Research Article

Co-composting of Olive Mill Waste and Wine-Processing Waste: An Application of Compost as Soil Amendment

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In order to decrease the environmental harm produced by the agro industries' wastes', an investigation of the co-composting of olive mill waste (olive mill wastewater (OMW), olive mill sludge (OMS)) and wine by-products (grape marc and winery wastewater) was done. Three aerated windrows of variable compositions were performed; these windrows differ in terms of their initial composition and the liquid used for their humidification; OMW and wastewater winery were used for humidification to replace water for windrow moistening. Moreover, the main physicochemical parameters (temperature, pH, electrical conductivity, and C/N) were monitored to evaluate the co-composting process. The latter lasted around three months. The elaborated composts were characterized by low C/N ratio, and they were rich in fertilizing and nutriment elements and of low heavy metal contents. The humidification of the windrows with OMW showed effectiveness in improving the windrows temperature, reflected by the high temperatures monitored during the composting process in comparison with the windrow humidified with winery wastewater. Furthermore, a longer thermophilic phase was held in windrows carrying OMS. The valorization of the produced composts for soil amendment significantly improved the soil fertility. Indeed, field experiments showed an increase in radish yield by 10%, the composts were harmless and did not have any phytotoxic effect on radish growth.

1. Introduction

The activity of the agro-food industries causes a lot of harm to environment due to the production of huge amounts of waste. In Morocco, the industrial sector generates yearly more than 1.2 million tons of wastes, among which the agro-food industry represents 67% [1]. Consequently, the management and the valorization of these harmful residues are necessary for the environment's preservation.

The olive oil extraction is an important activity that generates a huge amount of effluent, namely, olive mill wastewater, estimated to 400 000 m³ per year [2]. The OMW is an acidic and dark liquid effluent, with a high organic matter load and high conductivity [3, 4]; its composition varies both qualitatively and quantitatively according to the olive variety, climate condition, cultivation and harvesting practices, the olive storage time, and the olive oil extraction system [3].

In Morocco, the OMW management practice regulated and adopted by the majority of olive oil industries is the storage in evaporation ponds. However, this technique does not reduce the OMW toxicity since large quantities of olive mill sludge (OMS) are produced. These huge quantities represent a significant problem in olive oil industries; therefore, more effective solutions must be developed to remedy this problem.

The wine industry is another activity that generates significant quantity of waste; this activity produces large amount of by-products that are represented by solid organic residues (grape marc) and wastewater. The production of one hectoliter of wine produces 18 kg of grape marc [5], which gives an annual production of 7200 tons [6]. Still, the quantity of wine wastewater depends largely on the process used in the industry. The different residues from the wine industry are characterized by low pH and electrical conductivity and high organic matter content [7]. The richness of these by-products in organic matter allows their use for soil fertility improvement [8, 9]. The direct incorporation of these residues could cause serious environmental problems if they are added excessively to the soil in an uncontrolled manner. Consequently, their treatment is crucial before their discharge or use for agricultural purposes. The suitable management of these agro-food wastes is an important strategy for the environment protection. Many investigations have focused on the study of treatment and valorization techniques of these residues at an experimental scale [10–16]. At the industrial level, the treatment of these residues is rare, and the problem of all the available wastes has not yet been solved.

Recently, researchers have shown that composting is one of the effective alternatives for the recycling and the valorization of organic wastes [8, 17–22]. It is a degradation process of the organic matter, allowing the achievement of a stable product, rich in humic substances and in fertilizing elements, serving as soil organic amendment [23–25]. The application of compost can have strong ecological environmental values, allowing not only the removal of very expensive chemical fertilizers but also the improvement of the quality of agricultural soils and carbon sequestration [8, 9, 26].

Diverse studies had developed composting of organic waste, such as OMW [19, 24, 27–30], sludge from wastewater treatment [31], and agro-industrial waste [32]. While other studies on the co-composting of olive mill wastewater with poultry manure [25, 27, 33], olive mill wastewater with solid organic waste [20, 34], and date palm with activated sludge [35] have been carried out. The evaluation of the composting process and the study of its environmental impact due to gas emissions have also been the subject of numerous studies [35–38].

Considering the fact that co-composting olive oil waste (OMW and OMS) and wine industry have not gained much attention in previous studies; the aims of this work are as follows: firstly, to valorize the olive mill wastes (OMW and OMS) and by-products wine industry by co-composting; secondly, to study the evolution of the parameters describing the co-composting of mixtures of OM, OMS, and green waste; and lastly, to test the effect of the different composts produced on the performance and yield of radish in the field.

2. Materials and Methods

2.1. Raw Materials. The organic wastes used as substrates for the composting procedure included OMW, OMS, grape marc, winery wastewater, and household waste. The OMW and OMS were collected from a natural evaporation basin of continuous three-phase olive oil extraction unit located in Meknassa Ben Ali (8 km from Taza, Morocco). The basin was made of concrete with a storage capacity of 240 m³. The collected OMW had a maximum storage time of 30 days; the OMS were collected at the end of August, after a total evaporation of the quantities of water contained in the OMW. The grape marc and the winery wastewater were collected from an industrial company “Celliers de Meknès” located in Meknes (Morocco), and the green waste was gathered from markets located in Taza (Morocco). They are composed mainly of fruit and vegetable residues. The waste physicochemical characterization is presented in Table 1.

2.2. Composting Procedure. The raw materials were mixed in order to be co-composted in open area, using 3 windrows of 0.6 m height, 2 m length, and 1.2 m diameter base [30]. These windrows differed in terms of their initial composition and the liquid used for their humidification (Table 2). The proportions of the raw materials were calculated according to the methods described by Proietti et al. and Soudi [26, 39] to obtain a physicochemical characterization of the initial mixture for starting the composting process.

The moisture content was adjusted to around 50–60% (optimal moisture content for composting); this operation was performed during the turning of the windrows. The windrows were turned once every 3 days at the beginning of the process, then once every 7 days, and once every 15 days for the remaining composting period.

Representative and homogenous samples were collected during the composting procedure from the windrows for analysis. Each sample was obtained by mixing 6 subsamples taken from six different points of windrows, according to ISO 8633 [40].

2.3. Physicochemical Analysis. The raw materials and compost samples were analyzed for pH and electrical conductivity (EC) in a 1:5 (w/v) water soluble extract in [41, 42]. The dry matter content was assessed by drying at 105°C for 24 h using a drying oven type WTB Binder ED 115, and the OM was determined by measuring the loss of ignition at 550°C for 4 h using muffle furnace type Lenton EF11/8B. The organic carbon (OC) was calculated using the following equation:

$$\text{CO (\%)} = \frac{\% \text{MO}}{1,724} \quad (1)$$

The total Kjeldahl nitrogen (TKN) was analyzed using the Kjeldahl method according to AFNOR standard (1981) [43]. The determination of heavy metals and oligoelements were first extracted by heating 2 g of compost with HNO₃, and then the filtrate was analyzed using ICP-AES method (inductively coupled plasma atomic emission spectroscopy).

TABLE 1: Physicochemical characteristics of the raw materials used for composting process.

Parameters	OMS	OMW	Grape marc	Winery wastewater	Green waste
pH	5.42 ± 0.03	5.43 ± 0.05	7.42 ± 0.20	6.66 ± 0.02	5.88 ± 0.02
EC (mS·cm ⁻¹)	8.72 ± 0.02	8.02 ± 0.02	4.81 ± 0.03	0.76 ± 0.02	1.63 ± 0.02
Moisture (%)	22.66 ± 3.69	92.47 ± 0.56	72.42 ± 0.58	99.86 ± 0.01	36.98 ± 4.59
Dry matter (%)	77.34 ± 3.69	7.53 ± 0.56	27.58 ± 0.58	0.14 ± 0.01	63.02 ± 4.59
Mineral matter (%)	15.05 ± 5.91	25.25 ± 5.46	30.19 ± 1.65	45.20 ± 6.63	35.15 ± 1.95
Organic matter (%)	84.95 ± 5.91	74.75 ± 5.46	69.81 ± 1.65	54.80 ± 6.63	64.85 ± 1.95
Organic carbon (%)	49.28 ± 3.43	43.36 ± 3.16	40.49 ± 0.96	31.78 ± 3.84	37.61 ± 1.13
TKN (%)	0.74 ± 0.12	0.86 ± 0.15	0.97 ± 0.10	3.75 ± 0.30	1.53 ± 0.06
C/N	67.77 ± 9.73	ND	42.06 ± 3.47	ND	24.69 ± 1.19
COD (g·O ₂ /l)	ND	96.07 ± 0.5	ND	14.07 ± 0.1	ND
BOD (g·O ₂ /l)	ND	25.67 ± 0.51	ND	14.40 ± 0.4	ND

ND: not determined.

TABLE 2: Composition of the different windrows.

Windrow	A (600 kg)	B (600 kg)	C (1 ton)
Proportion of solid raw materials*			
(i) OMS	1/3	1/3	0
(ii) Grape marc	1/3	1/3	1/2
(iii) Green waste	1/3	1/3	1/2
Humidification effluent	OMW	Winery wastewater	OMW
Total volume used (m ³)	0.8	1.0	1.3

*Proportion expressed in weight/weight.

The characterization of the effluents was performed according to the experimental protocols described by Rodier et al. [44]. pH and electrical conductivity were directly measured using multiparameter consort C335. The chemical oxygen demand (COD) was determined according to NF T90-101 standard [45]. The biological oxygen demand (BOD) was determined using BOD meter-type OxiTop.

The windrow temperature was measured using multiparameter consort C535 daily during the mesophilic and the thermophilic composting phases and weekly until its maturity. The presented temperature value consists of a mean of 6 measurements at different points and depths of the windrow.

2.4. Phytotoxicity Test. The compost phytotoxicity was determined by evaluating its aqueous extract on seed germination. The germination test was carried out on 10 seeds of cress (*Lepidium sativum*) experimented at different dilutions in Petri dishes, including filter paper soaked in the compost extract. The test was conducted in dark at 25°C for 72 hours [32, 33]. Three repetitions were performed.

The germination index (GI) was determined considering the number of sprouts and root growth, using the following equation:

$$GI(\%) = \frac{GB}{GT} * \frac{LB}{LT} * 100, \quad (2)$$

where GB is the number of germinated seeds in the case of aqueous extract, GT is the number of germinated seeds in the

case of the control where the distilled water was used, LB is the root length on compost extract, and LT is the root length control.

2.5. Radish Production. The effects of the different produced composts were assessed on radish (*Raphanus sativus*), of a *National* variety. Nine parcels of 1.5 m² arranged in a random design were tested using compost and different combinations of compost mixed with farm manure, according to Table 3. These various combinations of composts and manure were used for the soil amendment at a rate of 6 t/ha; they were incorporated in the soil not only spread on the surface. The parcel, which was only amended with farm manure, was considered as the control. No mineral fertilization has been done in the parcels.

The radish seeds were sowed on 29 November 2015, and the radish was harvested on 24 January 2016. The composts effects were assessed by measuring radish yield at the end of the crop cycle.

2.6. Statistical Analysis. Statistical analyses of data were made by IBM SPSS Statistics Version 20. The analysis of the means comparison was carried out by Student's *t*-test to compare the evolution profiles of the composting parameters for the different windrows. The Duncan test was used to calculate the variance and to compare the average values of germination indices for the different composts produced.

All analyses were performed in triplicate. However, for agronomic yields, the results were obtained in the form of one replicate due to the limitation of the plots to a single field. Also, minerals and fertilizing elements and heavy metals were in only one replicate because of the limited cost of investigation of the analysis.

3. Results and Discussion

3.1. Composting Process

3.1.1. Temperature Evolution. During the composting process, the evolution of the temperature in the different windrows is presented in Figure 1. The temperature profiles allow the observation of four conventional stages: a mesophilic, a thermophilic, a cooling, and a maturation stage.

TABLE 3: Treatments used for parcel amendment.

Parcel	P ₀	P ₁	P ₂	P ₃	P ₄	P ₅	P ₆	P ₇	P ₈	P ₉
Compost (%)	0	100	100	100	75	75	75	50	50	50
Compost type		C _A	C _B	C _C	C _A	C _B	C _C	C _A	C _B	C _C
Farm manure (%)	100	0	0	0	25	25	25	50	50	50

C_A, C_B, and C_C are the different mature composts prepared, respectively, from the windrows A, B, and C.

FIGURE 1: The windrows' temperature profiles during the composting process.

In the first stage, an increase of the temperature to 47°C was recorded during the first week. This observation resulted from the rapid colonization and activity of the mesophilic microorganisms, which degraded organic matter and released heat, thus increasing the windrows' temperature. Then, the temperature rose progressively to reach values above 60°C, corresponding to the second stage, the thermophilic phase, and remained approximately 6 weeks. This result was due to the intense microbial activity reflecting high degradation rates occurring during the first stage [20]. The high temperatures may result in the reduction of pathogens and enhanced windrow sanitation [18]. At the 6th week, the third stage began, and the temperature decreased progressively to reach 30°C. These low temperatures were the result of the less intense microbial activity due to the depletion of easily degradable organic matter [21]. Finally, at the maturity stage, the temperature stabilized at 30°C, the process of composting reached its end, and the produced compost was mature. Moreover, the windrow temperature was highly related to the ambient temperature at this stage.

The long phases observed in windrows A and B can be attributed to the initial composition of the mixtures, in particular the presence of OMS. Indeed, the OMS presents a source of organic matter and therefore an availability of additional degradable substances in these mixtures (windrows A and B) contrary to mixture C. Hachicha et al. have found the similar results suggesting that the self-insulating capacity of OMS during the degradation process can lead to a long thermophilic phase [27]. Such a phase reflects an abnormal degradation process and a delayed transition to the stabilization phase [46].

Additionally, these results also show a significant difference between the temperature profile of windrows A and C and windrows B and C, confirmed by the means comparison test by Student's *t*-test ($p < 0.05$). This difference can be attributed to the activity of microorganisms during the degradation process depending on the initial composition of the substrates.

3.1.2. pH Evolution. The pH evolution of the composted materials followed the same trend in the different windrows (Figure 2). At the beginning of the process, a slight decrease in pH was noted. This result could be explained by the production of organic acids, dissolved CO₂ in the medium and by-products from the degradation of easily biodegradable compounds [46]. Then, an increase in pH from 6.5 to 6.7, from 6.44 to 6.59, and from 6.48 to 7.66 was observed for windrows A, B, and C, respectively. This could be the result of ammonia production from the degradation of amines [35, 47]. Finally, the pH decreased progressively and stabilized at a neutral pH for windrows A and B and alkali pH for C. These results suggest the formation of humic substances which act as buffers, as confirmed by Zenjari et al. and Amir et al. [48, 49].

The comparison between the three pH profiles shows a significant difference ($p < 0.05$) between windrows A and C and windrows B and C. This can be explained by the nature of the initial substrates put to compost. However, the addition of OMW during the composting process influences the pH of the mixtures.

3.1.3. EC Evolution. The progress of the electrical conductivity (EC) during the composting process is presented in Figure 3. During the first weeks of composting process, the EC values increased from 2.11 to 3.42 mS·cm⁻¹, from 2.04 to 3.95 mS·cm⁻¹, and from 2.09 to 3.87 mS·cm⁻¹, respectively, in the windrows A, B, and C. This increase revealed the extent of mineralization of the organic substrate and the release of ions [35]. Then, a progressive decrease in the EC was observed, even in windrows moistened by OMW exhibiting salts loss by the leaching phenomenon as well as by decreasing their extractability because of their fixation on the stabilized organic matter [49].

Moreover, the EC was measured on the extract of organic matter which is sensitive to the solvent extraction ratio and the temperature at which the method was carried out. It could be noticed that each windrow composition presented a particular progress, with significant differences attributed to phenomena of mineralization, leaching, and fixation depending on the composition of the substrates and the activity of microorganisms.

3.1.4. Organic Matter Degradation. The evolution of the organic matter content of the mixture during the composting process is considered as an essential parameter of biodegradation and transformation of organic matter during composting. Figure 4 presents the organic matter contents of the three mixtures at the beginning and end of the

FIGURE 2: pH evolution during the composting process.

FIGURE 3: EC evolution during the composting process.

FIGURE 4: Organic matter degradation during the composting process.

composting process. These results show that all the experimented windrows were characterized by a high rate of organic matter (>55%) at the beginning of the composting process. It could be noticed that the windrow C has the highest organic content. During the composting process, the organic matter content gradually decreased, resulting in a degradation rate exceeding 40% for the three mixtures.

This degradation is highlighted during composting by a mass loss of the initial mixtures or even a remarkable

reduction in the volumes of co-composted waste. The organic matter decomposition was the result of the microbial activity, allowing the transformation of the organic matter into stable humic substances [50]. The similar results were observed by El Fels et al., indicating a good degree of compostability of the mixtures [35].

Furthermore, no significant difference was shown by Student's *t*-test between the three windrows. So, one can conclude that the degradation of organic matter does not depend on the composition or nature of the co-composted waste.

3.1.5. C/N Ratio Evolution. The evolution of the C/N ratio is directly related to the biodegradation of organic matter, resulting in the lowering of the total carbon rate associated with the increase in nitrogen concentration. The initial C/N for the three windrows represented values between 25 and 35 (Figure 5). These C/N ratios were ideal for co-composting process [26, 51]. The C/N ratio evolution showed decreases of the ratios from 27.7 to 12.0, from 34.5 to 13.45, and from 34.3 to 12.2 for the windrows A, B, and C, respectively (Figure 5). This is closely related to the loss of organic carbon due to mineralization of the organic material, with a CO₂ production increasing the nitrogen concentration in the windrows during the biodegradation.

At the end of the co-composting, the C/N ratio reached values exceeding 8 for all windrows, indicating the maturity of the final compost [52]. Moreover, according to many authors, the C/N ratio is considered to be a maturity index; generally, the compost is mature if it presents a C/N ratio less than 20 [53–55].

The three profiles of the C/N ratio evolution are of the same speed and represent no significant difference between the three windrows confirmed by Student's *t*-test ($p > 0.05$).

3.2. Maturity and Quality of the Produced Composts

3.2.1. Characterization of the Produced Composts. The quality of the composts was assessed by comparing the final characteristics of the conceived composts to those of the French standard NF U44-051 (Table 4). The composts produced were neutral, except compost C was characterized by alkaline pH. This result is attributed to the nature of the co-composted waste. Also, they were characterized by C/N ratio around 12, exhibiting their stability.

The comparison of the three produced composts to the French standard showed significant amendment properties. Indeed, the produced composts were rich in fertilizing and mineral elements and low contents of heavy metals. These results have the same meaning with other previous studies valorizing OMW by co-composting, and the produced composts showed significant properties of organic fertilizers compared with the manure [18, 53, 56].

3.2.2. Phytotoxicity. The phytotoxicity of the produced composts was evaluated by the germination test; the result of this test is shown in Figure 6. This figure revealed the effects

FIGURE 5: The C/N ratio evolution during the composting process.

TABLE 4: Physicochemical characteristics of produced composts.

Parameters	Compost A	Compost B	Compost C	NF U44-051
pH	7.02 ± 0.02	7.09 ± 0.03	8.30 ± 0.02	ND
CE mS·cm ⁻¹	1.75 ± 0.02	3.04 ± 0.03	1.92 ± 0.03	ND
C/N	12.00 ± 1.23	13.45 ± 2.08	12.24 ± 1.15	>8
<i>Minerals and fertilizing elements (mg/kg)</i>				
P	855.99	158.37	346.15	ND
K	2763.56	2368.35	2264.55	ND
Mg	897.68	901.90	810.35	ND
Ca	10153.95	9237.85	9760.55	ND
Fe	5414.84	5101.55	6121.70	ND
Na	225.67	259.65	309.45	ND
Mn	49.65	31.65	33.35	ND
<i>Heavy metals (mg/kg)</i>				
Zn	57.83	11.6	12.2	600
Cu	22.26	5.95	6.55	300
Cr	9.35	7.60	5.95	12
Ni	<0.01	0.06	0.06	2
Cd	1.5	1.0	1.2	3
As	<0.01	<0.01	<0.01	18
Se	<0.01	<0.01	<0.01	12

ND: not determined.

of the extracts from produced composts on the germination of cress seeds at different dilutions.

The comparison between the different dilutions shows that a dilution rate of 25% shows the best germination for the different composts produced, marked by a higher rate for the compost and a significant difference between the different compost products C. However, a dilution rate of 50% shows a nonsignificant effect between the different compost products, whereas a dilution of 75% and the crude extract shows significant effects whose compost C represents the best germination index.

These differences can be explained by the nature of the raw materials, by the germination conditions of the watercress seeds and by the high conductivity of the compost extract. Indeed, high values in electrical conductivity inhibit the germination of watercress seeds, which results in low germination indices in raw compost extracts. According to

Lasaridi et al., compost that is too saline can be harmful to plants; acceptable EC values should not exceed 2 to 3 mS/cm [57]. However, the three composts do not represent any phytotoxic effect because all these results showed a germination rate higher than 50%, indicating a maturity of the compost produced [38, 58].

3.2.3. Effect of the Produced Composts on Radish Production.

The radish yields were assessed at the end of the crop cycle, while harvesting. The yields are presented in Figure 7. On farm manure, the yield was equal to 52.5 t/ha, but on the other parcels amended with the composts, the yield ranged from 52.8 t/ha to 57.7 t/ha. The produced composts significantly improved the productivity of the radish crop. However, the highest yields remained in parcels amended with the composts obtained from windrows C and A of 10%. It could be concluded that the OMW used for humidification played an important agronomic role. Indeed, the positive effect of composts based on olive mill waste on radish yields depends particularly on their richness in nutrients and fertilizers element particulates N, P, K, Ca, Mg, and Fe [19, 25]. These elements play a very important role in the growth of plants, thus promoting their vegetative activity, which results in an improvement in radish production yield.

However, Regni et al. suggest that long-term application of composted OMW to the soil improves vegetative activity and olive yield. They confirm that the contribution of nutrients to composted olive mill waste through their amendments associated with other indirect factors such as organic and dynamic matter content, water retention, and microbial biomass activity stimulates olive tree growth and improves olive yield and quality. These results are consistent with those found by Hachicha et al. showing the significant effect of composts based on OMS and poultry manure on potato yield [27].

4. Conclusion

The co-composting of olive mill wastes and the wine by-products with green waste has proved to be an effective means of producing an organic amendment for agricultural soils. The monitoring of the physicochemical parameters during this process has revealed a good progress of the co-composting process, a biodegradation of organic matter and a bioconversion of unstable matter into a stable product rich in humic substances. This biotransformation was also confirmed by the phytotoxicity test of the compost extracts produced, which showed that the various composts produced are mature and show no phytotoxic effect.

Moreover, the physicochemical characterization has proved that these composts are of good quality, rich in nutrients and particularly N, P, K, Ca, Mg, and Fe, and conforms to the standards of an organic amendment NF U44-051. The application of compost produced in the fields as organic soil improvers for radish cultivation has had a positive effect by increasing radish production yield by 10% compared to manure. Given the slow and complex dynamics

FIGURE 6: Effect of the compost extract on germination of cress seeds at different dilutions ($n = 5$, $p = 0.05$). Different letters (a–f) indicate significant differences ($p < 0.05$) between compost and dilution rates.

FIGURE 7: Production yield of harvested radish.

of organic substance transformation, long-term amendment experiments would be necessary and will be the subject of further research.

To conclude, one could say that the valorization of olive mill waste and the wine by-products is a promise strategy for the sustainable management of this type of waste allowing the transformation of environmental threats into a valuable product assuring fertility to agricultural soils.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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