba of CA variety (64.69 %) showed highest percentage and for benzaldehyde was fig of CUMH variety (43.45 %) the one with the highest percentage. In addition, breba SA showed a high linalool content up to 84.42 times higher than the content detected in fig CA, although fig CA variety stood out for its content in ketone compounds, especially 3,5-Octadien-2-one and 2-Nonanone with percentages 50.01 and 21.68 times higher, respectively for other varieties such as breba SF. All the varieties studied have shown a greater amount of 2-hexenal in breba than figs, in addition the SA variety showed a percentage 9.67, 23.66 and 10.82 times higher in fig pulp compared to breba pulp for the compounds hexanal, 2,4- heptadienal and 3,5-octadien-2-one, while for the compounds copaene and caryophyllene it was the opposite, higher values were obtained in breba than in fig. Regarding the CA and CUMH variety, both have shown higher results in breba than in fig for hexanal and 3,5-octadien-2-one, but the results of breba CUMH were 16.08 times higher than those detected in fig for 3,5-octadien-2-one. In summary, the composition of these compounds varies between breba and figs, as well as among different fig varieties studied. The genotype factor has a more prominent influence on the composition of volatile compounds in breba and figs compared to the different environmental conditions at the time when the brebas and figs are harvested. This highlights the importance of genetics in determining the sensory characteristics of brebas and figs varieties.

In other hand, attend to peel (Table 5), the fig of the variety CA was the fruit that obtained the highest percentage in the highest number of compounds (nonanal, benzenepropanal, decanal, methyl hexanoate, 1-octen-3-ol, 1-tetradecanol, aromandendrene and pinocarveol) followed by breba CA (hexanal, 2,4-heptadienal, octanal, 2-octenal, 2,6-nonadienal, methyl hexadecanoate and β-ionone) and follow by fig CUMH (undecanal, methyl octanoate and linalool). Although for the main compounds, benzaldehyde, 2-hexenal and hexanal the highest percentages were detected for fig SF (65.63 %), breba CDN (54.86 %) and breba CA (52.72 %), respectively. Hexanal and 2-hexenal contribute to different flavors, with hexanal giving a fresh, cut grass aroma, and 2-hexenal providing almond, apple, green, sweet, and vegetable notes (SAFC, 2012). In addition, fig of the SA variety obtained one of the highest percentages for the compound 2-decenal (0.38 %), while the breba SF showed the highest percentage for 2-phenethyl acetate (0.57 %) and phenylethyl alcohol (1.03 %) and fig CDN showed the highest percentage for copaene compound (0.84 %). On the other hand, it has been detected that the compounds benzaldehyde, undecanal, 1-tetradecanol, aromandendrene and linalool the percentage obtained in fig was higher than in breba in all varieties. However, the SA variety showed higher percentages in breba than in fig for the compounds 2,4-heptadienal, methyl hexanoate and methyl hexadecanoate 9.01, 5.49 and 8.90 times higher, respectively. In addition, the SF variety showed higher values in breba than in fig up to 12.51 times for the compound 2-phenethyl acetate. The peel of brebas and figs exhibits significant differences in the main compounds detected. The findings imply that, unlike

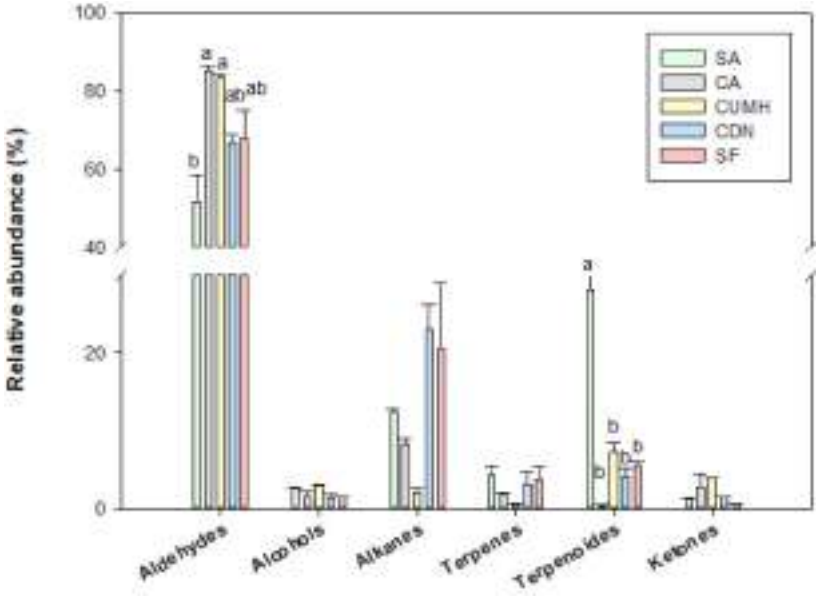

Fig. 3. Group of volatile compounds of fig pulp. SA – San Antonio; CA – Colar Albatera; CUMH – Colar UMH; CDN – Cuello Dama negro; SF – Superfig.

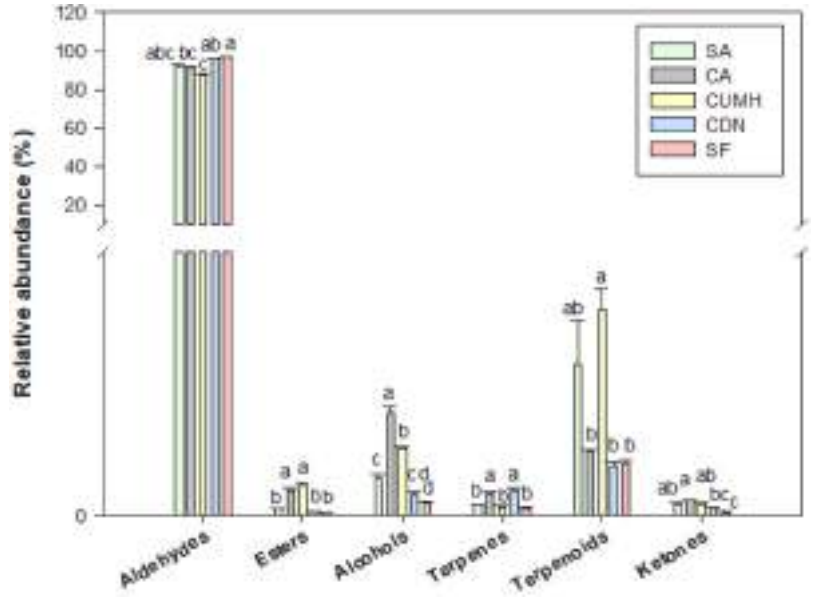

Fig. 4. Group of volatile compounds of fig peel. SA – San Antonio; CA – Colar Albatera; CUMH – Colar UMH; CDN – Cuello Dama negro; SF – Superfig.

the pulp fruit composition where genotype played a more prominent role, the peel composition is more susceptible to variations influenced by environmental conditions (temperature, humidity, sunlight exposure) during the time of harvest. As previously reported by Najafian et al. (2022) who studied the phytochemical diversity in lavender plants across different seasons. The variation in phytochemical content across seasons is a common phenomenon in many plant species. In addition El-Zaeddi et al. (2020) reported that there was a significant effect of harvest date on the volatile compounds of four aromatic herbs (dill, parsley, coriander, and mint) and suggests that the timing of harvest can influence the chemical composition, flavor, and potentially the medicinal properties of these herbs. No previous studies have been found that compare volatile compounds between breba and fig, which makes it difficult to discuss these results (Figs. 2,3 and 4).

4. Conclusion

This is the first study comparing peels and pulps of breba and figs fruits to help improve the knowledge of volatile profile in four different Spanish varieties. The CA variety demonstrated a higher content of key volatile compounds, suggesting that different varieties may exhibit distinct aromatic characteristics. Notably, the peel was richer in key volatile compounds compared to the pulp, especially in the CA variety. This variation could be attributed to genetic biotypes and pedo-climatic differences related to the location. Therefore, it is underscoring the importance of considering the entire fruit for consumption fresh, including its non-edible parts. The findings from this study have implications for selecting varieties with desirable volatile profiles and potentially minimizing food waste. In fact, understanding how volatile compounds impact brebas and figs fruits can have implications for agriculture, horticulture, and the pharmaceutical industry.

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Candela Teruel-Andreu: Writing – review & editing, Writing – original draft, Formal analysis. **Hanán Issa-Issa:** Writing – review & editing, Formal analysis. **Luis Noguera-Artiaga:** Writing – review & editing, Methodology, Conceptualization. **Esther Sendra:** Writing – review & editing, Methodology, Conceptualization. **Francisca Hernández:** Writing – review & editing, Methodology, Conceptualization. **Marina Cano-Lamadrid:** Writing – review & editing, Supervision, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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TECHNO-FUNCTIONAL PROPERTIES AND ENHANCED CONSUMER ACCEPTANCE OF WHIPPED FERMENTED MILK WITH *FICUS CARICA* L. BY-PRODUCTS.

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Techno-functional properties and enhanced consumer acceptance of whipped fermented milk with *Ficus carica* L. By-products

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ABSTRACT

The development of new fermented milks formulations enriched with fruit by-products may widen the existing market offer of products matching consumer demands on novel, and "*no artificial added sugars*" products. Unmarketable fig fruit, food by-product, could be considered as a potential ingredient to develop a new dairy product. The aim of this study was to study the consumer acceptance of fermented milk enriched with different percentage of pasteurized fig purée (by-products) and their technological properties. It was found that the quantity of fig puree added influenced fermented milks texture and spontaneous syneresis. Formulations containing 40% fig puree showed the highest values of firmness, consistency, cohesiveness, and viscosity index with improvements seen from 20% fig puree addition. Furthermore, the inclusion of fig puree in fermented milks reduced the levels of lactic acid bacteria comparing with control samples, but the microbial load was higher than 10^6 UFC g^{-1} LAB (estimated counts in MRS) and 9 UFC g^{-1} LAC (estimated counts in M17). Polyphenolic content increased with fig puree percentage, enhancing antioxidant activity. Volatile compound analysis identified hexanoic acid, acetoin, and butanoic acid as predominant in enriched fermented milks. It is also worth highlighting that sensory evaluation revealed better ratings for texture and sweetness acceptance in formulations containing 30% and 40% of fig puree, correlating with instrumental data. Overall, the quality parameters were maintained and even improved, leading to high consumer acceptability ratings.

1. Introduction

Dairy products are foods with great potential for fortification due to their ability to act as vehicles for bioactive ingredients. Recent years have seen an increased interest in fortifying dairy products with ingredients with functional and techno-functional properties. In particular, fermented milks, including yoghurt, stands out for its ability to increase the bioavailability of nutrients (Machado et al., 2022). Fermented milk is a milk product obtained by fermentation by the action of suitable microorganisms and resulting in reduction of pH with or without coagulation (*iso*-electric precipitation). These starter microorganisms should be viable, active, and abundant in the product to the date of minimum durability (FAO, 2011). Different strategies are currently being applied to incorporate fibre, bioactive compounds and functional ingredients that allow the clean label trend to be applied to fermented milks, becoming very important within the dairy industry (Du et al., 2022). Apart from that, sustainable strategies have also been incorporating in the development of dairy products, specially enriched fermented milks with improved nutritional, functional, and physical properties during last decades (Oliveira et al., 2022). Among the possibilities, the addition of fruit by-products is a good option to widen the commercial product offer and/or to develop new dairy products as a source of fibre, colouring and antioxidants (El-Said et al., 2014). In this sense, innovation based on the use of fruits not suitable for fresh consumption is a way of adding value while reducing food loss and companies take advantage of this resource to turn their by-products into profitable (Cano-Lamadrid and Artés-Hernández, 2022; Cassani et al.,2022; Teruel-Andreu et al., 2021). Several fermented milks enriched with ingredients and extracts from fruits have been previously reported such as grape (Silva et al., 2022), pomegranate (Cano-Lamadrid et al., 2017; Trigueros et al., 2012), cherry (Sánchez-Bravo et al.,2018), mango (Saeed and Ramzan, 2021), mulberry pomace (Du et al., 2023), and date (Almusallam et al., 2021), among others.

On the other hand, the main figs-producing countries in the world

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are Turkey with 350,000 t in 2022. In Europe, 74,460 t was produced in 2022, being Spain the major producer of figs/brebas (43,500 t), followed by Italy (9,480 t) (FAOSTAT, 2024). Fig cultivation produces large quantities of figs which are not all marketed fresh, around 50 %, due to inadequate or excessive ripening, spoilage, small size, soft texture or low quality as table fruit, but which can be used as an ingredient in other products, such as dairy products. Not only fig fruits are notably abundant in fiber, amino acids (such as aspartic acid and glutamine), vitamins (including thiamine and riboflavin), carotenoids (like lutein, cryptoxanthin, lycopene, and β-carotene), minerals (such as iron, calcium, and potassium), antioxidant polyphenols (cyanidin, chlorogenic acid, rutin, luteolin and catechin), sugars, and organic acids, but also by-products, mainly unmarketable fruits and leaves (Teruel-Andreu et al., 2021; Teruel-Andreu et al., 2023a, 2023b).

Therefore, the development of new products based on figs fruits which are unmarketable in fresh, can also be interesting from a nutritional, sensorial and techno-functional point of view. The aim of this study was to study the consumer acceptance of fermented milk enriched with different percentage of pasteurized fig purée (by-products) and their technological properties such as microbial counts, texture, gel stability, physicochemical parameters, total polyphenol content, antioxidant activity and volatile compounds in fig purée enriched fermented milk with different percentage (10, 20, 30, and 40 %). The expected outcomes are: i) To determine the level of consumer acceptance for fermented milk enriched with different percentages of pasteurized fig purée (by-products); ii) To assess the estimated microbial counts of lactobacilli in MRS and lactococci in M17 in the enriched fermented milk; iii) To evaluate the gel stability and texture properties, including firmness, consistency, cohesiveness, and viscosity index, of the fermented milk with varying fig purée concentrations; iv) To analyse the colour and pH levels of the fermented milk containing different percentages of fig purée; v) To quantify the total polyphenol content and antioxidant activity (DPPH) in the enriched fermented milk; and, vi) To identify and quantify the volatile compounds present in the fermented milk enriched with different percentages of fig purée. These outcomes will provide a comprehensive understanding of the technological properties and consumer acceptance of fermented milk products enriched with fig purée, highlighting their potential sensorial, functional, and techno-functional benefit.

2. Materials and methods

2.1. Preparation of the pasteurized fig purée

For the preparation of the fig puree, figs of the Colar variety, which were not harvested on farms for commercial propose, in Albatera (Alicante, Spain) in the 2023 season were collected, and stored frozen until the time of processing. More information about the composition of this by-product can be found in previous study (Teruel-Andreu et al., 2023b). Initially, a disinfection process was carried out by immersing the fruits in a solution of 200 ppm of peracetic acid (Citrocide® PC, Citrosol, Valencia, Spain) at 15 °C for 10 min, followed by rinsing with running water at 15 °C for 5 min (5 L s^{-1}). The puree was prepared using a Thermomix® TM5, starting with the crushing of the fruits (1 min, speed 5: 2000 rpm; 1 min, speed 7: 4200 rpm), assuring a homogeneous fig paste (no sieve was used), followed by thermal treatment (specifically pasteurization). Different combinations of time and temperature of thermal treatment were applied to different batches of puree to achieve a microbiologically stable puree with the minimal treatment necessary. It was based on previous research (Cano-Lamadrid et al., 2018a; Cano-Lamadridet al., 2018b) into fig smoothies with important modifications, ensuring the microbiological stability of the final product for use as an ingredient. Microbiological counts of enterobacteria, *E. coli*, mesophilic aerobic bacteria, anaerobic bacteria, moulds, and yeasts were conducted to determine the optimal thermal treatment to achieve a microbiologically stable puree. Finally, a disinfection process with peracetic acid in combination with heat treatment (100 °C, 20 min) was conducted to obtain a microbiologically stable puree for fig puree production. Data was not shown. The pasteurized puree was distributed in sterile containers, immediately cooled, and stored for 24 h before being added to fermented milk. Sugar profile was also analysed in the pasteurized fig purée. The content of glucose and fructose in pasteurized fig puree was quantified, being 15.5 and 15.6 g/100g, respectively. The addition of pasteurized fig purée was added after fermentation.

2.2. Fermented milks manufacture

Fig. 1 shows the experimental design to produce 3 independent batches of whipped fermented milk (2 for techno-functional and functional parameters and 1 for sensory analysis) made from UHT whole cow's milk (3.6 % fat content, Hacendado, Spain) to which lyophilized starter culture containing *Streptococcus thermophilus* (Lac), *Lactobacillus delbrueckii* ssp. *lactis*, and *Lactobacillus delbrueckii* ssp. *bulgaricus* (Lab) (CHOOZITTM MY800 LYO 5 DCU, Rhodia Food-Danisco A/S, Sassenage, France). Fermented milk was made following previous studies (Cano-Lamadrid et al., 2017; Jiménez-Redondo et al., 2022; Muelas et al., 2022) with some modifications. To facilitate dosage according to manufacturer instructions the lyophilized culture was poured into 20 mL of sterile peptone water (Merck KGa, SigmaAldrich, United States) and kept in a water bath at 43 °C for 20 min to hydrate. One litre capacity Pyrex bottles were sterilised, and UHT milk was added under hygienic conditions (laminar flow cabinet) adding milk volumes according to the formulation to be developed (ranging from 1 L for control and 600 ml for those formulations to have 40 % fig puree). Rehydrated culture dosage was 1000 Âµl per litre of milk. Once the starter culture was inoculated into the bottles, they were manually shaken for 1 min and incubated at 43 °C until they reached a pH of 4.6. Once this pH was reached, they were refrigerated for 24 h before being mixed and shaked with the fig puree. Finally, a determined amount of pasteurized puree (0, 10, 20, 30, and 40 %) was weighed and added to each fermented milk bottle, which were manually shaken until obtaining a homogeneous - drinking fermented milk. It was dispensed into sterile containers and kept refrigerated for 24 h at 4 °C before the start of the analyses (T1).

2.3. pH

The pH was measured using a pH-meter (model pH/Ion 510, Eutech Instruments Pte Ltd., Singapore). The measurements were taken at 20 °C-. There were 3 replicates of the two batches of each formulation, i. e. a total of 6 replicates.

2.4. Microbial load

Microbial load was analysed as previously described by Trigueros et al. (2012). MRS (Merck KGa, SigmaAldrich, United States) agar was used for estimating Lactobacilli counts (*Lactobacillus delbrueckii* ssp. *lactis*, and *Lactobacillus delbrueckii* ssp. *bulgaricus*, Lab, 37 °C, microaerophilia, 48 h), M17 (Merck KGa, SigmaAldrich, United States) was used for estimating Lactococci counts (*Streptococcus thermophilus*, Lac, 30 °C, aerobiosis, 24 h) and Rose Bengal Agar for Molds and Yeasts (Merck KGa, SigmaAldrich, United States) (26 °C, aerobiosis, 72 h). MRS medium is formulated for estimating Lactobacillus spp. counts and contains a combination of carbon sources, nitrogen, vitamins and salts that favour the growth of Lactobacillus. The acidic pH of MRS favours the growth of Lactobacillus, which thrives in slightly acidic conditions and helps suppress the growth of many other bacteria, in fact, in the 70's the scientific community realized that streptococci were not able to grow well in MRS. On the other hand, M17 medium was designed for the estimation of the growth of Streptococcus and other related microorganisms (lactococci) which contains a mixture of tryptone peptone or polypeptone, peptone, yeast extract and salts, which support the growth of Streptococcus mainly aided by the buffering capacity of M17

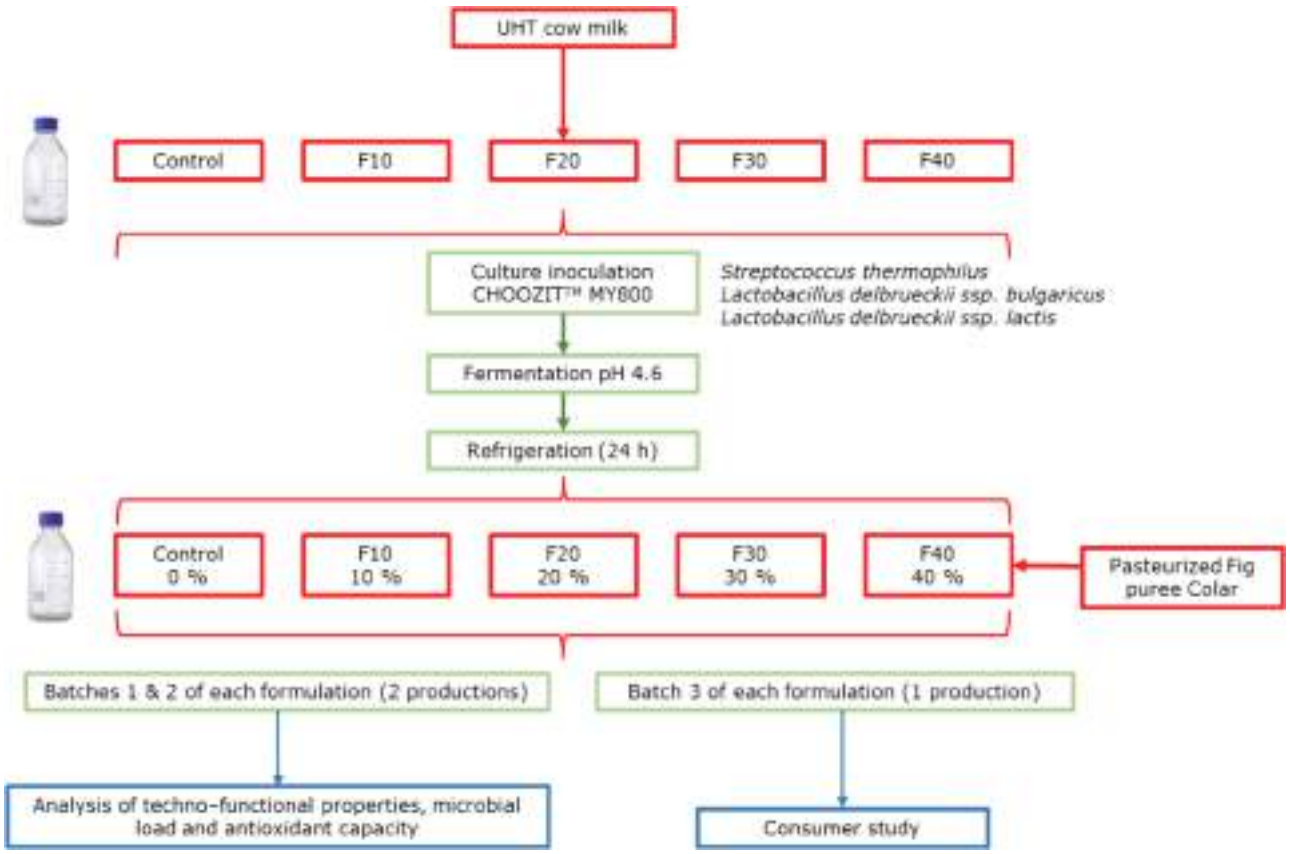

Fig. 1. Experimental design.

provided by disodium-β-glycerophosphate that buffers the medium as acid is produced from the fermentation of lactose. The presence of ascorbic acid stimulates the growth of lactic streptococci, and magnesium sulfate provides essential ions for growth. Maintaining a stable pH, which is crucial for the growth and metabolic activities of Streptococcus cannot be provided in MRS. Therefore, the gold standards for the estimation of *S. thermophilus* and *L. bulgaricus* counts are M17 and MRS respectively. This ensures that the culture conditions are optimal for the growth of each specific bacteria, allowing for accurate and meaningful counts and due to the use of standardized procedures. Moulds and yeasts were not detected in the samples; therefore, the results are not shown. The data was shown as a estimation of LAB in MRS and LAC in M17 counts. There were 3 replicates of the two batches of each formulation, i.e. a total of 6 replicates.

2.5. Cielab coordinates

The CIEL*a*b* color space of fermented milks was studied, and the following color coordinates were evaluated: lightness (L*), redness (a*, green–red coordinate), and yellowness (b*, blue-yellow coordinate). Color determinations were made at 12 ± 2 °C by means of a Minolta CM-2002 (Minolta Camera Co., Osaka, Japan) spectrophotometer, with a liquid accessory CR-A70 (Minolta Camera Co., Osaka, Japan), with illuminant D65 and an observer of 10°. The equipment was daily calibrated with the white plate provided by Minolta. pH was determined. There were 9 replicates of the two batches of each formulation, i.e. a total of 27 replicates.

2.6. Texture test and gel stability

Penetration test was performed with a Texture Analyser TA-XT2 (Stable Micro Systems, Surrey, England) and a 5 kg load cell was used. Constant speed penetration tests were performed directly on cylindrical containers (4.5 cm diameter, 4 cm height) (Trigueros et al., 2012). All instrumental texture analyses were conducted at 8 °C and spontaneous syneresis was removed previously. Exponent software was used for texture data curation, calculating Firmness, Consistency, Cohesiveness, and Viscosity Index. This is a 'destructive' test as no structure recovery is allowed. A cylindrical probe 10 mm diameter ebonite (P-10) was introduced 15 mm into the samples at a speed of 1 mm/s. Triplicate measures for each yogurt were performed.

Gel stability was visually assessed after incubation (Spontaneous syneresis) and determined by quantifying the volume of whey removed from curd after centrifugation (syneresis). There were 3 replicates of the two batches of each formulation, i.e. a total of 6 replicates.

2.7. Organic acid and sugar identification and quantification

Samples (5 g) were homogenized in 10 mL ultrapure water acidified with 0.1 % phosphoric acid H_3PO_4 and shaken vigorously (IKA® T25 digital ULTRA-TURRAX®, IKA® Werke Staufen, Germany) for 20 s at 13,500 rpm and centrifuged for 20 min at 15,000 rpm at 4 °C (Centrifuge Sigma 3–16 PK 10330 rotor 12158-H 25° angle, Shropshire, United Kingdom). The supernatants fluids were filtered through 0.45 μm membrane filters (Millipore Corporation, Bedford, USA). Samples (10 μL) were injected into a cation exchange column (Supelcogel C-610H, 300 × 7.8 mm, Supelco, Bellefonte) with a precolumn (Supelguard-H, 50 × 4.6 mm, Supelco, Bellefonte), using 0.1 % H_3PO_4 as mobile phase, at an operating flow rate of 0.5 mL min^{-1}. A Hewlett Packard HP-1100 instrument (Woldbronn, Germany) coupled with two detectors: DAD G1315A (set at 210 nm) and RID G-1362 A was used. Standards of organic acids and sugars were obtained from Supelco. Samples were run at 30 °C and the run time was 30 min. Peaks were identified by comparison with retention time of the standards and

quantified by regression formula obtained with the standards. There were 3 replicates of the two batches of each formulation, i.e. a total of 6 replicates.

2.8. Total polyphenolic content (TPC) and antioxidant capacity

Methanol extract (Merck KGa, SigmaAldrich, United States) was prepared as follows: sample (1 mL) were mixed with 10 mL of MeOH/water (80:20, v/v) + 1 %HCl, sonicated at 20 °C for 15 min, and left for 24 h at 4 °C. Then, the extract was again sonicated for 15 min, and centrifuged at 15 000 × g for 10 min. Total phenolics were extracted according to the protocol by other authors (Singleton et al.,1999) Absorption was measured using a UV–Vis Uvikon XS spectrophotometer (Bio-Tek Instruments, Saint Quentin Yvelines,France). For quantification, 5 concentrations of gallic acid (Merck KGa, SigmaAldrich, United States) (50, 100, 150, 200 and 250 mM) were prepared and the results were expressed in mg gallic acid equivalents per gram g of fresh weight (fw).The antioxidant capacity (DPPH scarvening activity) was carried out following previous study (Brand-Williams et al., 1995). Antioxidant activity was expressed as mM Trolox g^{-1} of fresh weight as calculated from a Trolox calibration curve (Merck KGa, SigmaAldrich, United States) (0.15, 0.30, 0.5, 0.75 and 1 mM). There were 3 replicates of the two batches of each formulation, i.e. a total of 6 replicates.

2.9. Volatile composition profile

The extraction of the volatile compounds of fermented milks enriched figs puree was carried out by headspace solid phase microextraction (HS_SPME) method. Briefly, 3 g of sample was weighted and added to a hermetic vial with polypropylene cap and PTFE (polytetrafluoroethylene)/silicone septa, together with 1 g NaCl. The vial was placed in an AOC-6000 Plus autosampler (Shimadzu Corporation, Kyoto, Japan) and, after 5 min of equilibration time, a 50/30 μm DVB/CAR/PDMS fiber (1 cm) was exposed to the sample headspace for 50 min at 50 °C (with agitation, 250 rpm). The separation and identification of compounds was done by GC2030 (Shimadzu Scientific Instruments, Inc., Columbia, MD, USA), in a Sapiens X5MS column (Teknokroma, Barcelona, Spain), of 30 m x 0.25 mm x 0.25 μm (length, diameter, and film thickness, respectively), and coupled with a mass spectrometer detector (TQ8040 NX triple quadrupole mass spectrometer; Shimadzu Scientific Instruments, Inc., Columbia, MD, USA). Only the single quadrupole acquisition mode was used on the TQ8040 NX (Q3 Scan; scan speed 2000 amu s^{-1}; mass range 35–300 m z^{-1}; event time 0.100 s). The oven temperature program was as follows: (i) initial temperature of 50 °C; (ii), increment of 2 °C min^{-1} up to 130 °C min^{-1} and hold for 5.00 min; (iii) increment of 10 °C min^{-1} up to 180 °C, and hold for 5.00 min; (iv), increment of 20 °C min^{-1} up to 280 °C, and hold 5.00 min. Helium column head pressure was 53.5 kPa (constant linear velocity mode of 36.3 cm s^{-1}). Injector, ion source, and interface were at 220, 230, and 280 °C, respectively. Helium was used as carrier gas, column flow 1.00 mL/min, split less and purge flow of 6 mL min^{-1}. Retention indexes of a commercial alkane standard mixture (Sigma-Aldrich, Steinheim, Germany) were used to identify the compounds, as well as the NIST 17 Mass Spectral and Retention Index Libraries. The identification was considered tentative when it was based only on mass spectral data, and only compounds with spectra similarity > 90 % were considered as correct hits. Linear retention similarity filter was set at ± 10 units. This volatile compound extraction method has been previously used for the analysis of different food matrices, including fig fruits (Teruel-Andreu et al., 2024). There were 2 replicates of the two batches of each formulation, i.e. a total of 4 replicates.

2.10. Sensory analysis

A sample group of 60 consumers was recruited at CIAGRO (Miguel Hernández University, Orihuela, Spain) aged between 18 and 70 years. The main requirement for their recruitment was that they regularly consumed yoghurt/fermented milks and added fruit. The consumer study was conducted at the UMH facilities in Orihuela. Consumers were asked about their global satisfaction degree using a 9-point hedonic scale (1 = dislike extremely, 5 = neither like nor dislike, and 9 = like extremely), together with questions regarding attributes intensity using a Just About Right (JAR) scale. Each consumer was served ~ 25 mL of each sample coded with 3-digit numbers, together with the questionnaire. Water and unsalted crackers were provided to consumers between samples for palate cleaning. Before starting the evaluation of the enriched fermented milks, each consumer was asked to taste the control sample without the addition of pasteurised fig puree. They did not have to evaluate it, they simply had to know the milk matrix in which the different percentages of pasteurised fig puree were added. The consumer study was performed according to the principles established by the Declaration of Helsinki, and the survey was approved by the UMH research ethics committee.

2.11. Statistical analysis

Statistical analysis and comparison among means were carried out using the statistical package SPSS 24.0 (IBM SPSS Statist cs, Chicago, IL, USA). The mean value shown for pH, microbial load, organic acids and sugars, texture parameters and functional analysis is based on the 3 replicates of the two batches of each formulation, i.e. a total of 6 replicates. The mean value shown for CIELab coordinates is based on the 9 replicates of the two batches of each formulation, i.e. a total of 27 replicates. The mean value shown for volatile composition is based on the 2 replicates of the two batches of each formulation, i.e. a total of 4 replicates. The mean value shown for volatile composition is based on the 60 responses of consumers. One –way ANOVA test was first used as formulation as a factor, and then used storage as a factor. Tukey test was used for means comparison (95 % confidence level). Principal component analysis (PCA regression map) was conducted to project the samples depending on the techno-functional parameters and microbial load.

3. Results and discussion

3.1. Microbial load and metabolic products

All formulations reached pH<4.5 (Table 1 and Fig. 2). As it has been detailed in the methodology section, fig purée was mixed with fermented milk after 24 h of fermentation in different concentrations (10, 20, 30 and 40 %). Glucose and fructose content is also shown in Table 1 and Fig. 2. It can be observed that the main detected sugars coming from fig purée, in agreement with previous studies (Teruel-Andreu et al., 2023b). It is essential to highlight the residual galactose only was detected in control sample, being 0.68 g/100 g. The results of the statistical analysis pointed out significant differences ($p < 0.05$) for pH. Control and F10 presented the lowest pH, followed by F20, and F30 and F40. In this sense, it can be said that the inclusion of figs resulted in elevated pH values when compared to the control samples of fermented milks, slightly increased the initial pH of 4.48 in control samples (Fig. 2). Previous research indicated that pH values ranged from 4.8 to 5.3 for figs grown in the Mediterranean region (Polat and Caliskan, 2008).

After fermentation, the microbial counts were greater than 6.5 Log LAB CFU/g (estimated counts in MRS) and 9 Log LAC CFU/g (estimated counts in M17). No significant differences were observed among the samples, although it can be said that estimated microbial counts of LAC (in M17 medium) in F20-F40 were lower than the counts in control samples, and LAB (in MRS medium) in F20 and F40 were lower than the counts in control samples, Similar results were found by Almusallam et al. (2021), which studied the effect of date palm spikelet's extract on the physicochemical and microbial properties of set-type yogurt and reported that at the beginning of storage, there was no difference in the viable counts of both strains (*L. bulgaricus* and *S. thermophilus*) among

Table 1
pH. microbial load (estimated L*actobacilli and Streptococci* counts in MRS and M17 medium), organic and sugar content. texture parameters and gel stability. color. antioxidant capacity and total phenolic content of developed fermented milks.

Sample	Units	Tukey test*	Control	F10	F20	F30	F40
pH		*	4.48 ± 0.03^{b}	4.49 ± 0.01^{b}	4.70 ± 0.18^{ab}	4.84 ± 0.18^{a}	4.81 ± 0.01^{a}
LAB [¥]	Log CFU/g	NS	6.53 ± 0.27	6.53 ± 0.18	6.45 ± 0.18	6.69 ± 0.10	6.43 ± 0.04
LAC [≈]		*	9.00 ± 0.06^{a}	8.94 ± 0.07^{ab}	8.71 ± 0.12^{b}	8.73 ± 0.04^{ab}	8.90 ± 0.01^{ab}
Lactic acid	g/100 g	*	0.51 ± 0.01^{a}	0.48 ± 0.01^{a}	0.44 ± 0.03^{ab}	0.42 ± 0.05^{b}	0.43 ± 0.02^{b}
Malic acid		*	nd	0.01 ± 0.00^{b}	0.02 ± 0.00^{b}	0.03 ± 0.00^{ab}	0.05 ± 0.00^{a}
Lactose		**	2.37 ± 0.12^{a}	2.21 ± 0.06^{ab}	2.04 ± 0.03^{b}	1.89 ± 0.07^{c}	1.73 ± 0.06^{c}
Glucose		**	nd	0.17 ± 0.01^{d}	0.33 ± 0.01^{c}	0.47 ± 0.01^{b}	0.62 ± 0.01^{a}
Fructose		**	nd	0.27 ± 0.01^{d}	0.39 ± 0.00^{c}	0.53 ± 0.02^{b}	0.66 ± 0.01^{a}
Firmness	g	***	15.25 ± 3.21^{c}	15.74 ± 2.48^{c}	16.02 ± 0.85^{bc}	17.79 ± 1.74^{b}	19.74 ± 1.26^{a}
Consistency	g seg	***	378.46 ± 72.25^{c}	403.54 ± 87.30^{bc}	395.87 ± 29.50^{bc}	449.25 ± 50.06^{b}	511.82 ± 33.19^{a}
Cohesiveness	g	***	−11.77 ± 2.31^{c}	−10.94 ± 1.57^{c}	−12.40 ± 0.73^{c}	−14.04 ± 1.53^{b}	−16.42 ± 0.57^{a}
Viscosity index	g seg	***	−5.54 ± 1.01^{c}	−2.44 ± 0.94^{c}	−5.26 ± 0.58^{c}	−11.25 ± 1.78^{b}	–22.83 ± 3.26^{a}
Syneresis	%	***	71.38 ± 0.50^{a}	65.98 ± 2.27^{b}	59.34 ± 0.70^{c}	56.65 ± 0.40^{d}	56.52 ± 0.62^{d}
L*		***	78.59 ± 0.61^{a}	70.50 ± 0.08^{b}	64.86 ± 1.20^{c}	59.34 ± 1.70^{d}	56.11 ± 0.26^{e}
a*		***	−1.85 ± 0.03^{e}	1.65 ± 0.08^{d}	2.65 ± 0.05^{c}	3.34 ± 0.08^{b}	4.06 ± 0.22^{a}
b*		***	5.05 ± 0.03^{d}	5.41 ± 0.08^{cd}	5.82 ± 0.08^{bc}	6.04 ± 0.21^{b}	6.54 ± 0.33^{a}
AE		***	–	8.82 ± 0.07^{d}	14.50 ± 0.08^{c}	19.98 ± 0.09^{b}	23.30 ± 0.25^{a}
TPC	mg GAE g^{-1}	**	36.30 ± 2.23^{c}	45.64 ± 9.88^{bc}	50.06 ± 8.79^{abc}	57.09 ± 6.75^{ab}	64.42 ± 3.64^{a}
DPPH•	mmol T g^{-1}	*	13.11 ± 0.70^{b}	13.85 ± 0.93^{ab}	14.56 ± 0.31^{ab}	14.31 ± 0.97^{ab}	15.28 ± 0.74^{a}

[∞] NS=not significant ($p < 0.05$); *. **. and ***. significant at $p < 0.05$. 0.01. and 0.001. respectively. [≠] Among formulation values followed by the different letter within the same column were significant different ($p > 0.05$). Tukey's multiple-range test; GAE: gallic acid equivalents; T: Trolox; [¥]LAB=estimated *Lactobacillus delbrueckii* ssp. *lactis*, and *Lactobacillus delbrueckii* ssp. *Bulgaricus* counts in MRS medium; [≈]LAC=estimated *Streptococcus thermophilus* counts in M17 medium.
The mean value shown for pH, microbioal load, organic acids and sugars, texture parameters and functional analysis is based on the 3 replicates of the two batches of each formulation, i.e. a total of 6 replicates. The mean value shown for CIELab coordinates is based on the 9 replicates of the two batches of each formulation, i.e. a total of 27 replicates.

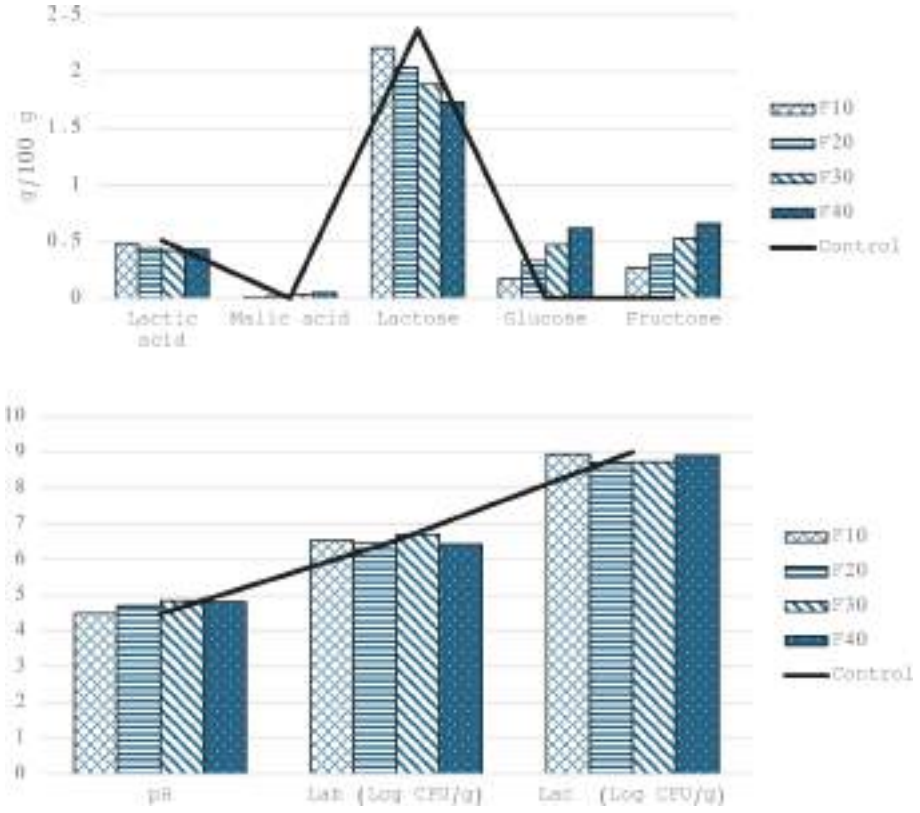

Fig. 2. Sugars and organic acids content (upper chart) and pH and microbial load (below chart) of enriched fermented milks. LAB=estimated *Lactobacillus delbrueckii* ssp. *lactis*, and *Lactobacillus delbrueckii* ssp. *Bulgaricus* counts in MRS medium; LAC=estimated *Streptococcus thermophilus* counts in M17 medium.

yogurt samples. On the other hand, our results agreed with previous study about cinnamon enriched yoghurt before fermentation being reported counts ranging from 6.82 to 7.54 Log LAB CFU/g (estimated counts in MRS) and from 8.26 to 9.01 Log LAC CFU/g (estimated counts in M17). The *Codex alimentarius* indicated that at least the sum of microorganisms constituting the starter culture in a fermented milk should be a minimum of 10^7 CFU/g, in total. The healthy probiotic properties of fermented milks reported by scientists are well known, but it should be noted that only one health claim in consumer information is allowed in the European Union: 'Live cultures present in yoghurt or fermented milk improve the digestion of lactose in the product for people with lactose digestion difficulties'; the conditions of use of this claim are as follows: 'To qualify for inclusion in the claim, the yoghurt or fermented milk must contain at least 10^8 Colony Forming Units of live starter microorganisms (*L. bulgaricus and S. thermophilus*) per gram' (EFSA 2010; R(EU) 432/2012).

On the other side, significant changes of lactic acid were detected with results in a range of 0.42 to 0.51 g/100 g, being control, F10 and F20 the samples that presented the highest content. It is necessary to highlight that the difference among samples is due to the addition of fig purée after fermentation, once the 30 % and 40 % of fig purée was added, a dilution effect happened. In this sense, malic acid was also detected but in lower quantities than lactic acid and was not detected in the control fermented milks as expected. Previous studies (Teruel-Andreu et al., 2023b) that analysed "Colar" figs detected malic acid with values of 26.61 g/Kg dw in peel and 29.47 g/Kg dw in pulp.

Regarding sugars, control samples presented the highest content in lactose 2.37 g/100 g. Our residual lactose results were lower than those reported by Jiménez-Redondo et al., 2022 who found values ranged of 2.53 and 3.25 g/100 g in cinnamon enriched yoghurt at 24 h after fermentation. As expected, F40 presented the highest glucose (0.62 g/100 g) and fructose content (0.66 g/100 g), followed by F30, F20 and F10. Previous studies (Teruel-Andreu et al., 2023b) showed that the Colar fig, which was used in this work, stood out for its total sugar content among several varieties with values of 379.33 g/Kg dw and 363.97 g/Kg dw for glucose and fructose, respectively. It is well known that *L. bulgaricus* only metabolizes lactose, while *S. thermophilus* metabolizes both lactose and glucose. Furthermore, post-lactic acid fermentation, the product boasts an increased probiotic content and reduced allergens, such as lactose (Markowiak and Śliżewska, 2017). Previous studies indicated that adding fruit to dairy products helps maintain or enhance the growth and survival of probiotics in those products. It would be interesting to know the evolution during the shelf life (Sendra et al., 2008; Trigueros et al., 2012).

3.2. Texture parameters and gel stability

Among formulations, significant differences ($p < 0.001$) were observed in "firmness", "consistency", "cohesiveness" and "viscosity index" (Table 1). It is important to highlight that from the fermented milks with 40 % fig puree, F40 showed.

the highest values of firmness, consistency, cohesiveness, and viscosity index. A reduction of all texture parameters was noticed when the percentage of fig purée was reduced, being values of firmness, consistency, cohesiveness and viscosity index in F10 and F20 like the ones of control samples. It was also observed that the percentage of added fig purée from 20 % significantly improved the firmness, consistency, cohesiveness, and viscosity index of fermented milks, comparing with control sample. This may be due to the fact that the branched chain structure of polysaccharides can facilitate covalent cross-linking between casein molecules, leading to the formation of larger casein aggregates (McClements and Decker, 2018). In addition, polyphenols present in the gel can bind with amino acid side chains in the protein, forming complexes that fill the voids within the protein matrix. This stabilizes the casein network, consequently enhancing the texture of fermented milk (Trigueros et al., 2012). Similar results were observed by other authors (Shahein et al., 2022) when date syrup was added to fermented camel milk resulting in higher viscosity. Additionally, another study (Du et al., 2023) examined the effect of adding mulberry pomace polysaccharide to yogurts and showed that a moderate addition of mulberry pomace polysaccharide could improve yogurt texture. Another relevant fact is the dramatic difference among percentage of syneresis by centrifugation among fermented milks analyzed. On the other side, a significant decrease in syneresis was observed in F30 and F40 formulations. The natural pectin presents in figs could increase water holding capacity, resulting in a lower percentage of syneresis. This is because pectins assist in creating a three-dimensional network within the gel formed in fermented milks, resulting in increased viscosity (Arioui et al., 2017). The values obtained by other authors (Çavdaroğlu and Yemenicioğlu, 2022) were 11.68 % and 9.68 % in fig stems and whole figs, respectively. The incorporation of fig purée in fermented milk could be a good strategy to obtain creaminess matrix reducing the addition of additives.

3.3. Colour coordinates

As expected, the results showed that L^* parameter, lightness, was significantly affected by the incorporation of fig puree, being lower in formulations with higher concentration of fig puree (Table 1). As to a^* coordinate (green–red coordinate) and b^* coordinate (blue-yellow coordinate), an increase was observed when the concentration of fig purée was increasing. Solomon et al. (2006) examined the quantity of anthocyanins in six commercial varieties of figs and concluded that in all the analyzed figs, anthocyanins were concentrated in the fruit skin and constituted the main colouring compounds. The tendency observed meant an increase of brown colors, characteristic of pasteurized fig purée (degradation of anthocyanins after heat treatment). Colour difference among formulation was better observed in AE parameter, in response to the proportion of fig puree incorporated, being the highest value found in F40, followed by F30, F20 and F10.

3.4. Total polyphenolic content (TPC) and antioxidant capacity

The total polyphenol content (TPC) showed significant differences (Table 1) among fermented milks, presenting the highest content which 20 %, 30 %, and 40 % of fig purée was incorporated. TPC in F10, F20, F30, and F40 was 1.3-fold, 1.4-fold, 1.6-fold, 1.8-fold more than control sample. Fig fruits is a rich in polyphenols (Meziant et al., 2021; Teruel-Andreu et al., 2021) such as flavonoids (Meziant et al., 2021). Recently, previous studies conducted with figs from various Spanish varieties highlight the polyphenolic content (Teruel-Andreu et al., 2023b) of the "colar" variety and its antioxidant capacity (Wojdyło et al., 2016). It is known that polyphenols have strong antioxidant properties (Wu et al., 2004), and in this study was observed the same tendency as TPC in DPPH antioxidant capacity values, being the highest values found in F40. The fermented milks showed an increase in polyphenolic content and antioxidant activity with an increase in the percentage of added fig puree. But it is necessary to highlight that the differences among formulations, including control, were small, due to the possible interference of peptide compounds with antioxidants compounds. Our results are consistent with other studies in which fruit was added to dairy matrices, and they also obtained higher antioxidant activity with the increase in the percentage of added fruit. (Ning et al., 2021; Taheur et al., 2023; Trigueros et al., 2012). Cutrim and Cortez (2018) showed the beneficial effects and application of polyphenols in dairy products with conclusions from previous studies about the interactions between various polyphenols and milk components in the bioactivity and bioavailability of these compounds. Some of these conclusions were polyphenol-casein interactions significantly alter the structure of caseins and how the addition of milk proteins to fat increased the bioavailability of polyphenols.

3.5. Volatile compounds

Twenty-eight compounds were isolated, identified (Table 2) and quantified as a relative abundance (%) (Table 3).

The volatile compounds were categorized based on their chemical families, being acids the most prevalent group (Table 2). It is noteworthy that none of the samples had measurable volatiles compounds related to off-flavours notes, supporting the high quality of the products. It is essential to indicate that the headspace technique is based on the equilibrium between the volatile compounds present in the food matrix and their gas phase in the headspace of the vessel. This equilibrium depends on several factors, such as the volatility of the compounds, the interaction between them and the food matrix, as well as the equilibrium temperature and time. Many of these factors have been controlled in all samples in the same way as temperature and time. When different concentrations of an aromatic ingredient are added, as in our case pasteurised fig puree, volatile compounds can compete for the available headspace. This can lead to a saturation of certain volatile compounds in the gas phase, preventing a linear response with respect to the concentration added. On the other hand, each volatile compound has a different partition coefficient between the solid/liquid phase (feed matrix) and the gas phase (headspace). These coefficients may not be proportional to the added concentration, especially if there are interactions between the volatile compounds or with the food matrix. Furthermore, it is important to note that at higher concentrations of the aromatic ingredient (pasteurized fig purée), certain volatile compounds may reach a saturation point in the headspace. This means that even if more of the compound is added to the matrix, its concentration in the gas phase does not increase proportionally. Therefore, linearity of all components was not observed and discussion was focused on the main ones.

Among the 28 compounds identified, hexanoic acid was the main compound detected in all fermented milk, which is characteristic of fermented milks (no differences among them were detected) (Table 3) and can be related to cheesy, sour, and fatty aroma. The second and third abundant volatile compounds detected in enriched fermented milk were acetoin and butanoic acid (Table 3). Acetoin compound is a common volatile compound which is originated during fermentation by Lactobacillus species (Roncal et al., 2017). The lowest percentage was observed in control fermented milk (21.602 %) and F10 (18.193 %) and the higher percentage was obtained for F40 (26.778 %). Also, acetoin could come from pasteurized figs, due to the amount of acetoin increased as the pasteurized fig purée increased. Sertkaya et al., (2021) analysed the volatile compounds in the peel and pulp of two varieties of Turkish figs and detected high amounts of acetoin in both the skin and pulp. Also, butanoic acid is a characteristic volatile compound in fermented milks, being the highest percentage of this compound detected in the control (20.94 %). 11 volatile compounds (V1, V8, V10, V11, V12, V16, V18, V19, V22, V23 and V24) were detected in higher percentages in F40, most of them related to fruity and sweet notes. In a study where the volatile compounds of several Spanish varieties of brebas and figs were analysed, including the 'Colar' variety used in this study, some of these volatile compounds were found (V8, V11, V12, V19, V23), the rest of volatile compounds detected in greater quantity in F40, which were not identified in fresh figs, may have formed during the pasteurization of the fig puree. Although the main volatile compounds detected in the fruits such as hexanal, 2-hexenal, and benzaldehyde, all of them belonging to the aldehyde family, have not been detected in the analysed fermented milks (Teruel-Andreu et al., 2024). It may have been modified by the heat treatment. In a study (García-Parra et al., 2020) where they analysed the volatile compounds of pumpkin puree processed by high-pressure heat treatment, they detected that the levels of 2-hexenal decreased significantly after processing. Other authors Cosmai et al. (2013) who have studied the influence of the thermal stabilization process on the volatile profile of canned tomato-based foods reported that the concentration of hexanal showed a significant increase during thermal stabilization, as a result of thermal degradation of

Table 2
Aromatic compounds found in fermented milk enriched with pasteurized fig purée using headspace solid phase microextraction (HS-SPME).

Code	Volatile Compounds	Chemical Family	RT (min)	Retention Indexes		Descriptors
				Exp	Lit	
V1	**Acetoin**	Ketone	3,13	745	743	Sweet, buttery, creamy
V2	**Butanoic acid**	Acid	4,44	798	804	Cheesy, buttery
V3	**2-Hexanol, 3-methyl-**	Alcohol	6,68	887	890	–
V4	**2-Heptanone**	Ketone	7,28	905	900	Cheesy, fruity, ketonic
V5	**Hexanoic acid**	Acid	12,46	999	997	Cheesy, sour, fatty
V6	**Limonene**	Terpene	14,04	1025	1028	Sweet, citrus
V7	**Cyclopentane, pentyl-**	Alkane	14,46	1032	1033	Herbal, flower
V8	**2-Nonanone**	Ketone	17,81	1088	1091	Fruity, sweet, cheese, herbal
V9	**Linalool**	Terpene	18,39	1097	1101	Citrus, floral
V10	**Undecane**	Alkane	18,55	1100	1100	–
V11	**Nonanal**	Aldehyde	18,71	1102	1102	Citrus, waxy, aldehydic
V12	**Phenylethyl Alcohol**	Alcohol	19,01	1107	1110	Sweet, floral
V13	**Cyclodecane**	Alkane	19,58	1115	1125	–
V14	**1-Undecene, 2-methyl-**	Alkane	22,87	1165	1170	–
V15	**Octanoic acid**	Acid	23,79	1179	1186	Fatty, waxy, rancid, cheesy
V16	**Ethanol, 2-(2-butoxyethoxy)-**	Alcohol	23,97	1181	1189	–
V17	**Octanoic acid, ethyl ester**	Ester	24,84	1194	1196	Waxy, sweet, fruity, musty
V18	**Dodecane**	Alkane	25,17	1199	1199	–
V19	**Decanal**	Aldehyde	25,40	1203	1203	Sweet, aldehydic, citrus
V20	**Linalyl acetate**	Terpene	28,36	1246	1256	Sweet, green, floral, spicy, woody
V21	**Nonanoic acid**	Acid	29,68	1265	1267	Fatty, waxy, cheesy
V22	**2-Undecanone**	Ketone	31,18	1287	1291	Waxy, fruity, ketonic
V23	**1-Dodecanol**	Alcohol	42,12	1473	1473	Earthy, soapy, waxy, fatty
V24	**2H-Pyran-2-one, tetrahydro-6-pentyl-**	Ketone	42,50	1481	1481	Sweet, creamy, lactonic
V25	**2-Tridecanone**	Ketone	42,80	1488	1496	Fatty, waxy, earthy
V26	**2,4-Di-*tert*-butylphenol**	Phenol	43,11	1494	1502	–
V27	**2,2,4-Trimethyl-1,3-pentanediol diisobutyrate**	Ester	45,09	1578	1587	–

(*continued on next page*)

Table 2 (*continued*)

Code	Volatile Compounds	Chemical Family	RT (min)	Retention Indexes		Descriptors
				Exp	Lit	
V28	**Hexadecanoic acid, ethyl ester**	Ester	53,37	1991	1992	Waxy, creamy, fruity

Table 3
Relative abundance (%) of volatile compounds in yogurts prepared by different percentage of fig purée.

	Tukey test*	CTRL	F10	F20	F30	F40
V1	***	21.6 ± 1.5^{b}	18.2 ± 1.4^{b}	22.8 ± 1.2^{ab}	22.2 ± 1.9^{ab}	26.8 ± 1.7^{a}
V2	***	20.9 ± 2.1^{a}	18.1 ± 1.8^{ab}	11.9 ± 1.8^{c}	15.9 ± 2.2^{bc}	15.0 ± 1.9^{bc}
V3	NS	1.2 ± 1.1	9.6 ± 4.5	7.6 ± 3.9	5.8 ± 3.2	1.7 ± 0.8
V4	NS	3.9 ± 0.5	4.5 ± 1.1	3.8 ± 0.8	3.9 ± 0.7	3.8 ± 1.0
V5	NS	42.3 ± 4.1	39.4 ± 3.2	40.6 ± 3.1	40.5 ± 3.5	40.8 ± 2.9
V6	NS	0.62 ± 0.01	0.57 ± 0.01	0.69 ± 0.01	0.78 ± 0.01	0.75 ± 0.01
V7	**	0.04 ± 0.01 ^{ab}	0.04 ± 0.01^{b}	0.08 ± 0.01^{a}	0.07 ± 0.01 ^{ab}	0.08 ± 0.01 ^{ab}
V8	***	0.70 ± 0.05 ^{bc}	0.61 ± 0.08^{c}	0.78 ± 0.04 ^{ab}	0.79 ± 0.02^{ab}	0.85 ± 0.03^{a}
V9	NS	0.01 ± 0.01	0.01 ± 0.01	0.01 ± 0.01	0.01 ± 0.01	0.02 ± 0.01
V10	***	0.07 ± 0.02 ^{bc}	0.06 ± 0.03^{c}	0.10 ± 0.01^{ab}	0.10 ± 0.01 ^{ab}	0.13 ± 0.03 ^{a}
V11	**	0.330 ± 0.04^{b}	0.327 ± 0.03^{b}	0.311 ± 0.06^{b}	0.343 ± 0.08^{b}	0.660 ± 0.08^{a}
V12	***	0.20 ± 0.05^{c}	0.21 ± 0.07^{c}	0.45 ± 0.9^{bc}	0.69 ± 1.1 ^{ab}	0.94 ± 0.8 ^{a}
V13	NS	0.07 ± 0.01	0.07 ± 0.02	0.08 ± 0.02	0.09 ± 0.03	0.09 ± 0.02
V14	*	0.03 ± 0.01^{b}	0.03 ± 0.0^{b}	0.05 ± 0.01^{a}	0.05 ± 0.01^{a}	0.05 ± 0.02^{a}
V15	**	6.85 ± 0.8^{b}	7.23 ± 0.7 ^{ab}	9.86 ± 0.6^{a}	7.67 ± 1.0^{ab}	6.99 ± 0.8^{b}
V16	**	0.14 ± 0.01^{ab}	0.14 ± 0.02 ^{ab}	0.10 ± 0.02^{b}	0.20 ± 0.01 ^{a}	0.21 ± 0.02^{a}
V17	NS	0.18 ± 0.01	0.13 ± 0.02	0.12 ± 0.02	0.14 ± 0.03	0.17 ± 0.03
V18	**	0.12 ± 0.01^{c}	0.11 ± 0.01^{c}	0.132 ± 0.02 ^{bc}	0.16 ± 0.01^{ab}	0.18 ± 0.02^{a}
V19	**	0.09 ± 0.02 ^{ab}	0.06 ± 0.03^{b}	0.06 ± 0.01^{b}	0.08 ± 0.02^{ab}	0.12 ± 0.02^{a}
V20	NS	0.11 ± 0.01	0.12 ± 0.01	0.10 ± 0.02	0.12 ± 0.02	0.16 ± 0.04
V21	NS	0.09 ± 0.01	0.06 ± 0.01	0.04 ± 0.03	0.05 ± 0.02	0.11 ± 0.06
V22	**	0.15 ± 0.01^{ab}	0.13 ± 0.02^{b}	0.17 ± 0.02^{a}	0.15 ± 0.03^{ab}	0.17 ± 0.01^{a}
V23	*	0.02 ± 0.01^{ab}	0.02 ± 0.01^{ab}	0.01 ± 0.01^{b}	0.02 ± 0.02 ^{ab}	0.04 ± 0.01^{a}
V24	**	0.06 ± 0.01^{b}	0.09 ± 0.01^{ab}	0.12 ± 0.02^{a}	0.09 ± 0.02 ^{ab}	0.10 ± 0.01^{a}
V25	**	0.02 ± 0.01^{b}	0.02 ± 0.01^{b}	0.03 ± 0.01^{a}	0.02 ± 0.01^{b}	0.03 ± 0.01^{ab}
V26	***	0.03 ± 0.01^{a}	0.01 ± 0.01^{b}	0.01 ± 0.01^{b}	0.01 ± 0.01^{b}	0.01 ± 0.01^{b}
V27	**	0.06 ± 0.01^{a}	0.01 ± 0.01^{c}	0.02 ± 0.01^{bc}	0.01 ± 0.01^{c}	0.05 ± 0.01^{b}
V28	*	0.024 ± 0.03 ^{a}	0.012 ± 0.01^{b}	0.002 ± 0.001^{c}	0.002 ± 0.001^{c}	0.007 ± 0.001^{c}

$^{\infty}$ NS=not significant ($p < 0.05$); *. **. and ***. significant at $p < 0.05$. 0.01. and 0.001. respectively. $^{\neq}$ Among formulation. values followed by the different letter within the same column were significant different ($p > 0.05$). The mean value shown for volatile composition is based on the 2 replicates of the two batches of each formulation, i.e. a total of 4 replicates.

hydroperoxides originating from the oxidation of linoleic acid. Conversely, the level of (E)-2-hexenal decreased significantly according to the results of this study.

In general, the differences in these compounds, could be responsible for the differences in the odour, aroma and flavour of the fermented milks and can be related to sweet notes. It is crucial to note that while there have been statistically significant differences in certain volatile compounds identified, it is challenging to ascertain whether these variances directly translate to differences in the odour and aroma of the fermented milks. Odor and aroma are influenced by synergistic interactions among all volatile compounds (Sánchez-Bravo et al., 2020).

3.6. Consumer study

In Table 4, the consumer acceptance scores of different attributes of fermented milks are shown. As can be seen from the data, no significant differences in consumer acceptance of the colour were detected, but there is a slight tendency for the colour acceptance rating to increase with increasing fig content. It would be an inverse tendency with respect to the CIELab system parameter L*. On the other hand, texture is the main attribute driving to consumers acceptance. Among the parameters related to texture, the most important one for consumer preference of dairy products are commonly firmness and creaminess (Duboc and Mollet, 2001). Consumers scores of firmness, creaminess, and viscosity were better rated as the content of fig puree increased (F30 and F40), which was correlated with the increase in these parameters observed in instrumental texture analysis. Higher firmness, higher consumer acceptance. Additionally, F40 was in the one with the highest sweetness and fig ID acceptance. A higher sugar profile, such as that found in fig purée, is associated with greater consumer acceptance. As it was above observed pasteurized fig purée is rich in glucose, and fructose. According to consumers, F30 and F40 presented the highest score for overall linking. Generally, increasing the percentage of fig puree improved the consumer acceptance in all parameters and overall. After Pearson

Table 4
Mean scores and ANOVA of developed yoghurts at T1 (24 h) for appearance. flavor notes. basic taste. and overall liking for consumers.

	Tukey test* $^{\infty\neq}$	F10	F20	F30	F40
Appearance	*	5.2 ± 2.1^{b}	5.5 ± 2.3^{ab}	6.3 ± 1.4^{a}	6.0 ± 2.2^{ab}
Fig at the bottom	**	5.1 ± 2.0^{b}	5.8 ± 1.8^{ab}	6.1 ± 1.4^{a}	6.2 ± 2.0^{a}
Firmness	***	3.9 ± 1.9^{b}	4.6 ± 2.1^{b}	5.7 ± 1.6^{a}	6.2 ± 1.9^{a}
Creaminess	***	3.5 ± 1.4^{c}	4.5 ± 2.1^{b}	5.8 ± 1.7^{a}	6.5 ± 1.7^{a}
Appearance after blending	**	4.9 ± 2.1^{b}	5.3 ± 2.0^{ab}	6.1 ± 1.4^{a}	6.1 ± 2.1^{a}
Color	NS	5.4 ± 2.0	5.9 ± 1.7	6.1 ± 1.3	6.2 ± 2.1
Viscosity	***	3.9 ± 1.8^{b}	4.5 ± 2.0^{b}	5.9 ± 1.6^{a}	6.0 ± 2.1^{a}
Particles	**	4.7 ± 1.9^{b}	5.1 ± 1.7^{ab}	5.6 ± 2.0^{ab}	6.1 ± 2.3^{a}
Sourness	**	5.0 ± 2.0^{b}	5.9 ± 1.7^{a}	5.9 ± 1.4^{a}	6.3 ± 1.8^{a}
Sweetness	***	4.9 ± 1.7^{c}	5.3 ± 1.7^{bc}	5.8 ± 1.4^{ab}	6.4 ± 1.9^{a}
Fig ID	***	4.5 ± 1.7^{c}	5.3 ± 1.8^{bc}	6.1 ± 1.8^{ab}	6.5 ± 2.2^{a}
Aftertaste	***	4.7 ± 1.5^{b}	5.6 ± 1.5^{a}	5.6 ± 1.4^{a}	6.1 ± 1.6^{a}
Overall liking	***	4.2 ± 1.8^{b}	4.9 ± 2.0^{b}	6.2 ± 1.5^{a}	6.4 ± 2.1^{a}

$^{\infty}$ NS=not significant ($p < 0.05$); *. **. and ***. significant at $p < 0.05$. 0.01. and 0.001. respectively. $^{\neq}$ Values followed by the different letter within the same row were significant different ($p > 0.05$). Tukey's multiple-range test. The mean value shown for volatile composition is based on the 60 responses of consumers.

correlation analysis, it can be said that overall liking is well-correlated with glucose (p-value: 0.034; R^2: 0.933) and fructose content (p-value: 0.030; R^2: 0.942), a* coordinate (p-value: 0.040; R^2: 0.914) and AE (p-value: 0.013; R^2: 0.974). Contrary, overall liking was inversely proportional to lactose content (p-value: 0.034; R^2: 0.933), and L* coordinate (p-value: 0.012; R^2: 0.977).

3.7. Principal component analysis (PCA)

For an easy visualization of the relationships among all variables of the developed fermented milks with figs puree, a PCA was run for all five samples, including only significantly different variables: pH, microbial load, organic content, sugar content, texture parameters, gel stability, color, antioxidant capacity, total phenolic content and volatile compounds. Fig. 3 shows the principal component analysis which explained 81.48 % (PC1 = 67.60 % and PC2 = 20.88 %) of the total variation of the experimental data. As observed, fermented milks containing 10 % fig puree were grouped together with the control sample, characterized by higher lactose and lactic acid content, as well as a lighter colour. In contrast, fermented milks containing 30 % and 40 % fig puree were grouped separately, distinctly different from both the first group and from each other. Fermented milks with 30 % and 40 % fig puree exhibited a firmer texture and higher sugar content. Additionally, they showed an increase in the red-yellow colour coordinates and enhanced functionality compared to the other samples.

4. Conclusion

The amount of figs puree added affects the technological properties of the developed fermented milks (the internal structure of the gel and spontaneous syneresis). Among all the fermented milks developed, variations were observed in microbial content, texture, colour, functionality, volatile compounds, and sensory attributes. The addition of 30 % and 40 % fig puree post-fermentation improved texture, sensory qualities, and functional parameters without negatively affecting the viability of lactic acid bacteria. It can be concluded that formulations containing 30 % and 40 % fig puree are particularly desirable for techno-functional properties and for consumer acceptance. These formulations are suitable for producing enriched fermented milks without added sugar. It can be said that in Spain, the addition of 40 % of fig purée would not be possible to develop "yoghurt enriched with fruits" because it requires a minimum of 70 % fermented milk to be called yoghurt, apart from other requirements (RD 271/2014). By comparison, it can be catalogued as a "Flavoured Fermented Milks" as Codex Alimentarius (Codex Stan, 243–2003) being possible a maximum of 50 % (m/m) of non-dairy ingredients such as fruit purees. Future research would include the behaviour during 30 days of refrigerated storage.

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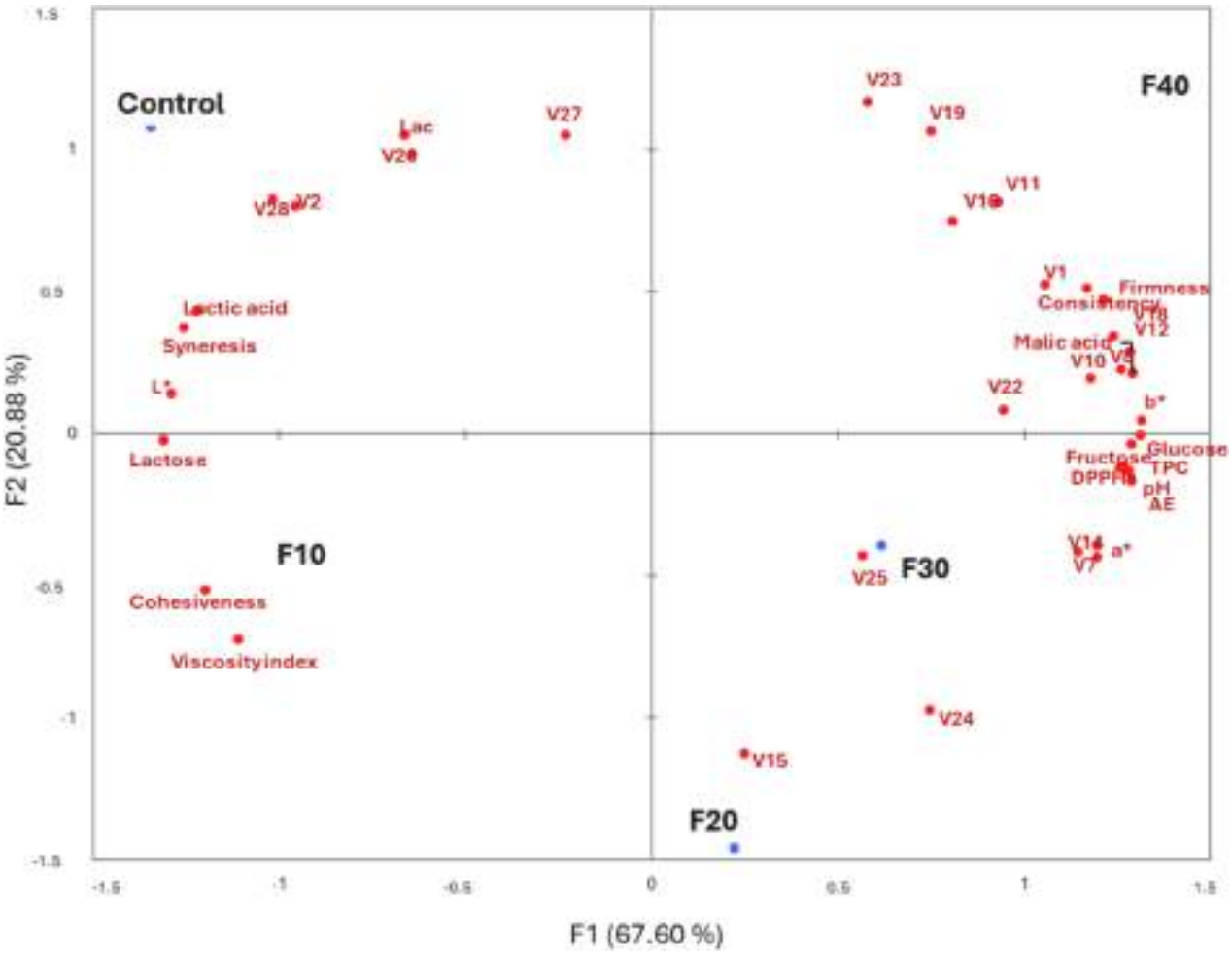

Fig. 3. Principal Component Analysis (PCA) of enriched fermented milks and parameters with statistical differences (88.5 %). LAB=estimated *Lactobacillus delbrueckii* ssp. *lactis*, and *Lactobacillus delbrueckii* ssp. *Bulgaricus* counts in MRS medium; LAC=estimated *Streptococcus thermophilus* counts in M17 medium.

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C. Teruel-Andreu: Writing – review & editing, Writing – original draft, Formal analysis. **N. Jiménez-Redondo:** Writing – review & editing, Formal analysis. **R. Muelas:** Formal analysis. **A.A. Carbonell-Pedro:** Formal analysis. **F. Hernández:** Writing – review & editing, Methodology, Conceptualization. **E. Sendra:** Writing – review & editing, Methodology, Conceptualization. **M. Cano-Lamadrid:** Writing – review & editing, Supervision, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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FLAVONOIDS, MICROBIAL LOAD AND QUALITY PARAMETERS CHANGES DURING SHELF-LIFE OF FERMENTED MILK ENRICHED WITH PASTEURIZED FIG PURÉE.

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Flavonoids, microbial load and quality parameters changes during shelf-life of fermented milk enriched with pasteurized fig purée

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ABSTRACT

Fig puree is rich in natural bioactive substances including flavonoids and can be useful to produce functional foods. This study investigated the effects of incorporating fig puree into fermented milks formulations on various physicochemical properties, and nutritional and functional compounds during 30 days of refrigerated storage. Fig puree was incorporated into cow fermented milks at 0 g 100 g^{-1} (control), 10 g 100 g^{-1}, 20 g 100 g^{-1}, 30 g 100 g^{-1} and 40 g 100 g^{-1}, respectively after fermentation. Initial pH values ranged from 4.48 to 4.84, with no significant changes observed during storage. Microbial counts remained above the acceptable threshold until 30 days in refrigeration. Color parameters indicated a decrease in lightness (L*) and hue (h) values with increasing fig puree content, while a* and b* values increased with increasing fig puree. The fig puree concentration dependently affected the texture analysis and revealed enhanced firmness, consistency, cohesiveness, and viscosity in yogurts with 40 g 100 g^{-1} of fig puree. Overall, texture stability was maintained during storage period, though slight softening was noted in some formulations. The addition of fig puree reduced syneresis compared to the control which could be attributed to the increase of pectin content. Total phenolic content and DPPH values increased with fig puree addition, with detectable anthocyanins and flavonols, predominantly quercetin-3-galactoside. Levels of these bioactive compounds increased during storage, with the highest amounts found in yogurts containing 40 g 100 g^{-1} of fig puree after 30 days of storage. These results suggest that fig puree enhances fermented milks properties and stability, offering potential health benefits due to increased bioactive compound content.

1. Introduction

Figs which is an important fruit in the Mediterranean diet are rich in fibers, minerals, sugars, organic acids, and phenolics with antioxidant capacity (Solomon et al., 2006). In this sense, fig-based products have been used historically in traditional cuisine and medicine. Over the decades, there has been an increase in the development of fig-based products (Teruel-Andreu et al., 2021) such as wine (Liu et al., 2021; Lu et al., 2021), smoothies (Cano-Lamadrid et al., 2018a, 2018b; Issa-Issa et al., 2020), fig powders (Viuda-Martos et al., 2015), biscuits (Bölek, 2021), jam (Rababah et al., 2011), and fig-milk desserts (Jahromi & Niakousari, 2018; Zare et al., 2024), among others. One of the most important qualities of figs in the development of new food products is focus to provide them with an attractive colour, enhance their taste and techno-functional properties, and beneficial qualities (Backes et al., 2018, 2020).

With regard to the environmental footprint of fig cultivation, the aim is to avoid wastage. Therefore, it is necessary to develop products with non-commercial figs (food loss by FAO definition) for different reasons such as undersized figs, over-ripening and harvest-related damage. Apart from “food loss”, the functional properties of fig by-products during processing (food loss by FAO definition), have been widely studied and stands out for its content of fibre, organic acids, sugars, and anthocyanins (Teruel-Andreu et al., 2023; Wojdyło et al., 2016), being a good change to develop fig-based food products. Therefore, the development of new fig-based products could not only be an increase of their functional and techno-functional value, but could also represent an opportunity to mitigate "food loss" (Teruel-Andreu et al., 2021).

Fermented milks have been defined as an accurate food matrix to incorporate fruit by-products (FAO, 2007) such as citrus fibers (Sendra

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Table 1
pH, microbial load (estimated *Lactobacillus* and *Lactococcus*) and CIE*Lab* color parameters of developed fermented milks at T0 (24 h) and T30 (30 days, refrigerated storage).

	pH	Lactobacillus (log UFC g^{-1})	Lactococcus (log UFC g^{-1})	L*	a*	b*	C	h
ANOVA [a,b]	***	NS	***	***	***	***	***	***
CTRL T0	4.48 ± 0.03 b	6.53 ± 0.27	9.00 ± 0.06 a	78.6 ± 0.6 a	−1.85 ± 0.03 e	5.05 ± 0.03 d	5.37 ± 0.02 d	110 ± 12 a
F 10 T0	4.49 ± 0.01 b	6.53 ± 0.18	8.94 ± 0.07 ab	70.5 ± 0.1 b	1.65 ± 0.08 d	5.41 ± 0.08 cd	5.66 ± 0.04 cd	73.0 ± 6.1 b
F 20 T0	4.70 ± 0.18 ab	6.45 ± 0.18	8.71 ± 0.12 b	64.9 ± 1.2 c	2.65 ± 0.05 c	5.82 ± 0.08 bc	6.39 ± 0.03 bc	65.6 ± 4.2 c
F 30 T0	4.84 ± 0.18 a	6.69 ± 0.10	8.73 ± 0.01 ab	59.3 ± 1.7 d	3.34 ± 0.08 b	6.04 ± 0.21 b	6.91 ± 0.04 ab	61.0 ± 6.0 d
F 40 T0	4.81 ± 0.01 a	6.43 ± 0.04	8.90 ± 0.01 ab	56.1 ± 0.3 e	4.06 ± 0.21 a	6.54 ± 0.33 a	7.70 ± 0.02 a	58.2 ± 6.0 e
CTRL T30	4.51 ± 0.04 b	6.50 ± 0.03	9.01 ± 0.05 a	78.8 ± 0.6 a	−1.85 ± 0.21 e	4.67 ± 0.15 e	5.02 ± 0.03 de	112 ± 13 a
F 10 T30	4.53 ± 0.02 b	6.49 ± 0.04	8.89 ± 0.04 ab	70.6 ± 0.7 b	1.70 ± 0.17 d	4.93 ± 0.11 e	5.21 ± 0.05 d	71.0 ± 7.0 b
F 20 T30	4.68 ± 0.01 ab	6.58 ± 0.16	8.77 ± 0.04 ab	64.5 ± 0.3 c	2.74 ± 0.04 c	5.43 ± 0.03 cd	6.09 ± 0.03 c	63.2 ± 4.0 cd
F 30 T30	4.88 ± 0.03 a	6.54 ± 0.15	8.64 ± 0.03 b	58.6 ± 0.6 d	3.41 ± 0.08 b	5.73 ± 0.18 cd	6.67 ± 0.21 b	59.2 ± 3.1 de
F 40 T30	4.86 ± 0.02 a	6.59 ± 0.16	8.61 ± 0.04 b	55.9 ± 0.8 e	4.05 ± 0.02 a	6.33 ± 0.07 ab	7.51 ± 0.03 a	57.4 ± 7.2 e

*, **, and ***, significant at $p < 0.05$, 0.01, and 0.001, respectively.
[a] NS = not significant ($p < 0.05$).
[b] Values followed by the different letter within the same column were significant different ($p > 0.05$), Tukey's multiple-range test.

et al., 2008), date syrup (Shahein et al., 2022), pomegranate peel (El-Said et al., 2014), among others. Apart from knowing the functional and techno-functional properties during food processing and the consumer acceptance of developed product, it is also important to understand the behaviour of fermented milk enriched with fruit by-products during refrigerated shelf-life.

One of the key parameters to consider is the stability of the gel, which may fluctuate under refrigeration (Saint-Eve et al., 2008). Changes in lactic acid bacterial load during refrigeration could be changed affecting the functional properties (probiotic levels, stability of flavonoids) (Jakobek & Matić, 2019) and the main quality parameters during shelf-life (Gris et al., 2007).

Taking all the aforementioned factors into consideration, the objective of the study was to investigate the changes of functional aspects (microbial load and flavonols) as well as techno-functional properties (colour, texture, organic acids, and sugars) of fermented milks enriched with different concentrations of pasteurized "Colar" fig puree (came from fig by-products) during refrigerated storage.

2. Materials and methods

2.1. Pasteurized fig puree and fermented milk manufacture

To prepare the fig puree, we utilized Colar variety figs sourced from Albatera farms in Alicante, Spain, harvested during the 2021 season and stored frozen until processing. Figs were harvested in the fields, only clean, whole fruits where collected. Figs were daily delivered to a fruit processing plant where they were classified according to size, ripening stage and postharvest damage. The discarded fruits were either damaged, under commercial size, or not in the proper ripening stage. Such fruits were stored under refrigeration conditions till the following morning when we collected them from the fruit processing plant. Once in our facilities figs were frozen at −20 °C until higienization and use. Following this procedure, figs were frozen within less than 48 h after harvesting. From now on, the figs underwent a disinfection process by immersing them in a solution of 200 mg L^{-1} of peracetic acid (Citrocide® PC, Citrosol, Valencia, Spain) at 15 °C for 10 min, followed by rinsing with running water at 15 °C for 5 min (5 L s^{-1}). The puree was prepared using a Thermomix®, starting with fruit crushing (1-min, speed 5; 1-min, speed 7), followed by thermal treatment (100 °C, 20 min). Pasteurized fig purée was distributed in sterile cups and cooled in a water bath with ice to decrease the temperature below 4 °C as fast as possible to enhance the efficiency of the pasteurization treatment. From previous experiments with fruit added fermented milks authors concluded that the best procedure to reduce anthocyanins decay due to microbial degradation is to add the fruit after fermentation (Cano-Lamadrid et al., 2017). Additionally, and to better yogurt texture development yogurts were stored under refrigeration overnight. This is the reason why yogurts as well as purees were cold stored overnight (24 h) to mix them when cooled. It is known that contamination by molds because of damage to figs could reduce quality and increase the risk of aflatoxin and ochratoxin. Therefore, triplicated samples from two harvest days were sent to an external laboratory for mycotoxins analysis by LC MS-MS and the following results were obtained. All tested mycotoxins were under the detection limits of the technique (Aflatoxin B1 <1.0 μg kg^{-1}; Aflatoxin B2 <1.0 μg kg^{-1}; Aflatoxia G1 <1.0 μg kg^{-1}; Aflatoxin G2 <1.0 μg kg^{-1}; Desoxinivalenol <50 μg kg^{-1}; Fumonisins sum (B1 + B2) <20 μg kg^{-1}; Ocratoxin-A <1.0 μg kg^{-1}; Patulin <10 μg kg^{-1}; and, Zearalenone <10 μg kg^{-1}) and analyzed fig by-products fulfilled the requirements of EU regulations as indicated below (Maximum Residue Level MRL sumatory of Aflatoxins 10 μg kg^{-1} and Ocratoxin A 8.0 μg kg^{-1} by R396/2005 of the European Parliament and of the Council of February 23, 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending; Council Directive 91/414/EEC. Current consolidated version: May 11, 2024).

For producing fermented milk were using UHT whole cow's milk (3.6 g 100 mL^{-1} fat content, Hacendado, Spain) and lyophilized concentrated lactic ferment containing *Streptococcus thermophilus, Lactobacillus delbrueckii* ssp. *lactis, and Lactobacillus delbrueckii* ssp. *bulgaricus* (CHOOZITTM MY800 LYO 5 DCU, Rhodia Food-Danisco A/S, Sassenage, France). Fermented milk was made following previous studies (Cano-Lamadrid et al., 2017; Jiménez-Redondo et al., 2022; Muelas et al., 2022). with some modifications. To facilitate dosage according to manufacturer instructions the lyophilized culture was poured into 20 mL of sterile peptone water (Merck KGa, SigmaAldrich, United States) and kept in a water bath at 43 °C for 20 min to hydrate. One litre capacity Pyrex bottles were sterilised, and UHT milk was added under hygienic conditions (laminar flow cabinet) adding milk volumes according to the formulation to be developed (ranging from 1 L for control and 600 mL for those formulations to have 40 g 100 g^{-1} fig puree). Rehydrated culture dosage was 1000 μL per litre of milk. Once the starter culture was inoculated into the bottles, they were manually shaken for 1 min and incubated at 43 °C until they reached a pH of 4.6. Once this pH was reached, they were refrigerated for 24 h before being mixed and shaked with the fig puree. Finally, a determined amount of

pasteurized puree (0, 10, 20, 30, and 40 g 100 g^{-1}) was weighed and added to each fermented milk bottle, which were manually shaken until obtaining a homogeneous drinking fermented milk. It was dispensed into sterile containers and kept refrigerated for 24 h at 4 °C. Finally, the fermented milks were kept refrigerated for analysis at two sampling times: i) T0 (24 h of refrigerated storage) and ii) T30 (30 days of refrigerated storage).

2.2. Microbial load and techno-functional properties analysis

MRS agar was utilized for enumerating *Lactobacilli* counts (LAB) at 37 °C under microaerophilic conditions for 48 h, while M17 agar was employed for *Lactococci* counts (LAC) at 30 °C under aerobic conditions for 48 h. Rose Bengal Agar was employed for detecting molds and yeasts at 26 °C under aerobic conditions for 72 h. The color characteristics of fermented milks were investigated within the CIELab* color space, evaluating parameters such as lightness (L*), redness (a*, green-red coordinate), and yellowness (b*, blue-yellow coordinate). Color determinations were conducted at 12 ± 2 °C using a Minolta CM-2002 spectrophotometer, equipped with a liquid accessory CR-A70, with illuminant D65 and an observer of 10°. The equipment underwent daily calibration with the provided white plate by Minolta. pH was measured in all batches to monitor the fermentation process and to ensure that the pH reached the value of 4.6 as indicated above. Additionally, the pH was measured in the fortified fermented milks after incorporation of the fixed g 100 g^{-1} of pasteurized fig puree at time 0 and at time 30 (these values are shown in Table 1). Three replicates were conducted for pH and microbiology, while nine replicates were performed for color assessment. A penetration test was executed using a Texture Analyser TA-XT2 with a 5 kg load cell. Constant speed penetration tests were conducted directly on cylindrical containers (4.5 cm diameter, 4 cm height). All instrumental texture analyses were carried out at 8 °C after removing spontaneous syneresis. This test is 'destructive' as it does not allow structure recovery. A cylindrical probe with a diameter of 10 mm (P-10) was inserted 15 mm into the samples at a speed of 1 mm s^{-1}. Triplicate measures were taken for each yogurt. Gel stability was visually assessed post-incubation (spontaneous syneresis) and determined by quantifying the volume of whey removed from the curd after centrifugation (syneresis). A Hewlett Packard HP-1100 instrument (Woldbronn, Germany) coupled with two detectors: DAD G1315A (set at 210 nm) and RID G-1362 A was used for analysing organic acids and sugars in fermented milks (Jiménez-Redondo et al., 2022). Chromatographic analysis was performed in isocratic gradient with a flow of 0.5 mL/min and a mobile phase consisting of ultrapure water acidified with 0.1 % phosphoric acid. The column used was a Supelcogel C-610H, 30 cm × 7.8 mm (Supelco Park, Bellefonte, PA, USA). Sugars were detected with a refractive index detector and lactic acid with a diode array (DAD) at a wavelength of 210 nm. The quantification was carried out using external calibration curves prepared with pure standards of sugars and lactic acid (Merck KGaA, Darmstadt, Germany).

2.3. Total polyphenolic content (TPC), antioxidant capacity and flavonoids identification and quantification

0.5 g of fresh sample were used and 4 mL of extracting solution methanol/water/formic acid (80:19.9:0.1, v/v) was added for phenolic compounds extraction. The tubes were shaken in an orbital bath (Unitronic 320 OR, Selecta, Barcelona, Spain) with ice for 10 min at 250×*g* and 4 °C sonicated (Model 3000512, Selecta, Barcelona, Spain) for 10 min and centrifugated at 4000×*g* for 10 min at 4 °C (Sigma 3−18 K; Sigma Laborzentrifugen, Osterode and Harz, Germany). Supernatants collected after centrifugation are re-extracted additionally 2 times and quantified in triplicate using the Folin−Ciocalteu reagent, with results expressed as mg gallic acid equivalent per gram of fresh weight (fw). These extracts were used for antioxidant capacity (DPPH) as per the method by (Brand-Williams et al., 1995), with results expressed as mmol Trolox equivalents (TE) per gram of fresh weight (fw). For flavonoids determination, extracts were filtered through a 0.45 μm pore size membrane filter before injection in the LC–MS/MS system (LC-MS/MS 8050, Shimadzu, Kyoto, Japan). The method of LC–MS/MS analysis was performed according to the procedure described by (Uysal et al., 2023) with a slight modification. The column temperature (Mediterranea SEA 18, 10 mm L x 0.21 mm i.d., 2.2 μm, Teknokroma, Barcelona, Spain) was set at 50 °C. The mobile phase consisted of two solvents: (i) Solvent A, water/formic acid (99.9:0.1, v/v) and (ii) Solvent B, acetonitrile/formic acid (99.9:0.1, v/v). Anthocyanin compounds were eluted as following conditions: 0.4 mL/min fow rate and 30 °C, isocratic conditions for 1 min with 99 % A, from 1 to 15 min linear gradient of 1–40 % acetonitrile with 0.1 g 100 mL^{-1} formic acid (B), solvent B was increased to 100 %, between 15 and 23 min, then returned to initial conditions of 99 % A in 2 min, and isocratic conditions with 99 % of 1 g 100 mL^{-1} aqueous formic acid for 5 min followed by washing and reconditioning the column. The sample volume injected was 10 μL. The sample volume injected was 10 μL. The ultraviolet visible (UV–visible) spectra were scanned from 200 to 600 nm for all peaks. The analysis was performed in triplicate for each sample. The identification was acquired using authentic standards and comparing the retention times and UV–visible spectra with those found in the literature. The characterization of the single components was carried out via the retention time and the accurate molecular masses. The PDA spectra were measured over the wavelength range of 200–600 nm. The runs were monitored at the following m/z: cyanidin 3,5-diglucoside at 611.10, quercetin-3-galactoside at 465.00, and quercetin-3-glucoside at 463.25. Retention times (Rt) and spectra were compared with pure standards.

2.4. Statistical analysis

Statistical analysis and comparison among means were carried out using the statistical package SPSS 24.0 (IBM SPSS Statist cs, Chicago, IL, USA). One–way ANOVA test was carried out, followed by Tukey's test (95 % confidence level). Principal component analysis (PCA regression map) was conducted to project the samples depending on the techno-functional parameters and microbial load.

3. Results and discussion

3.1. Microbial load and metabolic products

Table 1 shows the pH values, observing statistically differences among formulations, but no difference was reported between sampling time (T0-30). The initial pH values of the control, F10, F20, F30 and F40 fermented milks samples were 4.48, 4.49, 4.70, 4.84 and 4.81, respectively. As expected, the addition of pasteurized fig puree increased the pH of the final fermented milks. The range of values of fig fruits reported by other authors is above the value of the fermented milk (between 5.2 and 6) (Pereira et al., 2017). Our results are in accordance with previous studies; Feng et al. (2019) observed no significant differences in the pH values between jujube juice enriched yogurt formulations during monitored storage period. It could be said that the main reason is the zero or low production of lactic acid by lactic acid bacteria during refrigeration conditions.

As to microbial load, different behaviour was observed between *Lactobacillus* and *Lactococcus*. In terms of formulation, although no statistically significant differences were reported for *Lactobacillus*, statistically significant variations were observed for *Lactococcus*. It is important to highlight that no differences in *Lactobacillus* (above 6.5 Log LAB CFU g^{-1}) and *Lactococcus* (above 8.6 Log LAB CFU g^{-1}) were noted between sampling time (T0-30). The microbial load was adequate, being above 6 Log CFU/g the level established by the International Recommendations for Fermented Milks (FAO, 2003). The observed trend aligns with the findings reported in the pH section. Contrary, previous studies in which fermented milks was enriched with different fruits reported a reduction

Table 2
Texture parameters and gel stability of developed fermented milks at T0 (24 h) and T30 (30 days, refrigerated storage).

	Firmness (g)	Consistency (g·seg)	Cohesiveness (g)	Viscosity index (g·seg)	Syneresis
ANOVA [a,b]	**	**	**	**	**
CTRL T0	15.3 ± 3.21 c	378 ± 72 c	−11.8 ± 2.3 a	−5.54 ± 1.01 c	71.4 ± 0.5 a
F 10 T0	15.7 ± 2.48 c	404 ± 87 bc	−10.9 ± 1.6 a	−2.44 ± 0.94 a	66.0 ± 2.3 b
F 20 T0	16.0 ± 0.85 bc	396 ± 30 bc	−12.4 ± 0.7 a	−5.26 ± 0.58 bc	59.3 ± 0.7 c
F 30 T0	17.8 ± 1.74 b	449 ± 50 b	−14.0 ± 1.5 bc	−11.3 ± 1.78 d	56.5 ± 0.4 d
F 40 T0	19.7 ± 1.26 a	512 ± 33 a	−16.4 ± 0.6 c	−22.8 ± 3.26 e	56.7 ± 0.6 d
CTRL T30	14.5 ± 1.24 d	351 ± 21 d	−11.1 ± 0.4 a	−4.15 ± 0.52 b	73.8 ± 0.4 a
F 10 T30	14.6 ± 0.98 cd	360 ± 17 cd	−11.6 ± 0.2 a	−2.42 ± 0.41 a	65.8 ± 0.7 b
F 20 T30	15.8 ± 0.24 c	389 ± 11 c	−11.9 ± 0.9 a	−2.85 ± 0.33 a	61.5 ± 0.2 bc
F 30 T30	17.3 ± 0.11 b	437 ± 57 b	−13.1 ± 1.2 ab	−9.08 ± 1.11 d	60.9 ± 0.6 cd
F 40 T30	19.3 ± 1.10 a	498 ± 49 b	−15.8 ± 1.1 c	−20.8 ± 2.10 e	56.1 ± 0.7 d

*, **, and ***, significant at $p < 0.05$, 0.01, and 0.001, respectively.
[a] NS = not significant ($p < 0.05$).
[b] Values followed by the different letter within the same column were significant different ($p > 0.05$), Tukey's multiple-range test.

in the microbial load after 20–30 days of refrigerated storage (Almusallam et al., 2021; Feng et al., 2019; Mahmoudi et al., 2021; Ning et al., 2021; Silva et al., 2022; Taheur et al., 2023). This could be due to post-acidification throughout refrigerated storage in fermented milks. According to the results obtained, it can be concluded that fig by-products could be suitable to the development of fermented products with lactic acid bacteria, maintaining their viability and ensuring probiotic effects.

Significant changes in organic acids and sugars changes were observed as an effect of formulation and sampling time. Figs from the "Colar" variety employed in developed fermented milks was rich in sugars was reported by Teruel-Andreu et al. (2023) had values for glucose and fructose in fresh pulp of "Colar" figs were 379 g kg^{-1} and 364 g kg^{-1} dried weight, respectively. Similarly, the content of these sugars in pasteurized fig puree was quantified, being 155 g kg^{-1} and 156 g kg^{-1}, respectively. As expected, the addition of fig puree resulted in increased glucose and fructose content, with the highest levels detected in F40, followed by F30, F20, and F10, compared to the CTRL. Galactose was also detected in CTRL sample, but it was not possible to estimate the galactose content in the fermented milk enriched with fig puree due to the large size of area of fructose and the proximity of their retention times (12.86 min for galactose and 13.05 min for fructose). Changes in the content of lactic acid was observed between sampling time, being in all samples higher at T30 given lactic acid metabolism during refrigerated storage.

3.2. CIELab coordinates

As to CIELab* coordinates, significantly differences ($p < 0.05$; Table 1) was found among formulations and between sampling times. Compared with the control, the a* and b* and C values of fermented milks developed increased with increasing amounts of puree fig, whereas their L* and h values decreased. Generally, incorporation of FP in fermented milks significantly decreased L* values compared with the control, indicating that decreased the lightness and a light pink colour formation. After 30 days of storage, the color of FP fermented milks samples kept constant for L* and a* coordinates, although the b* and C values showed a decreasing tendency in all formulations except the C value for formulation F40 at T30, where the result remains constant. For h, the results obtained among the formulations after 30 days of storage were variable, with formulations F20 at T30 and F30 at T30 decreasing, while the rest of the formulations remained constant. Our results were similar to these authors that reported a decreasing trend during storage, especially for the luminosity (L*) in enriched yogurt with edible anthocyanin-rich plant materials such as mulberry pomace (Du et al., 2021, 2023), pomegranate (Cano-Lamadrid et al., 2017), cherry (Sánchez-Bravo et al., 2018), grape (Silva et al., 2022). The decreasing trend during cold storage of certain color parameters is mostly caused by the degradation of the bound pigments during storage (Du et al., 2023). In addition, the pH of the medium has been reported as the main cause of anthocyanin color changes (Cheynier, 2012), but in our study, no changes in pH were detected during the storage period. Other authors have reported that there was no statistically significant effect of storage time on the color of natural plain yogurts (Jakubowska & Karamucki, 2019).

3.3. Texture parameters and gel stability

Table 2 shows texture parameters and syneresis of developed fermented milks. Statistically differences among formulations and between sampling time (T0-T30) was reported in this study. Taking the formulation into account, the highest values of firmness (19.74 g), and consistency (511.82 g seg) were observed when 40 g 100 g^{-1} of fig puree was added (F40), followed by the other percentages in both sampling times (T0 and T30). The lowest values of cohesiveness (−16.42 g) and viscosity index (−22.83 g seg) were observed when 40 g 100 g^{-1} of fig puree was incorporated (F40), followed by the other percentages. When considering the sampling time, it is important to note that formulations with 30 g 100 g^{-1} and 40 g 100 g^{-1} fig puree showed no statistically significant changes in firmness values between T0 and T30. In addition, statistically differences of consistency were observed in CTRL, F10 and F20 between sampling time, while consistency was maintained in the rest of the formulations. However, the cohesiveness values remained constant, while there was fluctuation in viscosity results with lower results for CTRL and F20 respect initial time, but constant for the rest of formulations. Overall, F30 and F40 remained constant during the 30 days of storage, with only changes slightly in consistency and cohesiveness for F40 and F30, respectively. Our results indicate that incorporating fig puree enhanced certain techno-functional parameters, particularly when 30 g 100 g^{-1} and 40 g 100 g^{-1} of fig puree were added. This may be due to figs, especially the peel, have a high content of pectin (Gharibzahedi et al., 2019). Pectin or plant polysaccharide has proven effective protein aggregation and reduces serum separation in yogurt (Foley & Mulcahy, 1989). Additionally, its use increases viscosity in acidic milk gels and contributes to textural stabilization in stirred yogurt (Amice-Quemeneur et al., 1995). Previous studies reported an improvement in texture and increased viscosity in yogurts fortified with plant material high in pectin such as lemon peel powder (Rahman et al., 2024), apple pomace (Wang et al., 2019), and orange fibre (Kieserling et al., 2019).

In terms of syneresis, statistically significant differences were observed among formulations; as the percentage of fig puree increases, the syneresis of fermented milk decreases. Also, statistical difference was reported between sampling time (T0-T30). A low incidence of syneresis suggests an enhanced capacity to retain water, which usually correlates with higher gel strength (Huang et al., 2021). Fermented milks with 20 g 100 g^{-1} and 30 g 100 g^{-1} of FP at T30 showed higher syneresis values compared to the ones at T0. The increase in g 100 g^{-1}

Table 3
Organic acid and sugars (g kg^{-1}), total phenols content (TPC) and antioxidant capacity (DPPH assay) of developed fermented milks at T0 (24 h) and T30 (30 days, refrigerated storage).

	Malic acid	Lactic acid	Lactose	Galactose	Glucose	Fructose	TPC	DPPH
							(g GAE kg^{-1})	(mmol Trolox kg^{-1})
ANOVA [a,b]	*	*	**	**	**	**	**	*
CTRL T0	nd	51.3 ± 1.1 ab	237 ± 12 a	68 ± 2 a	nd	nd	36.3 ± 2.2 c	13.1 ± 0.7 b
F 10 T0	1.0 ± 0.1 b	48.0 ± 1.0 bc	221 ± 6 ab	nd	17.2 ± 1.0 de	26.3 ± 1.1 d	45.6 ± 9.8 bc	13.9 ± 0.9 ab
F 20 T0	2.0 ± 0.1 b	43.8 ± 3.1 cd	204 ± 3 b	nd	32.7 ± 1.1 c	39.3 ± 0.9 c	50.1 ± 8.8 abc	14.6 ± 0.3 ab
F 30 T0	3.0 ± 0.1 ab	42.2 ± 5.2 d	189 ± 7 c	nd	46.7 ± 0.9 b	52.7 ± 2.1 b	57.1 ± 6.8 ab	14.3 ± 1.0 ab
F 40 T0	5.0 ± 0.1 a	42.8 ± 2.3 d	173 ± 6 c	nd	61.7 ± 1.0 a	66.7 ± 1.3 a	64.4 ± 3.6 a	15.3 ± 0.7 a
CTRL T30	nd	60.7 ± 2.0 a	248 ± 5 a	51 ± 4 b	nd	nd	32.1 ± 1.9 d	12.6 ± 0.5 c
F 10 T30	1.0 ± 0.1 b	60.1 ± 1.9 a	235 ± 8 a	nd	13.7 ± 0.8 e	21.3 ± 0.9 e	45.2 ± 2.5 bc	12.6 ± 0.2 c
F 20 T30	2.5 ± 0.1 b	52.7 ± 1.8 ab	216 ± 10 ab	nd	34.7 ± 1.0 c	32.5 ± 1.1 cd	51.3 ± 3.7 abc	13.0 ± 0.8 b
F 30 T30	3.3 ± 0.1 ab	49.0 ± 1.7 b	192 ± 4 c	nd	48.0 ± 1.1 b	43.7 ± 1.4 cd	54.8 ± 2.5 abc	13.3 ± 0.6 b
F 40 T30	4.1 ± 0.1 a	47.7 ± 2.2 bc	162 ± 6 c	nd	60.3 ± 1.7 a	63.8 ± 1.3 ab	65.5 ± 2.7 a	13.2 ± 0.7 ab

*, **, and ***, significant at $p < 0.05$, 0.01, and 0.001, respectively.
[a] NS = not significant ($p < 0.05$).
[b] Values followed by the different letter within the same column were significant different ($p > 0.05$), Tukey's multiple-range test.

syneresis during storage is in agreement with other studies (Ramirez-Santiago et al., 2010) but Sánchez et al. (2020) reported that yogurt would be less susceptible to syneresis during storage if its water retention capacity is improved.

As mentioned earlier, the pectin found in the fig puree may contribute to enhanced gel stability, resulting in reduced syneresis. Other aspects in which the fig puree may have influenced the texture include increase the solids and increase the phenolic compounds. It is common to increase the solids of commercial yogurts to improve the texture of the final yogurt (Jaster et al., 2018) and the phenolic compounds in FP could interact with caseins to form soluble complexes and further enhance the gel strength of set yogurts (Kumar & Mishra, 2003).

On the other hand, it is worth mentioning that figs and fig latex contain several proteases with high specificity for casein as a substrate and so able to coagulate milk. Most of them could be inactivated at temperatures above 60 °C which is much lower than the used in the present study to pasteurize fig pure. Overall, authors expect most textural changes to be due to fruit polysaccharides but to proteolytic changes due to fig enzymes that were mostly inactivated. Crude fig protease extract has been reported to have stable proteolytic activity in a pH range of 6.5–9.0 (optimal at pH 7–8) but lose activity, at pH 2–3. The proteolytic activity of the fig extract is stable up to 60 °C but declines at higher temperatures (Kim et al., 2011). The main protease in fig is ficin (Cysteine Proteases), and when isolated maintains activity up to 72 °C, but the stability declines at higher temperatures, and it is active within a pH range of 6.5–8.5, with maximum activity observed at pH 7.0 (Devaraj et al., 2008). Ficin Isoforms (A, B, C, D1, D2) from fig latex exhibit varying degrees of thermal stability, however, all of them are prone to autolysis at high temperatures (Zare et al., 2013).

3.4. Functionality

The total phenolic content (TPC) and radical scavenging rate (DPPH) are presented in Table 3. The developed fermented milks exhibited a rising trend in both total phenolic content (TPC) and DPPH radical scavenging activity with increasing amounts of added fig puree. This finding was attributed to the abundant polyphenols and high antioxidation potential of figs; thus, recent studies have highlighted the high antioxidant potential of figs (Teruel-Andreu et al., 2021, 2023; Wojdyło et al., 2016). After 30 days of storage, the TPC of FP fermented milks samples kept constant except for CTRL and F30 formulations that decreases slightly. While, for DPPH all formulations showed a decreasing tendency, obtaining values between 1 and 1.2 times lower. Muniandy et al. (2016) studied the influence of green, white and black tea addition on the antioxidant activity of probiotic yogurt during refrigerated storage and indicated that TPC of all tea yogurts analyzed decrease after 21 days storage. In contrast, these authors Du et al. (2021) indicated the content TPC increase of mulberry pomace-fortified stirred yogurts during cold storage at 4 °C. These differences in results may be due to the interactions between the compounds of the added plant material and the components of the milk, which affect the bioavailability or degradation of the antioxidant compounds.

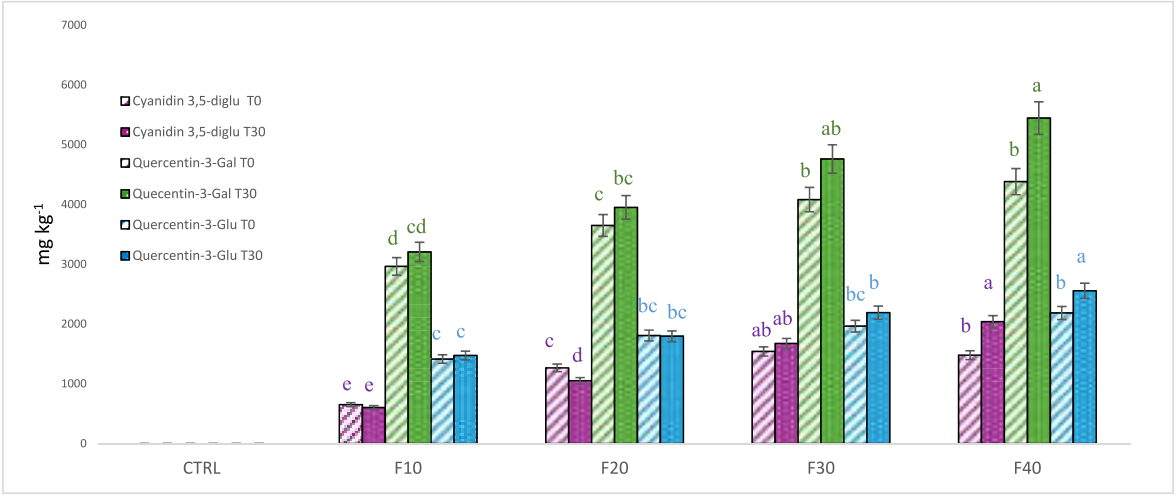

Fig. 1. Concentration (mg kg^{-1}) of the anthocyanins identified in developed fermented milks.

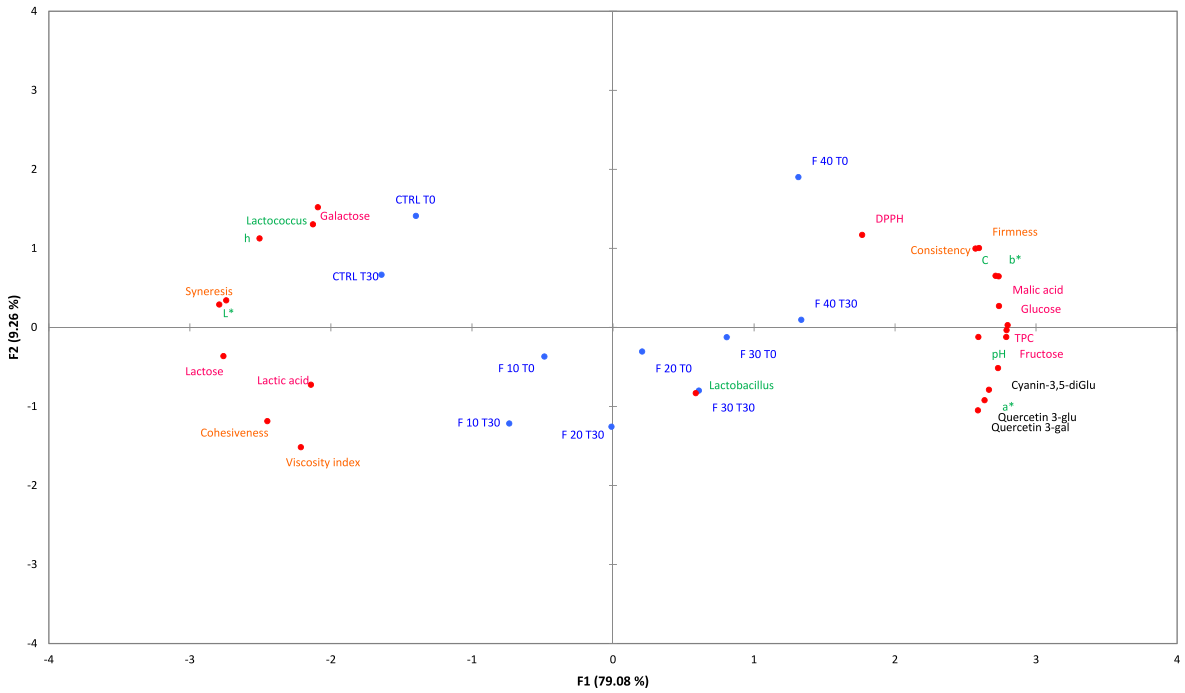

Fig. 2. Principal Component Analysis (PCA) of developed fermented milks and parameters with statistical differences (89 %).

3.5. Flavonoids content

Three flavonoids were identified (2 flavonols and 1 anthocyanin) in the fermented milks (Fig. 1). At T0, quercentin-3-galactoside (mean value of 3770 mg kg^{-1}) was the highest flavonol identified and quantified followed by quercentin-3- glucoside (mean value of 1844 mg kg^{-1}) and cyanidin 3,5-diglucoside (mean value of 1235 mg kg^{-1}). Significant differences were found among samples with different fig percentage at both T0 and T30 (Fig. 1). As expected, any of them was detected in CTRL and as the fig puree content increased, the detected compounds increased. The fig puree incorporated was previously pasteurized, which likely accounts for the absence of the rich anthocyanin profile typically found in fresh figs. These authors Wojdyło et al. (2016) researched phenolic compounds, antioxidant and antidiabetic activity of different cultivars of *Ficus carica* L. fruits and detected the content of cyanidin 3, 5-diglucoside (mean value of 1.0 mg 100 g^{-1} dm) and quercentin-3-galactoside (mean value of 8.3 mg 100 g^{-1} dm) in different cultivars figs. While our results of the pasteurized puree showed contents of cyanidin 3,5-diglucoside (53.9 μg g^{-1}), quercetin 3-galactoside (160 μg g^{-1}) and quercetin 3-glucoside (48.8 μg g^{-1}). In addition, previous research (Sakhale et al., 2015) reported significant losses in anthocyanin content during heating in the fig jam-making process.

Significant differences were found between sampling time in each formulation. It is essential to highlight that a higher content was extracted using the same methodology at T30 compared to samples at T0. The highest amounts of all detected compounds, in particular quercetin-3-galactoside, were found in milks fermented with 40 g 100 g^{-1} fig puree at T30. After 30 days of storage, cyanidin 3,5-diglucoside of FP fermented milks samples kept constant for F10 and F30 formulations, although F20 showed a result decreasing and formulation F40 obtained increased values. For quercetin 3-galactoside, the results increase in all formulations after 30 days of storage. Quercetin 3-glucoside remained constant with formulations F10 and F20 during 30 days of storage while the F30 and F40 formulations showed higher values at T30. The dairy matrix could contribute for stability of some phenolic compounds (Oliveira et al., 2018). Contrary (Du et al., 2022), that studied antioxidant activity in yogurt supplemented with mulberry pomace, reported that especially anthocyanins gradually degraded due to oxidation and utilization during the refrigerated storage. Interactions between proteins and flavonoids like anthocyanins and flavonols have been extensively studied. Higher numbers of hydroxyl groups (-OH) in flavonoids result in stronger hydrogen bonds with proteins, as evidenced other authors (Arts et al., 2002; Li et al., 2023; Yuksel et al., 2010). Caseins, being hydrophobic molecules with regions of negative and positive charge, form stable bonds with flavonoids. Also, due to this factor, the presence of multiple -OH groups and a positive charge, the time stability of cyanidin 3,5-diglucoside is ensured (Trigueros et al., 2014) copigmentation between cyanidin 3,5-diglucoside and studied flavonols through ionic interactions and hydrogen bonds, particularly with quercetin 3-glucoside, results in more stable complexes over time (Cavalcanti et al., 2011). These interactions, observed by Cao et al. (2023) in Dashi blackberry juices, contribute to maintaining anthocyanin concentrations, especially when flavonols like kaempferol, rutin, quercetin, and isoquercetin are involved, mediated by hydrogen bonds and van der Waals forces. In other hand, malic acid was protective in bread samples with grape seed proanthocyanidins, reducing thermal degradation and enhancing final product content (Zhang et al., 2023). In addition, fermenting cultures, notably LAB, influence phenolic compound stability via pH changes and metabolic processes. The specific LAB combination in fig fermented milks production-maintained anthocyanin levels, as seen in blueberry and pomegranate yogurts (Cano-Lamadrid et al., 2017; Ścibisz et al., 2012). Finally, Temperature and sugar content also affect flavonoid stability, refrigeration and sucrose enhance stability (Nikkhah et al., 2007). The increase in bioactive compounds in fermented milks with fig puree shows that lactic acid bacteria have not metabolized the bioactive compounds. These positive results indicate the feasibility of adding fig puree to fermented milks, although there is a need to improve the preservation of bioactive compounds in the fig puree.

3.6. Principal Component Analysis (PCA)

Principal component analysis (PCA) is an important tool used for visualizing the relationship between the analyzed data and its composition. Therefore, the first two principal components (Fig. 2) explained 88.34 % (F1 = 79.08 % and F2 = 9.29 %, respectively) of the total

Table 4
Pearson's correlation coefficient showing the strength of relationship between variables. Values in bold are significant at 95 % confidence limit.

	Malicacid	Lacticacid	Lactose	Galactose	Glucose	Fructose	Cyanin	Quercetin3 − gal	Quercetin3 − glu	TPC	DPPH	L*	a*	b*	C	h	Syneresis	Firmness	Consistency	Cohesiveness	Viscosity	pH	*Lactobacillus*	*Lactococcus*
Malic acid	**1**																							
Lactic acid	**−0.669**	**1**																						
Lactose	**−0.949**	**0.762**	**1**																					
Galactose	**−0.671**	0.422	0.619	**1**																				
Glucose	**0.988**	**−0.711**	**−0.972**	**−0.713**	**1**																			
Fructose	**0.969**	**−0.718**	**−0.949**	**−0.786**	**0.983**	**1**																		
Cyanin	**0.912**	**−0.681**	**−0.946**	**−0.767**	**0.962**	**0.946**	**1**																	
Quercetin 3-gal	**0.864**	−0.553	**−0.849**	**−0.911**	**0.905**	**0.926**	**0.948**	**1**																
Quercetin 3-glu	**0.879**	−0.592	**−0.868**	**−0.911**	**0.918**	**0.942**	**0.953**	**0.998**	**1**															
TPC	**0.968**	**−0.698**	**−0.967**	**−0.75**	**0.98**	**0.987**	**0.948**	**0.926**	**0.939**	**1**														
DPPH	0.593	**−0.865**	−0.602	−0.41	0.58	0.618	0.474	0.411	0.467	0.575	**1**													
L*	**−0.968**	**0.698**	**0.951**	**0.781**	**−0.991**	**−0.981**	**−0.978**	**−0.945**	**−0.955**	**−0.978**	−0.554	**1**												
a*	**0.9**	**−0.636**	**−0.869**	**−0.914**	**0.93**	**0.959**	**0.941**	**0.986**	**0.993**	**0.946**	0.54	**−0.963**	**1**											
b*	**0.932**	**−0.858**	**−0.971**	−0.604	**0.943**	**0.942**	**0.883**	**0.793**	**0.824**	**0.944**	**0.766**	**−0.916**	**0.848**	**1**										
C	**0.964**	**−0.796**	**−0.986**	−0.567	**0.97**	**0.946**	**0.906**	**0.797**	**0.822**	**0.952**	**0.675**	**−0.937**	**0.84**	**0.984**	**1**									
h	**−0.83**	0.582	**0.792**	**0.953**	**−0.867**	**−0.91**	**−0.899**	**−0.982**	**−0.985**	**−0.894**	−0.488	**0.917**	**−0.989**	**−0.771**	**−0.753**	**1**								
Syneresis	**−0.911**	**0.78**	**0.923**	**0.798**	**−0.948**	**−0.973**	**−0.95**	**−0.925**	**−0.942**	**−0.96**	**−0.638**	**0.963**	**−0.957**	**−0.924**	**−0.912**	**0.923**	**1**							
Firmness	**0.934**	**−0.739**	**−0.962**	−0.467	**0.923**	**0.891**	**0.833**	**0.715**	**0.74**	**0.915**	**0.639**	**−0.877**	**0.758**	**0.955**	**0.976**	**−0.658**	**−0.826**	**1**						
Consistency	**0.92**	**−0.732**	**−0.955**	−0.478	**0.909**	**0.881**	**0.82**	**0.716**	**0.741**	**0.911**	**0.639**	**−0.866**	**0.757**	**0.949**	**0.962**	**−0.661**	**−0.812**	**0.996**	**1**					
Cohesiveness	**−0.903**	**0.653**	**0.914**	0.383	**−0.876**	**−0.842**	**−0.772**	**−0.645**	**−0.673**	**−0.875**	−0.585	**0.823**	**−0.692**	**−0.904**	**−0.939**	0.584	**0.785**	**−0.966**	**−0.95**	**1**				
Viscosity	**−0.835**	0.574	**0.861**	0.258	**−0.798**	**−0.75**	**−0.68**	−0.533	−0.562	**−0.793**	−0.534	**0.729**	−0.575	**−0.845**	**−0.886**	0.454	**0.665**	**−0.948**	**−0.939**	**0.979**	**1**			
pH	**0.903**	**−0.681**	**−0.908**	−0.589	**0.939**	**0.88**	**0.944**	**0.823**	**0.829**	**0.875**	0.454	**−0.939**	**0.832**	**0.843**	**0.902**	**−0.766**	**−0.879**	**0.838**	**0.806**	**−0.803**	**−0.718**	**1**		
Lactobacillus	0.104	−0.26	−0.212	−0.108	0.196	0.197	0.282	0.211	0.185	0.199	−0.16	−0.224	0.176	0.133	0.161	−0.178	−0.254	0.159	0.149	−0.082	−0.022	0.318	**1**	
Lactococcus	**−0.657**	0.49	**0.729**	**0.656**	**−0.751**	**−0.72**	**−0.888**	**−0.833**	**−0.818**	**−0.72**	−0.163	**0.793**	**−0.775**	−0.617	**−0.657**	**0.768**	**0.785**	−0.531	−0.506	0.477	0.364	**−0.837**	−0.382	**1**

variation of the experimental data. It can be noted different groups on the figure being a clear one CTRL samples at both T0 and T30 which was characterized by the highest value of L*, content of lactose, galactose, lactic acid, *Lactococcus*, h value and syneresis. Pearson's coefficient between L* coordinate showed negative correlations with glucose and fructose ($r^2 = -0.99, -0.98$, respectively; $p < 0.05$), meaning that the samples with higher percentage of fig puree was less light. Fermented milks without fig puree or low percentage were characterized by low C value which presented positive correlations with lactose and b* ($r^2 = 0.99, 0.98$; $p < 0.05$) (Table 4). As expected, the higher pasteurized fig puree, the higher values of several parameters such as antioxidant capacity, texture parameters DPPH, CIELab coordinates (C and b*), identified and quantified flavonoids, malic acid, glucose, fructose and identified flavonoids. The a* coordinate showed positive correlations with quercetin 3-galactoside ($r^2 = 0.99$; $p < 0.05$), which meant that fermented milks with more content of fig puree had more content of this flavonols. Consistency showed positive correlations with firmness ($r^2 = 1.00$; $p < 0.05$) and viscosity showed positive correlations with cohesiveness ($r^2 = 0.98$; $p < 0.05$).

4. Conclusion

The addition of pasteurized fig puree to the fermented milk showed significant effects on several parameters, such as pH, microbial count, colour, texture, syneresis, sugar content, antioxidant properties and the presence of bioactive compounds. Despite slight variations observed in some parameters during the 30 days of cold storage, overall stability was maintained, indicating the potential of these dairy products. The viability of the lactic acid bacteria was ensured during the shelf life established for the product. In particular, the addition of 30 g 100 g^{-1} to 40 g 100 g^{-1} fig puree improved the texture of the yoghurt, reduced syneresis and increased the presence of bioactive compounds, in particular quercetin-3-galactoside. Fig puree played an important role in improving yoghurt quality and could be used as a valuable ingredient in yoghurt formulations, promoting the value of by-products from *Ficus carica* fruit processing and increasing consumer benefits.

CRediT authorship contribution statement

C. Teruel-Andreu: Writing – review & editing, Writing – original draft, Formal analysis. **N. Jiménez-Redondo:** Formal analysis. **R. Muelas:** Formal analysis. **A. Almansa:** Formal analysis. **F. Hernández:** Writing – review & editing, Methodology, Conceptualization. **M. Cano-Lamadrid:** Writing – review & editing, Supervision, Methodology, Conceptualization. **E. Sendra:** Writing – review & editing, Methodology, Conceptualization.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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Bioactive compounds (LC-PDA-Qtof-ESI-MS and UPLC-PDA-FL) and *in vitro* inhibit α-amylase and α-glucosidase in leaves and fruit from different varieties of *Ficus carica* L.

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ABSTRACT

The aim of the study was to analyze the potential health-promoting and nutritional components of leaves and figs from several Spanish varieties of *Ficus carica* L. The study focused to identify (by LC-PDA-QTof/MS) and quantify (by UPLC-PDA-FL) various components including carotenoids, chlorophylls, tocols, amino acids, phenolic acids, flavonols, anthocyanins. Besides, the sugar profile, the antioxidant capacity (ORAC, FRAP and ABTS) and the *in vitro* hypoglycaemic potential *via* inhibition of α-amylase and α-glucosidase were evaluated. The leaves were found to have significant antioxidant properties. This is attributed to their high content of carotenoids (3170.77-6763.77 mg/ 100 g dm), chlorophylls (405.58–744.23 mg/ 100 g dm), tocols (59.35–115.61 mg/ 100 g dm), and polyphenols (1150.34 mg/100 g dm). Additionally, figs presented greater sources of amino acids (725.07 mg/ 100 g dm) and exhibited higher anti-diabetic activity than leaves. Figs of "*Cuello Dama Negra*" variety presented the highest content of anthocyanins (108.22 mg/100 g dm). The study suggests that incorporating these vegetal materials into another food matrix could have potential health benefits, especially in terms of antioxidant and anti-diabetic effects.

1. Introduction

The cultivation of figs dates back thousands of years, making it one of the oldest fruits to be cultivated by humans. Fig trees have spread to various parts of the world due to their adaptability to various climates, from the Mediterranean's temperate regions to the subtropics areas (Ben Abdallah et al., 2023). According to the official data belonging to 2021, the total world fig production is 1,348,254.74 t (FAOSTAT, 2021). Turkey (320,000 t), Egypt (299,000 t), Algeria (107,266 t), Iran (83,899 t), and Morocco (144,153 t) are the major producer countries, yielding almost 80 % of the total production (FAOSTAT, 2021).

The fig tree (*Ficus carica* L.) is one of the earliest fruit trees cultivated globally. Under optimal growing conditions with sufficient water and nutrients, the common fig tree produces buds throughout the growing season, which extends until autumn. These buds have the potential to develop into fruits. Fig trees can bear two crops a year. The first crop, known as "brebas," develops from buds on growth of the preceding year, often from the axils of leaves. These buds start developing with the onset of the following spring, and fruit usually matures within the first month of summer. The main crop of figs, in contrast, arises from buds in the axils of leaves on shoots that have grown during the current season (Micheloud et al., 2023).

Ben Abdallah et al. (2023) describe four distinct types of figs based on their pollination and fruit characteristics. These types are commonly found in fig cultivation worldwide: *i*) Common Fig (Parthenocarpic Type): the most familiar type of fig and includes many popular varieties; *ii*) the San Pedro type: the first crop (breba) that pollination is not needed and the second crop (the main crop: fig) in which pollination is required to achieve full maturation; *iii*) Smyrna type: production of fruit with viable seeds, pollination is required to set fruit; *iv*) The Caprifig type: the syconia (figs) contains pollen that will pollinate the Smyrna and San Pedro types.

Ficus carica is notably important for its extensive use in food, industry, and medicine and recent research examines the bioactive

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compounds and therapeutic properties of medicinal plant extracts, exploring their action mechanisms and toxicological effects (Ramadan, 2023). Some of these studies (Abdel-Rahman et al., 2021) have reported significant inhibition of cancer cell lines, such as Hep2 and HepG2, indicating the potential role of fig leaves in cancer prevention or treatment. In addition, fig extracts have also been studied for their potential to help manage diabetes and antioxidant potential (Teruel-Andreu et al., 2023). In the case of figs, the presence of polyphenols, along with other nutrients and bioactive compounds, contributes to their reputation as a health-promoting fruit. Including figs in a balanced diet can be a delicious way to incorporate these beneficial compounds into nutrition (Cano-Lamadrid & Artés-Hernández, 2022).

For all of the above mentioned, this research aimed to quantify and identify the individual carotenoids, chlorophylls, tocotrienols, tocopherols, free amino acids, and polyphenols profile (phenolic acids, flavonoids including anthocyanins, flavonols, and polimeric procyanidins) in leaves and figs of four Spanish varieties "San Antonio" (SA), "Colar" (CA, CUMH), "Cuello Dama Negra" (CDN), and "Superfig" (SF) of *F. carica.* In addition, *in vitro* biological activity (antioxidant and antidiabetic capacity) was analyzed. This is the first study in which key bioactive compounds are indented and quantified in *Ficus carica* leaves and fruit of figs in Spanish cultivars. Hence, this research contributes to the knowledge of the nutritional and bioactive composition of leaves and figs of *Ficus carica* and explores their potential health benefits.

2. Material and methods

2.1. Standards, compounds and chemicals

Standards used for the quantification of carotenoids, chlorophylls, tocotrienols, amino acids and polyphenols were purchased from Extrasynthese (Lyon, France) and Carote*Nature* GmbH (Münsingen, Switzerland). The aminoquinolyl-*N*-hydroxysuccinimidyl carbamate (AQC) reagent was from Synchem (Felsberg-Altenburg, Germany). UPLC-grade water, prepared by using an HLP SMART 1000 s system (Hydrolab; Gdańsk, Poland), was additionally filtered through a 0.22 μm membrane filter immediately before use. Acetonitrile, formic acid, and methanol for UPLC (gradient grade) were from Merck (Darmstadt, Germany). Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid), α-amylase from porcine pancreas (type VI-8), α -glucosidase from *Saccharomyces cerevisiae* (type I), hexane, ethyl acetate, acetone, and BHT were purchased from Sigma-Aldrich (Steinheim, Germany).

2.2. Plant materials

For this study, leaves and figs of the following varieties were used: San Antonio (SA), Colar (CA, CUMH), Cuello Dama Negra (CDN), and Superfig (SF) of *Ficus carica* were collected from the experimental field of the Universidad Miguel Hernández de Elche (UMH) in the province of Alicante Spain (02°03′50″ E, 38°03′50′ N). The Colar variety was collected in two localities: (*i*) in the above-mentioned coordinates (CUMH) and, (*ii*) in a commercial plot in Albatera (CA), Alicante, southern Spain (0°55′49′ W, 38°1317″ N). The leaves and figs were collected from 20-year-old trees with a plant density of 8 m × 5 m. Standard cultural practices (pruning, thinning, fertilization, and pest control treatments) were performed. Thirty leaves and figs were collected on two separate occasions, leaves were collected in May 2021 while figs were collected in July 2021. Both leaves and figs were collected and then transported to a laboratory for freeze-dried and ground to obtain the homogeneous dry material for further analysis.

2.3. Estimation of carotenoids and chlorophylls by LC-PDA-Qtof-ESI-MS (Identification) and UPLC-PDA (Quantification)

To analyze carotenoid and chlorophyll compounds, approximately 0.35 g of freeze-dried powdered samples were combined with 10 % magnesium carbonate. These samples were then agitated in darkness for 30 min at 300 rpm using a DOS-10 L Digital Orbital Shaker (ELMI; Riga, Latvia). The solvent mixture used consisted of 6 mL of hexane:acetone: methanol (2:1:1, *v/v/v*), following the method described previously by Wojdyło et al. (2020).

The mixture is centrifuged at 4 °C for 7 min at 19,000 ×g (MPW-55; Warsaw, Poland), supernatants collected after centrifugation are evaporated to dryness (XcelVap®, Horizon Technology, Inc.; Salem, MA, USA) and residue was re-extraction additionally 5 times. The pellet was diluted using 5 mL of 100 % methanol. The diluted sample is filtered through hydrophilic polytetrafluoroethylene (PTFE) 0.20-μm membrane (Millex Samplicity Filter; Merck, Darmstadt, Germany) and used for analysis.

Analysis of carotenoids was performed using ultra performance liquid chromatography (Acquity UPLC System) with a binary solvent manager and a photodiode array (PDA) detector (Waters Corp.; Milford, MA, USA). The separation was performed on an Acquity UPLC BEH reversed-phase (RP) C18 column (2.1 × 100 mm, 1.7 μm, Waters Corp.; Milford, MA, USA). The elution solvents were 0.1 % formic acid (A) and ACN:MeOH (7:3, *v/v*) (B). The detection wavelength for carotenoid compounds was 450 nm. Calibration curves (0.05 to 5 mg/mL; $R^2 \geq 0.998$) were constructed using reference standards of *trans*-β-carotene, α-carotene, all-*trans*-lutein, all-*trans*-β-cryptoxantin, chlorophyll *a*, and pheophorbide *a*. Chlorophyll *b*, chlorophyllide and pheophytin derivatives were expressed as chlorophyll *a* (Extrasynthese, Lyon, France; Carote*Nature* GmbH, Münsingen, Switzerland). The results were expressed as mg per 100 g of dry matter (dm).

2.4. Estimation of tocopherols and tocotrienols was carried out by using ultra-performance liquid chromatography with a fluorescence detector (UPLC-FL).

For the determination of tocopherols and tocotrienols. Lyophilized powdered samples (approx. 0.35 g) mixed with 5 mL of ethanol with 0.05 % butylated hydroxytoluene (BHT). The samples are then incubated overnight at a temperature of 4 °C. After, adding 10 mL 60 % CaOH and maintain 50 °C for 2 h for saponification. Then, the samples were mixed with hexane:ethyl acetate (9:1) with 0.05 % BHT. After that, NaOH (saturated solution) was added. The supernatant was collected, evaporated, and dissolved in methanol with 0.05 % BHT. The filtration was done using a Hydrophilic PTFE 0.20 μm membrane.

The analysis of tocopherols and tocotrienols was performed as described previously by Wojdyło et al. (2022) with some modifications, using UPLC (Acquity UPLC Waters; Milford, MA, USA) fluorescence detector (FL). The separation was performed on an Acquity UPLC BEH RP C18 column (1.7 μm, 2.1 mm × 100 mm, Waters Corp.; Milford, MA, USA). The wavelengths used for excitation and emission were 290 nm for excitation and 330 nm for emission. Identification and quantification were performed based on reference standards and calibration curves. Calibration curves (0.05 to 20 mg/mL; $R^2 \geq 0.998$) were constructed using α-, β-, γ-, δ-tocopherol and tocotrienol (Extrasynthese, Lyon, France; Sigma-Aldrich, Steinheim, Germany). The results were expressed as mg per 100 g of dm.

2.5. Estimation of free amino acids by LC-PDA-Qtof-ESI-MS (Identification) and UPLC-PDA (quantification)

For the determination of amino acids were performed as previously described Wojdyło et al. (2020). Freeze-dried powder of samples (approx. 0.08 g) were vortexed with 0.5 mL methanol:water (1:1, *v*/v), sonicated (Sonic 6D; Polsonic, Warsaw, Poland) for 15 min and centrifugated at 19000 g for 10 min at 4 °C (MPW-350; MPW Med. Instruments; Warsaw, Poland). The supernatant was collected and derivatized with borate derivatization buffer (pH 8.8). They were then incubated for 10 min at 55 °C with continuous vortexing using a thermo mixer (Thermomixer C; Eppendorf, Hamburg, Germany). After this

process, added 50 μL sample extract to a vial with 100 μL of a solution containing 10 mM AQC in acetonitrile (ACN), after mixing, the vials are then placed into a thermo mixer (ThermoMixer; Eppendorf, Hamburg, Germany) and heat at 55 °C for 10 min.

Estimation of free amino acid were performed with an ACQUITY Ultra Performance LC system equipped with a photodiode array detector with a binary solvent manager (Waters Corp.; Milford, USA) series with a mass detector G2 Qtof micro-mass spectrometer (Waters; Manchester, UK) equipped with an electrospray ionization (ESI) source operating in positive modes. The separation was performed using a amino column (2.1 × 100 mm, 1.7 μm) (Waters Corp.; Milford, USA). The gradient elution at a flow rate of 0.50 mL/min over 15 min. For gradient elution the following chemical was used: solvent A (50 mL of solution: acetonitrile, formic acid, and ammonium acetate (10:6:84, *v/v/v*) in 950 mL water) and solvent B (acetonitrile and formic acid; 99.9:0.1, *v/v*). The gradient program was: 99.0 % A at 0–0.30 min, 97.0 % A at 3.20 min, 88.0 % at 6.80 min, 82.0 % A at 8.95 min, 74.0 % A at 9.50 min, 67 % A at 9.80 min, 40.0 % A at 10.65 min, and 99.0 % A at 14.50–15.00 min. The PDA spectra of amino acids were measured at wavelength $\lambda = 260$ nm. All analysis was done in three repetitions. The results were expressed as mg per 100 g of dm.

2.6. Estimation of phenolic compounds by LC-PDA-Qtof-ESI-MS (identification) and UPLC-PDA-FL (quantification)

To determine polyphenolic compounds, the methodology used was the described by Wojdyło et al. (2020) For extraction, a mixture of methanol, water, ascorbic acid, and 1 % hydrochloric acid (30:68:1:1, *v/v/m/v*) was used. The extraction was repeated after the samples were stored for 24 h at 4 °C. Following this, the samples were centrifuged at 19,000 *g* for 10 min at 4 °C using an MPW-350 centrifuge (Warsaw, Poland). The resulting supernatants were filtered through a hydrophilic PTFE membrane (0.20 μm; Millex Samplicity Filter, Merck, Germany) and then prepared for analysis.

Phenolic compounds were analyzed using an ACQUITY Ultra Performance Liquid Chromatography system (Waters Corporation; Milford, MA, USA) equipped with a binary solvent manager and a photodiode array (PDA) detector. This system was coupled to a G2 Qtof micro-mass spectrometer (Waters; Manchester, UK) featuring an electrospray ionization (ESI) source operating in both negative and positive ionization modes. The injection volume was 5 μL sample in a BEH C18 column (2.1 × 100 mm, 1.7 μm; Waters Corporation; Milford, MA, USA) at 30 °C, using a gradient elution at a flow rate of 0.42 mL/min. The eluents included 2 % formic acid (solvent A) and 100 % acetonitryle (solvent B). The PDA spectra for phenolic acids flavonols and anthocyanins were measured at 320, 360 nm and 520 nm, respectively. The optimized MS parameters were as follows: *m/z* range of 100 to 1200; capillary voltage of 2000 V; cone voltage of 35 V; source temperature of 100 °C; desolvation temperature of 250 °C; and a nitrogen desolvation gas flow rate of 300 L/h. Component characterization was achieved by measuring retention times and accurate molecular masses in both negative and positive ion modes using mass spectrometry, with results presented as base peak intensity (BPI) chromatograms. Data collection was managed by MassLynxTM 4.1 ChromaLynx Application Manager (Waters Corp.; Milford, MA, USA). Quantification was performed by injecting solutions of known concentrations (0.05 to 5 mg/mL, $R^2 \leq 0.9998$) of caffeic and ferulic acids, quercetin, and kaempferol-3-*O*-rutinoside, −glucoside, and -galactoside, pelargonidn-3-*O*-glucoside as standards. The remaining flavonol and anthocyanin derivatives were expressed as the corresponding -3-*O*-glucoside derivatives. Phloroglucinol method (Wojdyło et al., 2020) was used for the analysis of polymeric procyanidins content. All analysis was done in three repetitions and the results were expressed as mg per 100 g of dm.

2.7. Analysis of in vitro biological activity

To evaluate antioxidant and anti-diabetic activities, approximately 1 g of lyophilized powdered samples is used. These samples are combined with 5 mL of a methanol, water, and hydrochloric acid mixture (80:19:1, *v/v/m*). The mixture is then incubated overnight at 4 °C. Post-incubation, the samples undergo sonication for 20 min using a Sonic 6D sonicator (Polsonic; Warsaw, Poland). After sonication, they are centrifuged at 19,000 ×*g* for 10 min at 4 °C using an MPW-55 centrifuge (MPW Med. Instruments; Warsaw, Poland). The antioxidant capacity was tested as oxygen radical absorbance capacity (ORAC), ferric reducing ability of plasma (FRAP) and 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) antioxidant assays were determined as previously described by Wojdyło et al. (2020). The results were expressed as mM Trolox per 100 g of dm.

The anti-diabetic activity was measured as inhibition of α-amylase and α-glucosidase were determined as previously described by Wojdyło et al. (2020). Acarbose solution (at the concentration of 1.02 mg/mL) was used as a positive control for anti-diabetic activities. The results are presented as IC_{50} (mg of dried sample per mL of enzyme) were performed in triplicate using a using a Synergy H1 plate reader (BioTek; Winooski, VT, USA).

2.8. Statistical analysis

Analyses were conducted in triplicate and results were expressed as the mean value ($n = 3$) ± SE (standard error). Data was analyzed using XLSTAT 2016: Data Analysis and Statistical Solution for Microsoft Excel (Addinsoft; Paris, France). A one-way analysis of variance (ANOVA) was performed. After ANOVA, Tukey's multiple range test was used. Principal component analysis (PCA) was used to create a regression map that projects the samples for “leaves” and “figs” based on the variables that showed significant differences.

3. Results and discussion

3.1. Identification and quantification of carotenoids

The results regarding the identification and quantification of carotenoids analyzed by UPLC-PDA-Qtof-ESI-MS system are summarized in Table 1. Seven carotenoids were detected in the leaves and fruits of *F. carica*. In general, leaves were richer in carotenoids than figs. The content of carotenoids in leaves ranged from 6763.77 to 3170.77 mg/ 100 g dm, with the highest values recorded for SF variety. The content of carotenoids in figs ranged from 60.62 to 87.00 mg/ 100 g dm and decreased in the following variety order: CDN > SA > CUMH > SF > *CA*. Previous study by Wojdyło et al. (2021) indicated that the total carotenoid content was 4 times higher in apple, pear, and quince leaves than in fruits. However, our results indicated that the total carotenoid content was 73 fold more in leaves than fruits. The main carotenoids found in both leaves and figs were β-carotene followed by lutein, being leaves content 69 and 103 folder more than figs. According, Bashir et al. (2023) identified carotenoids in figs include β-carotene, α-carotene, β-cryptoxanthin, lutein, and zeaxanthin. Previous studies (Kamiloglu & Akgun, 2023) show that the abundance of carotenoids varies by variety. Australian figs primarily contain lycopene (0.32 mg/100 g), while Turkish yellow figs and Israeli green-purple figs have lutein as the most abundant carotenoid (6.14–7.15 μg/g DW and 4.08–12.58 μg/g FW, respectively). Algerian white figs mainly contain β-carotene (4.32 μg/ 100 g DW). Additionally, the literature (Bashir et al., 2023) shows that variability in carotenoid content, also which can be influenced by several factors, the drying process (which can reduce total carotenoid content by up to 80 %), and a decline in carotenoid levels during fig maturation.

Overall, the total carotenoid content in fresh figs is low compared to other fruits (Kamiloglu & Akgun, 2023). But, the consumption of

Table 1
Content of carotenoids and chlorophyll (mg/ 100 g dm) in leaves and fruits of figs of studied cultivars.

	Carotenoids							
Variety	Violaxanthin	Lutein	Zeaxhatin	β-Crypoxanthin	Lycopene	α-Cartotene	β-Carotene	∑ Individual carotenoids
				Leaves				
SA	10.1 ± 0.6 ab	1034.0 ± 59.7 a	73.8 ± 4.3 ab	168.1 ± 9.7 ab	4.8 ± 0.3 a	97.8 ± 5.6 cd	5030.0 ± 290.4 a	6418.6 ± 370.6 a
CA	5.1 ± 0.3 c	589.4 ± 34.0 b	36.6 ± 2.1 c	98.7 ± 5.7 c	2.4 ± 0.1 c	65.8 ± 3.8 d	2372.7 ± 137.0 c	3170.8 ± 183.1 c
CUMH	8.5 ± 0.5 b	922.8 ± 53.3 a	62.7 ± 3.6 ab	149.7 ± 8.6 b	1.6 ± 0.1 cd	159.1 ± 9.2 b	4098.6 ± 236.6 ab	5403.0 ± 311.9 ab
CDN	9.2 ± 0.5 b	907.0 ± 52.4 a	57.6 ± 3.3 b	153.2 ± 8.8 ab	1.2 ± 0.1 d	121.3 ± 7.0 bc	3479.7 ± 200.9 bc	4729.1 ± 273.0 b
SF	12.6 ± 0.7 a	1147.2 ± 66.2 a	75.2 ± 4.3 a	193.1 ± 11.1 a	3.3 ± 0.2 b	221.2 ± 12.8 a	5111.3 ± 295.1 a	6763.8 ± 390.5 a
				Figs				
SA	0.2 ± 0.0 a	13.0 ± 0.8 a	2.0 ± 0.1 a	2.6 ± 0.1 a	1.8 ± 0.1 a	1.4 ± 0.1 a	58.8 ± 3.4 ab	79.9 ± 4.6 ab
CA	0.2 ± 0.0 ab	6.7 ± 0.4 cd	0.8 ± 0.0 d	1.4 ± 0.1 c	0.6 ± 0.0 c	0.7 ± 0.0 b	50.3 ± 2.9 b	60.6 ± 3.5 b
CUMH	0.1 ± 0.0 cd	8.9 ± 0.5 bc	1.2 ± 0.1 bc	1.8 ± 0.1 bc	0.9 ± 0.0 b	1.6 ± 0.1 a	56.4 ± 3.3 ab	71.0 ± 4.1 ab
CDN	0.2 ± 0.0 bc	10.7 ± 0.6 ab	1.5 ± 0.1 b	2.1 ± 0.1 b	0.6 ± 0.0 bc	1.3 ± 0.1 a	70.7 ± 4.1 a	87.0 ± 5.0 a
SF	0.1 ± 0.0 d	5.3 ± 0.3 d	0.9 ± 0.1 cd	0.9 ± 0.0 d	0.5 ± 0.0 c	1.4 ± 0.1 a	55.3 ± 3.2 ab	64.3 ± 3.7 b
				Tukey's Multiple Range Test for mean values				
Leaves	9.1 a	920.1 a	61.2 a	152.5 a	2.7 a	133.0 a	4018.4 a	5297.1 a
Figs	0.2 b	8.9 b	1.3 b	1.7 b	0.9 b	1.3 b	58.3 b	72.5 b
				Chlorophylls				
	CH *b*	CH *b*′	CH *a*	CH *a*′	Pheophytin *a*	Pheophytin *a*′	∑Total	CH *a*:CH*b* ratio
				Leaves				
SA	43.66 ± 2.52 c	10.50 ± 0.61 b	230.81 ± 13.33 c	52.61 ± 3.04 b	63.33 ± 3.66 b	12.04 ± 0.69 b	412.94 ± 23.84 b	5.23 a
CA	49.00 ± 2.83 c	9.56 ± 0.55 b	248.90 ± 14.37 c	31.19 ± 1.80 c	55.88 ± 3.23 b	11.05 ± 0.64 b	405.58 ± 23.42 b	4.78 ab
CUMH	86.64 ± 5.00 b	19.80 ± 1.14 a	363.59 ± 20.99 b	53.70 ± 3.10 b	103.79 ± 5.99 a	22.37 ± 1.29 a	649.88 ± 37.52 a	3.92 b
CDN	78.14 ± 4.51 b	17.91 ± 1.03 a	398.56 ± 23.01 b	67.44 ± 3.89 a	48.77 ± 2.82 b	10.52 ± 0.61 b	621.34 ± 35.87 a	4.85 ab
SF	111.67 ± 6.45 a	16.14 ± 0.93 a	505.86 ± 29.21 a	37.43 ± 2.16 c	62.51 ± 3.61 b	10.63 ± 0.61 b	744.23 ± 42.97 a	4.25 ab

Mean ± standard deviation; value in the same columns followed by different letters are significantly different at $p \leq 0.05$ according to Tukey's test. Legend_ SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negro; SF: Superfig; CH: Chlorophylls.

carotenoids has been demonstrated to bring health benefits, especially three carotenoids α-carotene, β-carotene, and β-cryptoxanthin are precursors of provitamin A (Jaswir et al., 2011).

3.2. Identification and quantification of chlorophylls

UPLC-PDA-FL analysis of methanolic extracts of leaves is summarized in Table 1. Six chlorophylls were identified in leaves and significant differences were found in the total content of chlorophylls between the varieties studied ranging from 405.58 to 744.23 mg/ 100 g dm for CA and SF, respectively. The main compounds in the analyzed samples were CH *b* followed by CH *a*. SF variety showed the highest content for the compounds CH *b* (111.67 mg/ 100 g dm) and CH *a* (505.86 mg/ 100 g dm). Literature sources (Jokar et al., 2021) indicate levels 9.9, 12.1 and 15.1 (mg/g FW) of total chlorophylls content in a study with fig tree grew in different conditions (control, covert with blue net and covert with yellow net). Between the analyzed leaves of selected varieties of *Ficus carica* the ratio of chlorophyll *a* to *b* was at significative different. The results show that in SA variety, chlorophyll *a* is up to 5.23 times more abundant than chlorophyll *b*. While, in CUMH variety chlorophyll *a* is 3.92 times more abundant than chlorophyll *b*. As previously reported by Gholami et al. (2012) in their study with drought-stressed *F. carica* plants were detected a decrease in chlorophyll significantly in severe drought figs trees (Siah and Shah varieties). Also show how the ratio of Chl*a*/Chl*b* decreased significantly in other of varieties studies (Deym and Sabz), therefore it could be because no decrease of peripheral light-harvesting complexes and a higher stress tolerance in the two varieties. In our study were obtained lower values (405.58 to 744.23 mg/ 100 g dm) of total chlorophyll content between varieties. In addition, Ben Amor et al. (2021) reported that, in another study done with four Mediterranean species (*Phoenix dactylifera* L., *Olea europaea* L., *Punica granatum* L. and *Ficus carica* L.) in two different sites (pollution site and control site). The total chlorophylls content was affected by the impact of the pollution and results decreased by 60 and 44 % for *Punica granatum* and *Ficus carica* respectively while the reduction *Phoenix dactylifera* and *Olea europaea* was lower.

It is interesting to note that the assessed *in vitro* antioxidant and antimutagenic activities of dietary chlorophyll derivatives have yielded positive results and suggests a wide range of potential health benefits associated with their consumption in both fresh and processed foods, as well as dietary supplements (Ferruzzi et al., 2002).

3.3. Identification and quantification of tocols

The tocols (tocotrienols, TT; and tocopherols, TF) composition of the tested leaves and figs is presented in Table 2. The content of tocotrienols and tocopherols detected was higher in the leaves (87.03 mg/ 100 g dm) samples than in figs. (7.68 mg/ 100 g dm). Significant differences were found in the ∑TT&TF among studied varieties in leaves, being SF variety, which showed the highest value for the ∑TT&TF (115.61 mg/ 100 g dm). In general, the ∑TF (52.15 mg/ 100 g dm) value was higher than the ∑TT (34.88 mg/ 100 g dm) for all varieties, except for the CA variety. The main TT detected was δ-TT, while the main TF detected was α-TF in all varieties, being the highest values in the SF variety (30.43 and 65.54 mg/ 100 g dm, respectively). Significant differences were found between the ∑TF in figs of several varieties. CUMH variety accumulated the greatest amounts of TF (1.75 (mg/ 100 g dm) followed by SA variety (1.62 (mg/ 100 g dm). The content of TF in figs decreases in the following order: α-TF > γ-TF > δ-TF > β-TF. Regarding values of tocols between leaves and figs, ∑TF was 39 times fold in leaves than in figs and β-TF was 130 times fold in leaves than in figs. The content of tocopherols in figs was similar to that reported by other authors (0.2 and 0.3 for α-TF and γ-TF (mg/100 g dm) (Pande & Akoh, 2010). According to Konyalıoğlu et al. (2005) the content of α-TF in *Ficus carica* leaves was higher than species such as *Q. ilex, Myrtus communis*, *Rhamnus alaternus*, and *Phillyrea angustifolia.* Therefore, confirm that these leaves could be used as a potential new source of α-TF. Additionally, Wojdyło et al. (2022) analyzed the composition of tocopherol and tocotrienol isomers in the leaves of various fruit trees. They reported that the total tocopherol content varied from 203.34 to 260.86 μg/ g dry weight in spring leaves, and from 23.83 to 235.62 μg/ dry weight in autumn leaves. Gholami et al. (2012) indicated that the content of α-TF in fig leaves increased when fig trees were affected for the water stress period. Consequently, these strong variations in tocopherol isomer contents are related to different growth stages and environmental conditions such as light intensity, season, and water stress.

Table 2
Content of tocotrienols and tocopherols (mg/ 100 g dm) in leaves and fruits of figs of studied cultivars.

Variety	δ-TT	β-TT	γ-TT	α-TT	δ-TF	β-TF	γ-TF	α-TF	∑TT	∑TF	∑TT&TF
						Leaves					
SA	23.97 ± 1.38 a	1.77 ± 0.10 ab	0.51 ± 0.03 a	4.84 ± 0.28 ab	0.51 ± 0.03 b	7.14 ± 0.41 bc	0.32 ± 0.02 b	42.39 ± 2.45 b	31.09 ± 1.80 a	50.35 ± 2.91 b	81.45 ± 4.70 bc
CA	24.29 ± 1.40 a	1.54 ± 0.09 b	0.18 ± 0.01 b	4.39 ± 0.25 b	0.62 ± 0.04 ab	4.76 ± 0.27 d	0.40 ± 0.02 ab	23.17 ± 1.34 c	30.40 ± 1.76 a	28.95 ± 1.67 c	59.35 ± 3.43 c
CUMH	29.41 ± 1.70 a	2.00 ± 0.12 ab	0.18 ± 0.01 b	5.56 ± 0.32 ab	0.57 ± 0.03 b	8.03 ± 0.46 ab	0.49 ± 0.03 a	42.00 ± 2.43 b	37.16 ± 2.15 a	51.10 ± 2.95 b	88.26 ± 5.10 b
CDN	28.74 ± 1.66 a	2.00 ± 0.12 ab	0.27 ± 0.02 b	5.64 ± 0.33 ab	0.62 ± 0.04 ab	5.90 ± 0.34 cd	0.32 ± 0.02 b	46.97 ± 2.71 b	36.66 ± 2.12 a	53.82 ± 3.11 b	90.48 ± 5.22 b
SF	30.43 ± 1.76 a	2.27 ± 0.13 a	0.51 ± 0.03 a	5.90 ± 0.34 a	0.78 ± 0.04 a	9.72 ± 0.56 a	0.47 ± 0.03 a	65.54 ± 3.78 a	39.10 ± 2.26 a	76.51 ± 4.42 a	115.61 ± 6.67 a
						Figs					
SA	4.82 ± 0.28 a	0.24 ± 0.01 b	0.02 ± 0.00 ab	0.64 ± 0.04 a	0.10 ± 0.01 b	0.07 ± 0.00 a	0.33 ± 0.02 a	1.11 ± 0.06 ab	5.72 ± 0.33 a	1.62 ± 0.09 a	7.34 ± 0.42 a
CA	5.12 ± 0.30 a	0.26 ± 0.02 ab	0.02 ± 0.00 a	0.80 ± 0.05 a	0.12 ± 0.01 ab	0.03 ± 0.00 c	0.11 ± 0.01 c	0.76 ± 0.04 c	6.21 ± 0.36 a	1.02 ± 0.06 b	7.23 ± 0.42 a
CUMH	6.02 ± 0.35 a	0.33 ± 0.02 a	0.02 ± 0.00 a	0.78 ± 0.04 a	0.14 ± 0.01 a	0.08 ± 0.00 a	0.16 ± 0.01 b	1.37 ± 0.08 a	7.15 ± 0.41 a	1.75 ± 0.10 a	8.90 ± 0.51 a
CDN	5.33 ± 0.31 a	0.28 ± 0.02 ab	0.02 ± 0.00 bc	0.76 ± 0.04 a	0.12 ± 0.01 ab	0.04 ± 0.00 bc	0.19 ± 0.01 b	0.80 ± 0.05 c	6.38 ± 0.37 a	1.15 ± 0.07 b	7.53 ± 0.43 a
SF	5.26 ± 0.30 a	0.27 ± 0.02 ab	0.01 ± 0.00 c	0.70 ± 0.04 a	0.11 ± 0.01 ab	0.05 ± 0.00 b	0.10 ± 0.01 c	0.88 ± 0.05 bc	6.25 ± 0.36 a	1.14 ± 0.07 b	7.39 ± 0.43 a
						Tukey's Multiple Range Test for mean values					
Leaves	27.37 a	1.92 a	0.33 a	5.27 a	0.62 a	7.11 a	0.40 a	44.02 a	34.88 a	52.15 a	87.03 a
Figs	5.31 b	0.28 b	0.02 b	0.74 b	0.12 b	0.05 b	0.18 b	0.98 b	6.34 b	1.34 b	7.68 b

Mean ± standard deviation; value in the same columns followed by different letters are significantly different at $p \leq 0.05$ according to Tukey's test. Legend_ TT: Tocotrienols; TF: Tocopherols; SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negro; SF: Superfig.

Tocopherols are well-established for their antioxidant properties and role in sensory preservation, tocotrienols remain an area of ongoing research with potential distinct benefits. The overall importance of tocols in preventing lipid oxidation underscores their significance in maintaining the quality and safety of fat-rich food products. Further research into tocotrienols and their unique properties could provide additional insights into their applications in the food industry (Delgado et al., 2020).

3.4. Identification and quantification of free amino acid

Amino acids have been traditionally classified as nutritionally "indispensable" (whose carbon skeletons are not synthesized *de novo* by animal cells) or "dispensable" (synthesized *de novo* in animals). However, the new classification of amino acids includes "Conditionally Indispensable amino acids" (amino acids that are typically synthesized by the body, but under certain conditions, the body may not produce them in adequate amounts) (Wu, 2013).

Twenty-one amino acids were identified, of which, eight belong to indispensable amino acids, six belong to conditionally indispensable amino acids and seven belong to dispensable amino acids as present in Table 3. Two of the most abundant indispensable amino acids in leaves were MET and ILE, being CA variety, which showed the highest values (30.18 and 9.57 mg/100 g dm, respectively). Additionally, two of the most abundant conditional amino acids in leaves were GLU and PRO ranged from 4.37 to 21.61 and 6.00 to 28.94 (mg/100 g dm), respectively. ALA, which belongs to dispensable amino acids, was the highest amino acid detected in leaves, especially for SF variety (43.02 mg/100 g dm).

In Figs, the highest total amino acid content was shown by the SA variety (1814.03 mg/100 g dm). Indispensable amino acids were detected in the following decreasing order: THR > VAL > ILE > LEU > PHE > MET > TRP > LYS. For conditionally amino acids the total amount ranged from 199.36 to 354.19 mg/100 g dm being CA varietal which presented the highest total amount. The highest dispensable amino acids were detected for figs were ASN followed by LSOP amino acids, being SA variety, which showed the highest values for both (550.18 and 503.08 mg/100 g dm). Generally, the dispensable amino acids fraction is dominant in all fruits and leaves. Additionally, according to Núñez-Gómez et al. (2021) that breba analyzed several Spanish varieties, and detected a total of 11 amino acid, the amino acid TYR was only detected in SF variety while in our study were detected in all varieties studies. In other hand the highest value of total amino acids was showed by SA variety in both studies. In addition, as previously research reported (Allegra et al., 2018; Byeon & Lee, 2021) ASP was the amino acid detected in greater quantity in figs of the varieties "Masui Dauphine" and "Dotatto" figs varieties.

Figs content in amino acids was generally higher than in leaves, especially for some dispensable amino acids such as LSOP and SAP which showed values 253.38 and 157.21-fold in figs than leaves. As previously reported by Wojdyło et al. (2021), other fruits such as apple, pear, and quince, also obtained a higher content of amino acids than in leaves. However, according to Lianju et al. (2003) the amino acid content in leaves was higher than in fruits. Amino acids play a crucial role in immune responses by regulating the activation of various immune cells such as T lymphocytes, B lymphocytes, natural killer cells, and macrophages and also regulate cellular redox state, gene expression, lymphocyte proliferation, and the production of antibodies, cytokines, and other cytotoxic substances. Adequate dietary provision of all amino acids is emphasized as necessary for maintaining normal immunocompetence and protecting the host from various diseases across different species (Li et al., 2007).

3.5. Identification and quantification of phenolic compounds

LC-PDA-Qtof-ESI-MS (identification) and UPLC-PDA-FL (quantification) analysis of the leaves and figs polyphenols compounds revealed eight phenolic acids, five flavonols and two anthocyanins (Table 4). Polyphenol profiles differed significantly across leaves of the varieties analyzed. SF (1933.56 mg/100 g dm) and CDN (1828.98 mg/100 g dm) leaves showed the highest polyphenols total content. Phenolic acids such as caffeic acid (345.77 mg/100 g dm) and ferulic acid (194.10 mg/100 g dm), were predominant in all leaves analyzed. On the other hand, the main flavonols detected was apigenin-*C*-hexoside-pentoside (294.50 mg/100 g dm). Leaves obtained TPC in a range between 572.80 and 1933.56 (mg/100 g dm). In addition, in figs two phenolic acid

Table 3
Content of free amino acids (mg/100 g dm) in leaves and fruits of figs of studied cultivars.

	Leaves					Figs					Tukey's Multiple Range Test for mean values	
	SA	CA	CUMH	CDN	SF	SA	CA	CUMH	CDN	SF	Leaves	Figs
Indispensable Amino Acids												
THR	1.09±0.06 b	3.82±0.22 a	3.12±0.18 a	3.93±0.23 a	3.96±0.23 a	35.86±2.07 a	11.79±0.68 b	15.57±0.90 b	14.41±0.83 b	12.80±0.74 b	3.18 b	18.08 a
LYS	0.33±0.02 d	0.10±0.01 d	1.29±0.07 b	0.79±0.05 c	2.16±0.12 a	0.53±0.03 a	0.27±0.02 b	0.27±0.02 b	0.27±0.02 b	0.16±0.01 c	0.94 a	0.30 b
MET	1.19±0.07 c	30.18±1.74 a	9.89±0.57 b	13.82±0.80 b	14.08±0.81 b	4.49±0.26 a	2.17±0.13 b	4.13±0.24 a	3.82±0.22 a	3.95±0.23 a	13.83 a	3.71 b
VAL	2.83±0.16 b	1.19±0.07 c	3.89±0.22 b	5.70±0.33 a	5.60±0.32 a	22.94±1.32 a	8.76±0.51 b	11.56±0.67 b	11.18±0.65 b	8.04±0.46 b	3.84 b	12.50 a
ILE	2.13±0.12 c	9.57±0.55 a	5.92±0.34 b	6.10±0.35 b	8.94±0.52 a	17.06±0.99 a	3.59±0.21 b	5.90±0.34 b	5.77±0.33 b	5.13±0.30 b	6.53 a	7.49 a
LEU	1.49±0.09 c	9.51±0.55 a	3.69±0.21 b	3.40±0.20 b	3.97±0.23 b	11.02±0.64 a	2.20±0.13 c	4.68±0.27 b	4.47±0.26 b	3.85±0.22 b	4.41 a	5.25 a
PHE	1.73±0.10 c	2.54±0.15 c	5.94±0.34 ab	4.98±0.29 b	7.24±0.42 a	8.64±0.50 a	1.68±0.10 d	3.27±0.19 bc	3.54±0.20 b	2.26±0.13 cd	4.49 a	3.88 a
TRP	0.80±0.05 b	5.53±0.32 a	5.05±0.29 a	5.67±0.33 a	5.39±0.31 a	2.43±0.14 a	0.38±0.02 b	0.60±0.03 b	0.60±0.03 b	0.57±0.03 b	4.49 a	0.92 b
Sum	11.60±0.67 d	62.44±3.60 a	38.78±2.24 c	44.38±2.56 bc	51.33±2.96 ab	102.96±5.94 a	30.85±1.78 b	45.98±2.65 b	44.06±2.54 b	36.76±2.12 b	41.71 a	52.12 a
Conditionally Indispensable Amino Acid												
ARG	0.97±0.06 bc	0.01±0.00 d	1.58±0.09 b	5.00±0.29 a	0.49±0.03 cd	nd	nd	nd	nd	nd	1.61 a	0.000 b
GLN	1.57±0.09 c	4.26±0.25 b	5.23±0.30 b	4.70±0.27 b	7.09±0.41 a	123.62±7.14 a	38.34±2.21 c	56.35±3.25 bc	61.89±3.57 b	64.97±3.75 b	4.57 b	69.03 a
GLY	3.94±0.23 ab	1.91±0.11 c	3.10±0.18 b	4.79±0.28 a	4.20±0.24 a	9.54±0.55 a	4.78±0.28 c	6.54±0.38 bc	7.89±0.46 ab	6.89±0.40 b	3.59 b	7.13 a
GLU	4.37±0.25 c	21.61±1.25 a	12.86±0.74 b	15.01±0.87 b	13.68±0.79 b	4.28±0.25 b	8.79±0.51 a	3.08±0.18 bc	2.25±0.13 c	0.80±0.05 d	13.51 a	3.84 b
PRO	11.90±0.69 b	6.34±0.37 c	15.01±0.87 b	28.94±1.67 a	6.01±0.35 c	126.64±7.31 b	301.39±17.40 a	132.13±7.63 b	127.31±7.35 b	103.69±5.99 b	13.64 b	158.23 a
TYR	0.64±0.04 d	5.20±0.30 b	2.68±0.15 cd	3.16±0.18 bc	19.77±1.14 a	3.18±0.18 a	0.89±0.05 c	1.26±0.07 c	1.28±0.07 c	2.41±0.14 b	6.29 a	1.80 b
Sum	23.39±1.35 c	39.33±2.27 b	40.45±2.34 b	61.60±3.56 a	51.23±2.96 ab	267.26±15.43 b	354.19±20.45 a	199.36±11.51 c	200.62±11.58 bc	178.76±10.32 c	43.20 b	240.04 a
Dispensable Amino Acids												
L-SOP	0.61±0.04 c	0.02±0.00 d	0.35±0.02 c	1.98±0.11 a	1.33±0.08 b	503.08±29.05 a	73.56±4.25 c	213.70±12.34 b	153.26±8.85 b	146.30±8.45 b	0.86 b	217.98 a
ASN	2.60±0.15 a	0.16±0.01 d	1.75±0.10 bc	1.29±0.07 c	1.93±0.11 b	550.18±31.76 a	81.86±4.73 d	207.45±11.98 bc	231.56±13.37 b	144.45±8.34 cd	1.55 b	243.10 a
SER	0.57±0.03 d	1.91±0.11 c	2.70±0.16 ab	2.44±0.14 bc	3.06±0.18 a	54.60±3.15 a	28.67±1.66 bc	34.76±2.01 b	24.67±1.42 c	31.00±1.79 bc	2.13 b	34.74 a
ASP	3.32±0.19 b	0.58±0.03 c	12.20±0.70 a	11.11±0.64 a	10.31±0.60 a	57.48±3.32 a	20.83±1.20 c	41.67±2.41 b	34.73±2.01 b	34.04±1.97 b	7.50 b	37.75 a
ALA	22.14±1.28 c	30.43±1.76 bc	33.24±1.92 b	35.39±2.04 ab	43.02±2.48 a	199.24±11.50 a	89.95±5.19 c	98.25±5.67 c	95.43±5.51 c	135.96±7.85 b	32.84 b	123.76 a
GABA	4.40±0.25 d	4.04±0.23 d	17.05±0.98 a	8.94±0.52 c	13.63±0.79 b	77.58±4.48 a	63.41±3.66 a	66.35±3.83 a	67.80±3.91 a	59.98±3.46 a	9.61 b	67.02 a
HCYS	1.20±0.07 c	0.54±0.03 d	0.26±0.02 d	2.71±0.16 a	2.23±0.13 b	1.64±0.09 a	0.33±0.02 c	0.40±0.02 c	0.33±0.02 c	0.88±0.05 b	1.39 a	0.72 b
Sum	34.84±2.01 b	37.67±2.17 b	67.55±3.90 a	63.86±3.69 a	75.52±4.36 a	1,443.80±83.36 a	358.60±20.70 c	662.58±38.25 b	607.77±35.09 b	552.61±31.90 bc	55.89 b	725.07 a
Total	69.82±4.03 b	139.44±8.05 a	146.78±8.47 a	169.83±9.81 a	178.08±10.28 a	1,814.03±104.73 a	743.63±42.93 b	907.91±52.42 b	852.44±49.22 b	768.13±44.35 b	140.79 b	1017.23 a

Mean ± standard deviation; value in the same columns followed by different letters are significantly different at $p \leq 0.05$ according to Tukey's test. Legend_ THR: Threonine; LYS: Lysine; MET: Methionine; VAL: Valine; ILE: Isoleucine; LEU: Leucine; PHE: Phenylalanine; TRP: Tryptophan; ARG: Arginine; GLN: Glutamine; GLY: Glycine; GLU: Glutamic acid; PRO: Proline; TYR: Tyrosine; L-SOP: Phospho-L-serine; ASN: Asparagine; SER: Serine; ASP: Aspartic acid; ALA:Alanine; GABA: Gamma aminobutyric acid; HCYS: Cysteine; SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negro; SF: Superfig.

Table 4
Content of polyphenols in leaves and fruits of figs of studied cultivars (mg/100 g dm).

	Leaves					Figs					Tukey's Multiple Range Test for mean values	
	SA	CA	CUMH	CDN	SF	SA	CA	CUMH	CDN	SF	Leaves	Figs
Phenolic acid												
Neochlorogenic acid	9.20 ±0.95 b	21.33 ±0.81 b	14.61 ±1.36 b	63.44 ±4.18 a	66.36 ±2.21 a	3.56 ±0.67 a	2.64 ±0.60 a	2.41 ±0.21 a	3.29 ±0.82 a	2.68 ±0.41 a	34.99 a	2.91 b
Chlorogenic acid	14.02 ±1.64 b	13.12 ±0.29 b	11.09 ±0.02 b	32.87 ±0.22 a	31.72 ±0.19 a	8.71 ±1.91 a	2.29 ±0.12 b	3.97 ±0.29 ab	2.34 ±0.07 b	3.46 ±0.21 b	20.56 a	4.15 b
Caffeic acid	136.09 ±13.18 b	147.17 ±5.74 b	167.99 ±13.49 b	663.55 ±24.54 a	614.06 ±22.05 a	nd	nd	nd	nd	nd	345.77 a	0.00 b
Ferulic acid	126.02 ±15.24 b	141.93 ±6.11 b	107.21 ±4.84 b	286.70 ±10.19 a	308.65 ±13.48 a	nd	nd	nd	nd	nd	194.10 a	0.00 b
Dihydrocaffeic acid hexoside4	20.13 ±1.66 b	102.24 ±6.97 a	30.28 ±15.76 b	33.09 ±6.13 b	41.88 ±2.59 b	nd	nd	nd	nd	nd	45.53 a	0.00 b
Syringic acid hexoside	25.98 ±3.68 bc	92.05 ±5.86 a	16.92 ±0.69 c	36.31 ±1.11 b	35.23 ±0.24 b	nd	nd	nd	nd	nd	41.30 a	0.00 b
Gallic acid di pentoside	13.98 ±2.02 ab	26.31 ±2.57 a	3.82 ±0.49 b	7.86 ±0.33 b	15.95 ±4.26 ab	nd	nd	nd	nd	nd	13.58 a	0.00 b
Sinapic acid	5.67 ±1.03 a	4.75 ±0.36 a	3.48 ±0.48 a	5.31 ±0.99 a	5.69 ±0.17 a	nd	nd	nd	nd	nd	4.98 a	0.00 b
Sum of phenolic acids	351.09 ±39.37 b	548.91 ±23.01 b	355.39 ±22.81 b	1,129.13 ±47.03 a	1,119.54 ±44.85 a	12.27 ±2.58 a	4.92 ±0.72 a	6.38 ±0.50 a	5.63 ±0.89 a	6.14 ±0.62 a	700.81 a	7.07 b
Flavonols												
Apigenin-*C*-hexoside-pentoside	136.83 ±18.62 b	144.54 ±6.49 b	134.67 ±14.04 b	489.80 ±14.06 a	566.66 ±21.71 a	13.48 ±3.67 a	3.07 ±0.47 b	5.37 ±0.54 ab	3.22 ±0.13 b	3.60 ±0.22 b	294.50 a	5.75 b
Quercetin-3-*O*-rutinoside	6.67 ±1.21 b	4.88 ±0.13 b	3.06 ±1.65 b	18.16 ±0.56 a	18.30 ±0.23 a	nd	nd	nd	nd	nd	10.21 a	0.00 b
Quercetin-3-*O*-glucoside	22.76 ±2.70 b	22.71 ±0.98 b	20.83 ±5.28 b	81.30 ±2.73 a	99.77 ±6.24 a	nd	nd	nd	nd	nd	49.48 a	0.00 b
Quercetin-3-*O*-(malonyl)-glucoside	23.49 ±3.32 ab	15.36 ±1.36 b	10.28 ±3.08 b	38.21 ±3.58 a	43.29 ±6.11 a	nd	nd	nd	nd	nd	26.12 a	0.00 b
Keampferol-3-*O*-rutinoside	14.48 ±1.71 ab	10.68 ±2.14 b	6.27 ±3.74 b	22.87 ±4.47 ab	31.60 ±3.80 a	nd	nd	nd	nd	nd	17.18 a	0.00 b
Sum of flavonols	204.22 ±27.56 b	198.17 ±6.83 b	175.10 ±27.79 b	650.34 ±24.28 a	759.61 ±38.09 a	13.48 ±3.67 a	3.07 ±0.47 b	5.37 ±0.54 ab	3.22 ±0.13 b	3.60 ±0.22 b	397.49 a	5.75 b
Anthocyanins												
Cyanidin-3,5-*O*-diglucoside	nd	nd	nd	nd	nd	55.28 ±12.49 ab	72.07 ±2.01 ab	17.14 ±2.64 b	91.02 ±20.63 a	17.29 ±1.43 b	0.00 b	50.56 a
Pelargonidin-3-*O*-rutinoside	nd	nd	nd	nd	nd	10.50 ±1.66 ab	13.42 ±0.63 a	2.79 ±0.20 b	17.21 ±3.40 a	3.57 ±0.17 b	0.00 b	9.50 a
Sum of anthocyanins	nd	nd	nd	nd	nd	65.78 ±14.15 ab	85.49 ±2.64 ab	19.93 ±2.84 b	108.22 ±24.03 a	20.86 ±1.60 b	0.00 b	60.06 a
PP	33.08 ±0.02 e	81.19 ±0.01 a	42.32 ±0.02 d	49.51 ±0.01 c	54.41 ±0.01 b	119.41 ±0.00 a	67.80 ±0.01 e	82.60 ±0.01 c	87.59 ±0.01 b	81.75 ±0.00 d	52.10 b	87.83 a
TPC	588.39 ±66.95 b	828.27 ±29.83 b	572.80 ±50.59 b	1,828.98 ±71.32 a	1,933.56 ±82.95 a	198.67 ±17.83 a	156.36 ±3.10 ab	107.90 ±3.39 b	199.03 ±23.89 a	106.21 ±1.82 b	1150.34 b	153.63 a

Mean ± standard deviation; value in the same columns followed by different letters are significantly different at $p \leq 0.05$ according to Tukey's test. Legend_ Sum1: Total phenolic acids; Sum2: Total flavonols; Sum3: Total anthocyanins; PP: Polymeric procyanidins; TPC: Total polyphenols content; SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negro; SF: Superfig.

compounds were detected neochlorogenic acid (2.91 mg/100 g dm) and chlorogenic acid (4.15 mg/100 g dm). Regarding flavanol only compound was detected apigenin-*C*-hexoside-pentoside (5.75 mg/100 g dm). Besides, also were detected two anthocyanins compounds cyanidin-3,5-*O*-diglucoside (50.56 mg/100 g dm) and pelargonidin-3-*O*-rutinoside (9.50 mg/100 g dm) in figs.

Significant differences were found in the total anthocyanin content among different fig varieties, in the following descending order: CDN > SA > CA > CUMH > SF. Is noted, that the total anthocyanins content in CDN figs (199.03 mg/100 g dm) is higher than the rest of the varieties studied.

Other authors (Naveed et al., 2018; Rashmi & Negi, 2020) have reported chlorogenic, caffeic, ferulic, p-coumaric, and sinapic acids among the predominant hydroxycinnamic acids in leaves of *Ficus carica*, and gallic acid, protocatechuic, vanillic, and syringic acids are among the predominant hydroxybenzoic acids. Nadeem and Zeb (2018) studied leaves of *Ficus carica* collected at four stages of ripening and detected quercetin-3-*O*-glucoside, caftaric acid, quercetin-3-*O*-, -7-diglucoside, and cumaroyl-hexose as the main phenolic compounds. In a study (Petruccelli et al., 2018) with leaves of *Ficus carica* from 10 Italian varieties, eighteen phenolic compounds were determined, and caffeoyl-malic acid and rutin were found to be the major components.

As previously reported Arvaniti et al. (2019) the most common phenolic acids present in figs are gallic acid, ferulic acid, syringic acid, chlorogenic acid, caffeic acid, and cinnamic acid. Besides, according to Kamiloglu and Akgun (2023) most of fig phenolic acid studies reported that the main phenolic acid in this fruit are chlorogenic acids. Wojdyło et al. (2016) assayed diverse *F. carica* fruit extracts and indicated that

chlorogenic acid was the only phenolic acid detected in fig fruits with values from less than 10 (mg/100 g dm) to 125 (mg/100 g dm) between varieties studied. Regarding anthocyanin content, according to Arvaniti et al. (2019) cyanidin-3-*O*-rutinoside and cyanidin-3-*O*-glucoside were the main anthocyanin compounds detected in figs. For comparison, the content of pelargonidin-3-*O*-rutinoside detected in our study was similar to that reported by Voća et al. (2014). They detected values in a range from 107.94 to 432.28 (mg/kg) between the different strawberry varieties in three harvest periods.

Previous studies (Wojdyło et al., 2021) indicated that a greater amount of polyphenols was observed in leaves than in fruits of different varieties of apple, pear and quince.

Fig. 1 shows the quantification of the polyphenols found in leaves and figs of several varieties of *F. carica*. The main group of polyphenols identified in the leaves was phenolic acid, followed by flavonols and polymeric procyanidins (PP). Significant differences were found between the leaves of the varieties for PP, phenolic acid, and flavonols. The CA leaves (0.81 g/kg) presented the highest PP content, while the varieties CDN and SF showed the highest content of phenolic acid (11.29 g/kg and 11.20 g/kg) and flavonols (6.50 g/kg and 7.60 g/kg). In leaves, the highest total polyphenolic content (TPC) was shown by the SF (19.34 g/kg) variety. Regarding figs, the highest TPC was obtained in PP and anthocyanin groups. SA shows the highest value in TPC for PP (1.20 g/kg) and flavonols (0.13 g/kg). Except for anthocyanins where the varieties followed the descending order CDN > CA > SA > CUMH > SF.

Determining the health benefits of phenolic compounds is complicated by their multifaceted nature and the potential presence of other active compounds in extracts. Besides, there are difficult establishing standardized procedures to determine the actual amounts of phenolic compounds in different samples. Finally, developing polyphenol-rich formulations for health-related applications is an area that requires urgent attention (Dias et al., 2021). Indeed, there has been a growing interest in using natural pigments, such as anthocyanins, in various industries including food, pharmaceuticals, and cosmetics. Fig peel are example that could serve as natural colorants and may offer additional benefits such as anti-inflammatory and anti-aging effects (Backes et al., 2020).

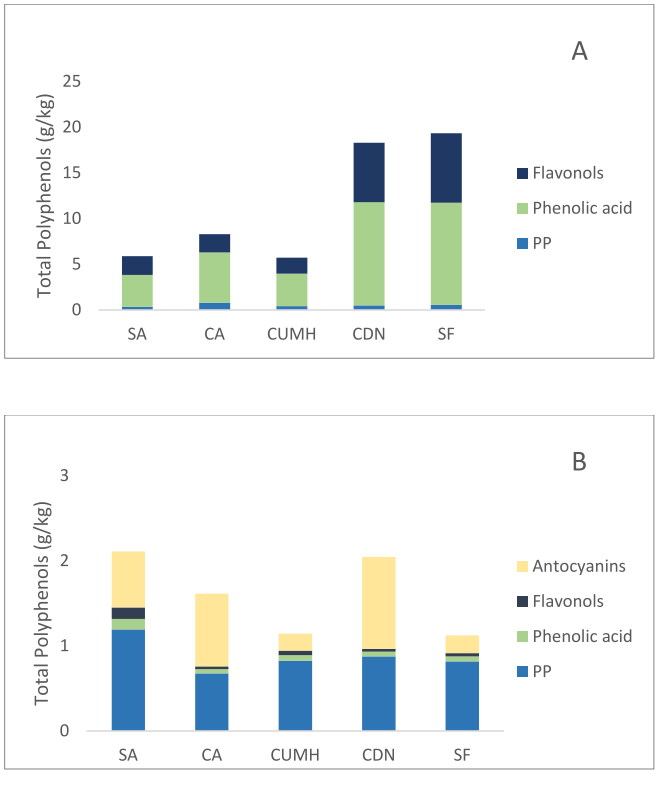

Fig. 1. Concentration (g/kg) of the main polyphenols families identified in leaves (A) and and fruits of figs. (B) (*Ficus carica* L.). Legend_ PP: Polymeric procyanidins; SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negro; SF: Superfig.

3.6. Principal component analysis of bioactive compounds

Principal component analysis (PCA) is an important tool used for visualizing the relationship between the analyzed data and its chemical composition. Therefore, the first two principal components (Supp. Fig. 2 A) explained 74.14 % (PC1 = 44.62 % and PC2 = 29.52 %, respectively) of the total variation of the experimental data. Three can be noted three groups on the figure the analyzed samples and their respective chemical analyses. The main group contains the leaves of almost all analyzed varieties and the majority of chemical compounds. The second group (visible in the upper right part of the biplot) was represented by GLU, MET, LEU and ILE amino acids as well as LCALB, which was the richest in these compounds. The last group included lycopene and LSA which were characterized (in comparison with the others) by a particularly high concentration of these compounds. In Supp. Fig. 2. B The first two principal components (PCs) explain a significant portion of the variance (81.34 %), while PC1 represented 66.87 % of the total variation and accounted mainly for chemical compounds and it contained SA figs variety, which was characterized by a particular high concentration of these compounds. PC2 explained 14.46 % of the total variation and accounted for the rest of the varieties analyzed. CDN, SF, and CUMH varieties were plotted at the upper of the chart while CALB was plotted at the bottom part. All varieties plotted in the PCA 2 part of the chart due to their lowest content in biochemical compounds than the SA variety.

3.7. Biological activity

Antioxidant properties were assessed using the ability to capture synthetic radicals (ABTS), ferric reducing ability of plasma (FRAP), and oxygen radical absorbance capacity (ORAC) assays. As shown in Table 5, for leaves CDN variety accumulated significantly higher amounts of antioxidant properties for ABTS (5.87 mmol Trolox/100 g dm), FRAP (4.84 mmol Trolox/100 g dm), and ORAC (21.16 mmol Trolox/100 g dm) methods assessed contrary to leaves SA variety showed a lower value. It is well known that variety is one of the factors which may influence antioxidant capacity and the antioxidant potential depends on the content of bioactive components such as polyphenols, isoprenoid, vitamins, or other molecules present in plants (Wojdyło et al., 2021). It is difficult to liken antioxidant capacity between different studies due to variations caused by different extraction methods. Nonetheless, for comparison the content ABTS and FRAP assay in our study was different from that reported by Hssaini et al. (2020) who studied figs of 11 different varieties and determined values in a ranged 1.75 to 8.04 (mmol TE/g dm) and between 1.09 and 10.65 (mmol TE/g dm) for ABTS and FRAP, respectively. Other researchers have evaluated the antioxidant capacity in the leaves of various fruit species (Pompeu et al., 2021) and reported that the mean oxygen radical absorbance capacity (ORAC) measured in the leaves of *Ficus carica* was 1214.3 (Expressed as μM TE /g dm).

Pearson's coefficient (Supp. 1) showed positive correlations in leaves between ABTS with VAL, L-SOP, neochlorogenic acid, chlorogenic acid, caffeic acid and apigenin-*C*-hexoside-pentoside (r = 0.91, 0.93, 0.91, 0.91, 0.96, 0.89 respectively; $p < 0.05$). Leaves also presented positive correlation between FRAP with VAL, L-SOP, neochlorogenic acid and caffeic acid (r = 0.93, 0.95, 0.84, 0.91 respectively; $p < 0.05$). Regarding ORAC showed positive correlations with neochlorogenic acid, chlorogenic acid and ferulic acid (r = 0.93, 0.88, 0.91 respectively; $p < 0.05$). In figs Pearson's coefficient showed positive correlations for ABTS with cyanidin-3,5-*O*-diglucoside (r = 0.92 $p < 0.05$) and pelargonidin-3-*O*-rutinoside (r = 0.92 $p < 0.05$). FRAP also showed positive

Table 5
Antioxidant activity of leaves and fruits of figs of studied cultivars (mmol Trolox/100 g dm) and antidiabetic activity (mg /mL dm).

Variety	Antioxidant activity			Antidiabetic activity	
	ABTS	FRAP	ORAC	α-amylase	α-glucosidase
			Leaves		
SA	2.72 ± 0.19 b	1.29 ± 0.08 d	10.03 ± 0.58 c	45.55 ± 11.68 a	3.18 ± 0.88 a
CA	2.81 ± 0.23 b	0.75 ± 0.03 e	16.15 ± 0.93 b	45.42 ± 11.65 a	3.17 ± 0.88 a
CUMH	3.66 ± 0.15 b	2.23 ± 0.02 c	10.24 ± 0.59 c	45.43 ± 11.65 a	3.17 ± 0.88 a
CDN	5.87 ± 0.39 a	4.84 ± 0.06 a	21.16 ± 1.22 a	46.02 ± 11.80 a	3.21 ± 0.89 a
SF	4.94 ± 0.14 a	3.28 ± 0.02 b	19.78 ± 1.14 ab	46.98 ± 12.05 a	3.28 ± 0.91 a
			Figs		
SA	0.64 ± 0.01 b	0.69 ± 0.00 ab	4.59 ± 0.26 ab	69.32 ± 14.11 a	43.99 ± 14.42 a
CA	0.60 ± 0.02 b	0.60 ± 0.07 bc	4.88 ± 0.28 a	68.41 ± 13.92 a	43.41 ± 14.23 a
CUMH	0.43 ± 0.06 c	0.45 ± 0.05 bc	4.66 ± 0.27 ab	67.25 ± 13.69 a	85.36 ± 27.98 a
CDN	0.97 ± 0.05 a	0.97 ± 0.12 a	3.57 ± 0.21 b	46.65 ± 11.96 a	86.43 ± 32.67 a
SF	0.37 ± 0.02 c	0.33 ± 0.04 c	3.55 ± 0.21 b	71.80 ± 14.61 a	86.99 ± 32.88 a
			Tukey's Multiple Range Test for mean values		
Leaves	4.00 a	2.48 a	15.47 a	45.88 b	3.20 b
Figs	0.60 b	0.61 b	4.25 b	64.69 a	69.24 a

Mean ± standard deviation; value in the same columns followed by different letters are significantly different at $p \leq 0.05$ according to Tukey's test. Legend_ SA: San Antonio; CA: Colar Albatera; CUMH: Colar UMH; CDN: Cuello Dama Negro; SF: Superfig.

correlation with cyanidin-3,5-*O*-diglucoside ($r = 0.91$ $p < 0.05$) and pelargonidin-3-*O*-rutinoside ($r = 0.91$ $p < 0.05$) the same than ABTS.

In fig fruits, antioxidant decreases in the order: CA > SA > CUMH > CDN > SF. Additionally, CDN variety showed the strongest antioxidant potential measured by ORAC (21.16 mmol Trolox/100 g dm).

Therefore, as can be seen in Table 5, the antioxidant activity in leaves (2.48–15.47 mmol Trolox/100 g dm) is higher than in figs. (0.60–4.25 mmol Trolox/100 g dm). The inhibition of the enzymes pancreatic α-amylase, intestinal α-glucosidase, and pancreatic lipase by leaves and fruits of figs was assessed and reported in Table 5. No significant differences were observed between the varieties studied for activity against pancreatic α-amylase and activity against, intestinal α-glucosidase. The highest potential towards pancreatic α-amylase ($IC_{50} = 64.69$ mg /mL dm) and intestinal α-glucosidase ($IC_{50} = 69.24$ mg /mL dm) was presented by fruits of figs. All analyzed sample were less active than acarbose activity $IC_{50} = 0.1$ and 2.4 mg /mL, for α-amylase and α-glucosidase, respectively. Similar results presented Turkiewicz et al. (2022) for *Chaenomeles* leaves.

Pearson's coefficient in leaves showed that pancreatic α-amylase presented positive correlation with ARG ($r = 0.96$, $p < 0.05$) and PRO ($r = 0.92$, $p < 0.05$). Intestinal α-glucosidase presented a weak negative correlation with TYR, δ- TF, α- TF, 3,

apigenin-*C*-hexoside-pentoside, quercetin-3-*O*-glucoside, quercetin-3-*O*-(malonyl)-glucoside and keampferol-3-*O*-rutinoside ($r = -0.90$, -0.88, -0.88, -0.89, -0.91, -0.93, -0.89, -0.95 respectively; $p < 0.05$). Pearson's coefficient in figs showed positive correlations between the pancreatic α-amylase and β-carotene ($r = 0.89$ $p < 0.05$) and between intestinal α-glucosidase and Violaxanthin ($r = 0.89$ $p < 0.05$). The capacity inhibition of enzymes α-amylase and α-glucosidase has positive impacts on the health of people with type 2 diabetes mellitus and obesity prevention and weight management (Wojdyło et al., 2021). Additionally, previous studies (El Enshasy et al., 2023) have reported that amylase is one of the most significant enzymes involved in regulating fruit development, growth, and ripening. During the ripening process, enzymes facilitate cell wall hydrolysis, particularly of polysaccharides. The activity of α-amylase increases during ripening and subsequently decreases during senescence, playing a key role in the conversion of starch to sugars. Meziant et al. (2021) determined α-glucosidase in extracts from peels from *F. carica* (*Bakor Noir, Bouankik, Azenjer,* and *Tazegaght*) local fig varieties (Bakor of Bejaia (north Algeria). The results show the moderate potential of extracts from the peel of *Ficus carica* to inhibit α-glucosidase and concluded anthocyanins present in the extracts could be primarily responsible for the inhibitory effects against α-glucosidase (Meziant et al., 2021). Wojdyło et al. (2016) also studied the potential health-promoting constituents of 10 different varieties of fig and determined moderate the *invitro* hypoglycaemic potential *via* inhibition of α-glucosidase (IC_{50} values ranging from 15.4 to 22.9 mg/mL).

4. Conclusions

In this study, the phytochemical profiling of leaves and fruits of *F. carica* were evaluated. The results of this work show that leaves of *F. carica* have strong antioxidant capacity due to high contents of carotenoids, chlorophyll compounds, tocotrienols, tocopherols, and polyphenols. Whereas figs are a better source of amino acids than leaves. In addition, figs exhibited moderate antidiabetic activity (α-amylase and α-glucosidase), which was higher than that of the leaves, although no significant differences were observed between the varieties. Regarding the differences in chemical composition between the various varieties CDN of both leaves and figs appear to have notable antioxidant properties and polyphenol content. While figs, especially the SA variety, showed a higher total amino acid content than leaves. The research findings show that leaves and fruits of *F. carica* could be an ingredient for functional foods. It is consumption could be beneficial for health, reducing the risk of suffering from diseases related to oxidative stress.

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CRediT authorship contribution statement

Candela Teruel-Andreu: Writing – review & editing, Writing – original draft, Visualization, Formal analysis, Data curation. **Marina Cano-Lamadrid:** Writing – review & editing, Writing – original draft, Visualization, Validation, Resources. **Francisca Hernández:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Funding acquisition, Conceptualization. **Aneta Wojdyło:** Writing – review & editing, Writing – original draft, Supervision, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2024.141977.

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07
RESULTS AND DISCUSSION

This section includes the main results and discussions from the published articles, organized into three blocks based on specific objectives. Comprehensive details are available in the previous section.

7.1 Know the applications of figs, leaves and other by-products in the food and pharmaceutical industry (Objective 1).

The results related to this objective are detailed in the first publication, which included:

- 1 st Publication: The analysis of 41 studies on the compounds in *Ficus carica* fruits and leaves, identified fig-based products and examined their total phenols and antioxidant capacity. Title: *Ficus carica* Fruits, By-Products and Based Products as Potential Sources of Bioactive Compounds: A Review.

Bioactive compounds in different fig parts

Identified compounds belong to different chemical families, such as phenolic acids (caffeic acid, chlorogenic acid, ferulic acid, coumaric acid, syringic acid, quinol and gallic acid) and flavonoids (catechin, kaempferol and quercetin). Quercetin-3-*O*-rutioside was reported as the major individual phenolic compound in whole figs, followed by polymeric procyanidins, quercentin-3-glucoside, chlorogenic acid and cyanidin-3-*O*-rutinoside. As for the bioactive compounds of the peel, cyanidin-3-rutinoside was the most abundant, followed by cyanidin-3,5-diglucoside, cyanidin-3-*O*-diglucoside, epicatechin, catechin and quercetin-rutinoside. Epicatechin and cyanidin-3-rutinoside were the main compounds found in fig pulp, while kaempferol 3-*O*-glucoside, was the main compound reported in fig leaves.

Bioactive content of fig-based products and their antioxidant activity

Figs are primarily consumed fresh or dried and are also processed into products like jams (Slavin, 2006), with an increasing range of fig-based products now available. The bioactive compound content and antioxidant activity of figs vary by cultivar and processing method (De Pilli et al., 2019). Studies indicate that fig jam processing

significantly reduces total phenolics (by 68.6%) and anthocyanin content (by 60.2%). Similarly, drying processes decrease phenolic content; for instance, Vallejo et al. (2012) reported a 15% reduction in total phenolics during the drying of Cuello Dama figs.

Recent research highlights the potential of fig peel and pulp by-products as food ingredients. For example, fig powder has been used as a colorant in buns and muffins (Chauhan & Tanwar, 2016). Fig seed powder added to cookie formulations improved fiber content, total phenolic content, and antioxidant activity (Bölek, 2021). Sweet extracts derived from fig by-products are also utilized in traditional desserts, such as *Shir Anjir*, an Iranian dessert made from whole cow milk and dried figs. This dessert plays a significant role in the valorization of underutilized agricultural figs. Furthermore, *Shir Anjir* presents noteworthy health benefits, serving as a source of soluble fiber, essential vitamins, amino acids, antioxidants, and natural sweeteners (Jahromi & Niakousari, 2018). Studies (Cano-Lamadrid et al., 2017) incorporating Mediterranean crop purées with pomegranate juice have reported that fig-based products enrich the antioxidant profile of smoothies while promoting the consumption of underutilized fruits. Fig-pomegranate smoothies exhibit notably higher anthocyanin content, thereby boosting their antioxidant potential and making them a viable alternative to fresh fruits. However, storage may lead to a reduction in total phenolic content (TPC). While several fig-based products have already been explored, including smoothies, powders, colorants, and biscuits, there remains significant potential for innovation. The development of novel products, such as fig coffee, dried figs processed using advanced techniques, and fermented milk incorporating fig by-products, has the potential to enhance the sustainability and economic viability of fig trees cultivation. Such efforts would not only diversify product offerings but also enhance the comprehensive understanding of the health-promoting properties associated with figs.

Conclusions of the 1 st objective

Despite growing interest in the bioactive compounds found in figs and their by-products, further standardized research is necessary to validate their health benefits.

Future investigations should emphasize both in vitro and in vivo studies to better understand their nutritional, functional, and techno-functional properties.

Additionally, the valorization of fig processing waste, such as leaves, peel, and pulp, holds promise for generating economic value, addressing environmental concerns, and improving consumer health.

7.2 Determine morphological, functional, and nutritional characterization of breba and fig fruits and by-products (Objective 2).

The results related to this objective are detailed in four publications, which included the following parameters:

- 2 nd Publication: Leaf characterization, sugar profile, crude fiber content, mineral content, antioxidant capacity and total phenolic content in leaves of fig trees. Title: *How Does Cultivar Affect Sugar Profile, Crude Fiber, Macro and Micronutrients, Total Phenolic Content, and Antioxidant Activity on Ficus carica Leaves?*
- 3 rd Publication: Morphological characteristics, sugar profile, crude fiber content, mineral content, antioxidant capacity and total phenolic content in fruits of fig trees. Title: *Nutritional and functional compounds and antioxidant activity of edible and non-edible fruit part of brebas and figs (Ficus carica L.) among different varieties*.
- 4 th Publication: Volatile compounds profile in fruits of fig trees. Title: *Volatile profile of breba and fig fruits (peel and pulp) from different Ficus carica L. varieties.*
- *7 th Publication: Carotenoids, Chlorophylls, tocols, amino acids, Polyphenolic compounds and biological activity* in leaves and fruits of fig trees*. Title: Bioactive compounds (LC-PDA-Qtof-ESI-MS and UPLC-PDA-FL) and in vitro inhibit α-amylase and α-glucosidase in leaves and fruit from different varieties of Ficus carica L.*

Leaf characterization

The CA cultivar showed the largest leaf dimensions, with lengths, widths, central lobe lengths, and petiole lengths of 27.46, 21.78, 14.55, and 10.54 cm, respectively. Other cultivars ranged from 19.65–22.71 cm (leaf length), 17.38–21.04 cm (leaf width), 10.16–12.64 cm (central lobe length), and 8.16–8.98 cm (petiole length). These differences may be due to the different genotypes and to environmental factors. Additionally, these results align with previous studies (Abdelsalam et al., 2019; Almajali et al., 2012; Khadivi et al., 2018) indicating leaf size varies by cultivar and location.

Sugar profile and crude fiber content in leaves

Fructose was the predominant sugar, exceeding glucose and sucrose levels by 1.10-fold and 1.84-fold, respectively (based on the average across cultivars). Although previous studies identified sucrose as the main sugar in *F. carica* leaves (Vemmos et al., 2013), significant differences were observed among cultivars, with the CA cultivar having the highest sugar total content (159.38 g/kg dw). The variations among the Colar cultivar located in different plots may be attributed to environmental factors such as soil type, irrigation water, or fertilization practices. Crude fiber content ranged from 6.53% in CUMH to 22.67% in CA. Consequently, *F. carica* leaves represent a valuable dietary fiber source, particularly for applications in food products designed to achieve "high in fiber" labeling claims (≥6 g per 100 g) (Regulation, 2006). Further research is warranted to analyze the soluble and insoluble dietary fiber fractions and fully elucidate their nutritional potential.

Mineral content in leaves

The mineral composition of *Ficus carica* leaves, revealing significant differences ($p < 0.05$) in macro- and micromineral concentrations across cultivars. The SA cultivar exhibited the highest macro- and micromineral contents, with the exception of manganese (Mn), which was most abundant in the CA cultivar. Calcium (Ca) was the predominant mineral, ranging from 19.97 g/kg dw (CUMH) to 68.04 g/kg dw (SA), followed by potassium (K), which ranged from 13.87 to 18.63 g/kg dw. These findings corroborate previous studies identifying Ca as the dominant mineral in *F.carica* leaves (El Dessouky Abdel-Aziz et al., 2020; Ghazi et al., 2012). The high calcium content in *F.carica* leaves positions them as a potential dietary calcium source, meeting the Codex Alimentarius, Guidelines (FAO, 2001) for Use of Nutrition Claims states that solid foods must contain a calcium content of 15% of the nutrient reference value (NRV) of 800 mg of calcium to be labelled as a source of calcium.

Antioxidant capacity and total phenolic content in leaves

The ABTS assay, revealing that cultivar SF exhibited the highest antioxidant capacity (52.43 mM Trolox dw). This was followed by cultivar CDN (52.07 mM Trolox dw), SA (44.91 mM Trolox dw), CUMH (42.46 mM Trolox dw), and CA (33.81 mM Trolox dw). Similarly, the DPPH assay showed that cultivar SF had the highest activity (72.45 mM Trolox dw), with cultivars CUMH (70.14 mM Trolox dw), CA

(68.84 mM Trolox dw), CDN (59.27 mM Trolox dw), and SA (52.54 mM Trolox dw) following.

In terms of FRAP, the cultivars ranked from highest to lowest as follows: CDN (124.79 mM Trolox dw), SF (115.66 mM Trolox dw), CUMH (67.15 mM Trolox dw), CA (60.70 mM Trolox dw), and SA (56.09 mM Trolox dw). For total phenolic content (TPC), the highest value was recorded for cultivar CUMH (18.86 g GAE/ kg dw).

There are few scientific manuscripts addressing the antioxidant activity and total phenolic content in *F. carica* leaves, specifically using the same solvent, which makes comparisons difficult. Furthermore, previous studies on *F. carica* leaves have reported that total phenolic compound content is influenced by variety (Mahmoudi et al., 2016) and extraction solvent (Ghazi et al., 2012).

Morphological characteristics of brebas and figs

SF Breba fruits exhibited the greatest weight, followed by CUMH. The findings of this study regarding Breba fruit weight aligned with those reported in earlier research (Núñez-Gómez et al., 2021). A similar pattern was observed for fig fruits, with weight ranking as SF > CUMH > CA. Breba fruits were 1.5–2.2 times heavier than figs across all varieties. Regarding morphologically, breba fruits were more elongated (length/width ratio: 1.3–1.8) compared to the rounder figs (1.1–1.4). Additionally, the pulp percentage ranged from 84.5–96.2% in Breba fruits and 88.7–93.8% in figs, with 4–15% of the fruit classified as by-products. According Ayuso et al. (2022) fig peels, accounting for approximately 27% of total weight, are a source of bioactive compounds.

The color of fig peel is a key characteristic that varies significantly among different cultivars. In this study, the Breba figs of the SF variety, which exhibit a purple-greenish peel, recorded the highest values for L*(34.83), a*(10.40), b*(10.00), and C*(15.23) CIELAB color coordinates among the varieties analyzed. Conversely, the figs of the CDN variety, characterized by a dark purple peel, showed the lowest values for the a*(3.48), b*(-0.15), and C*(3.7) coordinates. Additionally, significant differences were found for all color coordinates between CA and CUMH, with CUMH obtaining the highest values, these differences in fruit color within the same variety but grown in different locations may be influenced by various agronomic factors. One such factor is tree pruning, which can increase light exposure can enhance pigment synthesis, potentially leading to variations in peel coloration.

Sugar profile and crude fiber content in brebas and figs

The organic acids identified in the peel and pulp of brebas and figs were citric acid and malic acid, with malic acid being the predominant acid across all analyzed samples, consistent with previous findings (Núñez-Gómez et al., 2021). Significant differences in citric acid content were found in the pulp among varieties of brebas and figs. For brebas, the highest citric acid content was observed in the CA variety (18.48 g/kg dw), while for figs, the highest content was found in the CDN variety (12.43 g/kg dw).However, for malic acid, significant differences were only detected in fig pulp, with the CDN variety exhibiting the highest value (40.90 g/kg dw). Additionally, in figs, the malic acid content in the pulp of the CA and CDN varieties was 1.12 and 1.64 times higher, respectively, than in breba pulp.

Glucose and fructose were present throughout all parts of the examined fruits, aligning with previous research that identified these as the primary sugars in whole fruits from various Spanish fig varieties (Wojdyło et al., 2016). In breba, significant differences in glucose and fructose levels were only observed in the pulp, with the lowest values recorded in the SA variety 4.74 g/kg dw for glucose and 12.82 g/kg dw for fructose. Furthermore, figs demonstrated significant varietal differences in sucrose, glucose, and fructose levels across both the peel and pulp. The CA variety exhibited the highest sucrose content, measuring 32.84 g/kg dw in the pulp and 26.17 g/kg dw in the peel. The highest overall sugar concentration was found in the pulp of the CA variety, reaching 776.14 g/kg dw. Generally, fig pulp contained greater amounts of glucose and fructose compared to the peel.

The breba peel exhibited a total crude fiber content that was 1.21 times greater than that of the pulp, with statistical analysis also confirming that variety had a significant impact on this difference. The highest crude fiber content in breba pulp was found in the CA variety (11.57%), while the CUMH variety had the highest fiber content in breba peel (13.77%). For figs, the highest crude fiber content in the pulp was observed in the CA variety (8.93%), and in the peel, the highest value was found in the CDN variety (10.45%). Notably, breba peel contained significantly more fiber than fig peel.

Mineral content in brebas and figs

Potassium was the predominant macroelement in the analyzed samples, with the highest concentration found in the SA breba pulp (16.52 g/kg dw). Therefore, both brebas and figs are considered potassium-rich foods, as their potassium content

exceeds the minimum threshold (600 mg/100 g) established in Regulation (EU) No. 1169/2011 of the European Parliament and of the Council (Regulation, 2011).

The ranking of microelement concentrations in breba pulp, based on the average values across all varieties, followed this order: Zn (29.78 mg/kg dw) > Fe (18.48 mg/kg dw) > Cu (8.13 mg/kg dw) > Mn (5.07 mg/kg dw). In breba peel, the sequence was: Fe (41.98 mg/kg dw) > Zn (12.18 mg/kg dw) > Mn (6.84 mg/kg dw) > Cu (5.98 mg/kg dw).

For figs, similar trends were observed for macroelements, with potassium being the predominant macroelement. In fig pulp, the highest potassium concentration was found in the SA variety (8.54 g/kg dw). The trend for microelements in figs was consistent in both peel and pulp (mean values for all varieties): Fe (16.24–14.47 mg/kg dw) > Zn (12.55–8.93 mg/kg dw) > Cu (5.73–4.09 mg/kg dw) > Mn (3.38–2.69 mg/kg dw).

The values obtained in breba were higher than those recorded in fig. The SA and CA breba pulp exhibited 2.29- and 2.88-fold higher calcium (Ca), 9.17- and 7.20-fold higher sodium (Na), 3.24- and 4.52-fold higher copper (Cu), and 6.84- and 5.17-fold higher zinc (Zn) concentrations, respectively, compared to fig pulp. Similarly, SA breba peel demonstrated significantly higher concentrations of all macro- and microelements compared to fig peel, with Ca (2.54-fold), potassium (K; 3.07-fold), magnesium (Mg; 2.91-fold), Na (2.67-fold), Cu (1.93-fold), iron (Fe; 2.94-fold), manganese (Mn; 2.87-fold), and Zn (1.76-fold).

Antioxidant capacity and total phenolic content in brebas and figs

Firstly, the results for brebas will be discussed. The CDN variety exhibited the highest values for ABTS (93.00 mM Trolox dw), DPPH (151.16 mM Trolox dw), FRAP (195.74 mM Trolox dw), and TPC (18.09 g GAE/kg dw) in breba peel, whereas the SA variety showed the lowest values for ABTS (22.65 mM Trolox dw), DPPH (71.80 mM Trolox dw), FRAP (34.67 mM Trolox dw), and TPC (13.83 g GAE/kg dw).

In comparing breba peel and pulp, it was found that the ABTS, DPPH, FRAP, and TPC values in the peel were significantly elevated, ranging between 3- and 13-fold, 1- and 2.5-fold, 3- and 21-fold, and 2.6- and 3-fold higher, respectively, than in the pulp.

For fig pulp, the highest ABTS values were found in CA (6.56 mM Trolox dw), CUMH (5.29 mM Trolox dw), and CDN (6.54 mM Trolox dw). No significant differences were observed for DPPH values. The highest FRAP value was obtained in the SA

variety (12.60 mM Trolox dw). In terms of TPC, the highest values were observed in CA (3.53 g GAE/kg dw) and CDN (3.68 g GAE/kg dw), while the SF variety exhibited the lowest value (1.84 g GAE/kg dw).

Regarding fig peel, the highest values for ABTS, DPPH, FRAP, and TPC were observed in CDN (43.66 mM Trolox dw), SF (51.41 mM Trolox dw), CA (54.48 mM Trolox dw), and SA (15.05 g GAE/kg dw), respectively. Conversely, the lowest values were found in SF (16.48 mM Trolox dw) for ABTS, CDN (24.80 mM Trolox dw) for DPPH, SF (26.47 mM Trolox dw) for FRAP, and SF (8.09 g GAE/kg dw) for TPC.

In addition, for fig fruits, the ABTS, DPPH, FRAP, and TPC values in fig peel were 4–7 times, 0.5–1 times, 4–9 times, and 3–5.6 times higher than those in fig pulp, respectively. These findings align with previous research, as several authors have reported greater antioxidant activity in fig peels compared to the pulp (Del Caro & Piga, 2008; Harzallah et al., 2016). Additionally, varietal differences have been shown to influence antioxidant activity (Harzallah et al., 2016; Hssaini et al., 2021).

A comparative analysis of the results obtained for brebas and figs indicates that the pulp of SF brebas exhibited ABTS, FRAP, and TPC values that were 3-fold, 5.4-fold, and 2.9-fold higher, respectively, than those observed in SF fig pulp. Furthermore, the peel of CDN brebas demonstrated 6.1-fold and 4.3-fold higher DPPH and FRAP values, respectively, compared to CDN fig peel. Overall, the measured values in breba fruits were consistently higher than those recorded in figs.

Volatile compounds

Volatile compounds in *Ficus carica* L. were analyzed via HS-SPME combined with GC, identifying 35 compounds. The volatile compounds detected in the pulp of the analyzed fruits were categorized as aldehydes (*n* = 13), alcohols (2), alkanes (1), terpenes (4), terpenoids (1) and ketones (3). In contrast, those found in the peel were classified as aldehydes (13), esters (4), alcohols (3), terpenes (2), terpenoids (2) and ketones (1). This distribution aligns with findings reported by other researchers (Lachtar et al., 2022; Yao et al., 2021; Zidi et al., 2021).

For breba pulp aldehydes were the predominant group, with hexanal exhibited the highest percentage in the CA variety (57.86%) and benzaldehyde was predominant in the CUMH (43.45%) and SF (34.38%) varieties. Hexanal and benzaldehyde were the most abundant detected compounds overall. In figs pulp, hexanal showed the highest percentage hexanal with the CA variety reaching 64.69%. In breba peel, 2-

hexenal and benzaldehyde were dominant. Similarly, aldehydes were the most prominent chemical family in fig peel, with benzaldehyde showing the highest percentage in the SF variety (65.63%).

Significant differences were observed in 15 out of 22 volatile compounds when comparing the pulp of brebas and figs. Overall, figs exhibited higher concentrations of these compounds, with the CDN fig presenting the highest levels in six specific volatiles: 2-hexenal (7.35%), 2-decenal (0.78%), 3-phenyl 2-propenal (2.91%), 2,2,4,6,6-pentamethylheptane (24.19%), copaene (3.07%), and caryophyllene (3.48%). In contrast, breba CA showed the highest concentration of hexanal, reaching 64.69%.

Among the fig varieties analyzed, CA figs displayed the highest concentrations of most volatile compounds in the peel, including nonanal, benzenepropanal, decanal, methyl hexanoate, 1-octen-3-ol, 1-tetradecanol, aromadendrene, and pinocarveol. Following this, breba CA exhibited the greatest levels of hexanal, 2,4-heptadienal, octanal, 2-octenal, 2,6-nonadienal, methyl hexadecanoate, and β-ionone. Meanwhile, fig CUMH recorded the highest concentrations of undecanal, methyl octanoate, and linalool.

These results highlight that the peel of figs and brebas contains a higher concentration of volatile compounds compared to the pulp. Additionally, the quantity of volatile compounds is highly dependent on the variety, with the CA variety being the most aromatic. The primary chemical family contributing to the aroma of figs and brebas consists of aldehydes. Additionally, the aroma of brebas and figs is characterized by various aromatic descriptors, including, fruity, green, sweet, floral and fatty aroma. To date, no previous studies have directly compared volatile compounds between brebas and figs, making it challenging to contextualize these findings. Future studies should include sensory analyses to complement these chemical profiles and enhance understanding.

Carotenoids

The UPLC-PDA-Qtof-ESI-MS analysis identified seven carotenoids in the leaves and fruits of *F. carica*. The carotenoid content was significantly higher in leaves (6763.77–3170.77 mg/100 g dm) compared to fruits (60.62–87.00 mg/100 g dm). The highest total content carotenoids in leaves observed in the SF variety. β-Carotene and lutein were the predominant carotenoids, with concentrations in leaves exceeding those in fruits by 69–103 times. Previous studies (Kamiloglu &

Akgun, 2023) highlights variations in carotenoid composition depending on fig variety. Australian figs predominantly feature lycopene at 0.32 mg per 100 g. In contrast, lutein is the principal carotenoid in Turkish yellow figs and Israeli green-purple figs, with concentrations ranging from 6.14–7.15 µg/g dw and 4.08–12.58 µg/g fw, respectively. Algerian white figs primarily contain β-carotene at 4.32 µg per 100 g dw.

Chlorophylls

The UPLC-PDA-FL analysis of methanolic leaf extracts identified six chlorophylls, with total chlorophyll content showing significant variation across varieties, ranging from 405.58 mg/100 g dm in CA to 744.23 mg/100 g dm in SF. Chlorophyll *b* (CH *b*) and chlorophyll *a* (CH *a*) were the predominant compounds, with the SF variety exhibiting the highest concentrations (CH *b*: 111.67 mg/100 g dm; CH *a*: 505.86 mg/100 g dm). The chlorophyll *a*/*b* ratio also varied significantly, from 5.23 in SA to 3.92 in CUMH. According to Gholami et al. (2012), severe drought significantly reduced chlorophyll levels in fig trees (Siah and Shah varieties), while the chlorophyll *a*/*b* ratio decreased in Deym and Sabz varieties, suggesting that this could be attributed to the absence of a reduction in peripheral light-harvesting complexes and greater stress tolerance in the two varieties. Additionally, previous studies Ben Amor et al. (2021) reported substantial reductions in chlorophyll content under pollution, with decreases of up to 60% in *Ficus carica*.

Tocols

Leaves exhibited significantly higher tocols (tocotrienols, TT; and tocopherols, TF) concentrations (mean value 87.03 mg/100 g dm) compared to figs (mean value average 7.68 mg/100 g dm). In leaves, SF variety displaying the highest ∑TT&TF content (115.61 mg/100 g dm). The main TT detected was δ-TT, while the main TF detected was α-TF, with SF showing the highest concentrations (30.43 and 65.54 mg/100 g dm, respectively). In figs, the highest TF levels were observed in the CUMH variety (1.75 mg/100 g dm) and the content of TF in figs decreases in the following order: α-TF > γ-TF > δ-TF > β-TF. Tocols were markedly more abundant in leaves than in figs, with ∑TF and β-TF being 39- and 130-fold higher, respectively. The tocopherol levels found in figs were comparable to values previously reported by other researchers (Pande & Akoh, 2010), with α-TF and γ-TF concentrations measuring 0.2 and 0.3 mg per 100 g of dry matter. Additionally, studies by Konyalıoğlu et al. (2005) suggest that fig leaves may serve as a promising new source of α-TF.

Amino acids

A total of twenty-one amino acids were detected, categorized as follows: eight classifieds as indispensable, six as conditionally indispensable, and seven as dispensable. Among leaves, methionine (MET) and isoleucine (ILE) were the most prevalent indispensable amino acids, with the highest concentrations found in the CA variety (30.18 and 9.57 mg/100 g dry matter. Moreover, glutamine (GLU) and proline (PRO) stood out as the most abundant conditional amino acids in leaves, with concentrations varying between 4.37–21.61 mg/100 g dm and 6.00–28.94 mg/100 g dm, respectively. Among dispensable amino acids, alanine (ALA) was detected at the highest levels, particularly in the SF variety, where it reached 43.02 mg/100 g dm.

In figs, the SA variety exhibited the highest total amino acid content (1814.03 mg/100 g dm). The ranking of indispensable amino acids was threonine (THR) > valine (VAL) > isoleucine (ILE) > leucine (LEU) > phenylalanine (PHE) > methionine (MET) > tryptophan (TRP) > lysine (LYS), while conditionally indispensable amino acids ranged from 199.36 to 354.19 mg/100 g dm, with CA showing the highest levels. Asparagine (ASN) and phospho-L-serine (LSOP) were the most prevalent dispensable amino acids in figs, with SA displaying the highest values (550.18 and 503.08 mg/100 g dm). The fraction dispensable amino acids fraction predominates in the analyzed figs and leaves.

The amino acid content in figs was generally greater than that found in leaves, with certain dispensable amino acids, such as LSOP, exhibiting values that were 253 times higher in figs than in leaves. Similarly, previous findings by Wojdyło et al. (2021), indicated that other fruits, including apples, pears, and quinces, also contained significantly higher amino acid concentrations compared to their respective leaves.

Polyphenolic compounds

The polyphenolic composition of leaves and figs was elucidated, revealing eight phenolic acids, five flavonols, and two anthocyanins. Leaves from the SF (1933.56 mg/100 g dm) and CDN (1828.98 mg/100 g dm) varieties exhibited the highest total polyphenol content. Caffeic acid (345.77 mg/100 g dm) and ferulic acid (194.10 mg/100 g dm) were the predominant phenolic acids across all leaf samples, while apigenin-C-hexoside-pentoside (294.50 mg/100 g dm) emerged as the most abundant flavonol.

In figs two phenolic acid compounds were detected neochlorogenic acid (2.91 mg/100 g dm) and chlorogenic acid (4.15 mg/100 g dm). Additionally, two anthocyanins were detected: cyanidin-3,5-*O*-diglucoside (50.56 mg/100 g dm) and pelargonidin-3-*O*-rutinoside (9.50 mg/100 g dm). Notably, CDN figs exhibited the highest total anthocyanin content (108.22 mg/100 g dm) and total polyphenol content (TPC) (199.03 mg/100 g dm) among the varieties examined.

Phenolic acids were the predominant group of polyphenols identified in the leaves, followed by flavonols and polymeric procyanidins (PP). Significant differences between varieties were observed for PP, phenolic acids, and flavonols contents. CA leaves exhibited the highest PP content (0.81 g/kg), while CDN and SF leaves showed the highest levels of phenolic acids (11.29 g/kg and 11.20 g/kg, respectively) and flavonols (6.50 g/kg and 7.60 g/kg, respectively). The SF variety displayed the highest TPC in leaves, reaching 19.34 g/kg. In figs, the greatest TPC was primarily linked to the PP and anthocyanin groups. Among the varieties, SA exhibited the highest TPC for PP at 1.20 g/kg and flavonols at 0.13 g/kg. Regarding anthocyanin content, the varieties ranked in descending order as follows: CDN > CA > SA > CUMH > SF.

Biological activity

Antioxidant properties were assessed using ABTS, FRAP, and ORAC assays. The mean values of antioxidant activity for ABTS, FRAP, and ORAC were higher in leaves (4.00, 2.48, and 15.47 mmol Trolox/100 g dm) than in figs (0.60, 0.61, and 4.25 mmol Trolox/100 g dm). CDN leaves exhibited the highest antioxidant activity, as measured by ABTS (5.87 mmol Trolox/100 g dm), FRAP (4.84 mmol Trolox/100 g dm), and ORAC (21.16 mmol Trolox/100 g dm). In fig fruits, the highest antioxidant activity, as determined by the sum by the three methods (sum of ABTS, FRAP, and ORAC methods) follows the order: CA > SA > CUMH > CDN > SF. It is well established that variety influences antioxidant capacity, which depends on the content of bioactive components such as polyphenols, isoprenoids, vitamins, and other plant-derived molecules Wojdyło et al. (2021). Comparing antioxidant capacities across studies is challenging due to variations in extraction methods.

The evaluation of α-amylase and α-glucosidase inhibitory activity revealed no significant differences among the studied varieties. However, a significant difference was observed between the results obtained from leaves and fruits, with the fruits exhibiting the highest inhibition potential for both enzymes. These findings highlight the moderate inhibitory potential of figs. Meziant et al. (2021) analyzed

α-glucosidase inhibitory activity in extracts from peels of *Ficus carica* local fig varieties (varieties Bakor Noir, Bouankik, Azenjer, and Tazegaght) from northern Algeria (Bakor of Bejaia). Their findings revealed moderate inhibitory potential, attributed primarily to anthocyanins present in the extracts. Similarly, Wojdyło et al. (2016) investigated 10 fig varieties and reported moderate in vitro hypoglycemic potential via α-glucosidase inhibition, with IC_{50} values ranging from 15.4 to 22.9 mg/mL.

Conclusions of the 2 nd objective

- Fig leaves are a rich source of calcium, making them a potential dietary supplement.
- The CA leaves was linked to all sugar-related variables, including sucrose, glucose, and fructose.
- Differences in sugar content of leaves among CA and CUMH may be influenced by environmental factors such as soil type, irrigation, and fertilization.
- The SA leaves were associated with all mineral content variables.
- SF Breba fruits exhibited the highest weight among the studied varieties.
- Brebas and figs qualify as a valuable source of potassium.
- Antioxidant activity was mostly linked to the peel rather than the pulp in both brebas and figs.
-Breba fruits generally exhibited higher antioxidant activity, crude fiber, and mineral content than figs.
-Peels have higher antioxidant activity, fiber, and mineral content, while pulps contain more sugars.
- Peels generally have a richer volatile compound than pulps, emphasizing the importance of considering whole-fruit consumption.
- The CA variety was the most aromatic.
- The aroma of brebas and figs is mainly derived from aldehydes family compounds.
- Hexanal, 2-hexenal, and benzaldehyde were the most prominent volatile compounds in peel of brebas and figs.
- Leaves had significantly higher carotenoid content than fruits, with SF showing the highest levels.

- The SF variety leaves exhibited the highest chlorophyll levels

- Leaves had much higher tocols (tocotrienol and tocopherol) content than figs, with SF having the highest values.

- Figs contained higher amino acid levels than leaves, with the SA variety showing the highest total amino acid content.

- The leaves of *F. carica* have a high antioxidant capacity due to their rich content of carotenoids, chlorophyll, tocotrienols, tocopherols, and polyphenols.

- The SF and CDN varieties leaves had the highest total polyphenols content in leaves.

- The SA and CDN varieties had the highest total polyphenols content in figs.

-Leaves were rich in phenolic acids and flavonols, particularly caffeic acid and apigenin derivatives, while figs contained anthocyanins such as cyanidin-3,5-*O*-diglucoside.

- Figs extracts demonstrated moderate potential to inhibit α-glucosidase.

7.3 Development and optimization of fermented milk based on fig (Objective 3).

The results related to this objective are detailed in two publications, which included the following parameters:

- 5 th Publication: Microbial load and techno-functional properties analysis, Volatile compounds, Sensory analysis. Title: *Techno-functional properties and enhanced consumer acceptance of whipped fermented milk with Ficus carica L. By-products.*

- 6 th Publication: Analysis of microbial load and techno-functional properties after 30 days of storage, flavonoid compounds, total polyphenolic content, and antioxidant capacity: *Flavonoids, microbial load and quality parameters changes during shelf-life of fermented milk enriched with pasteurized fig purée.*

Microbial load and techno-functional properties analysis

Initial pH values for the control, F10, F20, F30, and F40 were 4.48, 4.49, 4.70, 4.84, and 4.81, respectively. The inclusion of fig puree increased the initial pH of fermented milks, consistent with the pH of figs (5.2–6) reported in other studies (Pereira et al., 2017). Sampling time did not significantly affect pH. After fermentation, microbial counts exceeded 6.5 Log CFU/g for *Lactobacillus* and 8.6 Log CFU/g for *Lactococcus*, surpassing the FAO (2003) minimum recommended threshold of 6 Log CFU/g for probiotic effects. Significant differences in *Lactococcus* counts were observed among formulations during storage, with higher levels in T0 (9.0–8.9 Log CFU/g) compared to T30 (9.0–8.61 Log CFU/g), while *Lactobacillus* counts remained stable. The viability of probiotics was unaffected by the addition of fig puree, suggesting compatibility with maintaining beneficial microbial properties, likely due to reduced post-acidification. Organic acid and sugar levels varied with formulation and time. Colar figs, rich in glucose (379 g/kg dw) and fructose (364 g/kg dw), increased sugar content in fig-enriched milks. F40 at T0 showed the highest glucose (61.7 g/kg) and fructose (66.7 g/kg). Lactic acid levels

increased in all samples at T30 due to lactic acid bacteria (LAB) activity during storage. Higher lactic acid concentrations were observed in the control, F10, and F20, with a dilution effect noted in F30 and F40 due to higher fig puree content. These findings suggest fig puree is a viable ingredient for enhancing fermented milk products, maintaining microbial viability, and ensuring probiotic effects.

Colour coordinates

The CIELab* coordinates of the fermented milks showed significant differences among formulations and between sampling times. The incorporation of fig puree (FP) had a notable impact on the color properties of the fermented milks. The L* parameter decreased with higher concentrations of fig puree, indicating reduced lightness. In contrast, the a* coordinate (green–red axis) and b* coordinate (blue–yellow axis) increased proportionally with the concentration of fig puree.

After 30 days of cold storage, the L* and a* coordinates remained stable across all formulations. However, the b* and chroma (C) values showed a decreasing tendency during storage for most formulations, except for the chroma of F40, which remained constant. Similar findings have been observed in yogurts enriched with anthocyanin-rich plant materials such as mulberry pomace (Du et al., 2023), pomegranate (Cano-Lamadrid et al., 2017), cherry (Sánchez-Bravo et al., 2018) and grape (Silva et al., 2022). This reduction in color intensity has been attributed to the gradual degradation of anthocyanin pigments during cold storage, as confirmed by other authors (Du et al., 2023).

Texture parameters and gel stability

In terms of texture parameters and syneresis of the developed fermented milks, significant differences were observed among formulations and between sampling times (T0–T30). Among the formulations, fermented milk with 40% fig puree (F40) exhibited the highest values for firmness (19.74 g) and consistency (511.82 g s). Sampling time also influenced the textural parameters. Formulations containing 30% and 40% fig puree showed no significant changes in firmness between T0 and T30. However, consistency decreased over time in the CTRL, F10, and F20 formulations but was maintained in the F30 and F40 formulations. Cohesiveness values remained constant across all formulations, while viscosity fluctuated, showing lower values for CTRL and F20 but remaining stable for the other formulations. Regarding syneresis, the percentage decreased as the fig puree content increased, with the F30 and F40 formulations exhibiting the lowest values.

These findings suggest that incorporating fig puree in fermented milks, particularly at higher levels (30% and 40%), enhances the techno-functional properties of fermented milk, likely due to the high pectin content in figs (Gharibzahedi et al., 2019). Pectin is known to enhance protein aggregation, reduce serum separation, and increase viscosity in acidic milk gels, contributing to textural stabilization (Amice-Quemeneur et al., 1995; Arioui et al., 2017; Foley & Mulcahy, 1989). Furthermore, polyphenols can bind with amino acid side chains in the protein matrix, stabilizing the casein network and enhancing the texture of fermented milk (Trigueros et al., 2012). Additionally, the branched-chain structure of polysaccharides can facilitate covalent cross-linking between casein molecules, forming larger aggregates (McClements & Decker, 2018). Therefore, the addition of fig puree minimizes the need for textural additives, offering a natural strategy to improve texture parameters and gel stability in fermented milk.

Total polyphenolic content and antioxidant capacity

The developed fermented milks exhibited an increasing trend in both total phenolic content TPC and DPPH with increasing amounts of added fig puree. TPC showed significant differences among the formulations, with the highest content observed in samples containing 20%, 30%, and 40% fig puree. Specifically, TPC in F10, F20, F30, and F40 was 1.3-fold, 1.4-fold, 1.6-fold, and 1.8-fold higher, respectively, than in the control sample. A similar trend was observed for DPPH activity, with the highest values found in the F40 formulation. After 30 days of cold storage, TPC in fig puree fermented milk samples remained constant, except for the CTRL and F30 formulations, which showed slight decreases. In contrast, DPPH activity in all formulations decreased, with values 1 to 1.2 times lower than the initial levels. The results align with studies on probiotic yogurt supplemented with tea, as reported by Muniandy et al. (2016), which documented a decline in TPC after 21 days of storage. In contrast, Du et al. (2021) noted an increase in TPC in mulberry pomace-fortified yogurt during cold storage. These differences may arise from interactions between plant-derived polyphenols and milk proteins. Cutrim and Cortez (2018) highlighted that polyphenol-casein interactions can significantly modify the structure of casein and enhance the bioavailability of polyphenols.

Flavonoids compounds

Three flavonoids (two flavonols and one anthocyanin) were identified in fermented milks with fig puree. The flavonoid levels increased with higher fig puree content, while no flavonoids were detected in the control (CTRL). At T0, quercetin-3-

galactoside (mean value of 3770 mg/kg) was the most abundant flavonol, followed by quercetin-3-glucoside (mean value of 1844 mg/kg) and cyanidin-3,5-diglucoside (mean value of 1235 mg/kg). After 30 days of storage, the cyanidin-3,5-diglucoside levels in fig puree fermented milk samples remained constant for the F10 and F30 formulations. However, the F20 formulation showed a decrease, while the F40 formulation exhibited increased values. For quercetin-3-galactoside, levels increased across all formulations after 30 days of storage. Quercetin-3-glucoside levels remained constant in the F10 and F20 formulations during the 30 days of storage, whereas the F30 and F40 formulations showed higher values at T30.

The interactions between proteins and flavonoids, particularly anthocyanins and flavonols, have been extensively studied, highlighting the role of hydrogen bonding in stabilizing these compounds. Flavonoids with a higher number of hydroxyl (-OH) groups form stronger hydrogen bonds with proteins, particularly caseins, which are hydrophobic molecules with both negatively and positively charged regions (Arts et al., 2002; Li et al., 2023; Yuksel et al., 2010).

Lactic acid bacteria (LAB), essential in fermentation, also play a crucial role in flavonoid stabilization. LAB-induced pH modulation and metabolic activities impact these compounds stability. Similar effects have been documented in fermented dairy products such as blueberry and pomegranate yogurts, where LAB fermentation preserved flavonoid levels (Cano-Lamadrid et al., 2017; Ścibisz et al., 2012).

Refrigeration and the presence of sucrose enhance flavonoid preservation, reducing oxidative and enzymatic degradation (Nikkhah et al., 2007). The combined effects of protein binding, microbial activity, and optimal storage conditions result in an increase in measurable flavonoid concentrations over time, supporting the use of fig puree in fermented milks and emphasizing the importance of optimizing processing conditions for maximum bioactive compound retention.

Volatile compounds

Twenty-eight volatile compounds were identified, with hexanoic acid being the dominant compound in all samples. Hexanoic acid contributed to cheesy and fatty aromas. The second and third most abundant volatile compounds detected in enriched fermented milk were acetoin and butanoic acid. Acetoin is a common volatile compound produced during fermentation by *Lactobacillus* species (Roncal et al., 2017). Our results show that acetoin levels increased with fig puree content,

with the lowest percentages observed in control fermented milk (21.602%) and F10 (18.193%) and the highest percentage in F40 (26.778%). This increase is likely due to the fermentation process and the composition of the fig puree. Butanoic acid, a characteristic volatile compound in fermented milk, was detected at the highest percentage in the control sample (20.94%). Eleven volatile compounds (V1, V8, V10, V11, V12, V16, V18, V19, V22, V23, and V24) were more abundant in F40, with most contributing to fruity and sweet aromas. Among these, five compounds (V8, V11, V12, V19, and V23) had previously been detected in analyses of the Colar fig variety (Teruel-Andreu et al., 2024).

The remaining volatile compounds found in greater quantities in F40, which were not identified in fresh figs, may have formed during the pasteurization of the fig puree. A study by García-Parra et al. (2020), which analyzed the volatile compounds in pumpkin puree processed by high-pressure heat treatment, showed that the levels of 2-hexenal decreased significantly after processing. This highlights how heat treatment alters volatile profiles. Differences in volatile profiles may explain variations in aroma and flavour, of the fermented milks. But, although variations in volatile composition provide valuable insights the presence of specific compounds does not necessarily guarantee a corresponding sensory effect, as aroma perception is influenced by complex interactions among multiple compounds. Notably, no off-flavor compounds were detected, indicating high product quality.

Consumer study

Texture plays a crucial role in consumer acceptance of fermented milk products. Among texture-related parameters, firmness and creaminess are typically the most influential factors in consumer preference (Duboc & Mollet, 2001). Consumer ratings for firmness, creaminess, and viscosity improved with higher fig puree content (F30 and F40), aligning with the increased values observed in instrumental texture analysis. Consumers identified F30 and F40 as having the highest overall liking scores, which showed a positive correlation with glucose, fructose, and the a* coordinate, while an inverse relationship was observed with lactose and the L* coordinate. The addition of fig puree contributed to enhanced consumer acceptance across all evaluated parameters.

Conclusions of the 3 rd objective

-Fig puree increased initial pH values and maintained probiotic viability while minimizing post-acidification.

- The higher pectin content in fig puree improved textural stability and minimized syneresis in fermented milks. Fermented milks containing 40% fig puree exhibited the greatest firmness and consistency over a 30-day period.

-Fig puree also enriched the nutritional profile by increasing phenolic content and antioxidant capacity.

-Quercetin-3-galactoside was the most abundant flavonol.

- Quercetin-3-galactoside levels increased in all formulations after 30 days of storage.

-The study identified 28 volatile compounds in fig-enriched fermented milk, with hexanoic acid being the dominant contributor to cheesy and fatty aromas.

-Enhanced consumer preference for formulations with 30–40% fig puree (F30, F40).

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CONCLUSIONS/ CONCLUSIONES

This research underscores the multifaceted nutritional and functional properties of figs and their derived products:

- Breba and fig fruits can be labeled as high in potassium, as their potassium levels exceed 600 mg/100 g, meeting EU regulatory standards.
- Breba fruits generally exhibit higher antioxidant activity, crude fiber, and mineral content than figs.
- The peels of both brebas and figs exhibited significantly higher levels of antioxidant activity, fiber, minerals, and volatile compounds than the pulp, which was richer in sugars. This underscores the importance of whole-fruit consumption to maximize health benefits.
- Aromatically, the CA variety exhibited the most pronounced volatile profile, characterized by a high concentration of the predominant volatile compounds. Among these, aldehydes such as hexanal, 2-hexenal, and benzaldehyde stood out, dominating the volatile composition of both brebas and figs.
- Figs contained anthocyanins like cyanidin-3,5-*O*-diglucoside.
- CDN and SA varieties had the highest total polyphenols content in figs.
- Fig extracts exhibited a moderate capacity to inhibit α-glucosidase.

Additionally, this study explored the potential of fig leaves.

- Leaves emerged as a promising dietary supplement, particularly due to their high calcium content.
- The leaves of the SA variety presented the highest mineral content.
- CA leaves were strongly associated with sugar-related compounds such as sucrose, glucose, and fructose. Environmental factors, including soil type, irrigation, and fertilization, were found to influence sugar content variations between CA and CUMH varieties.
- The leaves demonstrated exceptional antioxidant capacity due to their high concentrations of carotenoids, chlorophyll, tocotrienols, tocopherols, and polyphenols.
- Leaves were particularly rich in phenolic acids and flavonols, such as caffeic acid and apigenin derivatives
- The SF and CDN varieties exhibited the highest total polyphenols content in leaves, reinforcing the potential of fig tree leaves as a functional ingredient in nutraceutical applications.

This research also examined alternative strategies for utilizing figs that do not meet fresh market standards, such as smaller-sized fruits.

- Incorporating fig puree into fermented dairy products proved beneficial, as it increased the initial pH, maintained probiotic viability, and minimized post-acidification.
- The high pectin content improved textural stability and reduced syneresis, with formulations containing 40% fig puree (F40) demonstrating the best firmness and consistency over 30 days.
- Fig puree also enhanced the nutritional profile by increasing phenolic content and antioxidant capacity. Notably, quercetin-3-galactoside was the most abundant flavonol, with its levels increasing across all formulations after 30 days of storage.
- Formulations containing 30–40% fig puree exhibited higher consumer preference.

Overall, this research highlights the significance of *F.carica* cultivation in the Mediterranean basin as a key resource for developing efficient, resilient, and sustainable agri-food systems. The comprehensive utilization of fig fruits and leaves presents new opportunities in the food and pharmaceutical industries. Moreover, it offers economic and environmental benefits by promoting sustainable agricultural practices and reducing food waste.

Esta investigación destaca las multifacéticas propiedades nutricionales y funcionales de los higos y sus productos derivados:

• Las brevas y los higos pueden etiquetarse como ricos en potasio, ya que sus niveles superan los 600 mg/100 g, cumpliendo con las normas regulatorias de la UE.

• Las brevas generalmente presentan mayor actividad antioxidante, fibra cruda y contenido mineral que los higos.

• La piel de las brevas y los higos mostraron niveles significativamente mayores de actividad antioxidante, fibra, minerales y compuestos volátiles que la pulpa, que era más rica en azúcares. Esto subraya la importancia del consumo de la fruta entera para maximizar los beneficios para la salud.

• Aromáticamente, la variedad CA presentó el perfil volátil más pronunciado, caracterizado por una alta concentración de los compuestos volátiles predominantes. Entre estos destacan los aldehídos, como hexanal, 2-hexenal y benzaldehído, los cuales dominaron la composición volátil tanto de las brevas como de los higos.

• Los higos contenían antocianinas como la cianidina-3,5-O-diglucósido.

• Las variedades CDN y SA presentaron el mayor contenido total de polifenoles en los higos.

• Los extractos de higo mostraron una capacidad moderada para inhibir la α-glucosidasa.

Además, este estudio exploró el potencial de las hojas de higuera.

• Las hojas se perfilaron como un suplemento dietético prometedor, especialmente debido a su alto contenido de calcio.

• Las hojas de la variedad SA presentaron el mayor contenido mineral.

• Las hojas de CA mostraron una fuerte asociación con compuestos relacionados con el azúcar, como sacarosa, glucosa y fructosa. Se observó que factores ambientales, como el tipo de suelo, el riego y la fertilización, influyen en las variaciones del contenido de azúcar entre las variedades CA y CUMH.

• Las hojas demostraron una capacidad antioxidante excepcional gracias a sus altas concentraciones de carotenoides, clorofila, tocotrienoles, tocoferoles y polifenoles.

• Las hojas fueron particularmente ricas en ácidos fenólicos y flavonoles, como el ácido cafeico y derivados de la apigenina.

• Las variedades SF y CDN presentaron el mayor contenido total de polifenoles en las hojas, lo que refuerza el potencial de las hojas de higuera como ingrediente funcional en aplicaciones nutracéuticas.

Esta investigación también examinó estrategias alternativas para utilizar los higos que no cumplen con los estándares para su consumo en fresco, como los frutos de tamaño más pequeño.

• La incorporación de puré de higos en productos lácteos fermentados resultó beneficiosa, ya que aumentó el pH inicial, mantuvo la viabilidad probiótica y minimizó la postacidificación.

• El alto contenido de pectina mejoró la estabilidad de la textura y redujo la sinéresis, y las formulaciones con un 40 % de puré de higos (F40) mostraron la mejor firmeza y consistencia durante 30 días.

• El puré de higos también mejoró el perfil nutricional al aumentar el contenido fenólico y la capacidad antioxidante. Cabe destacar que quercetina-3-galactósido fue el flavonol más abundante, cuyos niveles aumentaron en todas las formulaciones después de 30 días de almacenamiento.

• Las formulaciones con un 30-40 % de puré de higos mostraron una mayor preferencia por parte de los consumidores.

En general, esta investigación destaca la importancia del cultivo de *F. carica* en la cuenca mediterránea como recurso clave para el desarrollo de sistemas agroalimentarios eficientes, resilientes y sostenibles. El aprovechamiento integral de los frutos y hojas de higo presenta nuevas oportunidades en las industrias alimentaria y farmacéutica. Además, ofrece beneficios económicos y ambientales al promover prácticas agrícolas sostenibles y reducir el desperdicio de alimentos.

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FUTURE RESEARCH

Once this research has been completed and based on the obtained results, further studies should focus on, ensuring the long-term sustainability of *F.carica* cultivation in southeastern Spain, considering the impact of climate change.

- It is suggested to continue exploring potential applications in the food industry for figs that are not commercialized due to their small size. Future research could focus on developing new fig-derived products or functional ingredients, to maximize their utilization and reduce waste.

- To complement the findings of this study, further research is recommended on the nutritional and physicochemical properties of unripen figs. This analysis would help determine their potential in the nutraceutical industry and assess whether their use is more advantageous compared to mature figs. Moreover, it could help optimize the yield of the fig tree by reducing stress factors during the second harvest, which coincides with high temperatures and water stress conditions. These conditions may benefit breba production in the following season, as brebas are figs that remain dormant on the fig tree until the next spring.

- Future studies should focus on the genetic improvement of fig trees to develop varieties with greater tolerance to high temperatures and water stress. This advancement would enhance their adaptation to adverse climatic conditions, ensuring crop stability and sustainability. Moreover, shortening the interval between breba and fig production would be highly beneficial for achieving continuous fruit production from early stages under favorable climatic conditions, while also ensuring fruits with optimal commercial characteristics for fresh consumption.

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