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Developing Organic Fertilizer Through Co-Composting Olive Mill Wastewater

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ABSTRACT

The main objective of this study was to evaluate the potential of olive mill wastewater (OMW) as an organic fertilizer through co-composting with various agricultural by-products. OMW was mixed with agricultural by-products, including maize silage, sugar beet pulp, and sugarcane bagasse, in controlled proportions and conditions. The study was conducted at the National Institute of Agricultural Research in Rabat, Morocco. The composting process was monitored over time, focusing on the evolution of key physicochemical parameters and phenol content of each mixture. The results showed that the performance of the composts varied, with the mixture containing sugar beet pulp (SBPO) exhibiting the most promising results, followed by maize silage (MSO) and sugarcane bagasse (SBO). These results suggest that co-composting OMW with agricultural by-products can produce high-quality organic fertilizers, thus reducing the need for inorganic alternatives and providing a sustainable waste management solution in the olive oil industry. It highlights the potential for reducing phenols characteristic of OMW and promoting sustainable agricultural practices. The application of the composts to crops was not tested, highlighting the need for further research in this regard. Future investigations should focus on evaluating the long-term effects of OMW-derived composts on soil health and crop productivity. This study explored a combination of materials that, to the authors' knowledge, has not been previously documented in scientific literature. The results underscore the importance of sustainable waste management practices and their potential role in improving soil fertility and reducing the environmental impact associated with olive oil production.

Keywords: olive mill wastewater, agricultural by-products, organic fertilizer, biodegradation, circular economy, composting, biomass.

INTRODUCTION

Over the course of two decades, there was a remarkable 53% increase in the global production of primary crops from 2000 to 2019 (Tortajada and González-Gómez, 2022). As agricultural production has grown, there has been an increase in the application of chemical fertilizers, resulting in the generation of nitrous oxide (N, O) from the application of synthetic fertilizer. Upon applying urea to soils, carbon dioxide (CO_2) is released, which was originally fixed during the industrial manufacturing process (Chataut et al., 2023). Thus, agriculture makes a significant contribution, constituting roughly 10–14% of the total global greenhouse gas (GHG) emissions (Shakoor et al., 2020). Concerning phosphorus fertilizer, a considerable amount is annually extracted

for fertilizer production, but a noteworthy proportion eventually makes its way into rivers, lakes, and oceans, resulting in eutrophication (Chataut et al., 2023). Regarding potassium fertilizer, potassium chloride (KCl) is the main source of potassium (K) used in agriculture. In China, the production of KCl results in the emission of 0.19 kg CO_2 -eq.kg⁻¹ of potassium oxide (K₂O), which is equivalent to 0.11 kg CO_2 eq.kg⁻¹ of KCl (Chen et al., 2018). Chemical fertilizers are contributing to substantial environmental pollution by diminishing the water-retaining ability and fertility of soil, elevating soil acidity, and reducing the microorganism population (Nosheen et al., 2021).

In the context of agricultural practices, essential types of amendments are employed to improve soil fertility and productivity. These include manure, compost, and biochar (Védère et al., 2022). Organic amendments, rich in organic matter (OM) and carbon, directly increase soil organic matter (SOM) concentrations and improve soil water contents. This enhancement in soil water is achieved through two main processes: first, the amendments absorb and retain water, and second, they enhance soil structure and porosity, leading to better water infiltration and storage. These organic amendments offer a promising solution to elevate SOM levels and improve soil water dynamics, contributing to more resilient and productive agricultural systems (Sisouvanh et al., 2021).

Another captivating opportunity arises in the valorization of organic waste, with a particular emphasis on the wastewater produced during olive oil extraction. Substituting inorganic fertilizers with organic amendments derived from olive mill wastes offers a promising and effective approach to mitigate the detrimental environmental impacts associated with olive oil production (El Joumri et al., 2023). Combining OMW with agricultural residues, and agricultural by-products, such as olive pomace, straw materials, or animal residues, can enhance composting. As a consequence, the end product acquires enrichment with essential nutrients and OM (Muktadirul Bari Chowdhury et al., 2013). In accordance with (Tapia-Quirós et al., 2020), the quantity of olive pomace and OMW produced from one ton of olives can vary based on the method utilized in the extraction of oil, typically resulting in approximately 400 kg of olive pomace and 1200 L of OMW. This wastewater holds a range of both organic and inorganic compounds, with the main components being

polyphenols, tannins, lipids, organic acids, suspended solids, and nutrients. Insufficient management of OMW can lead to the contamination of agricultural land, resulting in detrimental impacts on soil fertility and crop growth. Furthermore, the discharge of OMW not only affects the soil ecosystem but also has significant implications for the aquatic environment, as demonstrated by an investigation conducted by (Pavlidou et al., 2014) which revealed significant quantities of phenolic compounds and elevated levels of both ammonium and inorganic phosphorus in small streams. These contaminants actively contribute to the deterioration of these aquatic environments, especially prominent during the olive oil production peak in November and December. After the peak season of the olive oil production, it takes over five months for the ecosystem to recover.

The primary goal of this work was to explore the potential valorization of OMW and agricultural by-products for their utilization as soil amendments. On the basis of literature review analyzing 69 mixtures derived from olive mill by-products (EL Joumri et al., 2024), this study explored a combination of materials that, to the best of authors' knowledge, has not been previously documented in scientific literature. Therefore, this research aimed to bridge this gap by exploring the properties and interactions of these mixtures, thus providing an innovative contribution to the field. The study involved monitoring of changes in phenolic content and various physicochemical parameters throughout the experimental process. By investigating the suitability of these materials for soil amendment purposes, the study aimed to contribute to sustainable agricultural practices and enhance soil health.

MATERIALS AND METHODS

Composting procedure

Three mixtures were developed using OMW combined with different air-dried agricultural by-products. Maize silage (MS), sugar beet pulp (SBP), and sugarcane bagasse (SB) were airdried and used as raw materials in combination with OMW for the process of co-composting. These dried agricultural by-products were coarsely shredded, and soaked in OMW for 48 hours to reach saturation as the aim was to absorb the maximum amount of OMW for effective valorization. The fraction used is $(1:6.67)$ (w:v). The excess OMW was set aside, as it was not used in the composting process. The three composts displayed different OMW holding capacities, with absorption rates of 50%, 50%, and 75%, respectively, for agricultural by-products MS, SBP, and SB. The resultant products were laid in perforated plastic containers to allow aeration and stored at room temperature for a duration of 7 months. Manual agitation was performed consistently every week for the mixtures which ensured both homogenization and aeration. Additionally, they were moistened every 15 days to maintain the moisture level necessary for microbial activity and decomposition. To assess the physicochemical characteristics of both mixtures (agricultural by-products combined with OMW) and the individual agricultural by-products, representative samples of each type were subjected to drying in an oven at 60 °C, followed by grinding and sieving to achieve a particle size of 0.2 mm. The raw materials were individually analyzed before they were mixed together. Table 1 provides the physicochemical characteristics of agricultural by-products and OMW. Samples were collected at different time points (initial state after 48 hours of immersion in OMW, and at 7, 42, 70, 148, and 207 days) to monitor the composting process. In this research, the initial materials in the mixtures were established by considering their wet weight, and they are as follows:

- MSO: 23.08% maize silage + 76.92% OMW
- SBPO: 23.08% sugar beet pulp + 76.92% OMW

• SBO: 16.67% sugarcane bagasse + 83.33% OMW

Data^a of OMW on fresh weight basis; (K, Na, Ca, Mg expressed in mEq/L; TPC expressed in mg(GAE)/L).

Analytical methods

EC and pH measurements of the co-composted by-products were conducted in water suspensions at dilution ratios of 1:10 and 1:5 (w/v), respectively. The OM was determined by heating the dry sample at 550 \degree C in a muffle furnace for 4 hours. OC was calculated through the assessment of OM loss after ignition. Total nitrogen was determined using the Kjeldahl method. Following the 4-hour calcination at 500 °C, the resulting ashes were dissolved to analyze the concentrations of P, Na, K, Ca, and Mg. P was analyzed using the Olsen method (Olsen, 1954). The K and Na concentrations were determined using a flame photometer (Bower et al., 1952). Ca and Mg were measured using the method described by (Pelloux et al., 1971). The total phenolic content was determined using the Folin-Ciocalteu method, as described by (El Moudden et al., 2020), with slight modifications: 1 mL of the sample solution was mixed with 2 mL of Folin-Ciocalteu reagent diluted with distilled water at a 1:10 ratio. Then, 4 mL of $\text{Na}_2\text{CO}_3(7.5\%, \text{w/v})$ were added to the mixture. The absorbance at 765

Parameters	Maize silage	Sugar beet pulp	Sugarcane bagasse	Olive mill wastewater
рH	5.53	3.7	4.75	4.85 ^a
EC (mS/cm)	7.4	1.84	1.31	13.66a
OM (%)	96.43	97.91	98.76	82.23
Ash $(%)$	3.17	1.82	1.12	17.77
OC(%)	55.93	56.79	57.29	47.69
$TKN(\%)$	2.25	2.08	0.54	
C/N	24.85	27.25	106.57	
P(%)	0.20	0.05	0.042	
$K(\%)$	1.59	0.2	0.3	1.79a
Na (%)	0.11	0.13	0.03	$0.05^{\rm a}$
Ca (%)	0.32	0.36	0.04	20 ^a
Mg (%)	0.34	0.36	0.12	100 ^a
TPC (µg GAE/g DW)	1919.48	2255.69	1997.07	23.73^a

Table 1. Characteristics of the OMW, MS, SBP, and SB (dry weight basis)

Note: EC: electrical conductivity, OM: organic matter, TKN: total Kjeldahl nitrogen, P: phosphorus, K: potassium, Na: sodium, Ca: calcium, Mg: magnesium, TPC: total phenol content.

nm was determined using a UV-Vis spectrophotometer. The solution was left to stand in the dark for 30 minutes.

RESULTS

Table 2 illustrates the evolution in the physicochemical characteristics during the composting of OMW and agricultural by-products mixture. All the mixtures experienced a pH increase during composting. Throughout composting, the pH of the SBPO mixture displayed a continual rise, reaching its highest level by the end of the process, as shown in Figure 1. MSO and SBO exhibited a similar temporal evolution, with an initial increase in pH until day 42, followed by a decrease in pH at day 70, and a subsequent rise extending until the end of the process.

The EC of the three mixtures decreased throughout the composting process. The initial concentrations of OM were approximately the same for the three mixtures. However, the SBPO compost showed a higher rate of OM degradation. SBO has the highest value of OM. The ash content of the three composts increased during composting. During composting, the TKN values showed an increase in all mixtures. The highest final TKN content (4.37%) was observed in the SBPO mixture. The MSO compost takes the second position, showing TKN values of 3.75%. This substantial increase is most probably a result of the loss of compost mass. At the end of the list is SBO, showing a value of 2.14%. Mixture SBO exhibited the least significant N increase

In all three composts, the C/N ratio decreased substantially. It can be deduced that only the MSO and SBPO composts have achieved maturity, as their C/N ratios fall within the desired range. The C/N ratio of the SBO compost (24.93) is higher than the recommended range. The SBO mixture exhibited the least significant decrease in C/N ratio, as illustrated in Figure 2.

During composting, an increase in P was observed in the MSO, SBPO, and SBP mixtures. Throughout the composting process, the K, Na, Ca, and Mg content displayed irregular fluctuations, with both increases and decreases noted in all composts. Figure 3 shows that the phenols were completely degraded after 148 days of composting for MSO, and after 207 days for SBPO. However, there was no complete degradation of phenols for SBO; instead, SBO showed a significant decrease in phenol.

DISCUSSION

The pH increase observed in all the mixtures during composting was confirmed by (Tomati, 1996). In the initial phase of the process, a substantial increase in microorganisms and biological reactions occurs, resulting in an elevation of pH levels. As time advances, the process induces the mineralization of peptides, amino acids, and proteins into ammonia, leading to a subsequent rise in pH (Senesi, 1989, Paredes et al., 2002, Baeta-Hall et al., 2005).

Figure 1. Evolution of pH of composts over time

Figure 2. Evolution of C/N ratio of composts over time

Figure 3. Evolution of TPC of composts over time

Table 2. Evolution of the main parameters during the composting process (dry weight basis)

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Composting time (days)	pH	EC (mS/ cm)	OM $(\%)$	Ash (%)	OC $(\%)$	TKN (%)	C/N	P (%)	K (%)	Na (%)	Ca (%)	Mg (%)	TPC $(\mu g \text{ GAE/g})$
MSO													
0	5.85	8.8	94.53	4.69	54.83	2.48	22.11	0.2	3.14	0.22	0.4	0.38	4912.59
$\overline{7}$	6.44	8.45	92.82	6.29	53.84	2.6	20.71	0.21	2.78	0.17	0.4	0.38	3148.79
42	7.71	8.34	91.9	7.63	53.31	2.6	20.5	0.45	3.69	0.14	0.4	0.36	1876.38
70	6.84	6.75	91.47	8.28	53.06	2.59	20.49	0.42	1.57	0.14	0.4	0.36	1050.52
148	8.13	5.86	89.95	9.57	52.18	2.68	19.47	0.78	2.37	0.15	0.4	0.36	0
207	8.15	3.52	89	10.4	51.62	3.75	13.77	0.78	1.93	0.14	0.4	0.36	0
SBPO													
$\mathbf{0}$	4.43	5.01	95.86	3.51	55.6	2.49	22.33	0.09	1.84	0.18	0.4	0.46	4767.76
$\overline{7}$	5.05	4.65	94.34	4.67	54.72	2.72	20.12	0.12	1.65	0.15	0.4	0.46	4233.28
42	5.05	4.24	90.63	8.31	52.57	2.61	20.14	0.18	1.68	0.13	0.56	0.46	3135
70	5.65	4.15	90.84	8.69	52.69	2.52	20.91	0.19	1.35	0.2	0.6	0.46	2160.86
148	6.73	3.93	90.38	9.34	52.42	2.77	18.92	0.24	1.83	0.18	0.6	0.46	267.76
207	8.41	3.18	88.31	10.89	51.22	4.37	11.72	0.25	1.8	0.14	0.6	0.46	0
SBO													
0	4.9	5.3	96.89	2.5	56.2	1.18	47.63	0.11	2.32	0.07	0.16	0.22	4767.76
$\overline{7}$	4.94	4.93	96.2	2.95	55.8	1.37	40.73	0.13	2.07	0.06	0.16	0.22	3878.1
42	5.5	3.95	95.67	3.87	55.49	1.34	41.41	0.14	1.79	0.06	0.16	0.22	2685
70	4.5	3.78	95.48	4.36	55.38	1.41	39.28	0.15	1.09	0.13	0.12	0.22	1917.76
148	5.88	2.84	95.44	4.44	55.36	1.53	36.18	0.15	1.36	0.1	0.12	0.22	814.31
207	7.01	2.28	91.99	6	53.36	2.14	24.93	0.15	1.41	0.12	0.12	0.22	385

Note: EC: electrical conductivity, OM: organic matter, TKN: total Kjeldahl nitrogen, P: phosphorus, K: potassium, Na: sodium, Ca: calcium, Mg: magnesium, TPC: total phenol content.

The decline in pH observed on day 70 for MSO and SBO was similar to that reported in a study by (Paredes et al., 2005), where the composts derived from sewage sludge, cotton gin waste, and OMW displayed a similar trend. This observation could be linked to the initiation of the nitrification process.

The elevated EC is attributed to the inclusion of OMWs in the initial mixture (Hachicha et al., 2009). A study on a compost based on maize straw and OMW reported that the EC also decreased throughout the composting cycle (Paredes et al., 2002). Leaching during composting can result in a decrease in EC values due to the loss of soluble salts (Paredes et al., 2002, Abid and Sayadi, 2006, Said-Pullicino et al., 2007). Due to the high level of lignin, the residues exhibit a high OM and carbon content (Asses et al., 2018). Chowdhury et al. (2015) reported comparable findings in their study of composts. The initial percentage of OM of their composts ranged from 95.36% to 96.49%. By the end of composting, OM ranged from 88.10% to 89.0%. Regarding OC, the initial OC percentage in the four composts ranged from 55.31% to 55.96%. By the end of composting, the OC values varied within the range of 51.10% to 51.60% (Chowdhury et al., 2015). The SBO mixture showed ash content variations similar to the compost composed of OMW, solid olive mill waste, and household refuse (Barje et al., 2008). The MSO and SBPO mixtures, on the other hand, exhibited comparability to the compost studied by (Baddi et al., 2004), which included OMW, solid olive mill waste, and wheat straw.

Regarding TKN, The results of this study align with the outcomes documented in (Paredes et al., 2005, Chowdhury et al., 2015). This substantial increase is most probably a result of the loss of compost mass. The slight increase in nitrogen content observed in SBO could be attributed to its higher concentration of lignocellulosic materials. This finding aligns with previous studies by (Baddi et al., 2004, Tortosa et al., 2012, Siles-Castellano et al., 2020).

Compost maturity can be assumed when the C/N ratio falls below 20, as reported by (Golueke, 1982; Hachicha et al., 2012). On the other hand, the addition of compost to the soil with a C/N ratio not exceeding 15 is not likely to disrupt its microbiological balance (Bernal et al., 1998).

The slight increase in C/N observed in the SBO mixture is possibly attributed to its higher concentration of ligno-cellulosic materials. However, a similar result regarding the C/N ratio was observed in composts made from a mixture of olive husk (80%) and olive tree pruning (20%) where C/N decreased from 57.0 to 26.2 (Gigliotti et al., 2012).

The increase of P could be due to the biodegradation of OM present in these mixtures, which may release the incorporated P from the OM. Comparable values were found in the following studies: (Cegarra et al., 1996; Cayuelaet et al., 2006) for MSO, (Gigliotti et al., 2012; Chowdhury et al., 2015) for SBPO, and (Sánchez-Arias et al., 2008) for SBO. Regarding the fluctuations in the K, Na, Ca, and Mg levels, they may be attributed to the evaporation or retention of water during composting.The incomplete degradation of phenols in the SBO mixture can be attributed to the fact that SB, being an agricultural by-product, absorbed the highest amount of phenols (75%). However, it's noteworthy that the maturity of compost is closely linked to the content of polyphenols, as indicated by previous studies (Saviozzi, 1987; Dinel et al., 1996).

The physicochemical characteristics of composts MSO, SBPO, and SBO as well as those of certain commercial composts, are provided in Table 3. The pH of composts MSO, SBPO, and SBO varied from 7.01 to 8.41 after the composting process, and these values fall within the range recommended by (Mustin, 1987; Das, 2008). For an optimal decomposition of OM, bacteria and fungi require the conditions where the pH is between 5.5 and 8.5. The pH range of composts should be between 6.0 and 8.5 to be compatible with the majority of plants (Asses et al., 2018). The EC of all the mixtures examined in this work is below 12 mS/cm. Values exceeding 12 mS/cm caused toxicity in the majority of plants (Lasaridi et al., 2006). According to the XP CEN/TS 17730 standard, the composts analyzed in this study comply with the requirement of containing more than 20% OM. The composts studied have an organic carbon content of approximately 50%, as shown in table 3. These values are similar to those of the commercial composts studied by (Zmora-Nahum et al., 2007). The abundance of OM and OC in the composts is a reflection of the nature of the residues, containing substantial amounts of lignin (Asses et al., 2018). Composts MSO, SBPO, and SBO have the following respective total nitrogen values: 3.75%, 4.37%, and 2.14%. The C/N ratio ranged from 11.72 to 24.93. SBO presented the highest C/N ratio, having the lowest total nitrogen value. The incorporation of compost with a C/N ratio below 15 into the soil is unlikely to disturb its microbiological balance (Bernal et al., 1998). The Na and Ca values in all composts are comparable to those found in commercial composts. All composts have higher values of P and K than commercial composts, with the exception of P in SBO. However, (Silva et al., 2016) conducted a comprehensive study on different commercial composts, evaluating their physicochemical properties, stability, and maturity. The results showed that none of the analyzed commercial composts are suitable for soil amendment, as at least one parameter exceeded the recommended limits. Hence, it is important to be careful when using any commercial compost as a reference for soil improvement, particularly, when it comes to the levels of heavy metals and EC. However, the composts derived from solid olive mill waste, investigated by (Tortosa et al., 2012), met the requirements of Spanish fertilizer regulations (PRE/630/2011 2011). Phenols in SBPO and MSO underwent complete degradation, while SBO did not exhibit complete degradation of phenols. The

findings of this study reveal that the classification of composts, ranked from the most to the least performing, is as follows: SBPO > MSO > SBO.

Using organic waste, like OMW, as fertilizer can decrease dependence on costly commercial fertilizers that require substantial resources and energy for production. Additionally, integrating OMW into agriculture has the potential to lower waste treatment expenses. Though there is a wide selection of highquality composts on the market, their production sites are often distant from the plantation nurseries. This geographical gap leads to increased production costs due to transportation. Consequently, it becomes essential to consider locally produced available substrates near the nurseries as a means to reduce costs effectively (Manca et al., 2020).

Table 3. Physicochemical characteristics comparison of the MSO, SBPO, and SBO composts at final state and commercial products (dry weight basis)

Parameters	MSO	SBPO	SBO	Commercial compost
pH	8.15	8.41	7.01	6.0 (Manca et al., 2020) 8.0-9.0 (Silva et al., 2013) 7.01 (Romero et al., 2013) 7.97-8.88 (Tortosa et al., 2012) 5.27-8.36 (Zmora-Nahum et al., 2007)
EC(mS/cm)	3.52	3.18	2.28	4.9-8.1 (Silva et al., 2013) 1.69-2.44 (Tortosa et al., 2012) 0.56-8.70 (Zmora-Nahum et al., 2007)
OM (%)	89	88.31	91.99	25 (Manca et al., 2020) 33.2-64.5 (Silva et al., 2013) 56.12-76.03 (Tortosa et al., 2012) 18.0-86.4 (Zmora-Nahum et al., 2007)
Ash $(%)$	10.4	10.89	6	
OC(%)	51.62	51.22	53.36	16.6-32.2 (Silva et al., 2013) 25.19-40.31 (Tortosa et al., 2012) 8.2-49.2 (Zmora-Nahum et al., 2007)
$TKN(\%)$	3.75	4.37	2.14	0.5 (Manca et al., 2020) 1.6-2.4 (Silva et al., 2013) 1.86-2.11 (Tortosa et al., 2012) 0.67-3.79 (Zmora-Nahum et al., 2007)
C/N	13.77	11.72	24.93	28 (Manca et al., 2020) 8.7-20.1 (Silva et al., 2013) 13.5-19.5 (Tortosa et al., 2012) 9.8-21.6 (Zmora-Nahum et al., 2007)
P(%)	0.78	0.25	0.15	0.086 (Manca et al., 2020) 0.18-0.22 (Tortosa et al., 2012)
$K(\%)$	1.93	1.8	1.41	0.166 (Manca et al., 2020) 0.65-1.28 (Tortosa et al., 2012)
Na (%)	0.14	0.14	0.12	0.0235 (Manca et al., 2020) 0.6-0.83 (Tortosa et al., 2012)
Ca (%)	0.4	0.6	0.12	0.3 (Manca et al., 2020) 1.87-7.24 (Tortosa et al., 2012)
Mg (%)	0.36	0.46	0.22	1.3 (Manca et al., 2020) 0.49-1.27 (Tortosa et al., 2012)
TPC (µg GAE/g DW)	0	0	385	

Note: EC: electrical conductivity, OM: organic matter, TKN: total Kjeldahl nitrogen, P: phosphorus, K: potassium, Na: sodium, Ca: calcium, Mg: magnesium, TPC: total phenol content.

CONCLUSIONS

In light of the results and discussions, it appears that co-composting OMW with various agricultural by-products offers a promising pathway for producing high-quality organic fertilizers. The results of this study indicate variation in performance among the composts examined. In addition to the mixture with SBPO which stood out as the most promising, the compost made from MSO also demonstrated encouraging performance, followed by the one based on SBO. This ranking in compost performance underscores the diversity of materials used and the potential impact of different blends on the quality of the final compost. These conclusions highlight the importance of further research in this area to better understand the long-term impacts on soil health and crop productivity. It is also crucial to emphasize that this approach offers a sustainable solution for waste management in the olive oil industry, reducing dependence on inorganic fertilizers and promoting environmentally friendly agricultural practices. Additionally, this study paves the way for new research on previously unexplored material combinations, thus providing opportunities for innovation and ongoing improvement in sustainable agricultural practices.

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