



## Research article

## Valorization of date palm (*Phoenix dactylifera* L.) pruning biomass by co-composting with urban and agri-food sludge



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## ABSTRACT

In the Mediterranean countries, there is an increasing production of date palm wastes (*Phoenix dactylifera* L.), not only due to the raising production of date palm fruits, but also derived from the maintenance of urban and peri-urban green areas, especially in those affected by red palm weevil (*Rhynchophorus ferrugineus*). The management of this increasing volume of green wastes usually concludes with a controlled disposal that implies an important loss of resources, in terms of organic matter, nutrients and energy. In addition, the rise of wastewater generation and the incentive of the wastewater treatment processes have derived in an increase of the amount of the sludge produced, which makes difficult its management. This work studies the feasibility of co-composting palm wastes with sludge from the urban and agri-food sectors as alternative treatment to manage these organic waste streams and to obtain added-value compost. For this, four mixtures (P1, P2, P3 and P4) were prepared using as main component palm leave waste (PL) mixed with different types of sludge. In the piles P1, P2 and P3, sewage sludge (SS) was used as co-composting agent, while agri-food sludge (AS) was used in P4. Throughout composting, the thermal profile of the composting piles was assessed, as were physical, chemical, physico-chemical and maturity parameters. In addition, the changes in water-soluble organic matter were assessed using chemical analytical methods and the excitation–emission matrix (EEM) fluorescence spectroscopy. The results obtained showed the viability of the co-composting process to obtain end-products with adequate maturity degree and physical characteristics for their potential use as substrates, except for the salt contents that can limit their use in some agricultural sectors.

## 1. Introduction

Palm tree and especially, date palm (*Phoenix dactylifera* L.) is a tropical and subtropical tree of the family palmae (*Arecaceae*). Palm tree is widely used as an ornamental plant and constitutes one of the most cultivated palms in arid and semi-arid regions. This species has a significant role in the Mediterranean countries at different levels (economic, environmental, cultural and landscape). The world total number of date palms is about 105 million, 32,000 of those being located only in Europe, mainly in Spain (Agoudjil et al., 2011; Zaid and Arias-Jimenez, 2002). Concretely, *Phoenix dactylifera* is also extensively cultivated for date production (Abid et al., 2016), with a significant increasing production in the last years (Chandrasekaran and Bahkali, 2013). Therefore, palm pruning material represents a huge amount of agro-industrial by-products and wastes, such as leaves and palm fibre, whose management constitutes an environmental problem (Abid et al.,

2016). In addition, the strategies associated to the control and eradication of specimens affected by the pest caused by red palm weevil (*Rhynchophorus ferrugineus*) are also significantly increasing the volume of the palm wastes generated. As an example, and without considering any additional biomass related to the specimens affected by red palm weevil, each palm tree produces around 20 kg of dry leaves annually (Alkokaik et al., 2011; Chandrasekaran and Bahkali, 2013), which supposes a production of more than 0.2Mt only of palm leaves (El May et al., 2012). Therefore, the great volume of these organic wastes, principally due to their characteristics and the absence of an adequate management usually ends up with a controlled disposal that implies an important loss of resource in terms of energy, nutrients and organic matter. A traditional practice, considered as a cheap waste management technique for these wastes is the controlled and uncontrolled burning, which produces direct and indirect impacts in the environment, such as the emission of greenhouse gases.

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In addition, the generation of sewage sludge in the wastewater treatment plants is becoming an important problem, since the increasingly stringent wastewater treatment requirements have produced an important rise in the amounts of sewage sludge generated. The production in Europe is about 9.4 millions of tons per year, according to the European Environmental Agency, being used the 54 percent for agricultural purposes, the same trend being followed in USA (Martin-Mata et al., 2015). In the agri-food sector, aspects such as the increase of wastewater generation due to the rise of the production and the encouragement of the wastewater treatment processes have made worse the management and storage problems of the sludge generated. This fact has produced an important increase in the generation of sludge, which has made difficult its management (Morales et al., 2016).

Composting constitutes an environmentally-friendly and economically-viable technology for the treatment of organic wastes, which enhances their value by yielding a stabilized, mature, deodorized and sanitized product, free of pathogens and weeds and rich in humic substances, which is easy to store and is marketable as an organic amendment or fertilizer (Bernal et al., 2009). Different authors have reported the use of the composting process as a feasible technology to manage sewage sludge, with urban or agri-food origin (Lim et al., 2016; Morales et al., 2016). However, although the composting process has been widely studied for the management and recycling of agricultural wastes, the use of this treatment applied to palm tree wastes with another problematic organic waste, such as agri-food sludge, as co-composting agent has not been reported. Currently, there are only several works from the countries with the highest palm tree culture areas (Saudi Arabia, Egypt, Iran and Tunisia), in which different manures have been used as co-composting agents, such as cow, quail, goat and sheep manure (Sadik et al., 2010, 2012), sheep manure (Sghairoun and Ferchichi, 2011) and broiler chicken manure (Alkoaik et al., 2011).

Therefore, the main aim of this work was to evaluate the feasibility of co-composting palm waste with sludge from the urban and agri-food sectors to manage this type of organic wastes and to obtain an added-value organic fertilizer with a potential commercial value. For this, the study has been mainly focused on: a) the monitoring of the composting process in the mixtures through the thermal profile and the evolution of chemical parameters; b) the assessment of the quality and economic value of the end-products obtained based on their final characteristics and nutrient contents.

## 2. Material and methods

### 2.1. Characteristics of the initial materials

The raw materials used for the preparation of the four composting mixtures were constituted by palm leaves (PL), sewage sludge (SS) and agri-food sludge (AS). PL (*Phoenix dactylifera* L.) was collected after the park maintenance activities at the Orihuela municipality (Alicante, Spain) and prior to be used in the composting mixtures, was homogenized and crushed to < 5 cm particle size. SS1 and SS2 came from the municipal wastewater treatment plant of Torrevieja and Orihuela, respectively (Alicante, Spain) and SS3 was obtained from the municipal wastewater treatment plant of Elche (Alicante, Spain). SS1 and SS2 were obtained after the treatment of wastewater using an aerobic biological process with later stabilization by anaerobic digestion and dehydration using band filters; SS3 was obtained after treating wastewater by an aerobic process of active sludge with nutrient removal and later dehydration using centrifugal decanters. AS came from the treatment plant of an agri-food industry that principally treats wastewater derived from the processing of pear, located in Murcia (Spain). This sludge was obtained by flotation-cavitation, denitrification, forced aeration MTS, clarification in an UBSF reactor and later dehydration by centrifugation. Table 1 shows the main chemical properties of the initial organic wastes used in the compost preparation.

**Table 1**

Characteristics (on a dry weight basis) of the initial materials used in the composting piles.

Parameter	PL	SS1	SS2	SS3	AS
Dry matter (%)	64.9	26.1	24.5	19.0	8.20
pH	6.8	7.1	6.8	6.1	7.2
EC (dS/m)	7.3	3.3	2.1	4.4	5.1
OM (%)	91.4	68.7	64.8	66.8	52.4
C <sub>T</sub> (%)	44.1	37.8	35.8	43.6	34.5
N <sub>T</sub> (%)	2.0	5.9	5.8	5.5	3.3
C/N	24.2	6.4	6.2	7.9	10.4
P (g/kg)	1.7	17.8	27.8	26.2	5.9
K (g/kg)	19.9	1.7	2.2	2.4	5.7
Na (g/kg)	4.8	2.1	2.5	3.7	14.6

EC: Electrical conductivity; OM: Organic matter; C<sub>T</sub>: Total organic carbon; N<sub>T</sub>: total nitrogen.

PL: palm leaves; SS: sewage sludge; AS: agri-food sludge.

### 2.2. Compost elaboration

Four composts were prepared at the composting pilot plant (Compolab) of the Miguel Hernández University, placed in Orihuela (Alicante; 38° 5' 8" N, 0° 56' 49" W, elevation 23 m a.s.l.). The percentages of the organic wastes used to prepare the composting piles were established trying to use the maximum amount of PL with a sufficient proportion of sludge (on a dry weight basis) to balance the C/N ratio of the mixtures. Thus, the proportions of initial materials on a fresh weight basis were the following (dry weight basis between brackets):

- P1: 72.3% SS1 + 27.7% PL [46.6:53.4]
- P2: 67.7% SS2 + 32.3% PL [42.2:57.8]
- P3: 67.7% SS3 + 32.3% PL [36.2:63.8]
- P4: 82.5% AS + 17.5% PL [42.8:57.2]

The mixtures (about 15 m<sup>3</sup> each) were managed as trapezoidal windrows through the turning composting system. The piles were mechanically turned every week to improve the aeration of the composting mixtures and, during the thermophilic phase, to avoid an excessive increase in temperature. The temperature of the mixtures and that of the ambient were monitored throughout the composting process. These values were registered using several probes connected to data loggers (HOBO-Data Logger U12-006). The moisture content of each pile was maintained adding water at each turning time, within the optimal range of 55–65% to allow microbial metabolism. The bio-oxidative stage was considered finished when temperature in the composting mixtures was proximate to that of ambient and when temperature did not increase after turning. This phase lasted 139 days for P1, 152 days for P2, 116 days for P3 and 96 days for P4. After this, all the piles were allowed to mature approximately a month.

The composting samples were collected after mixing until homogenization seven sub-samples obtained from the top to the bottom of the pile, from seven places, according to the method used by Bustamante et al. (2012). The samples corresponded to the initial phase of the process (I), the thermophilic stage (T), when the bio-oxidative stage ends (EB) and the maturity period (M). Each representative sample was subdivided into two samples: i) the first sample was used to determine the moisture content, after drying the sample for 12 h at 105 °C; ii) the second was dried at 45 °C, ground to obtain a particle size < 0.5 mm and stored for the analytical determinations, according to Bustamante et al. (2012). All the analytical determinations were carried out in triplicate.

### 2.3. Chemical and physical determinations

In all materials, physico-chemical parameters (electrical

conductivity and pH) were assessed in a weight: volume (1:10) water-soluble extract, according to the standard method EN 12176 (1998). Samples moisture was assessed after drying at 105 °C until constant weight, following the standard method of FCQAO (1994). Organic matter (OM), total organic C ( $C_T$ ) and total nitrogen ( $N_T$ ), as well as the water-soluble fraction of organic carbon (WSC) were also assessed according to the methods used by Bustamante et al. (2012).

OM losses were determined using the ash concentrations at the beginning ( $X_1$ ) and end ( $X_2$ ) of the process, following equation (1) of Paredes et al. (2000):

$$OM \text{ loss}(\%) = 100 - 100 \frac{[X_1(100 - X_2)]}{[X_2(100 - X_1)]} \quad (1)$$

In addition, cation exchange capacity (CEC) was assessed using  $BaCl_2$ -triethanolamine, according to the method of Lax et al. (1986). The germination index (GI) was determined combining the measurements of seed germination and root elongation of cress seeds (*Lepidium sativum* L.), following the method described by Zucconi et al. (1981). After nitric-perchloric acid digestion, phosphorus (P) was determined colourimetrically as molybdovanadate phosphoric acid, while sodium (Na) and potassium (K) by flame photometry (Jenway PFP7 Flame Photometer, Jenway Ltd., Felsted, Dunmow, Essex, UK) (Morales et al., 2016). In the mature composts, the physical properties were assessed following the procedures described by Bustamante et al. (2008a).

#### 2.4. Fluorescence analysis

Prior to the determination, the composting samples were used after drying at 65 °C. Then, an extraction was carried out using deionized water in the proportion 1:20 (w:v) and shaking it at ambient temperature during 24 h. After filtration of the extracts (Whatman No. 2 paper), 1 ml of sample was centrifuged for 10 min at 15000 rpm (SIGMA 2K15 centrifuge (rotor Nr. 12143)) (Marhuenda-Egea et al., 2007). A Jasco Model FP-6500 spectrofluorometer was used to acquire the fluorescence spectra, following the detailed conditions described in Marhuenda-Egea et al. (2007).

In this study, fluorescence spectra from the water-soluble organic matter pool of the four composting piles were characterized obtaining three main components in wavelengths ranges associated with the humic, fulvic and peptide fractions, according to Sierra et al. (2005). In order to identify the component peaks corresponding to the different fluorophores present in the sample, it was necessary to use chemometric tools, such as PARAFAC (Parallel Factor Analysis) (Murphy et al., 2014; Ohno et al., 2008). Raman and Rayleigh scattering peaks were removed with the technique of “excision-interpolation” (Zepp et al., 2004; Morales et al., 2016), before creating the model with PARAFAC. This type of analysis using PARAFAC has been described in detail by Ohno et al. (2008). Data modelling and pre-processing were done with MATLAB © (2005), using different algorithms N-way toolbox and eemscat toolbox (<http://www.models.life.ku.dk>). The CORCONDIA diagnosis was used to estimate the model that best collects the trilinear information (Murphy et al., 2014). Fluorescence was expressed as percentage of each of the three main compounds in order to assess the evolution of these pools during the composting process.

#### 2.5. NPK equivalent economical value

Nutrient economic value of the composts obtained was determined following the procedure used by Jara-Samaniego et al. (2017a). The average marketable €/ton values considered have been of 298.8 €, 204.9 €, and 203.9 €, respectively, using the same sources considered by Jara-Samaniego et al. (2017a), provided by the World Bank (December 2016). In addition, the percentage of fertilizing unit in Urea, DAP and Chloride potassium were 46% N, 46%  $P_2O_5$  and 60%  $K_2O$ , respectively. Taking into account these values, the average N,  $P_2O_5$ ,  $K_2O$  fertilizing units might be estimated in 4.4, 14.8 and 4.1 €,

respectively. In addition, a concentration of 25% of fresh weight in all the mature composts was considered to establish the economic value of the compost main nutrients.

#### 2.6. Statistical analysis

The Marquardt-Levenberg algorithm was used to fit OM losses during the process to a kinetic function, with the software Sigmaplot 11.0. OM degradation during composting was fitted using a kinetic model of first order (2), according to Bernal et al. (1996):

$$OM \text{ losses}(\%) = A(1 - e^{-kt}) \quad (2)$$

Where t corresponds to the composting time (days), A is the maximum degradation of OM (% C), while k is the rate constant ( $d^{-1}$ ). Root mean square (RMS) and the significance values (F-values) were calculated to compare the curve fitting statistical significance and the fittings of different functions, respectively.

The EXI<sup>2</sup> Index, based on the thermal profiles obtained from the piles, was calculated as the quadratic sum of the daily difference between the temperature inside the pile and in the close environment during the composting bio-oxidative phase.

ANOVA was used for the statistical analysis of the data obtained, followed by the test of least significant difference (LSD) at  $P < 0.05$ . The Shapiro-Wilk and Levene tests were used to check normality and homogeneity of the variances, respectively, before ANOVA. All the statistics was done with the software SPSS 15.0.

### 3. Results and discussion

#### 3.1. Thermal and exothermic profile

The temperature evolution is considered as one of the principal parameters to control composting development, since degradation is more rapid in the thermophilic stage and the presence of high temperatures constitutes a key factor for the sanitization of the mixture (Chen et al., 2014). All the composting piles had a rapid increase of the temperature values, reaching in the first week values higher than 40 °C, which were maintained during more than two weeks (Fig. 1). Himanen and Hänninen (2011) and Morales et al. (2016) also reported this rapid temperature increase in experiments of co-composting using sewage and agri-food sludge with several bulking agents. However, the general trend of the temperature values in the composting piles was different, as it can be observed not only in Fig. 1, but also in the values of the exothermic indices studied (Table 2). In piles P1 and P2, elaborated using SS and PL, was observed a longer duration of the thermophilic phase, while piles P2 and P3 showed the highest number of days with thermophilic temperature values ( $> 40$  °C). All these aspects were reflected in the highest values of the Quadratic Exothermic Index (EXI<sup>2</sup>) observed in these composting piles, showing P1 the greatest EXI<sup>2</sup> value, indicating that the thermophilic phase was the most intense in this mixture (Table 2). The shorter thermophilic phase observed in P4 could be due to the effect of the different origin of the sludge used (agri-food sludge), with different characteristics than sewage sludge (Table 1), but also due to the lower proportion of the bulking agent compared to the rest of mixtures. The effect of the proportion and characteristics of the bulking agent on the temperature development have been reported by different authors. Yañes et al. (2009) observed a longer thermophilic phase in the mixtures with a greater proportion of bulking agent in an experiment of composting of sewage sludge with garden pruning. Santos et al. (2016) also reported in an experiment of composting of pig slurry a longer exothermic phase in the mixture with the highest proportion of cotton waste as bulking agent.

#### 3.2. Organic matter mineralization

An important decrease was observed in all the piles for all the

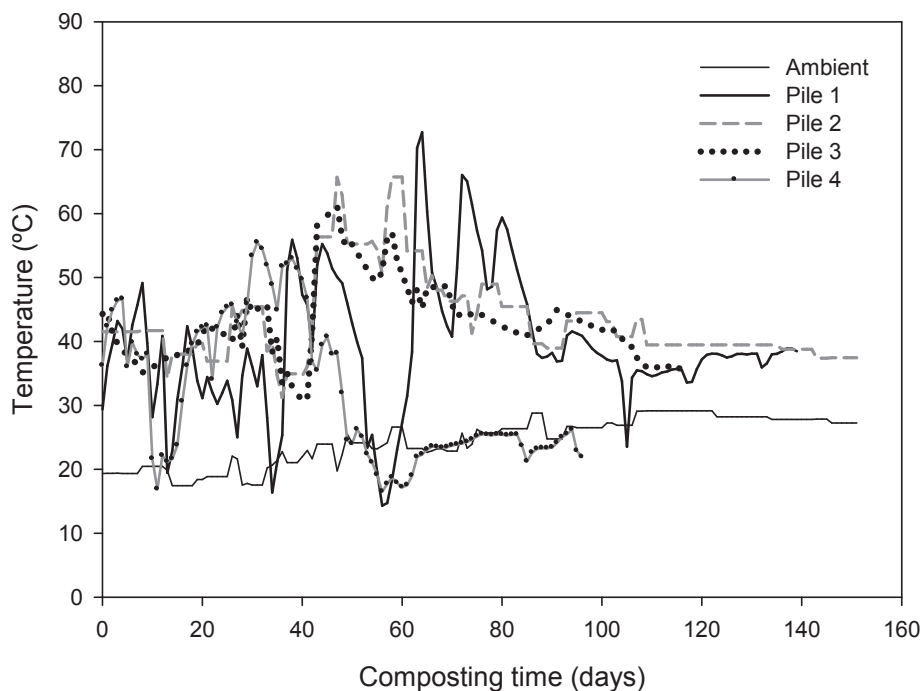


Fig. 1. Temperature profiles of Pile 1 (72.3% sewage sludge + 27.7% palm leaves); Pile 2 (67.7% sewage sludge + 32.2 palm leaves); Pile 3 (67.7% sewage sludge + 32.3% palm leaves); Pile 4 (82.5% agri-food sludge + 17.5% palm leaves).

**Table 2**  
Exothermic indices throughout the composting process.

Parameters	Pile 1	Pile 2	Pile 3	Pile 4
EXI <sup>2</sup> Index (°C <sup>2</sup> )	77,345	63,838	50,535	46,961
Ratio BP/TV <sup>a</sup>	139/49	152/71	116/80	96/27
Ratio EXI <sup>2</sup> /BP <sup>b</sup>	556	420	436	489

EXI<sup>2</sup>: quadratic exothermic index (quadratic sum of the daily difference between the average temperature of the pile and the ambient temperature).

Pile 1: 72.3% SS1 + 27.7% PL; Pile 2: 67.7% SS2 + 32.2 PL; Pile 3: 67.7% SS3 + 32.3% PL; Pile 4: 82.5% AS + 17.5% PL. SS: sewage sludge; AS: agri-food sludge; PL: palm leaves.

<sup>a</sup> Ratio BP/TV: Number of days of bio-oxidative phase/number of days with thermophilic values (temperature values > 40 °C).

<sup>b</sup> Ratio EXI<sup>2</sup> index/BP: values of the EXI<sup>2</sup> index/number of days of bio-oxidative phase.

mixtures throughout the composting process, from values of 82.4%, 78.6%, 80.4% and 80.8% for piles P1, P2, P3 and P4, respectively, to values of 44.3%, 63.7%, 61.5% and 70.1% (for piles P1, P2, P3 and P4, respectively), indicating the organic matter degradation process (Table 3). This decrease was especially significant during the thermophilic phase, when the microbial activity was maximum and the temperature reached the highest values (Bustamante et al., 2013). Organic matter losses were substantial in all the piles (Fig. 2), especially in the mixture P1, which reached the most intense thermophilic phase, as it was reflected in the highest value of the EXI<sup>2</sup>. The lowest degradation of OM was found in the maturity stage, which indicates the stabilization of the material after the bio-oxidative stage. This fact has been reported in previous co-composting studies using different raw materials, such as winery-distillery wastes and manures (Bustamante et al., 2008b), anaerobic digestates and vine shoot pruning (Bustamante et al., 2012) and agri-food sludge with different bulking agents (Morales et al., 2016).

The organic matter degradation profile throughout the composting process, determined by the losses of organic matter, were fitted to a kinetic equation of first-order (OM losses (%) = A (1 - e<sup>-kt</sup>)). The parameter values (standard deviation in brackets) obtained after fitting

the experimental results are shown in Table 4. All the equations had high significance ( $P < 0.001$ ), showing that all the piles fitted the first-order kinetic model. The A and k values obtained were slightly higher than those obtained by Alkoik et al. (2011), in an experiment of composting of date palm wastes, and those found in other composting experiments using sewage sludge (Banegas et al., 2007) and agri-food sludge (Morales et al., 2016). However, these values were close to those reported in composting studies using different organic wastes, such as pig slurry and anaerobic digestates (Santos et al., 2016; Bustamante et al., 2012). In addition, the origin of sludge (sewage or agri-food sludge) influenced the degradation rate (k), showing the lowest value for the mixture with AS.

### 3.3. Changes in the physico-chemical and chemical properties

At the beginning of composting, pH in all the mixtures was close to neutral values and within the range 6–8, considered adequate for composting (Bernal et al., 2009). Throughout the composting process, the pH values slightly decreased or practically did not change (Table 5), obtaining in the final phase of the process pH values in the interval 6.0–8.5, considered adequate for the agricultural use of compost (Hogg et al., 2002), and also within the range established (5.2–6.3) for their use as substrate, except for P4 (Bustamante et al., 2008a).

The initial values of the electrical conductivity were greater than 4 dS/m in the mixtures, probably as a consequence of the use of PL, which showed the highest salt contents (7.3 dS/m) (Table 1). During composting, this parameter increased due to the production of inorganic compounds as a consequence of OM mineralization and the rising relative contents of ions due to the pile weight loss (Bustamante et al., 2008b). In the mature composts, P1 and P2 had the highest values of EC, although all the composts had EC values higher than 7 dS/m, which could suppose a limitation for their agricultural use (Shak et al., 2014).

The C<sub>T</sub>/N<sub>T</sub> fell in all the piles during composting, especially in the bio-oxidative phase, coinciding with the maximum OM degradation (Bustamante et al., 2008b), this behaviour being also observed during the co-composting of date palm residues mixed with animal wastes

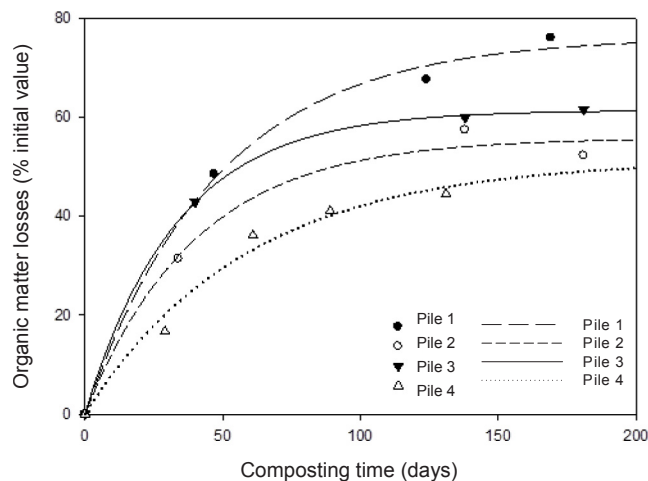
**Table 3**

Evolution of the physico-chemical parameters and of the main chemical parameters associated to organic matter evolution during composting (dry weight basis).

Composting phase	pH	EC (dS/m)	OM (%)	C <sub>T</sub> /N <sub>T</sub>	WSC (%)	Ch <sup>a</sup> (%)	Cf <sup>a</sup> (%)	Peptides <sup>a</sup> (%)
<i>Pile 1: 72.3% SS1 + 27.7% PL</i>								
I	6.40	5.3	82.4	23.1	4.75	42.0	22.2	35.7
T	6.00	8.6	70.7	11.1	1.69	53.0	22.4	24.6
EB	6.00	10.1	60.3	9.5	0.76	67.4	23.7	8.9
M	6.10	9.3	44.3	8.5	0.67	68.5	20.9	10.7
LSD	0.06	0.5	4.29	1.8	0.33	0.2	0.8	1.0
<i>Pile 2: 67.7% SS2 + 32.3% PL</i>								
I	6.70	4.7	78.6	11.6	2.41	43.1	20.8	36.1
T	6.20	6.3	71.6	11.3	1.38	50.0	23.6	26.4
EB	5.90	9.4	61.0	9.6	0.85	63.6	22.7	13.7
M	6.00	8.9	63.7	8.7	0.52	58.5	21.7	19.8
LSD	0.05	0.2	2.82	0.8	0.92	0.2	0.1	0.3
<i>Pile 3: 67.7% SS3 + 32.3% PL</i>								
I	6.60	5.0	80.4	15.1	2.24	32.8	20.6	46.6
T	6.20	8.1	70.1	11.5	1.53	54.8	22.3	22.9
EB	6.30	9.6	62.2	9.4	0.87	66.0	22.7	11.3
M	6.10	7.8	61.5	9.7	0.57	61.5	20.5	18.0
LSD	0.10	0.3	1.44	1.71	0.37	1.4	1.0	0.2
<i>Pile 4: 82.5% AS + 17.5% PL</i>								
I	7.40	5.7	80.8	16.0	1.14	40.5	9.5	50.0
T	7.50	6.5	77.9	14.7	1.01	46.6	9.3	44.1
EB	7.50	7.9	71.3	11.6	0.89	54.5	14.9	30.6
M	7.40	8.3	70.1	12.0	0.87	59.3	13.4	27.3
LSD	0.04	0.22	0.86	0.59	0.50	8.7	8.6	12.4

LSD: least significant difference at  $P < 0.05$ .

<sup>a</sup> Relative percentage in relation to the total sum of the three fractions (Cf, Ch and peptides). EC: electrical conductivity; C<sub>T</sub>: total organic carbon; N<sub>T</sub>: total nitrogen; OM: organic matter; WSC: water-soluble carbon; Ch: humic acid-like carbon; Cf: fulvic acid-like carbon. I: initial phase of composting; T: thermophilic phase of composting; EB: end of the bio-oxidative phase; M: maturity phase.



**Fig. 2.** Organic matter losses during the composting of Pile 1 (72.3% sewage sludge + 27.7% palm leaves); Pile 2 (67.7% sewage sludge + 32.2 palm leaves); Pile 3 (67.7% sewage sludge + 32.3% palm leaves); Pile 4 (82.5% agri-food sludge + 17.5% palm leaves). Lines represent curve-fitting.

**Table 4**

Parameter values obtained (standard deviation in brackets) after fitting organic matter losses to a first-order kinetic function (OM losses (%) =  $A(1 - e^{-kt})$ ).

Parameters	A	k	RMS	F-values
Pile 1	76.13 (2.94)	0.0208 (0.0028)	6.68	517***
Pile 2	55.78 (2.76)	0.0250 (0.0049)	10.32	195***
Pile 3	61.37 (0.37)	0.0299 (0.0008)	0.22	1092***
Pile 4	51.31 (5.67)	0.0171 (0.0042)	7.57	184***

A: maximum degradation of OM (%C); k: rate constant ( $d^{-1}$ ); RMS: residual mean square. \*\*\*: Significant at  $P < 0.001$ .

Pile 1: 72.3% SS1 + 27.7% PL; Pile 2: 67.7% SS2 + 32.2 PL; Pile 3: 67.7% SS3 + 32.3% PL; Pile 4: 82.5% AS + 17.5% PL. SS: sewage sludge; AS: agri-food sludge; PL: palm leaves.

**Table 5**

Main chemical and physical properties of the final composts.

	Pile 1	Pile 2	Pile 3	Pile 4
<i>Maturity parameters</i>				
CEC (meq/100 g MOT)	84	196	136	179
CEC/C <sub>T</sub> (meq/g C <sub>T</sub> )	1.24	4.10	2.70	3.51
GI (%)	60	114	68	87
<i>Nutrient contents</i>				
N <sub>T</sub> (g/kg)	35	35	32	30
P (g/kg)	13.9	15.5	16.3	5.4
K (g/kg)	14.6	14.8	14.2	23.5
<i>Physical properties</i>				
Bulk density (g/cm <sup>3</sup> )	0.20	0.20	0.22	0.17
TPS (%)	90.2	89.6	88.7	89.9
Shrinkage (%)	15.3	30.7	28.9	24.4
TWHC (mL water/L)	313	413	449	333
Air capacity (%)	58.8	48.3	43.8	56.6

CEC: cation exchange capacity; C<sub>T</sub>: total organic carbon; GI: Germination index; N<sub>T</sub>: total nitrogen; BD: Bulk density; TPS: total pore space; TWHC: total water holding capacity.

Pile 1: 72.3% SS1 + 27.7% PL; Pile 2: 67.7% SS2 + 32.2 PL; Pile 3: 67.7% SS3 + 32.3% PL; Pile 4: 82.5% AS + 17.5% PL. PL: palm leaves; SS: sewage sludge; AS: agri-food sludge.

(Alkoaik et al., 2011). This ratio is suggested as an index of maturity, with values  $< 20$ , which is considered as indicative of maturity (Bernal et al., 2009). However, piles P2, P3 and P4 showed values of this parameter lower than 20 at the beginning of the process, which shows that this ratio should not be used as absolute maturity indicator, but rather its changes throughout the process may indicate the degradation of OM during composting (Bustamante et al., 2008b, 2013).

All piles had an important drop of the water-soluble fraction of organic C, indicating the decomposition of the simple water-soluble organic fraction constituted by sugars, peptides and amino acids (Bustamante et al., 2012). At the end of composting, the WSC values in the end-products ranged from 0.52 to 0.87%, lower than the maximum values suggested in other studies as indicator of compost maturity ( $< 1\%$  or  $< 1.7\%$ ) (Bernal et al., 2009) and similar to the values found

in previous co-composting studies using sewage sludge (Banegas et al., 2007) and agri-food sludge (Morales et al., 2016).

### 3.4. EEM fluorescence spectra

The fluorescence excitation–emission matrix (EEM) spectroscopy provides detailed information concerning the fluorescence properties of water-soluble organic matter. This information allows the assessment of organic matter changes throughout the composting process. A three-dimensional picture is obtained from the fluorescence intensity as a function of excitation and emission wavelength (Marhuenda-Egea et al., 2007; Murphy et al., 2014).

The measured EEM data obtained were processed to remove Rayleigh and Raman dispersions (Zepp et al., 2004). In addition, the spectra obtained showed different fluorophores, defined by a wavelength pair of Ex/Em and that contribute to the EEM spectrum final contour. These fluorophores or components were detected using Parallel Factor Analysis (PARAFAC), since they may be related with bio-compounds (e.g. peptides and/or fulvic and humic substances) (Ohno and Bro, 2006). Using a mathematical model with these components, it was possible to obtain a good representation of the measured data. The difference between the measured and the modelled data constitutes the residual, i.e. the possible data that remain without modelling. If the model is correct, the residual correspond to noise and artifacts. However, it is important to revise them to confirm that important data were not out of the model. Fig. 3 shows a representation of the measured data, model and residual, for the initial sample and for the mature sample only of the pile P3, since the model was similar for the rest of piles. The PARAFAC model identified three components. These components were constituted by two principal ones: Component 1 ( $\lambda_{\text{ex}} \sim 245$  and  $335$  nm, and  $\lambda_{\text{em}} \sim 332$  and  $446$ ), and Component 2 ( $\lambda_{\text{ex}} \sim 225$  and  $280$  nm, and  $\lambda_{\text{em}} \sim 310$  and  $352$ ), and the secondary Component 3, associated to fulvic acid-like compounds ( $\lambda_{\text{ex}} \sim 240$  and

$290$  nm, and  $\lambda_{\text{em}} \sim 384$ ) (Fig. 4). Component 1 was associated to aromatic rings present in large organic molecules such as humic acids (Sierra et al., 2005; Ohno and Bro, 2006; Morales et al., 2016), and Component 2 was associated to peptides and proteins containing tyrosine and tryptophan (fluorescent amino residues) (Leenheer and Croué, 2003; Yamashita and Tanoue, 2003; Morales et al., 2016). Component 3 could be associated also with aromatic rings presented in fulvic acid-like molecules (Leenheer and Croué, 2003; Morales et al., 2016).

The selection of the number of fluorophores (or components) to construct the model constitutes an important factor to obtain a good model. A large number of fluorophores can create many mathematical artifacts, but a low number may leave important data outside the model. In composting processes, it has been possible to find two or three fluorophores (Marhuenda-Egea et al., 2007). However, for unknown samples the choice of fluorophores is optimized with the value of CORCONDIA, which must be close to 100%. In the PARAFAC model used, the three fluorophores selected provided a value of CORCONDIA of 91.2%. After reviewing the residuals, it can be confirmed that these three selected components allow to obtain a suitable model for the experimental data.

The changes considered with this technique mainly correspond to the water-soluble fraction. Table 3 shows the contribution of each fraction evaluated (humic and fulvic acid-like C, peptides). Each fraction showed a different evolution, since the fraction associated to Component 1 (humic acid-like C) increased during composting, while the fractions associated to Components 2 and 3 (peptides and fulvic acid-like C) decreased during the process, indicating the generation of humic acid-like compounds throughout composting (Antizar-Ladislao et al., 2006) and the degradation of the initial materials due to microbial activity (Marhuenda-Egea et al., 2007). In addition, the contribution of the Component 2 decreased more rapidly, as the peptides were rapidly metabolized by the microorganisms responsible for the composting process (Marhuenda-Egea et al., 2007). In the measured

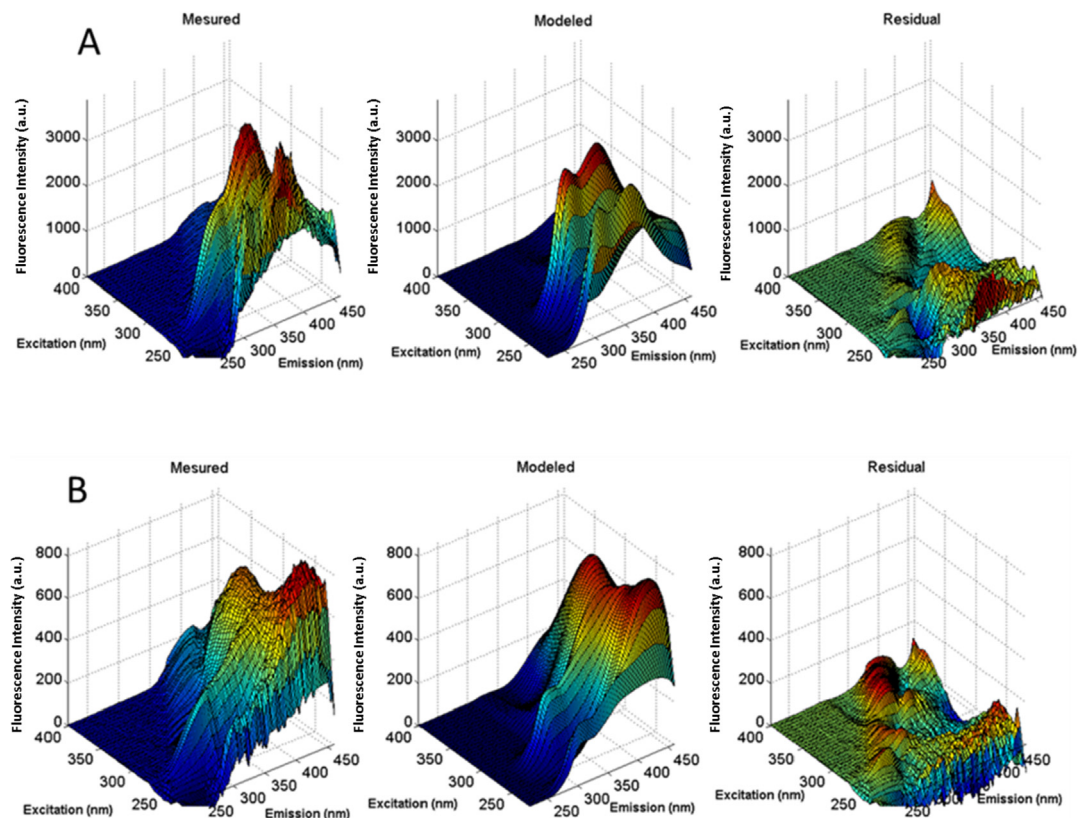


Fig. 3. Comparison of the measured (left), PARAFAC modelled (middle) and residual (right) for the initial sample (A) and maturity sample (B) of the Pile 3 (67.7% sewage sludge + 32.3% palm leaves). a.u.: arbitrary units.

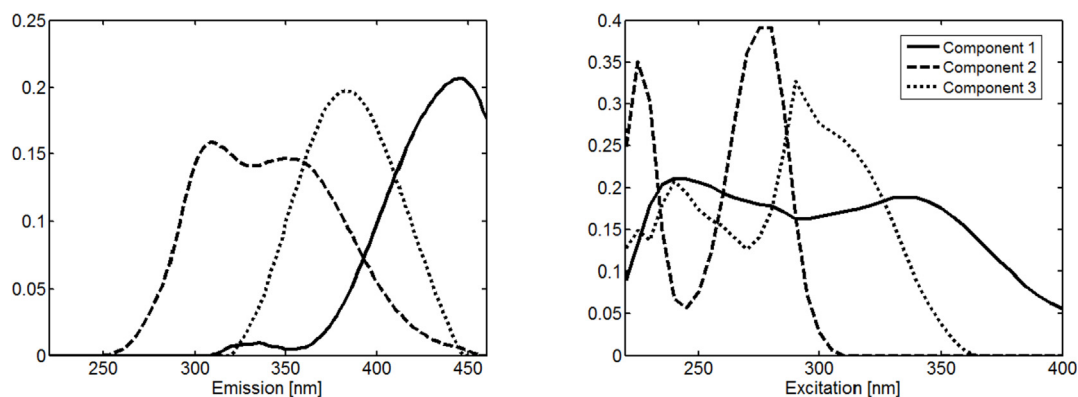


Fig. 4. Excitation and emission spectra of three EEM components (fluorophores) identified by the PARAFAC model.

data, it can be also observed this decrease in the peaks associated with this protein component (Fig. 4). The peptides contained in the raw materials used for the composting mixtures constitute a source of nitrogen easily-available for the microorganisms that participate in the composting process, which degrade rapidly these compounds in the first stages of the composting process.

### 3.5. Quality and economic value of the final composts

The principal chemical and physical characteristics of the final composts obtained are shown in Table 5. Concerning the parameters used to estimate compost maturity, all the composts fulfilled these indicators, since the values obtained were within the established range (Bernal et al., 2009). The CEC and the ratio CEC/C<sub>T</sub> are parameters usually considered as humification indicators (Bernal et al., 2009). In the final composts, the values of both parameters were higher than those established by different authors (CEC > 67 meq/100 g OM; Iglesias Jiménez and Pérez García, 1992a and CEC/C<sub>T</sub> > 1.9 meq/g C<sub>T</sub>; Iglesias Jiménez and Pérez García, 1992b), indicating an adequate maturity. In addition, the values of the germination index (GI) in all the composts were higher or similar to 60%, fulfilling the criterion that indicates maturity and absence of phytotoxicity in composts (Zucconi et al., 1981).

The macronutrient (NPK) concentrations (Table 5) were higher or similar to those found by other authors in studies of composting using municipal solid waste (Jara-Samaniego et al., 2017a), sewage sludge (Himanen and Hänninen, 2011; Doublet et al., 2011) and/or agri-food sludge (Morales et al., 2016), and similar to those reported in co-composting experiments with manures and other agro-industrial wastes (Bustamante et al., 2008b, 2012, 2013). P contents were higher in the piles elaborated with SS (P1, P2 and P3), while the contents of K were higher in the pile elaborated using AS (P4). The higher contents of K of P4 has been also found in previous experiments of composting using organic wastes with an agri-food origin (Bustamante et al., 2008b, 2010; Morales et al., 2016).

On the other hand, the physical properties of a material establish its suitability at a physical level for its use as growing medium. In general, the final composts showed adequate physical characteristics as growing medium components for soilless crop production. On the one hand, the values of the bulk density (BD) and the total pore space (TPS) were within the interval suggested for an “ideal substrate” (BD ≥ 0.40 g/cm<sup>3</sup> and TPS > 85%) (Abad et al., 2001) and the values of the shrinkage practically satisfied the limit established in all the composts (< 30%). On the other hand, the total water holding capacity (TWHC) showed lower values than the optimum value considered (550–800 mL/L), but this fact is according to the values observed of the air capacity, higher than the limiting range established by Abad et al. (2001) (20–30%). Morales et al. (2016) also reported this aspect in an experiment of co-composting agri-food sludge with different bulking agents. The higher

values of air capacity can favour their use as substrate since the lack of aeration is a limiting aspect, but the low TWHC can be solved with frequent irrigation, in small amounts to avoid leaching (Bustamante et al., 2012).

Concerning the economic value of the composts obtained considering their nutritional contents, Table 6 shows the value (€/ton) for each nutrient and the total value in the final composts. The results obtained show that the economic value in these materials is higher than that obtained in other previous studies using composts from municipal solid wastes (Jara-Samaniego et al., 2017a; b), probably due to the higher nutrient content present in sludge (sewage or agri-food sludge). These results show the added-value of the composts, related to their nutritional contents, and the different potential economic value depending on the raw materials used in their preparation.

## 4. Conclusions

The results obtained have shown that the co-composting of date palm waste with different types of sludge (sewage and agri-food sludge) is a viable treatment to manage these organic wastes. The suitable development of the process in all the mixtures allowed to achieve conditions for the sanitation of the materials. In addition, the composts obtained showed an additional economic value as a consequence of their high nutrient contents, and good physical characteristics, suitable for their potential use as growing medium and/or growing medium ingredient for soilless crop cultivation. However, the salt contents of these materials, especially in the composts elaborated with the highest proportions of sewage sludge (compost 1 and 2) could be a restricting issue for their agronomical use, so further studies are necessary to optimize the proportion of palm waste in the composting mixture to achieve a reduction of the salinity in the final compost.

Table 6

Economic value of each compost considering the nutrient content (€/ton) (N, P<sub>2</sub>O<sub>5</sub> and K<sub>2</sub>O).

Nutrient <sup>a</sup>	Pile 1	Pile 2	Pile 3	Pile 4	Average
Total N	11.8	11.7	10.7	10.0	11.1
P <sub>2</sub> O <sub>5</sub>	35.5	39.6	41.6	13.8	32.6
K <sub>2</sub> O	5.36	5.43	5.21	8.63	6.16
Total combined value	52.7	56.7	57.6	32.4	49.8

Pile 1: 72.3% SS1 + 27.7% PL; Pile 2: 67.7% SS2 + 32.2% PL; Pile 3: 67.7% SS3 + 32.3% PL; Pile 4: 82.5% AS + 17.5% PL. SS: sewage sludge; AS: agri-food sludge; PL: palm leaves.

<sup>a</sup> The economic value of the nutrient contents has been calculated considering a 25% of fresh matter in the composts.

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