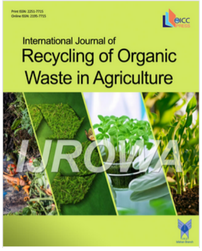

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Co-composting of olive mill waste cake residues (OMWC) and other wastes: Prediction and optimization using an optimal mixture design

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Abstract:
Purpose: This work was aimed to exploit the technique of design of experiments to evaluate the olive mill waste cake residue obtained after oil extraction while searching for an adequate composition of the initial substrate mixtures (RPM, DMOV, FV and EB for humidification), in addition to validate it experimentally.
Method: To this end, a mixture design using an efficient experiment methodology was constructed, with the aim of optimizing three responses of interest: pH, humidity, and organic matter of the 25 formulations.
Results: The optimal composition was comprised of organic and green household waste (56.7%), olive mill waste cake residues (21.7%) and poultry droppings (21.7%). These analyses were followed by the evolution of some physio-chemical parameters such as pH, temperature, organic matter, C/N ratio and polyphenols until they are stabilized. In addition, using spectroscopic analysis, maturation and phytotoxicity tests were carried out on the germinating cress.
Conclusion: The results of this research indicated the achievement of an ideal composition of an adequate mixture that had the characteristics of an organic amendment, non-phytotoxic and in conformity with the NFU-44-051 that led to mature compost.

Keywords: Olive mill waste; Olive mill waste cake; Extraction residues; Composting; Prediction; Optimal mixture design

1. Introduction

It is expected that the world population will increase by 2 billion people in the next 30 years, with the population increasing from the current 7.7 billion people to 9.7 billion people in 2050 (Battisti et al. 2022). Along with this increase in population, the quantities of household waste have undergone a similar evolution, mainly due to new production and consumption patterns and the progressive rate of rural migration and consequent urbanization, which is currently close to 60% (Bonkena et al. 2018).
In addition, according to world bank estimates in 2018, the global production of municipal waste was reported to be 2.01 billion tons/year (Chuenwong et al. 2022). The current production of household waste in urban areas of Morocco is estimated to be 5.3 million tons/year, with an average of 0.76 kg/inhabitant, and in rural areas 1.47 million tons/year, with an average of 0.28 kg/inhabitant/day. 23% of these wastes are of industrial type, especially olive oil crushing, which according to the ranking as the fifth largest producer with a volume of about 140,000 tons, occupies a significant proportion according to recent studies (Azenzem 2022; Barreca and Praticò 2020). This operation generates a significant amount of solid and liquid waste which in turn is

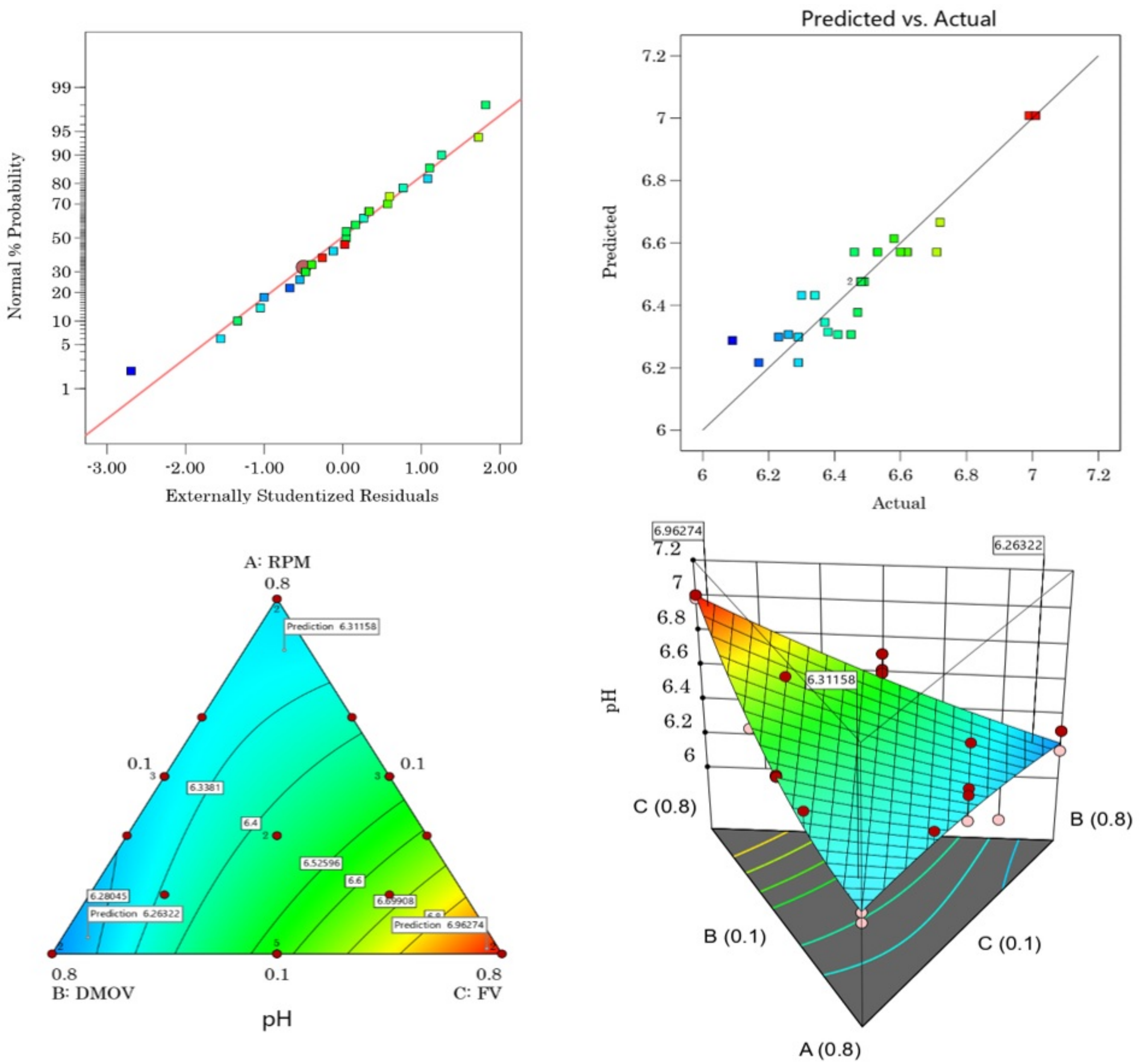

Figure 1. (a): Normal probability plot. (b): Predicted versus actual plot. (c): Contour plot and (d): 3D surface plot of the effect of the three components on pH response.

a serious threat to the environment and a major pollutant of water resources. For example, the disposal of more than 2000 crushing units in the Basin of Oum Er-Rbi to the region of Beni Mellal generates nearly 300 thousand tons of olive mill waste (Burri et al. 2019). In addition to the spread of diseases and the destruction of the landscape, this situation also generally damages the economic development of the country, especially in the tourism industry.

Many research works have been conducted to evaluate these co-products in different fields, for example the isolation of lipase, biophenols, and antioxidants from pomace (Ertuğrul et al. 2007; Kaleh and Geißen 2016), as well as their uses as a biopesticide, in producing bricks, as a spreading substance in soils (Barbera et al. 2013; Casa et al. 2009; El-Abbassi et al. 2017). Also, olive pomace has become especially important because of the isolation of omega 3 from it (Paz et al. 2020), as well as the production of lignin and sugars, ethanol and cellulase (Khdair and Abu-Rumman 2020; Leite et al. 2016; Mehdaoui et al. 2023b). As a result of the treatment of olive factory waste by natural evaporation, another waste is created, which is actually a paste called ”olive mill waste cake”. On the other hand, the co-composting of olive oil waste with other wastes can be considered as an affordable approach for amending these co-products and minimize their environmental impact. In fact, it is recommended to do a co-composting with manure (Gómez-Cruz et al. 2021).

Indeed, the statistical approach of using designs of experiments (DoE) is a powerful tool in mixture optimization when dealing with multi-components (López-Linares et al. 2020). In addition, mixture designs in DoE are a multifactor approach that is based on well-structured logics and provides fast and more reliable results in response optimization (Khdair and Abu-Rumman 2020; Leite et al. 2016).

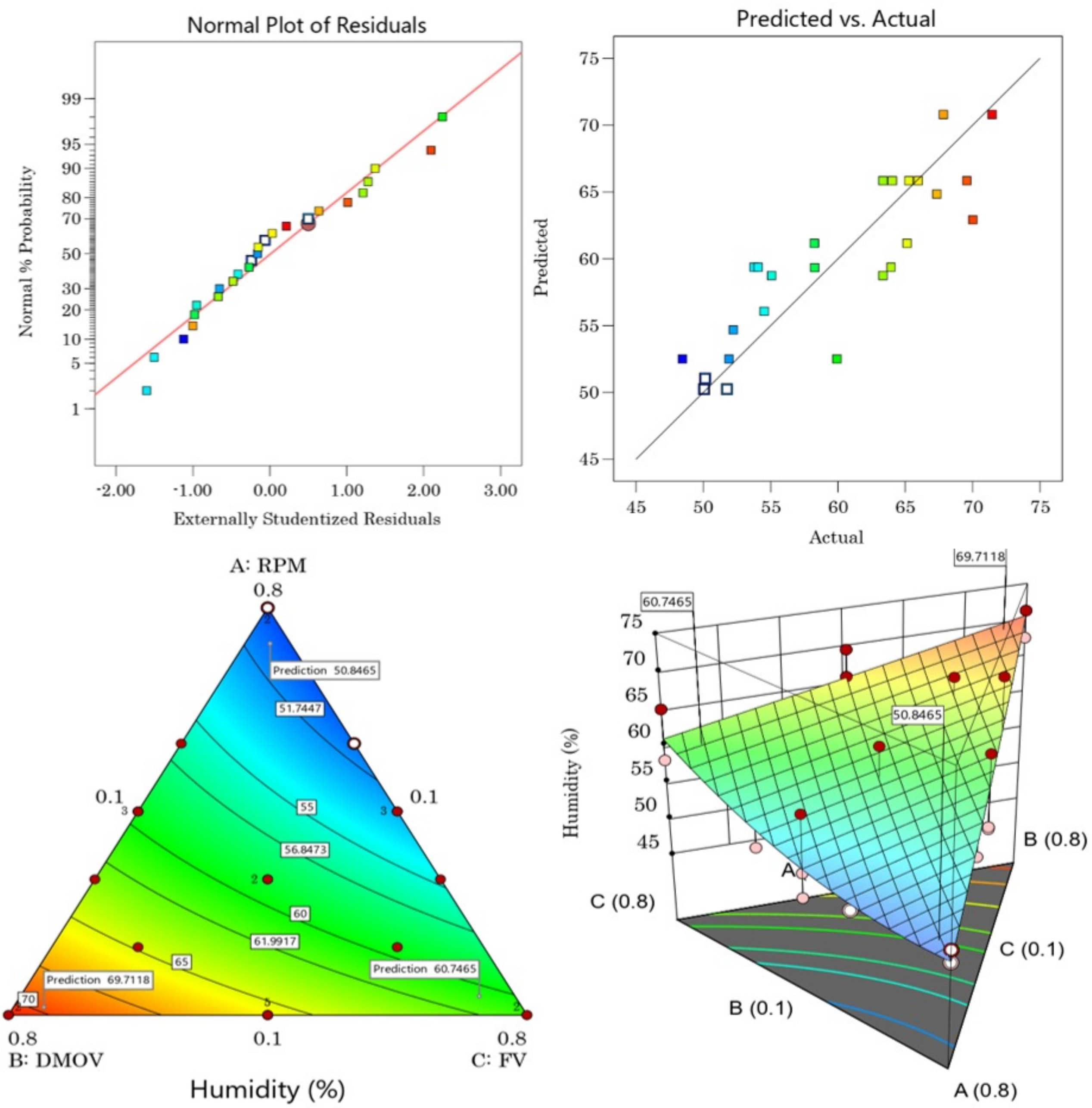

Figure 2. (a): Normal probability plot. (b): Predicted versus actual plot. (c): Contour plot and (d): 3D surface plot of the effect of the three components on Humidity response.

A recent study by the same team from our lab has contributed to the valorization of these cakes by extracting the oil that served not only as a source of bioenergy (Mehdaoui et al. 2023b); but also a corrosion bio-inhibitor (Mehdaoui et al. 2023a). However, this process generates about 3/4 of the initial mass of pastes in the form of residues which require a valorization.
The objective of this study was to valorize the residues of the oil extract from the olive mill waste cake by the die of composting while determining the ideal combination using an optimal plan of the mixture.

2. Material and methods

2.1 Initial substrates

The initial substrates used for the preparation of the compost were the residue of olive mill waste cake (RPM) obtained after extraction of vegetable oil from the olive mill waste cake which was supplied from the olive mill waste storage basin of the oil mills located in Meknassa Bni-Ali at 8 km from the city of Taza-Morocco, organic and green household waste (DMOV) supplied by the university restaurant Dhar El Mahraz in the city of Fez., poultry droppings (FV) collected from agricultural breeding farms in the region of Fez, and rainwater storage pond water (EB) taken from a rainwater collection basin located at the Faculty of Sciences Dhar El

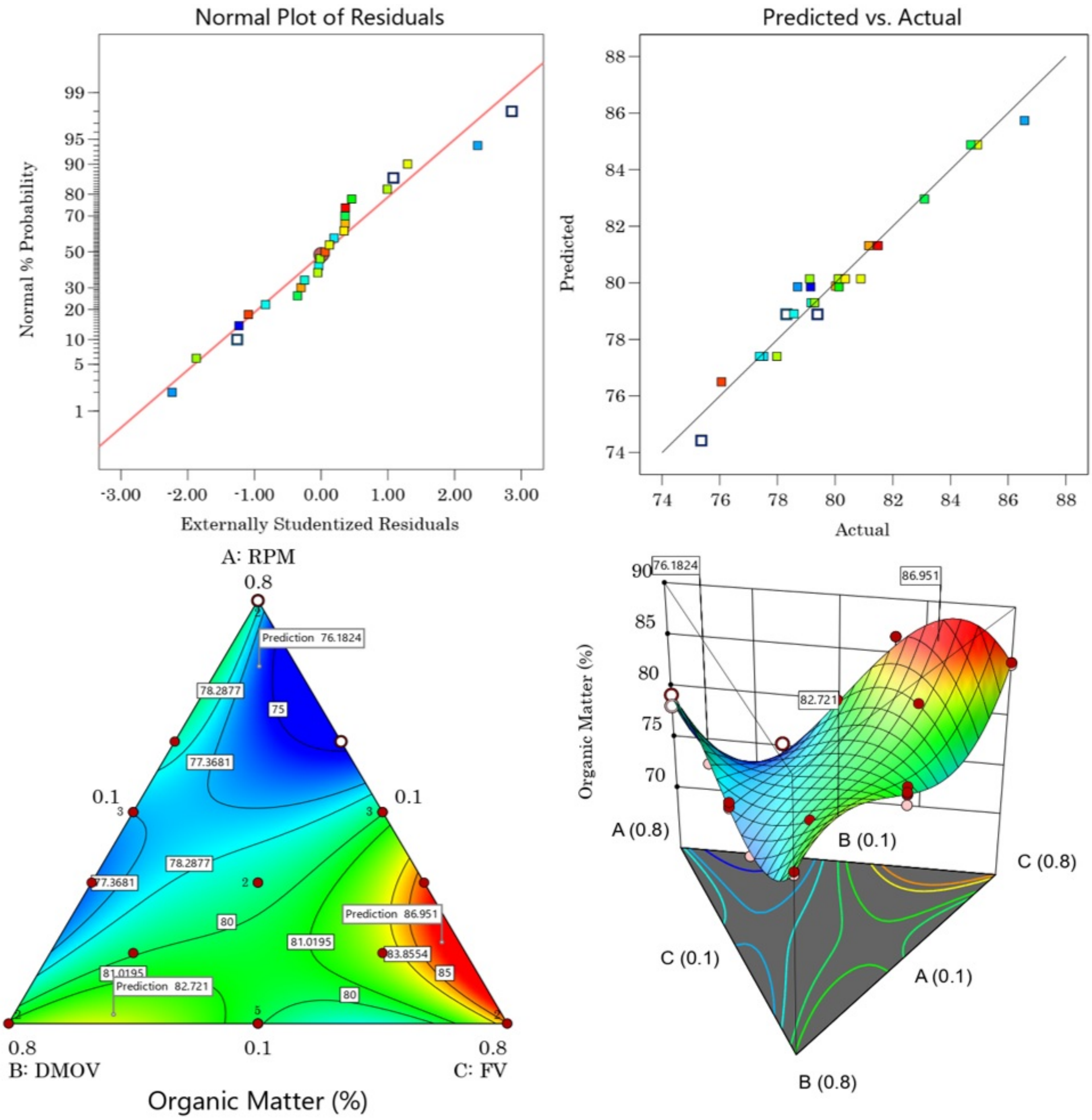

Figure 3. (a): Normal probability plot. (b): Predicted versus actual plot. (c): Contour plot and (d): 3D surface plot of the effect of the three components on MO response.

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2.2 Plant material

The cress (*Lepidum sativum*) was used to evaluate the phytotoxicity of the compost (Majbar et al. 2018).

2.3 Experimental design

The DoE approach is a robust tool to understand and optimize the experimental parameters, or components of a mixture, allowing a rational interpretation of their influence on the chosen responses with a considerable reduction in the number of experiments and a better understanding of the process mechanism (Weissman and Anderson 2015). In the current study, the impact of the proportion of RPM (A), DMOV (B), and FV (C) waste after their homogenization, on the pH (R_1), humidity (R_2), and organic matter (R_3) of the mixture being composted, has been described using some mathematical equations, so that predictions of the mixture could be made experimentally. In addition, the optimal mixed composition with the optimal criterion was developed using Design-Expert software version 12, with the assumption that there is no secondary constraint on the proportion of the components (Spessato et al. 2021). A total of 25 formulations (including 5 repetitions and two center points) were generated in random order to account for any randomized hidden effects (Table 1). The boundaries of the mixed composition and the geometric location of the experimental points in the mixture design are shown in Table 2 and Fig. 1. After completing the modeling, the final step in the study was optimization, which involved finding the

Table 1. Components of the mixture design and its limits.

Composants	Names	Units	Type	Coded Low	Coded High
A	RPM	%	Mixture	$+0 \leftrightarrow 0.1$	$+1 \leftrightarrow 0.8$
B	DMOV	%	Mixture	$+0 \leftrightarrow 0.1$	$+1 \leftrightarrow 0.8$
C	FV	%	Mixture	$+0 \leftrightarrow 0.1$	$+1 \leftrightarrow 0.8$

optimal proportions of each component based on the possible criteria and constraints. The results are shown in Figs 1, 2 and 3. For our application, the numerical optimization method was applied using the desirability function (D) as reported in a previous work (Ouadrhiri et al. 2021). This choice was considered based on the possibility of change weight and importance of both the mixture component and the responses (Weissman and Anderson 2015). The mixture design was performed by running 25 experiments with different compositions as defined by the software. The validated composition was replicated three times for the reliability of the results and experiments are presented in Table 9.

2.4 Physicochemical characterization of initial substrates

Characterization of the initial substrates was performed by measuring pH, electrical conductivity (EC), humidity (%H), organic matter (%OM), mineral matter (%MM), total organic carbon (%TOC), total kjeldhal nitrogen (%TKN) and C/N ratio.

2.5 Monitoring the composting process

The evaluation of the quality of the olive mill waste wake residue (RPM) compost was determined by the major factors of the process; especially the pH, temperature, organic matter, C/N ratio and polyphenols content. Sampling was carried out twice a week for almost three months until the compost was stabilized and matured.

2.6 Characterization of humidified water

The physicochemical characterization of the pond water intended for compost humidification was determined by measuring in-situ physical parameters (pH, temperature, electrical conductivity, and turbidity), chemical parameters: BOD_5, COD, nitrates, nitrites, ammonium, orthophosphates, sulphates, and chlorides; as well as bacteriological parameters: *faecal coliforms*, *salmonella* and *cholera vibrio*.

2.7 Phytotoxicity test

The phytotoxicity of the compost was evaluated by measuring its germination capacity. Germination tests were carried out on 10 Cress seeds in the compost extract at different dilutions (0.25, 0.5 and 0.75) compared to a brute solution and an indicator sample made with distilled water. All solutions were used to saturate filter paper in petri dishes. The tests were placed in the dark and at room temperature (25°C), for 72 h (Filho et al. 2021; Stevens and Anderson-Cook 2019). Three replicates were performed to ensure the reliability of the results.
The germination index (GI) was determined in relation to the number of germinated seeds and root growth according to the following formula (Stevens and Anderson-Cook 2019):

$$\mathrm{GI} = \left(\frac{\mathrm{GB}}{\mathrm{GT}}\right) \times \left(\frac{\mathrm{LB}}{\mathrm{LT}}\right) \times 100$$

With:
GB: Number of germinated seeds in the compost case,
GT: Number of germinated seeds in the control case,
LB: Root length in the compost case,
LT: Root length in the case of the control. The compost was

Table 2. Physico-chemical characterization of the initial substrates.

Paramètres	Olive mil waste cake residues (RPM)	Organic and green domestic waste (DMOV)	Poultry droppings (FV)
Ph	6.71	7.97	7.23
CE (Ms/cm)	6.2	2.65	8.91
H %	9.38	90.09	8.44
MM %	13.83	16.94	6.15
OM %	86.17	83.06	93.85
TOC %	49.98	65.19	73.66
TKN %	0.693	7.7	9.2
C/N	72.12	8.47	8.01
Polyphénols (g/L)	118.3	-	-

Table 3. Physico-chemical characterization of the FSDM humidified water.

Parametres	*EB*	(Mahmoud et al. 2020)
Ph	7.2	6.6 à 8.4
Temperature (°C)	20	35
EC (Ms/cm)	1.254	12
Turbidity		-
BOD_5 (mg(O_2) /L)	0	-
COD (mg(O_2)/L)	0	-
Nitrites NO_2^-(mg/L)	0.07	-
Nitrates NO_3^-(mg/L)	-	1.5
Ammonium NH_4^+(mg/L)	0.045	-
Sulfate SO_4^{2-}(mg/L)	-	250
Orthophosphates (PO_4^{3-})(mg/L)	-	-
NTK (%)	0	-

Table 4. Mineral and trace metal content.

Elements	Concentration (mg/L)	(Mahmoud et al. 2020)
Al	< 0.01	5
As	< 0.01	0.1
B	0.0106	-
Ca	79.386	-
Cr	< 0.01	1
Cu	< 0.01	2
Fe	0.0947	5
K	3.7433	-
Mg	30.862	-
Mn	0.0105	
Mo	0.0475	0.2
Na	83.437	0.01
Ni	< 0.01	-
P	< 0.01	2
Se	< 0.01	-
Si	7.4724	0.02
Zn	< 0.01	-

considered as mature if the GI was higher than 50.
The presence of humic acid was detected by measuring the characteristic absorbances (A) of these compounds based on the *Welt ratio* or E4/E6 = A465nm/A665 nm. If the ratio wasless than 5 the predominance was for humic acid and decomposition was advanced in the opposite case, the predominance was for fulvic acid and degradation was less advanced (Koriko et al. 2013).

3. Results and discussion

3.1 Physicochemical characterization of the initial substrates

The results of the physicochemical characterization of the initial substrates are reported in Table 2. The residue obtained after extraction of vegetable oil was endowed with a high mineral load, a rate of organic matter, and a very high C/N ratio with a pH close to neutrality, due to the decrease in the rate of nitrogen that was most likely the origin of the slight acidity (Zamboni et al. 2006). The extraction of vegetable oil from olive mill waste cake had almost no influence on its polyphenol load. Organic and green domestic waste was almost neutral and characterized by considerable percentages of nitrogen and total organic carbon. Poultry droppings, were neutral and very rich in organic matter compared to other substrates. They had a nitrogen composition and a C/N ratio close to those of the DMOV.

3.2 Physicochemical characterization of humidified water

The physicochemical and bacteriological characterization of the wetting substance is shown in Tables 3, 4 and 5. It showed that the pond water located at FSDM respects the Moroccan irrigation standard (Mahmoud et al. 2020) and did not present any risk of the soil-plant structure system. Moreover, the low presence of fecal coliforms favored the richness of the compost in organic matter which served for the nutrition of the already existing microorganisms and consequently ensured the good progress of the composting

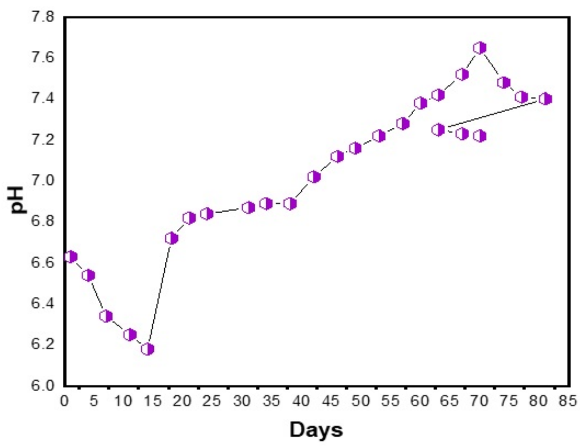

Figure 4. Evolution of pH as a function of time during the co-composting process.

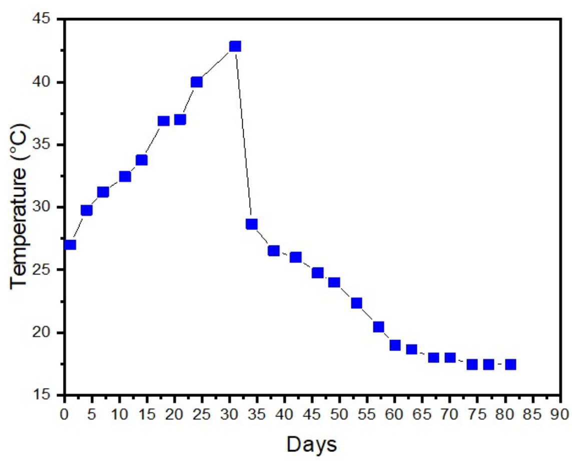

Figure 5. Evolution of temperature as a function of time during the co-composting process.

Table 5. Bacteriological characteristics of compost humidifying water.

Parametres	***Fecal coliforms*** (UFC/100mL)	***Salmonella***	***Cholera vibrio***
EB	10	0	0
SLV (NM 1276-01 2002)	1000 UFC/100mL	0	0

process.

3.3 Modeling and analysis of statistical properties of the mixture design

The optimal mixture, designed with the criterion of optimal, was used to describe the relationship of the responses of interest to fluctuations in the proportions of the mixture being composted and to determine the optimal proportions of the mixture according to the criteria listed in Table 7. The quality of the pH, humidity, and organic matter model was evaluated using Fishers' analysis of variance (ANOVA) test (F-value), probability value (p-value) and significance of lack of fit. However, the predictive property of the models was tested using the multilinear regression coefficients (R^2), the predicted coefficient ($R^2_{predicted}$) and the adjusted coefficient ($R^2_{adjusted}$). In addition, a plot of normal and predicted residual values against normal values was used to evaluate the distribution and normality of the residuals. The pH and moisture responses were the most consistent with quadratic model. While the organic matter response was more consistent with the cubic model.

3.4 pH model

The experimental values of the mixture design of the pH experiments presented in Table 9 were used to fit the quadratic model by the least squares procedure.
The analysis of variance ANOVA of the pH model, presented in Table 10 showed that the pH regression was significant with an F-value of 23.93 and a P-value < 0.0001, as well as the lack of fit (F-value of 2.59) was not statistically significant compared to the pure error. In addition, the R^2 coefficient of the predicted model was 0.8629, confirming the correlation and good fit between the experimental and calculated data. This was also proved by the difference between the adjusted R^2 (0.8269) and the predicted R^2 (0.7792), which was reasonable given that it was less than 0.2. The results of ANOVA analysis showed that only the interaction between olive mill waste cake residues and poultry droppings had a significant influence on pH as their P-value was 0.0099 (less than 0.05). In addition, the P-values of the interactions between the olive mill waste cake residues and the organic and green household waste (0.4390) as well as the interaction between the latter and the poultry droppings (0.4937) were higher than 0.05, thus revealing that these components were statistically not significant.
Therefore, only the effects of significant components were considered in Equation (1) in terms of real components to predict the pH model.

$$pH = 6.3A + 6.07B + 7.38C + 0.4AB - 1.44AC - 0.34BC \tag{1}$$

Indeed, the interactions between the AC, and BC components had a rather negative effect on pH with a negative coefficient of -1.44 and -0.34, respectively.
The normal plot of the residuals shown in Fig. 1(a) indicateed that the distribution of the residual points correctly followed the straight line, indicating the homogeneity of the distribution. In addition, the significance of the experimental values verified by plotting the predicted versus actual graph (Fig. 1(b)) showed that the set of values were distributed closely around the diagonal line, demonstrating the accuracy and validity of the predicted model. The 2D and 3D response surface graphs illustrated the effect of the mixture on pH. The results showed that the pH reached its maximum (6.96) in the area containing 0.8 of FV and 0.1 for RPM and DMOV.

3.5 Humidity model

The final quadratic model equation for the real component humidity model is presented in Equation 2.

$$\text{Humidity}(\%) = 48.9A + 75.8B + 63.7C - 9.4AB - 26.1^{*}AC - 1.1BC \tag{2}$$

The distribution of the points in the model was correct in terms of the validity of the model, that was reflected in

Table 6. Verification experiments under optimal conditions.

Optimal Conditions:	**XRPM=0.217**,	**XDMOV=0.567**,	**XFV=0.217**
	Predicted	**Experimental**	**Error**
pH	6.346	6.59	0.244(3.64%)*
Humidity	64.841	68.8	3.96(5.7%)*
OM	79.882	79.75	0.2(0.1%)*

the distribution of the points around the straight line in the normal plot (Fig. 2(a)), since it was homogeneous and the actual values were distributed uniformly from the diagonal in the predicted plot compared to the actual values (Fig. 2(b)).

The relationship between the actual components (RPM, DMOV and FV) was established via the 3D and 2D response surface plots. Indeed, the initial composition of compost showed a considerable influence on its humidity. In our case, the optimum moisture (69.68%) was observed in the region of 0.1158, 0.7468 and 0.1372 for components A, B and C, respectively. This corroborates the results of the characterization of the initial substrates since the high value was noted for B (DMOV) with 90.09%.

The results of ANOVA (Table 11) showed that, the predicted R^2 (0.5658), the adjusted R^2 (0.6807), and the R^2 coefficient (0.7472) indicated a good fit of the model to the experimental data. On the other hand, the P-values of the interactions between the real components, showed no significant effect. Moreover, these interactions showed negative effects for the model by large coefficients; 26.1 for AC, 9.4 for AB and 1.1 for BC.

3.6 Organic matter model

The experimental values of the mixture design of the organic matter presented a cubic model by the least squares procedure (Table 4).

The results of ANOVA of the organic matter model (Table 12) showed that the organic matter regression was highly significant (F-value = 41.31, P-value < 0.0001), as well as the lack of fit (F-value = 3.26) which was not significant compared to the pure error. In addition, the R^2 coefficient of the predicted model was 0.8632, confirming the correlation and good fit between the experimental and calculated data. This was also affirmed by the difference between the adjusted R^2 (0.9379) and the predicted R^2 (0.8632), and was consistent because it was less than 0.2.

The ANOVA analysis for the interaction of the real components showed that the interactions are significant due to their P-value which was less than 0.05 except the interaction between A, B and C because it recorded a P-value = 0.0605. Therefore, only the effects of the significant real components were considered in Equation (3) to predict the organic matter model.

$$\begin{aligned} \mathrm{OM}(\%) = {} & 92.4\mathrm{A} + 83\mathrm{B} + 82.5\mathrm{C} - 38.8\mathrm{AB} - 27.76\mathrm{AC} - \\ & 20.1\mathrm{BC} + 80.2\mathrm{ABC} + 63.3\mathrm{AB}(\mathrm{A}-\mathrm{B}) - \\ & 183.3\mathrm{AC}(\mathrm{A}-\mathrm{C}) + 95.13\mathrm{BC}(\mathrm{B}-\mathrm{C}) \end{aligned} \tag{3}$$

The interactions of components AB, AC and BC showed a negative effect on the studied parameters with a negative coefficient of -38.8, -27.76 and -20.1, respectively.

The normal plot of the residuals presented in Fig. 3(a) indicateed that the distribution of the residual points strongly followed the straight line, demonstrating a regular distribution. In addition, the experimental values were presented in the predicted versus actual plot presented in Fig. 3(b) showing that the set of values were distributed closely around the diagonal line, which confirmed the accuracy and validity of the predicted model.

Figs 3(c) and 3(d) show the 2D and 3D representation of the OM response (%). The results obtained (Figs 3(c) and 3(d)) showed that the poultry droppings cause a maximum increase of 65% in the amount of organic matter. This was consistent with the characterization results of FV before mixing, which recorded 93.85%.

3.7 Numerical optimization with the desirability function

Using the software, the highest value of the desirability function D was chosen as the optimum solution which corresponds to the proportions mentioned in Table 9. To assure the main objective of the study which was developing a mixture of three constituents (PM, DMOV and FV) with controlled properties according to the criteria mentioned in Table 6.

After making the compost with the ideal composition predicted by the software, 21.7% RPM, 56.7% DMOV and 21.7% FV, the initial mixture was slightly acidic (pH = 6.59), moderately humidity and very rich in organic matter.

3.8 Characterization of the initial optimized mixture to be composted

The physicochemical characterization of the initial mixture presented in Table 7 revealed it had a very low mineral load with a humidity and organic matter content at the upper limit of the norm which was closer to the natural state, as well as a low percentage of nitrogen characterized by a C/N ratio within the recommended range. It also had a high NPK and trace element content, which was beneficial to both the soil and the plants.

3.9 Monitoring the composting process

Evolution of pH

Fig. 4 represents the evolution of pH as a function of time during the co-composting process. The results showed that the compost possessed a slight acidification at the beginning of the process due to the release and accumulation of organic acid molecules produced by the first colonizers in anaerobic conditions established at the beginning of the composting process, as well as the decomposition of simple organic substrates, and the volatilization of the initial ammonia (Azim et al. 2018; Majbar et al. 2018).

At the end of the 3^{rd} week, an increase was detected, which was explained by the process of ammonification and ammonia production from the degradation of amines (proteins, nitrogenous bases...) (Himanen and Hänninen 2011) and the disappearance of easily degradable organic matter and mineralization.

At the end of the 6^{th} week, the process was characterized by a stabilization of the pH until neutralization revealing the buffer property of humus during the maturation phase.

In fact, the change in pH depended on several factors, such as the nature of the raw material used, the circulation of air that ensures effective ventilation and good degradation of organic matter in addition to the temperature that promoted the volatilization of ammonia (Azim et al. 2018). The pH value obtained corresponded to the limited value given by NFU44-051 Standard (between 5 and 8).

Table 7. Physicochemical characteristics of the initial RPMC mixture.

Parametrs	RPMC mixture	Standard NF U44-051
pH	6.63	$5 < pH < 8$
EC (mS/cm)	0.17	-
Humidity (%)	65.47	$40\% < H < 65\%$
OM (%)	77.33	$40 < MO < 70$
TOC (%)	60.69	-
NTK (%)	2.1	-
C/N ratio	28.9	$20 < C/N < 40$
Polyphenols (mg/mL)	0.1355	-
Oligo elements and nutrients (mg/L)		
P	5.4214	-
B	0.0339	-
K	4.542	-
Mg	4.2980	-
Ca	16.157	-
Fe	3.3614	-
Na	3.1129	-
Mn	0.1183	300
Cu	< 0.01	-
Zn	0.0558	600
Se	< 0.01	-
Ni	< 0.01	2
Other mineral elements (mg/L)		
As	< 0.01	18
Al	2.2748	-
Cd	-	3
Cr	< 0.01	12
Si	3.5046	-

3.10 Temperature evolution

The change in temperature during this process was the first sign that reflected the degradation level of the different substrates. Therefore, for rapid and successful composting, high temperatures for long periods are not recommended due to the effect of temperatures on the growth of microorganisms as the active agents to degrade organic matter (Atif et al. 2020).
The evolution of temperature with time during the co-composting process shown in Fig. 5, indicated the rapid elevation of internal temperature of the windrow from the mesophilic stage (20 – 40°C), which was characterized by rapid colonization of mesophilic microbial populations, to the thermophilic stage (> 40°C), which could conceivably be useful for neutralizing heat-sensitive pathogens.
Then the temperature started to decrease and most of the matter was decomposed and no longer recognizable. Temperatures were more stable (usually less than 40°C) even after the windrow was turned, and populations of microorganisms were replaced by others that prefer lower temperatures that favored the use of available nutrients. These results corroborated those found in organic matter, which begins to decrease after 30^{th} day. After the 70^{th} day, the temperatures stabilized, allowing the development of eumycetes and actinomycetes, the main decomposers of long chain polymers, cellulose, and lignin (Atif et al. 2020; Azim et al. 2018).
The value of the temperature of the mature compost is equal to 15°C while that mentioned by the standard is 23°C. This is due to the ambient temperature of the medium and the period in which the compost was elaborated.

3.11 Evolution of organic matter

Fig. 6 represents the evolution of organic matter as a function of time during the co-composting process. The monitoring showed a strong linear and progressive decrease until stability since the 20^{th} week, which followed by the decrease of the total organic carbon during the composting process due to the degradation by the microorganisms of the organic substances necessary for their metabolism, leading to their mineralization in the form of carbon dioxide (CO_2). The possible presence of anaerobic sites in the compost

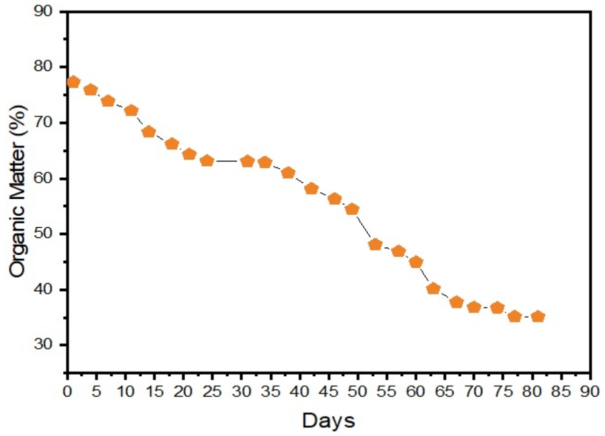

Figure 6. Evolution of organic matter as a function of time during the co-composting process.

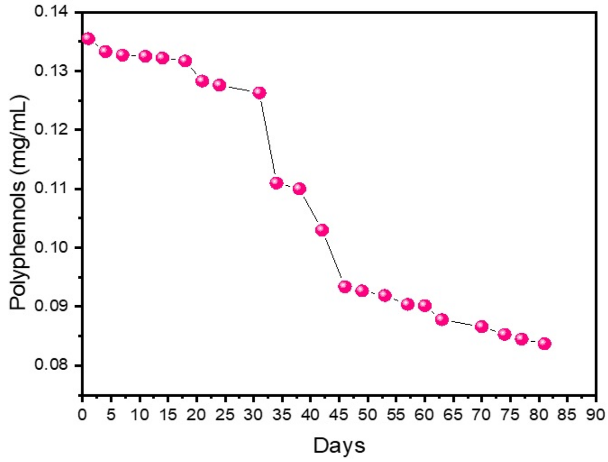

Figure 8. Evolution of polyphenols as a function of time during the co-composting process.

windrow resulted in releasing methane (CH_4) associated with fermentative metabolism (Said-Pullicino et al. 2007). In fact, the decomposition of organic matter resulted in producing carbon dioxide, the increase of which was correlated to a decrease in the oxygen level. Therefore, it is necessary to provide oxygen through aeration in the material to be composted to maintain the sufficient amount of oxygen which is done by turning the windows.
Indeed, organic carbon is one of the main constituents of composted organic waste and consists of total organic carbon (TOC) and inorganic carbon in the form of carbonates and bicarbonates.
In addition, TOC accounted for over 90% of the total carbon in composts due to CO_2 gas emission which occurred from the degradation of the organic matter derived from the initial substrates of the compost (waste of food industries, adventitious weeds, effluents of breeding as manure, droppings, urine, surface waters). Compared to raw green waste, household waste and sludge were comprised of $20-30\%$, $25-50\%$,$30-40\%$ respectively (Said-Pullicino et al. 2007).
In our experience, the degradation of the organic matter showed a normal trend since the initial value was 77.33% and it achieved 42.89% at the end of the composting process. And normal trend required a value between 40 and 70%.

3.12 Evolution of C/N ratio

The C/N (organic carbon/organic nitrogen) ratio decreases during composting process. This parameter was frequently measured to evaluate the maturity of the compost (Guo et al. 2012). The ratios lower than 20 and even 15 can imply the maturity of the compost. The ratios between 10 and 15 can be considered stable although the final ratio significantly depends on the initial substrates used.
Fig. 7 shows the evolution of the C/N ratio as a function of time during the co-composting process. The result obtained showed that the C/N ratio decreased over time until it stabilizes at the 11^{th} week, which proved the maturity of the elaborated compost. This indicated the biodegradation of the organic matter.

3.13 Evolution of the polyphenol composition

Fig. 8 shows the evolution of the dose of polyphenols as a function of time during the composting process. The results

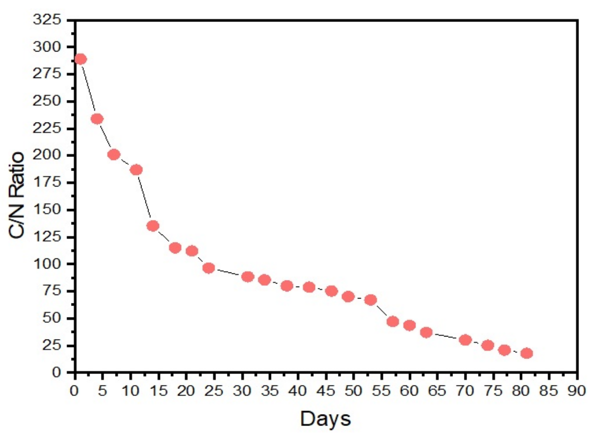

Figure 7. Evolution of the C/N ratio as a function of time during the co-composting process.

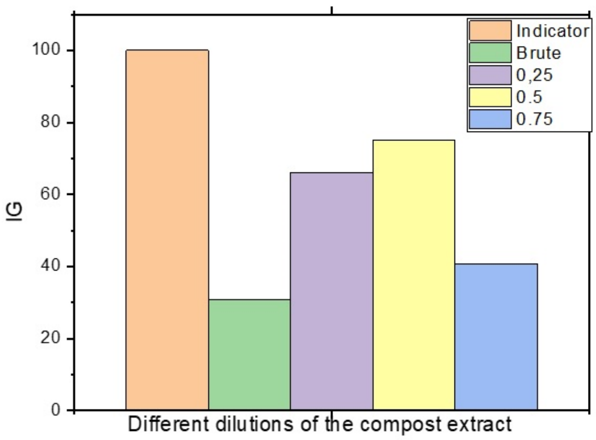

Figure 9. Effect of compost extract on cress germination at different dilutions.

Table 8. Physicochemical characteristics of RPMC mature compost.

Parametrs	CRPM	NFU44-051 Standard (Limits values)
Humidity (%)	38	40% < H < 65%
Temperature	15	23
pH	7.42	5 < pH < 8
NTK (%)	1.53	-
OM (%)	42.89	40 < MO < 70
C/N ratio	16.37	10 < C/N < 50
Polyphenols (mg/mL)	0.0837	-
Mineral elements and fertilizers (mg/L)		
P	4.1783	-
K	15.940	-
Mg	3.622	-
Ca	18.868	-
Fe	3.162	-
Na	14.571	-
Se	< 0.01	-
Other mineral elements (mg/L)		
As	< 0.01	18
Al	1.744	-
Cd	-	3
Ni	< 0.01	2
Cr	< 0.01	12

The final product could be used as an organic amendment to treat soils poor in mineral elements to improve their qualities and agricultural yields.

of analysis showed a significant degradation of polyphenols as a function of time. This decrease in polyphenolic compounds was probably associated with the formation of humic substances, which involved degradation and condensation reactions due to the phenols.
In addition, the mechanism under which this process is carried out is still not very clear, it is likely due to transform -monomers into polymers with high molecular weight, possess low solubility, and resist to biological degradation (Baddi et al. 2009). Indeed, several research has shown that polyphenols act as precursors for the formation of humic substances (Zhang et al. 2021; Zhu et al. 2021) and contribute to the formation of these substances in the soil (Fahimi et al. 2003).
In effect, excess polyphenols in agricultural soils can negatively affect microbial respiration, leading to lower microbial biomass and to reduce soil pH. In addition, a high enough dose can also increase the fungus/bacteria ratio in systems with high polyphenol content, especially with condensed tannin (Mutabaruka et al. 2007). This contributes to a physicochemical and biological imbalance of the soil, which can affect qualities of the agricultural yields, something that is not in accordance with the objective of the work.

3.14 Physicochemical characterization of mature compost

The physicochemical characterization of the compost is grouped in Table 8. The results were obtained after maturation which lasted about 3 months.
The compost elaborated was neutral, rich in nitrogen, and in organic matter that showed decreased trend by almost $1/2$ compared to the initial mixture. Its composition in mineral elements and C/N ratios was in accordance with the standard.
Regarding fertilizing elements, the validation compost of the ideal composition obtained by the mixture design was endowed with high doses of magnesium, potassium, calcium, iron, phosphorus, and sodium.

3.15 Phytotoxicity test

Germination index

Fig. 9 illustrates the effect of compost extract on cress germination. The observed results demonstrated that the germination index was 66% for a dilution of 0.25 of compost extract and 75% for that of 0.50. On the other hand, a low germination index for the 0.75 did register only 37.03%. These results showed that the dose of compost applied could influence the germination rate and consequently accelerate the maturation of the plants.

3.16 E4/E6 ratio

The ratio E4/E6 was 2.02, which indicated the presence of humic acid because the value was lower than 5. An advanced stage of decomposition was thus reached. The low ratio explained that the composts were mature, and the humus particles was large and complex. Composts lead to a reduction in particle size and an increase in this ratio (Lahlou et al. 2017).

4. Conclusion

This work aimed at the valorization of biowaste from the olive crushing industry, especially the olive mill waste cake by co-composting it with organic and green domestic waste and poultry droppings.

In fact, quadratic models of pH and moisture, and cubic organic matter predicted that the best composition of the initial mix of substrates to be composted was 56.7% DMOV, 21.7% RPM, and 21.7% FV, respectively. From the obtained results, it could be concluded that the mixing plan had the potential to be applied to predict the adequate composition of a mixture of substrates that can be turned into compost. This would help to develop a process design for composting and valorization of organic waste on an industrial scale.

The validation of the optimal compost and the evaluation of its quality was carried out by the follow-up the physicochemical parameters according to the time until its maturation. The stability of the biological system was determined after about 3 months. The final product obtained was in conformity with the organic amendment standard (NFU44-051).

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Author Contributions:
The authors confirm the study conception and design: I. Mehdaoui, I. Mehdaoui, F. EL Ouadrhiri; data collection: I. Mehdaoui, F. EL Ouadrhiri; analysis and interpretation of results: R. Mahmoud, EM. Saoudi Hassani, M. Ben Abbou; draft manuscript preparation: M.Taleb, Z.Rais, A.Lahkimi. The results were evaluated by all authors, and the final version of the manuscript was approved.

Compliance with ethical standards
Conflict of interest:
The authors declare that there are no conflicts of interest associated with this study.

Appendix:
Appendix includes Tables 9, 10, 11, 12, which are presented in the following.

Table 9. Experimental matrix of the optimal mixing design.

Run	A:(RPM)	Composants B:(DMOV)	C:(FV)	pH		Responses Humidity(%)		OM (%)	
	%	%	%	Experimental	Predicted	Experimental	Predicted	Experimental	Predicted
1	0.333	0.100	0.567	6.58	6.61	52.23	54.68	86.57	85.74
2	0.333	0.333	0.333	6.3	6.43	55.07	58.74	79.17	79.30
3	0.450	0.450	0.100	6.26	6.31	53.77	59.37	77.52	77.40
4	0.450	0.100	0.450	6.48	6.48	48.45	52.50	79.15	79.86
5	0.567	0.333	0.100	6.38	6.31	54.52	56.07	78.57	78.91
6	0.450	0.100	0.450	6.49	6.48	51.9	52.50	78.7	79.86
7	0.333	0.567	0.100	6.09	6.29	70.02	62.92	76.06	76.50
8	0.800	0.100	0.100	6.23	6.30	51.76	50.24	78.31	78.89
9	0.217	0.217	0.567	6.72	6.67	58.27	59.34	83.1	82.96
10	0.217	0.567	0.217	6.37	6.35	67.34	64.84	80.02	79.88
11	0.450	0.100	0.450	6.48	6.48	59.92	52.50	80.13	79.86
12	0.100	0.800	0.100	6.29	6.22	71.45	70.80	81.49	81.32
13	0.567	0.100	0.333	6.47	6.38	50.14	51.04	76.36	74.42
14	0.333	0.333	0.333	6.34	6.43	63.34	58.74	79.29	79.30
15	0.450	0.450	0.100	6.45	6.31	54.07	59.37	77.38	77.40
16	0.100	0.450	0.450	6.46	6.57	69.57	65.85	80.18	80.14
17	0.100	0.450	0.450	6.53	6.57	63.34	65.85	79.12	80.14
18	0.100	0.800	0.100	6.17	6.22	67.83	70.80	81.17	81.32
19	0.100	0.450	0.450	6.71	6.57	65.96	65.85	80.35	80.14
20	0.450	0.450	0.100	6.41	6.31	63.94	59.37	77.98	77.40
21	0.800	0.100	0.100	6.29	6.30	50.05	50.24	79.39	78.89
22	0.100	0.100	0.800	7.01	7.01	58.25	61.16	84.71	84.88
23	0.100	0.450	0.450	6.6	6.57	64.04	65.85	80.11	80.14
24	0.100	0.100	0.800	6.99	7.01	65.13	61.16	84.94	84.88
25	0.100	0.450	0.450	6.62	6.57	65.27	65.85	80.89	80.14

Table 10. ANOVA analysis for the pH model.

Source	Sum of Squares	df	Mean Square		F-value	p-value	
Model	1.04	5	0.2083		23.93	< 0.0001	Significant
(1)**Linear Mixture**	0.9518	2	0.4759		54.67	< 0.0001	
AB	0.0054	1	0.0054		0.6248	0.4390	
AC	0.0714	1	0.0714		8.20	0.0099	
BC	0.0042	1	0.0042		0.4869	0.4937	
Residual	0.1654	19	0.0087				
Lack of Fit	0.0995	7	0.0142		2.59	0.0707	Not significant
Pure Error	0.0659	12	0.0055				
R^2				0.8629			
Adjusted R^2				0.8269			
Predicted R^2				0.7792			
Adeq Precision				17.3188			

Table 11. ANOVA analysis for the humidity model (%).

Source	Sum of Squares	df	Mean Square		F-value	p-value	
Model	925.88	5	185.18		11.23	< 0.0001	Significant
[(1)]**Linear Mixture**	901.00	2	450.50		27.32	< 0.0001	
AB	3.01	1	3.01		0.1829	0.6737	
AC	23.26	1	23.26		1.41	0.2496	
BC	0.0443	1	0.0443		0.0027	0.9592	
Residual	313.28	19	16.49				
Lack of Fit	87.63	7	12.52		0.6657	0.6975	Not significant
Pure Error	225.65	12	18.80				
R^2				0.7472			
Adjusted R^2				0.6807			
Predicted R^2				0.5658			

Table 12. ANOVA analysis for the organic matter model (%).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	161.22	9	17.91	41.31	< 0.0001	Significant
Linear Mixture	83.01	2	41.51	95.72	< 0.0001	
AB	14.91	1	14.91	34.38	< 0.0001	
AC	8.42	1	8.42	19.42	0.0005	
BC	19.46	1	19.46	44.88	< 0.0001	
ABC	1.79	1	1.79	4.12	0.0605	
AB (A-B)	5.37	1	5.37	12.37	0.0031	
AC (A-C)	45.01	1	45.01	103.79	< 0.0001	
BC (B-C))	4.96	1	4.96	11.43	0.0041	
Residual	6.50	15	0.4336			
Lack of Fit	2.92	3	0.9737	3.26	0.0594	Not significant
Pure Error	3.58	12	0.2986			
R^2						0.9612
Adjusted R^2						0.9379
Predicted R^2						0.8632

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