




# Volatile composition of prickly pear fruit pulp from six Spanish cultivars

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**Abstract:** This research analyzed the volatile composition of the fruits pulp of six prickly pear cultivars (NT, NE, NO, NA, FR, and ORI) growing in Spain, by headspace solid-phase microextraction and gas chromatography (GC-MS and GC-FID). A total of 35 compounds were isolated, identified, and quantified, with aldehydes, alcohols, and terpenes being the predominant chemical families, and esters, ketones, linear hydrocarbons, and terpenoids being also found. Nonanol, 2,6-nonadienal, 1-hexanol, 2-hexenal, and D-limonene were the predominant compounds. NT and FR cultivars showed the highest concentration of total volatile compounds. On the other hand, NE and NO cultivars presented the lowest concentration. Future studies on sensory evaluation are required to determine the sensory quality of the fruits of these Spanish cultivars.

**Keywords:** alcohols, aldehydes, gas chromatography, HS-SPME, *Opuntia ficus-indica*

## 1. INTRODUCTION

*Opuntia ficus-indica* (L.) Mill., commonly known as prickly pear, cactus pear, or nopal cactus, is a tropical or subtropical plant which can grow in arid and semi-arid climates. Prickly pear is native to tropical and subtropical America but at present is naturalized in all continents (Sáenz, 2006). This plant is mainly known by their fruits, popularly named “figs” or “tunas”, but their cladodes are also consumed, principally in Mexico, which is the country with the largest area under cultivation (Reyes-Agüero, Aguirre, Carlín-Castelán, & González-Durán, 2013) and also the largest producer (FAO, 2018).

There is ample evidence of the health benefits of prickly pear: it is a source of nutrients and vitamins (Cherkaoui-Malki et al., 2014; FAO, 2018; Feugang, 2007), it shows antioxidant properties due to its phenolic content and antioxidant activity (Ammar, Ennouri, & Attia, 2015; Andreu, Nuncio-Jáuregui, Carbonell-Barrachina, Legua, & Hernández, 2017; Butera et al., 2002; Oumato et al., 2016) and presents medicinal use: anticancer effect (FAO, 2018; Feugang, 2007; Serra, Poejo, Matias, Bronze & Duarte, 2013), treatment of hyperglycemia (FAO, 2018; Frati, Jiménez, & Ariza, 1990; Lopez, 2007) and treatment of high levels of cholesterol (Cherkaoui-Malki et al., 2014; Ennouri et al., 2006) among others. Besides, prickly pear seed oil is rich in tocopherols, which are biologically highly active natural antioxidants, and essential and unsaturated fatty acids (Matthäus & Özcan, 2011; Özcan & Al Juhaimi, 2011). Prickly pear has also been studied for other uses and properties, for example CO<sub>2</sub> uptake (Nobel, Pimienta-Barrios, Hernández, & Ramírez-Hernández, 2002; Nobel, Valenzuela-Tapia, Zañudo-Hernández, Pimienta-Barrios, & Rosas-Espinoza, 2004), phytoremediation of soils (Bañuelos & Lin, 2010; Escobar-

Alvarado, Vaca-Mier, & Rojas-Valencia, 2018) and biofuel production (Sánchez-Godoy, 2012; Santos et al., 2016).

Sensory analysis data of prickly pear fruits in fresh is limited. By contrast, sensory analysis was performed in processed products from prickly pear fruits, like syrups (Sáenz, Estévez, Sepúlveda, & Mecklenburg, 1998), juices (Atef, Abou-Zaid, Ibrahim, Ramadan, & Nadir, 2013; El-Samahy, El-Hady, Habiba, & Moussa-Ayoub, 2007; Rothman, De Wit, Bothma, & Hugo, 2012), nectars (El-Samahy, El-Mansy, Bahlol, El-Desouky, & Ahmed, 2008), and sheets (Atef et al., 2013; El-Samahy et al., 2007). These studies evaluated color, aroma, acidity, taste, texture, and acceptability among other characteristics.

Volatile compounds directly affect the sensory quality of fruits, whose aroma is composed by a complex group of chemical substances such as alcohols, aldehydes, terpenes, ketones, and esters among others. Arena, Campisi, Fallico, Lanza, and Maccarone (2001) reported that the family predominating the aroma profile of this fruit was alcohols; however, Farag, Maamoun, Ehrlich, Fahmy, and Wesjohann (2017), in a more recent study, concluded that short chain aldehydes and acids were the major volatile classes. These compounds generally show a low concentration in fruits and their variability depends on climatological conditions, cultivar, maturity, and storage conditions among other factors (Vázquez-Araújo et al., 2011).

The aim of this study was to determine the volatile composition of fruits pulp of six cultivars of prickly pear, all grown in Spain under homogeneous farming conditions. The information generated will help farmers in selecting and growing those cultivars with the highest contents of volatile compounds.

## 2. MATERIALS AND METHODS

### 2.1 Plant material and sample processing

The fruits of six cultivars of *O. ficus-indica* were used for this study. Four of them (NA, NT, NE and NO) were collected at the experimental field station of Miguel Hernández University in the province of Alicante, Spain (02°03'50"E, 38°03'50"N, and 25 masl). FR and ORI cultivars were collected from private farms of Murcia and Alicante, respectively. Plant species were identified by an expert botanist from the Department of Plant Sciences and

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Microbiology, using the protocol by García-Rollán (1981). One voucher of each cultivar is kept in the Miguel Hernández University herbarium (#152019).

The harvest of the fruits was done during the summer of 2017 and 2018. Three different batches of samples were prepared using 10 uniform fruits of each cultivar; fruits were manually picked at the same ripening stage, and immediately transported to the laboratory for preparation and further analyses. In this way, a total of 30 fruits per cultivar were used for the analyses. Once on the laboratory, the spines of fruits were removed with a brush under tap water for 2 minutes, peeled, and fruits of each batch were cut, grinding for 10 s in a grinder (Taurus Aromatic Ver II; Taurus Group, Barcelona, Spain), and frozen at  $-80^{\circ}\text{C}$  until the time of analysis.

## 2.2 Extraction procedure of volatile aroma compounds

Headspace solid-phase microextraction (HS-SPME) was the method selected to study the volatile composition of the samples under analysis. After several preliminary test to optimize the extraction system, each sample (10 g of the mixture described above) was placed together with 10 mL of water, 1.5 g of salt, and  $\beta$ -ionone as internal standard (10  $\mu\text{L}$  of 1,000 mg/L) into 50 mL vials with polypropylene caps and a polytetrafluoroethylene/silicone septum. Then, a magnetic stirring bar was added, and the vial was placed in a water bath with controlled temperature and automatic stirring. The vials were equilibrated during 5 min at  $40^{\circ}\text{C}$  in the bath and after that a 50/30  $\mu\text{m}$  divinylbenzene/carboxen/polydimethylsiloxane fiber was exposed to the sample headspace for 30 min at  $40^{\circ}\text{C}$ . Later, desorption of the volatile compounds from the fiber coating was performed in the injection port of the GC-MS during 3 min. Extraction experiments were run in triplicate.

## 2.3 Chromatographic analyses

The isolation and identification of the volatile compounds was carried out on a gas chromatograph (GC), Shimadzu GC-17A (Shimadzu Corporation, Kyoto, Japan), coupled with a Shimadzu mass spectrometer detector (MS) QP-5050A. The GC-MS system was equipped with a SLB-5 ms capillary column, 95% dimethylpolysiloxane, and 5% diphenylpolysiloxane (Sigma-Aldrich, Spain; 30 m  $\times$  0.25 mm i.d., 0.25  $\mu\text{m}$  film thickness). Analyses were carried out using helium as carrier gas at a flow rate of 13 mL/min, in a split ratio of 1:20, and the following temperature program: (a) initial temperature  $80^{\circ}\text{C}$ ; (b) rate of  $3.0^{\circ}\text{C}/\text{min}$  to  $170^{\circ}\text{C}$  and hold for 1 min; (c) rate of  $25^{\circ}\text{C}/\text{min}$  from  $170$  to  $300^{\circ}\text{C}$  and hold for 1.8 min. Injector and detector temperatures were held at  $230$  and  $300^{\circ}\text{C}$ , respectively.

Three analytical methods were used for the identification of the volatile compounds: (1) retention indices of each problem compound (retention indices), (2) GC-MS retention times (authentic standard), and (3) mass spectra (authentic chemicals and NIST05 spectral library collection; NIST, 2011). Tentatively identified compounds, based on only mass spectral data, have been also included in this study.

The semiquantification of the volatile compounds was performed on a GC, Shimadzu GC-17A, with a fame ionization detector (FID). The column and chromatographic conditions were those previously reported for the GC-MS analysis. The injector temperature was  $300^{\circ}\text{C}$  and nitrogen was used as carrier gas (1 mL/min). The relative abundance was obtained from electronic integration measurements using FID.

For the semiquantification of the volatile compounds,  $\beta$ -ionone was added as internal standard (10  $\mu\text{L}$  of 1,000 mg/L) and the areas

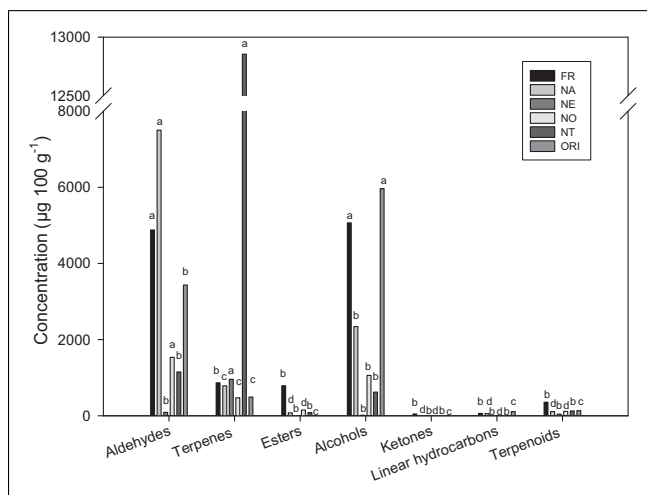


Figure 1—Main chemical families in the studied cultivars of prickly pear fruit pulp. Bars with the same letter within the same cultivar were not significantly different at  $P < 0.05$ , according to Tukey's multiple range test.

from all compounds were normalized using its area; this compound was chosen after checking that it was not present in the prickly pear cultivars under study. No standard curves were performed for each one of the quantified volatile compounds, so data included in this study should be considered as semiquantitative. However, relative values are suitable for comparing differences between prickly pear cultivars. All analytical analyses were run in triplicate.

## 2.4 Statistical analyses

One-way analysis of variance (ANOVA) and multiple-range tests were used for samples comparison. The method used to discriminate among the means (multiple range test) was the Tukey's least significant difference procedure. Significance was defined at  $P \leq 0.05$ . Statistical analysis was performed using StatGraphics Plus 5.0 software (2000) (Manugistics, Inc., Rockville, MD). Figure 1, which shows the concentration of each chemical family in the studied cultivars, was drawn using SigmaPlot 11.0 (Systat Software, San José, CA, USA). Besides, principal component analysis (Figure 2) was performed using XLSTAT software version 9 (Addinsoft, 2010).

## 3. RESULTS AND DISCUSSION

A total of 35 compounds were isolated, identified, and quantified using the HS-SPME technique combined with GC and two detectors (GC-MS and GC-FID). Table 1 shows these compounds with an assigned code and their sensory descriptors according to SAFC<sup>®</sup> Flavors and Fragrances Catalog (SAFC, 2011) and the Flavor and Extract Manufacturers Association of the United States (FEMA, 2018). Table 2 shows the concentration of these compounds in  $\mu\text{g}$  100/g and their total content in mg 100/g.

The cultivars which presented the highest concentration of volatile compounds were NT and FR (14.83 and 12.06 mg 100/g, respectively). By contrast, NE and NO cultivars were the cultivars with the lowest total volatile content (1.10 and 3.33 mg 100/g, respectively). The concentration found in Yellow and White cultivars (1.10 and 1.08 mg 100/g, respectively), studied by Arena et al. (2001), were similar to those found in NE cultivar, but the rest of cultivars showed higher values. The concentration of Red cultivar, studied by Arena et al. (2001), was lower than all the cultivars studied in this research.

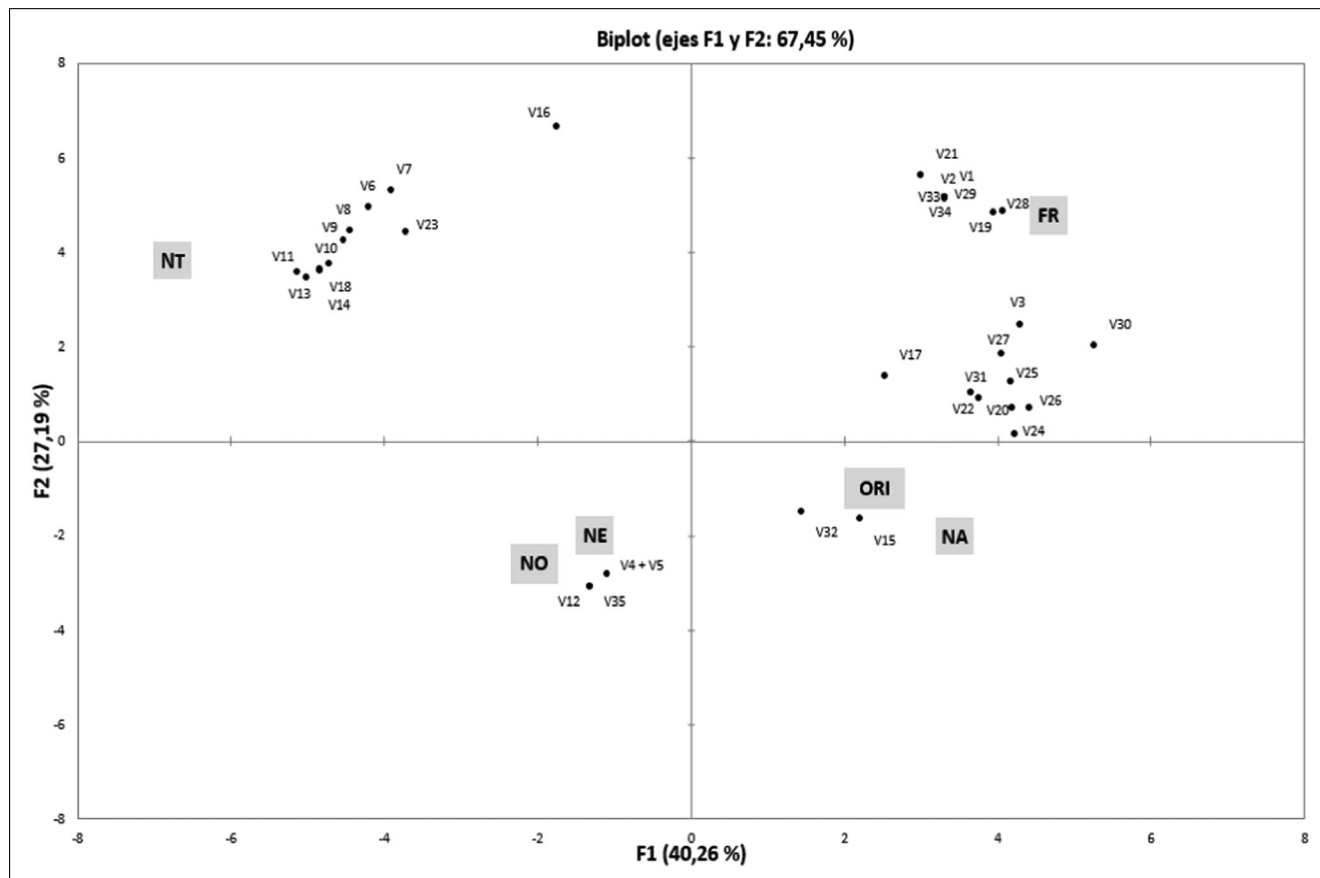


Figure 2—Principal component analysis (F1 and F2) of six cultivars of prickly pear fruit pulp.

The volatile compounds that were isolated can be grouped into seven chemical families:

- Aldehydes ( $n = 10$ ): 2-hexenal, heptanal, 2-heptenal, octanal, (*E*)-2-octenal, nonanal, (*Z*)-2-nonenal, (*E*)-2-nonenal, 2,6-nonadienal (two isomers were found), 2,4-decadienal.
- Terpenes ( $n = 7$ ):  $\beta$ -myrcene, *p*-cymene, D-limonene, (*E*)- $\beta$ -ocimene,  $\gamma$ -terpinene,  $\alpha$ -ocimene and  $\alpha$ -farnesene.
- Esters ( $n = 7$ ): 2-methylbutanoic acid methyl ester, methyl-3-hexenoate, ethyl hexanoate, hexyl acetate, ethyl octanoate, methyl-4-decenoate, methyl decanoate.
- Alcohols ( $n = 6$ ): 2-methyl-1-butanol, 1-hexanol, 1-octanol, (*E,Z*)-3,6-nonadien-1-ol, nonanol, 2-nonen-1-ol.
- Ketones ( $n = 1$ ): 1-Penten-3-one.
- Linear hydrocarbons ( $n = 1$ ): 5-undecene.
- Terpenoids ( $n = 2$ ): eucalyptol, linalool.

FR cultivar showed 30 compounds, of which four were cultivar-specific (1-penten-3-one, 2-methyl-butanol, 2-nonen-1-ol, and methyl decanoate). In fruits of the NE and NA cultivars 20 compounds were detected, of which eucalyptol was only detected in NA cultivar and hexyl acetate and  $\alpha$ -farnesene were only detected in the NO cultivar. NT cultivar presented 17 compounds and ethyl hexanoate was only found in this cultivar. In the fruits of the ORI cultivar, 16 compounds were detected and 2,4-decadienal was exclusive to this cultivar. NE cultivar showed only 11 volatile compounds.

Volatile compounds common to all cultivars were  $\beta$ -myrcene, *p*-cymene, D-limonene, (*E*)- $\beta$ -ocimene,  $\gamma$ -terpinene, linalool,

nonanal, and 2,6-nonadienal. The volatile profile of fruits of the FR, NO, NT, and ORI cultivars included many aldehydes, whereas those of the NA and NE cultivars had more terpenes. These results did not agree with other authors who reported alcohols (Arena et al., 2001; Flath & Takahashi, 1978; Oumato et al., 2016) and esters (Rodríguez, Díaz, & Nazareno, 2015) as the most numerous and abundant compounds.

Figure 1 shows the concentration of each chemical family in the studied cultivars. Aldehydes were the predominant compounds in NA and NO cultivars (69.0% and 46.1% of the total concentration of volatile compounds), followed by alcohols in NA cultivar (21.6%). FR showed alcohols and aldehydes as the predominant chemical families (42.0% and 40.5%, respectively), and ORI cultivar presented alcohols as the predominant compounds (58.9%) followed by aldehydes (33.9%). However, in NE and NT cultivars the predominant compounds were terpenes (86.7% in both cases). The results obtained in FR and ORI cultivars agreed with Flath and Takamashi (1978) and Arena et al. (2001), who reported alcohols as the most abundant chemical family. Oumato et al. (2016) studied three cultivars: *Dellahia* presented alcohols as the predominant chemical family followed by aldehydes, which also agreed with the results obtained in FR and ORI cultivar; however, *Aissa* and *Should* cultivars showed aldehydes as the primary chemical family, followed by alcohols, which agreed with the results in NA, FR, and NO cultivars. Rodríguez et al. (2015) obtained hydrocarbons as the most abundant chemical family; no cultivar followed this pattern in this study.

D-Limonene was the predominant compound in NE and NT cultivars, having a content ranging from 814  $\mu\text{g}/100\text{g}$  in the

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

Code	Compound	Retention time (min)	Retention indexes		Sensory descriptor
			Exp. <sup>a</sup>	Lit. <sup>a</sup>	
V1	1-Penten-3-one <sup>b</sup>	2.627	—	—	Vegetable <sup>d</sup>
V2	2-Methyl-1-butanol <sup>b</sup>	2.853	—	—	Green, malt, onion, wine <sup>d</sup>
V3	Methyl 2-methylbutanoate <sup>b</sup>	3.091	—	—	Apple, fruity, strawberry <sup>d</sup>
V4 + V5	1-Hexanol + 2-Hexenal	3.964	857	850	Green, herbaceous / Almond, fruity <sup>d</sup>
V6	Heptanal	4.464	901	901	Oily, fruity, woody, fatty, nutty <sup>d</sup>
V7	Methyl-3-hexenoate <sup>c</sup>	4.893	929	936	Fruity <sup>c</sup>
V8	2-Heptenal	5.449	954	954	Apple, citrus, fatty, spicy, vegetable <sup>d</sup>
V9	$\beta$ -Myrcene	6.103	988	987	Anise, fruity, herbaceous, sweet <sup>d</sup>
V10	Ethyl hexanoate	6.235	996	1000	Apple, banana, pineapple, wine-like <sup>d</sup>
V11	Octanal	6.426	1004	1006	Honey, fruity, fatty, citrus <sup>d</sup>
V12	Hexyl acetate	6.517	1007	1010	Apple, cherry, floral, pear, sweet <sup>d</sup>
V13	<i>p</i> -Cymene	7.115	1,029	1,027	Citrus <sup>d</sup>
V14	D-Limonene	7.242	1,033	1,033	Citrus, sweet <sup>d</sup>
V15	Eucalyptol	7.464	1,035	1,035	Citrus, herbaceous, fruity, sweet <sup>d</sup>
V16	trans- $\beta$ -Ocimene	7.558	1,044	1,041	Floral <sup>e</sup>
V17	2-Octenal, (E)-	7.934	1,058	1,059	Spicy, herbaceous, green <sup>d</sup>
V18	$\gamma$ -Terpinene	8.024	1,061	1,062	Herbaceous, citrus <sup>d</sup>
V19	1-Octanol	8.232	1,069	1,069	Citrus, fatty, woody, waxy <sup>d</sup>
V20	5-Undecene <sup>c</sup>	9.061	1,098	1,090	Not defined in the literature
V21	Linalool	9.181	1,102	1,101	Citrus, floral, sweet <sup>d</sup>
V22	( <i>E,Z</i> )-3,6-Nonadien-1-ol <sup>b</sup>	9.250	1,104	—	Melon, green, violet <sup>d</sup>
V23	Nonanal	9.334	1,106	1,104	Citrus, vegetable, nutty, waxy, fatty <sup>d</sup>
V24	( <i>Z</i> )-2-Nonenal	10.669	1,143	1,149	Waxy, fatty <sup>d</sup>
V25	( <i>E</i> )-2-Nonenal	10.823	1,148	1,156	Waxy, fatty <sup>d</sup>
V26	2,6-Nonadienal (isomer 1)	11.096	1,155	1,160	Vegetable, green <sup>d</sup>
V27	Nonanol	11.351	1,162	1,168	Melon, green, fatty <sup>d</sup>
V28	2,6-Nonadienal (isomer 2)	11.455	1,165	1,160	Vegetable, green <sup>d</sup>
V29	2-Nonen-1-ol <sup>b</sup>	11.551	1,168	—	Melon, waxy, fatty, sweet, violet <sup>d</sup>
V30	$\alpha$ -Ocimene <sup>b</sup>	12.166	1,185	—	Floral <sup>e</sup>
V31	Ethyl octanoate	12.858	1,203	1,206	Apricot, floral, pear, pineapple <sup>d</sup>
V32	2,4-Decadienal	16.685	1,297	1,309	Fatty, citrus, meaty <sup>d</sup>
V33	Methyl-4-decenoate <sup>b</sup>	17.172	1,309	—	Fruity <sup>c</sup>
V34	Methyl decanoate	17.807	1,324	1324	Oily, wine-like, fruity <sup>d</sup>
V35	$\alpha$ -Farnesene	26.220	1,523	1,522	Apple, lavender, lime, woody, green <sup>d</sup>

<sup>a</sup>Lit., literature (NIST 2011); Exp., experimental.

<sup>b</sup>Tentatively identified.

<sup>c</sup>Identified in DB-1 column.

<sup>d</sup>SAFC (SAFC, 2011).

<sup>e</sup>FEMA (FEMA, 2018).

NE cultivar to 11,026  $\mu\text{g}$  100/g in NT cultivar (these contents represented 69.2% and 72.9% of the volatile compounds profile, respectively). This compound can be described as having lemon, orange, citrus, and sweet notes. However, FR, NA, and ORI cultivars showed nonanol and 2,6-nonadienal (isomer 1) as the most abundant compounds. Nonanol represented 20.6% of the volatile profile in NA cultivar (2,251  $\mu\text{g}$  100/g), 34.0% in FR cultivar (4,146  $\mu\text{g}$  100/g), and 58.7% in ORI cultivar (5,946  $\mu\text{g}$  100/g). 2,6-Nonadienal (isomer 1) showed similar values to those of nonanol in FR cultivar (3,731  $\mu\text{g}$  100/g, 30.3% of the volatile compounds profile), higher values in NA cultivar (7,193  $\mu\text{g}$  100/g, 66.1% of the total volatile compounds) and lower ones in ORI cultivar (2926  $\mu\text{g}$  100/g, 28.7% of the volatile compounds profile). These compounds can be described as having green, melon, and fatty notes (nonanol) and vegetable and green notes (2,6-nonadienal). In NO cultivar, the most abundant compounds were 1-hexanol and 2-hexenal, with concentrations of 1,883  $\mu\text{g}$  100/g, represented 57.2% of the volatile profile both together. 1-Hexanol can be described as having green, herbaceous, wood, and sweet notes and 2-hexenal as having almond, apple, green, plum, sweet, and vegetable notes. These results do not agree with those obtained by Flath and Takahasi (1978), who obtained ethanol as the

predominant compound, but in the Should cultivar studied by Oumato et al. (2016). 2-Hexenal was the most abundant compound, which agreed with the results obtained in NO cultivar. Besides, Arena et al. (2001) identified 1-hexanol, together with 2-hexen-1-ol, as the most abundant compounds, which also agreed with the results for NO cultivar.

2,6-Nonadienal, one of the main volatile compound in the FR, NA, and ORI cultivars, has also been found as the principal volatile compound in cucumber (*Cucumis sativus* L.) (Buescher & Buescher, 2001; Kemp, Knavel, & Stoltz, 1974; Schieberle, Ofner, & Grosch, 1990) and it is an important contributor to mango (*Mangifera indica* L.) aroma (Engel & Tressl, 1983; Pino & Mesa, 2006). Nonanol, which was also an important component in FR, NA, and ORI cultivars, was also detected in black tea (*Camellia sinensis* L.; Chen et al., 2019) and *Arctium lappa* L. leaf (Golbazi, Zarei, Garakani, & Mojab, 2018), but in lower concentrations. 1-Hexanol and 2-hexenal, the principal compounds in NO cultivar, were also found in tropical fruits such as guava (*Psidium guajava* L.; Nishimura, Yamaguchi, Mihara, & Shibamoto, 1989; Soares, Pereira, Maio Marques, & Monteiro, 2007) and kiwi (*Actinidia chinensis* Planch.; Bartley & Schwede, 1989; Takeoka, Güntert, Jennings, Flath, & Wurz, 1986). The predominant compounds in NT

**Table 2**–Volatile composition found in fruits pulp of six cultivars of prickly pear ( $\mu\text{g } 100/\text{g}$ ).

Compound	ANOVA	Concentration ( $\mu\text{g } 100/\text{g}$ )					
		FR	NA	NE	NO	NT	ORI
1-Penten-3-one	*** <sup>a</sup>	47.15 a <sup>b</sup>	N.D. b	N.D. b	N.D. b	N.D. b	N.D. b
2-Methyl-1-butanol	**	9.91 a	N.D. b	N.D. b	N.D. b	N.D. b	N.D. b
Methyl 2-methylbutanoate	***	53.70 a	60.52 a	N.D. b	N.D. b	N.D. b	N.D. b
1-Hexanol + 2-Hexenal	***	123 b	N.D. d	N.D. d	1883 a	N.D. d	20.97 c
Heptanal	*	8.80 b	N.D. c	N.D. c	3.85 b	34.2 a	N.D. c
Methyl-3-hexenoate	**	16.39 b	N.D. c	N.D. c	2.77 b	54.56 a	N.D. c
2-Heptenal	***	16.13 b	N.D. c	N.D. c	5.08 b	117 a	N.D. c
$\beta$ -Myrcene	***	82.52 b	26.31 b	35.31 b	40.61 b	567 a	47.23 b
Ethyl hexanoate	**	N.D. b	N.D. b	N.D. b	N.D. b	26.24 a	N.D. b
Octanal	***	9.08 a	N.D. c	11.44 b	14.32 b	60.22 a	N.D. c
Hexyl acetate	***	N.D. b	N.D. b	N.D. b	138 a	N.D. b	N.D. b
<i>p</i> -Cymene	***	2.16 c	1.71 c	7.13 b	7.71b	79.12 a	1.2 c
D-Limonene	***	224 c	298 c	814 b	329 c	11026 a	10.14 d
Eucalyptol	*	N.D. b	11.3 a	N.D. b	N.D. b	N.D. b	N.D. b
( <i>E</i> )- $\beta$ -Ocimene	***	81.80 a	26.46 c	28.15 c	37.43 bc	95.43 a	50.87 b
( <i>E</i> )-2-Octenal	***	66.71 b	7.15 d	N.D. e	3.79 d	19.16 c	141 a
$\gamma$ -Terpinene	**	17.50 b	30.24 b	73.07 b	36.01 b	1086 a	N.D. c
1-Octanol	**	14.91 a	3.86 b	N.D. c	N.D. c	N.D. c	N.D. c
5-Undecene	*	63.46 b	55.81 b	N.D. c	N.D. c	N.D. c	107 a
Linalool	***	354 a	98.14 b	42.99 c	113 b	127 b	134 b
( <i>E,Z</i> )-3,6-Nonadien-1-ol	***	50.05 b	89.76 a	N.D. d	14.83 c	8.96 c	7.73 c
Nonanal	***	38.33 b	15.27 c	4.46 c	45.29 b	75.73 a	18.37 c
( <i>Z</i> )-2-Nonenal	**	52.60 b	121 a	N.D. c	N.D. c	N.D. c	37.78 b
( <i>E</i> )-2-Nonenal	***	67.58 b	34.22 c	N.D. d	N.D. d	N.D. d	106 a
2,6-Nonadienal (isomer 1)	***	3731 b	7193 a	69.02 c	491 c	829 c	2926 b
Nonanol	***	4146 b	2251 c	15.1 d	103 d	613 d	5946 a
2,6-Nonadienal (isomer 2)	***	828 a	132 b	4.23 b	29.80 b	15.48 b	166 b
2-Nonen-1-ol	***	785 a	N.D. b	N.D. b	N.D. b	N.D. b	N.D. b
$\alpha$ -Ocimene	***	457 a	403 a	N.D. b	N.D. b	N.D. b	384 a
Ethyl octanoate	**	6.78 a	13.52 a	N.D. b	N.D. b	N.D. b	N.D. b
2,4-Decadienal	***	N.D. b	N.D. b	N.D. b	N.D. b	N.D. b	28.59 a
Methyl-4-decanoate	***	679 a	N.D. c	N.D. c	9.16 b	N.D. c	N.D. c
Methyl decanoate	**	30.90 a	N.D. b	N.D. b	N.D. b	N.D. b	N.D. b
$\alpha$ -Farnesene	**	N.D. b	N.D. b	N.D. b	21.48 a	N.D. b	N.D. b
Total (mg 100/g)	***	12.06 ab	10.87 b	1.10 c	3.33 c	14.83 a	10.13 b

<sup>a</sup>\*, \*\*, and \*\*\*, significant at  $P < 0.05$ ,  $0.01$ , and  $0.001$ , respectively.

<sup>b</sup>Values are the mean of three replications. Values followed by the same letter, within the same row, were not statistically different according to the Tuckey's multiple range test. N.D., nondetected.

and NE cultivars, D-limonene, and  $\gamma$ -terpinene are also found in high contents in citrus fruits (Moufida & Marzouk, 2003; Reynes, Alter, Brat, Brillouet, & Rega, 2003; Rudaz et al., 2013).

Principal component analyses (PCA) was performed to obtain an easier and complete understanding of the relationship among the studied cultivars and their volatile compounds (Figure 2). The first principal component (F1) accounted for 40.26% and the second one for (F2) 27.19% of the total variance. It is important to remember that the higher the distance between two parameters, the lower their correlation.

F1 was positively linked with (*Z*)-2-nonenal, 5-undecene, (*E,Z*)-3,6-nonadien-1-ol, nonanol, 2-methylbutanoic acid methyl ester,  $\alpha$ -ocimene, 2,6-nonadienal (isomer 2), 1-penten-3-one, methyl decanoate, and 1-octanol, and negatively with ethyl hexanoate, *p*-cymene,  $\gamma$ -terpinene, D-limonene, octanal,  $\beta$ -myrcene, 2-heptenal, heptanal, nonanal, and methyl-3-hexenoate. F2 was positively linked with (*E*)- $\beta$ -ocimene and inversely with 1-hexanol + 2-hexenal, acetic acid hexyl ester and  $\alpha$ -farnesene.

The principal component F1 was able to establish differences among samples. FR, ORI, and NA cultivars, which were positioned on the right part of the graph, were correlated with the presence of volatile compounds with green and fatty notes, such as 2,6-nonadienal and nonanol. On the other hand, NT, NO, and NE cultivars were situated of the left of the graph and were positively linked with compounds having citrus,

fruity, and vegetable notes, mainly D-limonene, 1-hexanol, and 2-hexenal.

#### 4. CONCLUSION

The volatile composition of six cultivars of *O. ficus-indica* was analyzed. Even though prickly pears have not a strong aroma, a total 35 compounds of were isolated, identified, and quantified: 10 aldehydes (for example, 2,6-nonadienal), 8 terpenes ( $\beta$ -myrcene), 7 esters (methyl-3-hexenoate), 6 alcohols (nonanol), 1 ketone (1-penten-3-one), 1 linear hydrocarbon (5-undecene), and 1 terpene (linalool). The cultivars with the highest total concentration of volatile compounds were NT and FR, making them attractive for consumers because, in general, the more volatile content, the higher consumer acceptance. On the other hand, the fruits of the NO and NE cultivars showed the lowest concentration of volatile compounds. Nonanol and 2,6-nonadienal were the predominant compounds in FR, NA, and ORI cultivars, 1-hexanol + 2-hexenal in NO cultivar and D-limonene and  $\gamma$ -terpinene in NT and NE cultivars. *O. ficus-indica* fruits are highly valued for their high health-promoting benefits but sensory evaluation is needed to complete the knowledge of the aroma of this fruit and the effect of the cultivar. Thus, further investigation on the organoleptic attributes of prickly pear will be conducted and were not already done due to the lack of orchards in our surrounding area.

## AUTHOR CONTRIBUTIONS

L. Andreu-Coll and L. Noguera-Artiaga were responsible for laboratory work, statistical analysis, and writing the manuscript. A. Carbonell-Barrachina was responsible for experiment design, data processing, and reviewed the manuscript. P. Legua and F. Hernández reviewed the manuscript and were in charge of data analysis.

## CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

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