

Article

Valorization of Orange Peel Waste Using Precomposting and Vermicomposting Processes

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Abstract: The industrialization process of oranges generates waste, which is inadequately disposed of; this produces adverse effects on the environment. Among the alternatives for valorization is the vermicomposting process, which consists of the degradation of organic waste through the action of earthworms and microorganisms. Therefore, this research aimed to study this process using orange peel (OP) waste at the laboratory level. For this purpose, it was necessary to determine the degradation conditions through the monitoring of physicochemical parameters (temperature, pH, humidity, organic matter (OM), total organic carbon (TOC), total nitrogen (TN) and the carbon/nitrogen (C/N) ratio). To balance the substrate's nutrients, load material (LM) that included vegetable waste and eggshells was added to three different mixtures: M1 (50% OP + 50% LM), M2 (40% OP + 60% LM) and M3 (60% OP + 40% LM). To condition the substrate for earthworm (*Eisenia fetida*) activity, a previous precomposting process was performed. The results showed that all the mixtures fulfilled the requirements for a quality and mature vermicompost; however, the highest concentrations for TN were in the mixtures M1 and M2. The total time required for degradation of the OP waste was 13 weeks.

Keywords: precomposting; vermicomposting; orange peel waste; *Eisenia fetida*

1. Introduction

The orange (*Citrus × sinensis*) is a citrus fruit that is consumed all over the world in different ways [1]. In 2018–2019, the global production volume of fresh oranges was about 54.3 million metric tons [2]. Brazil is responsible for more than three-quarters of the orange juice produced worldwide and for 15% of the international exports of this product, followed by China, the United States, Mexico and some European countries [3,4].

During orange juice production, only around 50% of the weight of fresh oranges is transformed into juice. Subsequently, the process for obtaining juice generates large amounts of waste (peels, pulp, seeds, orange leaves and whole orange fruits that do not fulfill quality requirements). Orange peel (OP) waste constitutes approximately 50–60% of the weight of the processed fruit, composed of peels, tissue and the remaining portion of seeds [1,5]; it contains 75–85% water, 6–8% simple sugars (glucose, fructose and sucrose) and 1.53% polysaccharides (pectin, cellulose and hemicellulose). Its pH is 3–5, and there is a significant presence of essential oil that is mainly composed of d-limonene (83–97%) [6].

Different valorization routes have been proposed for OP waste, like biofuels, biorefinery, pectin extraction and animal feed, among others. Unfortunately, some of the aforementioned technologies are

not economically sustainable for implementation on a full scale and are generally only suitable for large citrus processing plants; therefore, OP waste is usually used as forage or disposed of in landfills [5]. Thus, vermicomposting emerges as a viable alternative for the degradation and stabilization of organic matter (OM) [7]. It is defined as the biochemical process of OM degradation through interactions between earthworms and microorganisms, obtaining a final product with higher nutrient content in ionic forms and a humic substrate rich in bioactivity [8].

It is an economical and sustainable option for the use of organic waste, and it seeks to improve the content of organic matter, so it is one of the best options to substitute for the introduction of harmful chemical fertilizers into the soil [9]. Vermicompost does not require additional processing to be incorporated into the soil as long as it meets the specifications of not containing foreign mineral materials, organic materials not digested by earthworms, inert materials, viable seeds and live earthworms according to quality specifications [10]. The addition of compost to the soil will improve any soil's physical, chemical and biological properties [11,12]. It is excellent-quality humus that provides immediate results due to the fact that the plants absorb nutrients faster [13].

Besides, this process significantly reduces greenhouse gas (GHG) emissions by avoiding waste reaching landfills; this determines the elimination of the methane-generating source that results from anaerobic decomposition as well as CO₂ and N₂O. These processes have very low emissions compared to those generated by untreated waste [14].

However, there are some negative environmental impacts that must be considered, such as transportation from the source to processing facilities as well as to vermicompost application sites, which could increase GHG emissions [15].

On the other hand, the literature consulted showed some studies related to the application of the vermicomposting process with OP waste, such as that by Favoretto et al., who considered this process an efficient technology to convert cattle manure waste mixed with OP and filter cake into organic fertilizer to reduce the incidence of soil pests [16]. Lee et al. investigated the suitability of orange skin to be reused as feedstock for *Eudrilus eugeniae* for 60 days, which revealed the potential of biotransforming orange skin into high-quality organic fertilizer [17]. Therefore, this research aims to study the operational conditions for the precomposting and vermicomposting processes using mixes of OP and vegetable waste in different proportions to obtain a quality vermicompost.

2. Materials and Methods

2.1. Precomposting Process

A total of 12 kg of OP waste was collected from a juice store and separated from the pulp. All the waste was finely chopped and disposed of in an 11.4 L capacity plastic container; this was placed on top of a table, and perforations were made in the bottom to collect the leachate generated. It was covered by a net to avoid the presence of pests such as insects or rodents. Subsequently, the container was tilted slightly for better drainage (Figure 1). During the precomposting process, the OP waste remained in this container for the necessary period of time before the vermicomposting process, and every third day, the container was sprayed with a sprinkler to maintain proper humidity levels [18,19].

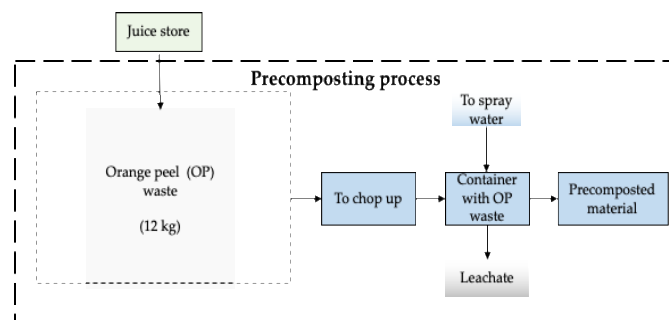


Figure 1. Precomposting process. Source: own elaboration.

2.2. Vermicomposting Process

The purpose of the precomposting process mentioned in Section 2.1 was to avoid a temperature increase that could affect the earthworms and facilitate OP waste biodegradation [20,21]; afterwards, the vermicomposting process was performed. In addition, 10 kg of green waste was collected from local markets (vegetable waste from lettuce, chard, cabbage, spinach, celery, cress and broccoli), and 1 kg of eggshells (from the bakery) were dried in sunlight for 24 h [20,22–24]; this is considered “load material” (LM). The addition of eggshells was due to their calcium (Ca^{+2}) content as well as proteins, glycoproteins and proteoglycans. In addition, Ca^{+2} is an important element in the worms’ diet and can help their reproductive process [22,25].

Experiments were conducted in four plastic containers, each with a 5.9 L capacity; they were adapted with holes that drained out to collect the leachates generated. All reactors were incubated in a dark place at environmental temperature. The first layer of soil (1 kg) was added to each of the four reactors, the second one was LM and the third layer was OP waste [26]. In the same way as was done during the precomposting process, water was sprayed every third day to maintain humidity levels. A total of 50 *Eisenia fetida* earthworms were collected in three plastic containers and transported to the site of the experiment (Figure 2).

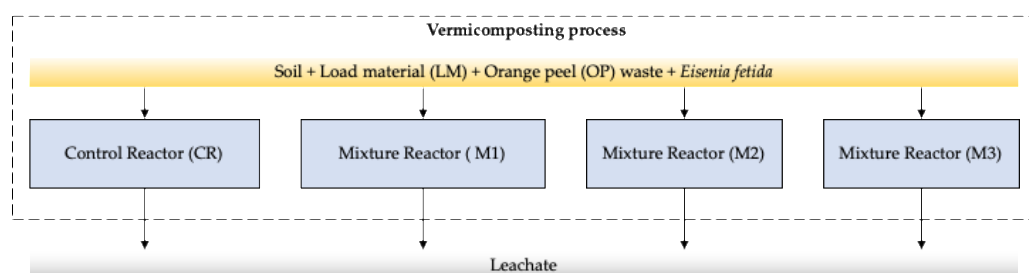


Figure 2. Vermicomposting process. Source: own elaboration.

The preparation for the reactors included the following combinations: (a) control reactor (CR)—50% OP waste (1 kg) + 50% LM (1kg), (b) mixture reactor (M1)—50% OP waste (1 kg) + 50% LM (1 kg) + 50 earthworms, (c) mixture reactor (M2)—60% OP waste (1.2 kg) + 40% LM (0.8 kg) + 50 earthworms and (d) mixture reactor (M3)—40% OP waste (0.8 kg) + 60% LM (1.2 kg) + 50 earthworms.

2.3. Physicochemical Parameters and Sensory Specifications

During the precomposting and vermicomposting processes, the following parameters were measured: temperature, pH, humidity, OM, total organic carbon (TOC), total nitrogen (TN), the carbon/nitrogen (C/N) ratio (Table 1) and sensory characteristics (smell and color). The procedures were determined by Mexican Standard NMX-FF-109-SCFI-2007, “Vermicompost (worm casting)—specifications and test methods”, and by local norms [10,27–29].

Table 1. Physicochemical parameters and tests.

Physicochemical Parameters	Test
pH	Potentiometer
Temperature	Environmental thermometer
Humidity	Gravimetric method
Organic matter	By calcination
Total organic carbon	From the percentage of organic matter, using the Van Bemmelen factor
Total nitrogen	Kjendahl method

Source: own elaboration.

2.4. Statistical Analysis

The physicochemical parameters (pH, temperature and humidity) are presented in Excel charts including error bars to show standard deviation. Data generated (OM, TOC and TN) were subjected to an analysis of variance (ANOVA) and pairwise comparison using Tukey's test ($p < 0.05$). The JMP 8.0 software package was used. All tests were performed in triplicate.

3. Results and Discussion

3.1. Precomposting Process

For the OP precomposting process, a period of 42 days of stabilization was required, which was in the range of 42 to 45 days reported by Favoretto et al., Mahaly et al. and Huang et al., highlighting the importance of time for this process using waste with similar chemical characteristics [16,26,30].

3.1.1. Temperature

Figure 3 shows the behavior of the environmental temperature and OP waste temperature, with values between 14 and 37.5 °C during the six weeks of this process.

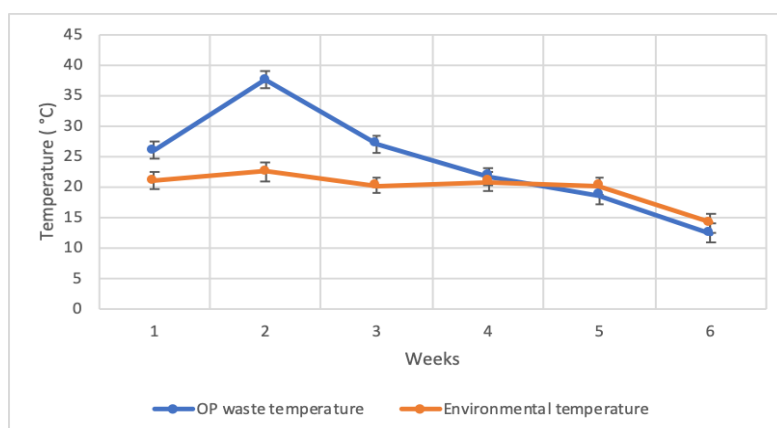


Figure 3. Temperature behavior in the precomposting process of orange peel (OP) waste.

This figure also shows that from weeks one to two, the OP waste temperature increased from 25.9 to 37.5 °C. This change was attributed to the biodegradation of organic substances; high temperatures constitute an indicator of success in the precomposting process [31–33]. This behavior has been reported in other studies, indicating that during the first five days of the precomposting process, temperatures increased from 20 °C to more than 55 °C, which assured the reduction of pathogens [34].

During week four, the OP temperature values were close to the environmental temperature (21.6 °C) due to the loss of biodegradable substances, which indicated the achievement of the maturation phase [35,36].

3.1.2. pH

Figure 4 shows the changes in the pH of the OP waste; its initial value was 5.93, and on week two, it increased to 6.09, remaining at a value of 6 during the last four weeks.

Hanc and Chadimova reported, during a two-week experiment consisting of precomposting apple pomace, an increase in pH from 4.0 to 6.7 due to the degradation of OM [37]. The literature consulted indicated the optimum pH range is 6 to 8.5; it is related to the formation of ammonium ions during the degradation of OM and the consumption of organic acids by microorganisms in the precomposting process [30,37,38].

The decrease in pH may be due to the formation of CO₂ and organic and humic acids [26,27,32]. When pH is 6 or above, it means that the OM is stabilized, and it is adequate for adding the earthworms into the vermicomposting process [39–41].

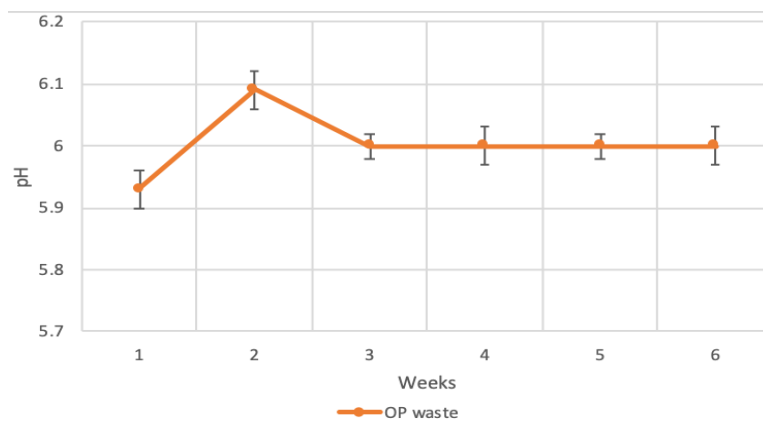


Figure 4. pH changes in precomposting OP waste.

3.1.3. Humidity

Figure 5 shows humidity behavior during OP waste precomposting. The humidity content was initially 84.12%, and at the end, it was 80.29%. Similar results have been reported for the precomposting process with fruit and vegetable waste, taking one to three weeks for humidity to decrease before conducting the vermicomposting process [32,42]. Based on the literature, humidity is an important factor, as it provides a medium for the transport of dissolved nutrients required for the metabolic and physiological activities of microorganisms. Nair et al. reported an initial humidity range between 80% and 85% for the precomposting process of food waste [43].

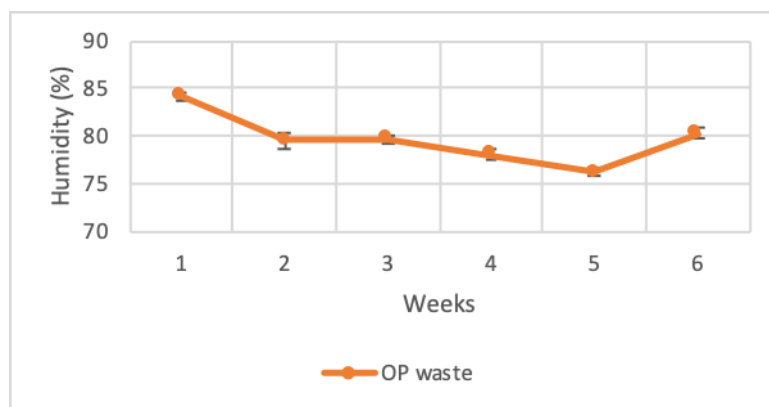


Figure 5. Humidity behavior during the precomposting process of OP waste.

3.1.4. Organic Matter (OM), Total Organic Carbon (TOC), Total Nitrogen (TN) and Carbon/Nitrogen (C/N) Ratio

The initial OM content for the OP waste was $78.33 \pm 1.84\%$, while the final OM value was $72.30 \pm 1.58\%$, representing the removal of 7.70%. FAO indicates that 50–70% is ideal for the OM parameter in the first 2–5 days at the beginning of the process [23]. Another study obtained a final OM value of 87.33% in vegetable and cow dung precomposting after 15 days [44]. The OM content decreased due to its mineralization by microorganisms to provide energy for microbial growth and metabolism [45,46].

The TOC for the OP waste began at $45.43 \pm 0.84\%$, coinciding with the 43.92% reported by Gelsomino et al. [47]. It is worth mentioning that TOC values can vary according to the parts integrated into the waste to be studied due to their physicochemical characteristics [43,48]. The final TOC for the OP waste was $41.96 \pm 0.69\%$, which was similar to the 38.1% reported for the precomposting of vegetables, fruits, green waste and tea leaves [49]. The carbon loss was the result of waste degradation by the action of microorganisms and the elimination of precomposted material as CO_2 and CH_4 [45,50].

The initial value of TN was $1.3 \pm 0.02\%$, similar to the 1.5% reported for OP waste [51]. The final value was $2.49 \pm 0.04\%$, which coincided with the 2.10% obtained in the precomposting process of orange waste [47,52]. The increase in TN could be attributed to the formation of NH_4^+ ions caused by the biodegradation of organic compounds [38,53]. The nitrogen flow is considered very efficient and effective when microorganisms are actively transforming the organic nitrogen; at the same time, plants are taking up the NH_4^+ and NO_3^- rapidly, which means the potential for nitrogen loss is relatively low [54].

The C/N ratio at the beginning was 33.65, which was in the recommended range of 25 to 30 [45,55]. At the end of the process, it decreased to the final value of 16.85. Kumar et al. reported a C/N ratio of 18.5 in a precomposting process of green waste and food waste [53]. According to several authors, a low C/N ratio is related to a stabilized product [48,55]. It has also been reported that the loss of OM decreases the weight of the precomposted material and reduces the C/N ratio [46]. Therefore, the final values obtained from this process were suitable for the vermicomposting process [56].

3.1.5. Sensory Characteristics of Precomposting

The change in the OP waste during the precomposting process is shown in Figure 6. On week one, the original color was orange, and its consistency was flexible, while on week six, it turned orange and brown and became rigid [32].

At the beginning of the experiment, the waste gave off penetrating odors; over time, they decreased, leading to an odor-free product. It is worth mentioning that the weight of the OP waste on week one was 10 kg, and on week six, it was 4 kg (a 60% reduction in weight) [57].



Figure 6. OP waste on weeks one and six of the precomposting process.

3.2. Vermicomposting Process

3.2.1. Temperature

Figure 7 shows the behavior of the temperature of the OP waste in the four mixtures (CR, M1, M2 and M3) during the vermicomposting process, which was performed for seven weeks. The four mixtures remained in the range of 20.96 to 22.2 °C during the first four weeks. From weeks five to seven, there were lower values in the range of 14.1 to 16.58 °C, mainly due to the climatic conditions.

Similar behavior was reported during the vermicomposting process using rice straw and cow manure waste, with an initial temperature of 27.5 °C and a final one of 17 °C, indicating that when the temperature decreased to values close to environmental temperature, the degradation of OM had almost completed and stabilized [58,59].

According to the literature, the temperature for earthworm development should not exceed 27 °C, and it must not be under 9 °C because it could cause their death [18,60]. The temperature values of the four mixtures stayed in this range, improving the microbial activity and the growth of earthworms, which could quickly stabilize the waste in the mixtures [18,61].

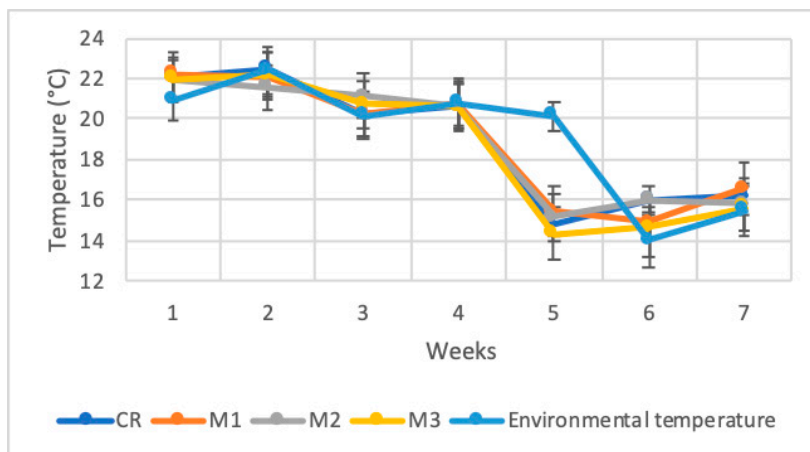


Figure 7. Temperature changes in the vermicomposting process of OP waste.

3.2.2. pH

Figure 8 shows the pH during the seven weeks of the vermicomposting process for the OP waste in the four mixtures registered values from 6.52 to 7.5. The pH at the end of the precomposting process was 6 (Figure 3); however, when the LM and the eggshells were added, the pH increased until it reached 6.5 to 7.2, corresponding to the initial pH values from this process. This change was probably due to the contribution of the eggshells, with a pH of 10.3 [17,62]. The final values from the process were from 7 to 7.3. The changes in pH were due to the degradation of OM through the release of organic acids and the mineralization of nitrogen [63,64].

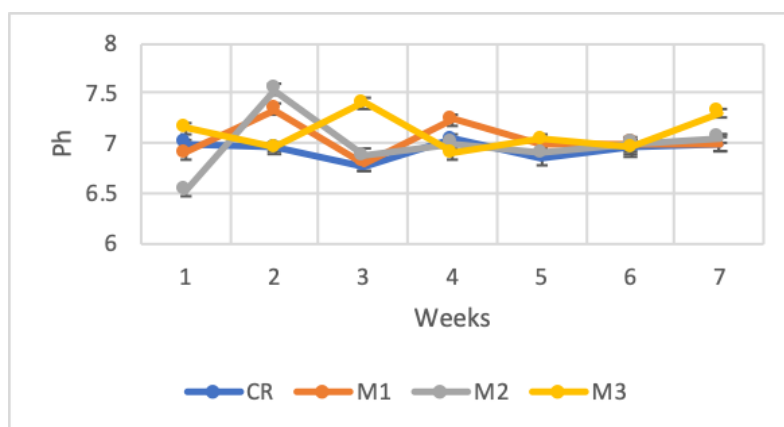


Figure 8. pH changes during the vermicomposting process of OP waste.

Similar results have been reported during the vermicomposting process with green waste, cattle manure and dried leaves, with an initial pH of 6.1 and a final pH of 7.10, indicating that this increase in pH was dynamic and dependent on the substrate due to the mineralization of the protein that produces alkaline ammonia or the loss of volatile acids with CO₂ release. The decreasing values could be because of the production of humic and fulvic acids [58,63]. Several authors have reported the adequate pH range for the vermicomposting process is between 5.5 and 8.0 [18,65].

3.2.3. Humidity

Figure 9 shows the humidity content in the four mixtures (CR, M1, M2 and M3) registered for the vermicomposting process, which remained within the range of 42.5% to 83.6%. CR and M1 had the lowest humidity content during the first four weeks. M2 and M3 showed the highest percentage of humidity during the whole process.

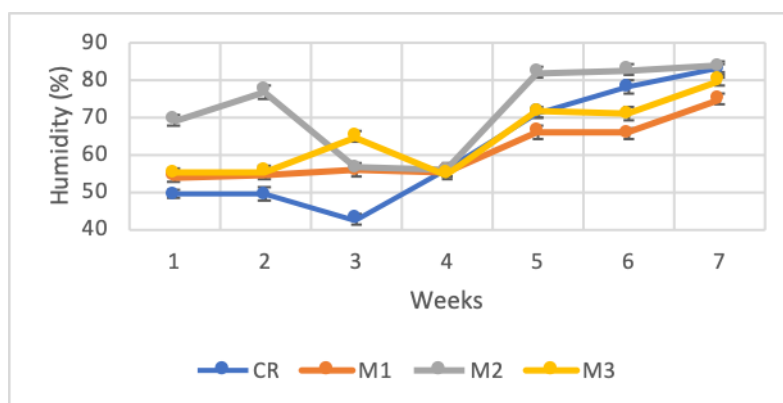


Figure 9. Humidity content during the process of vermicomposting of OP waste.

In the first four weeks, low humidity contents were registered, probably as a consequence of the biodegradation of OM during the vermicomposting process [66]. The humidity content is crucial for the controlled evolution of the biodegradation process [67]. According to the literature consulted, most of the authors reported humidity values from 60% to 80%, indicating a suitable environment for the rapid growth of *Eisenia fetida* to ensure their optimal survival [57,59].

3.2.4. Organic Matter (OM), Total Organic Carbon (TOC), Total Nitrogen (TN) and Carbon/Nitrogen (C/N) Ratio

The physicochemical characteristics, OM, TOC, TN and the C/N ratio, of the four mixes (CR, M1, M2 and M3) studied before the vermicomposting process are shown in Table 2. These were determined using the precomposted material added with the LM in the percentages indicated under the table.

Table 2. Characteristics of the four mixtures with OP waste before vermicomposting.

Parameters	CR	M1	M2	M3
OM (%)	76.97 ± 1.20 a	76.97 ± 1.20 a	73.93 ± 1.22 b	72.88 ± 1.16 b
TOC (%)	44.65 ± 0.71 a	44.65 ± 0.71 a	42.89 ± 0.91 b	42.28 ± 0.67 b
TN (%)	2.12 ± 0.04 a	2.12 ± 0.04 a	1.20 ± 0.02 b	1.60 ± 0.03 c
C/N Ratio	24.93	24.93	38.20	26.16

CR = control reactor—50% OP, 50% load material (LM); M1 = reactor—50% OP, 50% LM; M2 = reactor—60% OP, 40% LM; and M3 = reactor—40% OP, 60% LM. Mean values (mean ± SD, $n = 3$) followed by different letters are statistically different (analysis of variance (ANOVA); Tukey's test, $p < 0.05$).

Table 3 shows the values obtained at the end of the seven weeks of the vermicomposting process for the four mixes.

On the first week, the four mixtures, CR, M1, M2 and M3, initially registered an OM content of 76.97%, 76.97%, 73.93% and 72.88%, respectively (Table 2), ending with 63.79%, 64%, 65.80% and 64.88% (Table 3). M2 and M3 were the mixtures with the highest percentages of removal, with 11% and 10.97%, respectively.

Table 3. Characteristics of the four mixtures with OP waste after vermicomposting.

Parameters	CR	M1	M2	M3
OM (%)	63.79 ± 0.78 a	64 ± 1.14 a	65.80 ± 0.69 a	64.88 ± 0.86 a
TOC (%)	37 ± 0.64 a	37.13 ± 0.71 a	38.17 ± 0.53 a	37.64 ± 0.79 a
TN (%)	3.50 ± 0.08 a	4.50 ± 0.10 b	6.30 ± 0.13 c	3.78 ± 0.09 d
C/N Ratio	11.74	8.80	6.05	9.95

CR = control reactor—50% OP, 50% LM; M1 = reactor—50% OP, 50% LM; M2 = reactor—60% OP, 40% LM; and M3 = reactor—40% OP, 60% LM. Mean values (mean ± SD, $n = 3$) followed by different letters are statistically different (ANOVA; Tukey's test, $p < 0.05$).

Other authors have also reported a decrease in OM percentages, with an initial content of 95% and a final one of 55.3% for the vermicomposting process with fruit and vegetable waste [60,68]. The loss of OM during the vermicomposting process was attributed to the synergistic effect of the earthworms and microorganisms [19,68].

The OM percentage at the beginning of the vermicomposting process was significantly different between the mixtures CR and M1 in comparison to M2 and M3. However, the final OM values for the four mixtures did not have significant differences.

At the beginning, the mixtures CR, M1, M2 and M3 had TOC values of 44.65%, 44.65%, 42.89% and 42.28% (Table 2), and at the end, the values were 37%, 37.13%, 38.17% and 37.64% (Table 3), respectively, with percentages of removal of 17.13%, 16.84%, 11% and 10.97%. CR had the highest percentage of removal.

The initial TOC content values for the four mixtures decreased, coinciding with the information reported in the literature due to the combined action of earthworms and microorganisms, resulting in the homogenization, fragmentation and mineralization of organic materials, which in turn significantly reduced OM [65,69]. The decrease in TOC values can be attributed to the degradation of OM as a result of the conversion to carbon dioxide by earthworms and microbial respiration as well as assimilation in the form of biomass [63,70].

Initial TOC percentages for all four mixtures showed significant differences between the CR and M1 mixtures compared to M2 and M3. Regarding the final TOC values, there was no significant difference.

CR, M1, M2 and M3 had initial TN values of 2.12%, 2.12%, 1.20% and 1.60% (Table 2) and final contents of 3.50%, 4.50%, 6.30% and 3.78% (Table 3), respectively. In the four mixtures, the initial TN content indicated the existence of adequate conditions for the vermicomposting process. The increase in the TN concentrations at the end of the process was due to the loss of carbon in the form of CO₂ through the decomposition of the substrate. Besides, the excreta and polysaccharides of earthworms as well as tissue decomposition from dead earthworms enriched the TN concentration in the reactors [21,66,71,72].

The results obtained in this research also coincided with other experiments with OP waste, cattle manure and filter cake, reporting an initial TN content of 1.57% with variations throughout the process, finally obtaining 3.37% in 105 days [16,73].

The initial concentrations of TN among M1, M2 and M3 had significant differences. Among the CR and M1 mixtures, there was no such difference. Regarding the final values, the four mixtures had significant differences. It is important to highlight that the highest concentrations were in the mixtures M1 and M2.

The initial C/N ratio of the four mixtures was 24.93 (CR), 24.93 (M1), 38.20 (M2) and 26.16 (M3) (Table 2), coinciding with values of 23 (cow dung and green waste) and 29.10 (green waste, dry leaves, small branches and paper sheets) and corresponding to a great loss of carbon accompanied by a nitrogen increase [16,32,64,69].

The final values of the C/N ratio for the four mixtures were 11.74 (CR), 8.8 (M1), 6.05 (M2) and 9.95 (M3) (Table 3). The decrease in the C/N ratio was attributed to the continuous loss of carbon as CO₂ through microbial respiration and nitrogen production during the process [72]. The values registered in this experiment were under the value of 20 that is indicated by Mexican Standard NMX-FF-109-SCFI-2007 for quality vermicompost [10]. The decrease in the C/N ratio is indicative of

increased humification of OM, which is related to the quality and maturity of vermicompost [16,21,32,74]. Other studies reported that a C/N ratio lower than 20 was adequate in the vermicomposting process due to a stable evolution of the degradation process and mature vermicompost [74]. The application of vermicompost with a low C/N ratio has a beneficial impact on the soil [75].

4. Conclusions

Throughout this experiment, it was demonstrated that the vermicomposting process was feasible for degrading OP waste, obtaining a quality product according to the final physicochemical characteristics to be applied as a soil improver. The LM added to all the mixtures contributed to enriching the characteristics of the substrate to balance the nutrients required for a biological process.

The precomposting process was necessary to make the substrate adequate in such a way that the earthworms could degrade the OM efficiently, facilitating the vermicomposting process.

The precomposting and vermicomposting processes are extremely sensitive; for this reason, the monitoring of the parameters of temperature, pH, OM, TOC, TN and the C/N ratio was crucial to keep them under control.

All the mixtures had final values between the range established for quality and mature vermicompost; however, the highest concentrations for TN were in the mixtures M1 and M2.

The vermicomposting process is a viable alternative to valorize OP waste, contributing to an integral treatment to avoid inadequate final disposal that generates environmental impacts on the air, soil and water.

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