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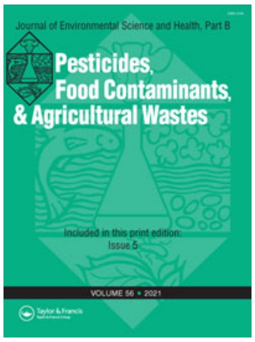

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Composting of winery waste and characteristics of the final compost according to Brazilian legislation

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ABSTRACT

The waste generated in the production of wine and grape juice is characterized by a high concentration of organic matter, when properly treated, can serve as sustainable strategies for its use and destination, and among these, the production of biocompost. Thus, the objective of this study was to evaluate the process of composting grape marc, sheep manure, and mango leaves, evaluating in the biocompost its physical-chemical, nutritional and microbiological characteristics for use in agriculture. The composting pile assembly followed the proportion of 30% of sheep manure as nitrogenous material and 70% of carbon-rich material (divided into 50% of grape marc and 20% of hose leaves), the initial C/N ratio was 33:1, and the process lasted 120 days according to legislation. When evaluating the results, the process occurred in an accelerated manner, where at 30 days the biocompost was already stabilized, and at the end of the process (120 days) it presented a C/N ratio of 5.85, as well as acceptable levels for the macronutrients K and P, and without risk of phytotoxicity, and could be used as organic fertilizer or as soil conditioner, reducing environmentally inadequate destination and generating savings with their reinsertion in the production chain.

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Agricultural reuse; agroindustry; biocompost; C/N; organic waste

Introduction

In terms of world fruit production, grapes stand among the five most-produced fruits[1] and, in 2018, according to data from the International Vine and Wine Organization (OIV),[2] this production was equivalent to nearly 78 million tons, with a world vineyard area of 7.4 million hectares. This, in relation to 2017, meant a growth of 42.5 million hectoliters (m hl) in production, reaching 292.3 million hl in 2018.

In the Northeast region, the Sub-Middle São Francisco is recognized by fruit farming, with viticulture characterized by the high production of grapes for both fresh consumption and for the production of wines and juices,[3] being the second main grape-producing region of Brazil.[4]

However, the wine sector has faced several questions related to the generation of grape wastes in the production process of juices and wines.[5] These residues originated from agro-industrial activities present, as their main characteristic, high concentrations of organic matter, which can cause major environmental impacts due to the formation of organic acids during the fermentation of the waste, generating foul smells, reducing the content of dissolved oxygen in water bodies, and contributing to soil contamination when improper disposal occurs.[6] In this perspective, the sustainable management of residues becomes an important ally with regard to their reduction or reuse.[7]

Regarding grape wastes produced in the vinification process, the quantity and quality vary considerably as a function of the vineyard size, methods, and equipment used in fruit pressing, as well as grape variability. All these factors collaborate in the production of distinct data on the generation of waste per ton of crushed fruit.[8]

Zhang et al.[9] cite that the main organic solid wastes in wine production include grape pomace, wine lees, stalks, and sludge. Grape pomace represented nearly 25% of the total weight of the grape used in the vinification process,[10] being usually composed of peel, stalks, seeds, and moisture, besides other components such as organic acids and polyphenols. The red grape pomace also presents residual sugars and some amounts of alcohol (ethanol).[9,11] And according to Tonon et al.,[12] it is estimated that only 3% of this waste is used for soil fertilization by the wineries themselves.

Requejo et al.,[13] mention that among the organic wastes generated in the winemaking process, grape marc stands out due to its high content of organic matter and nutrients, and can be used to meet part of the needs for use in agriculture. Thus, the high waste production demands the use of sustainable strategies for its use and destination, among them the production of organic fertilizers through composting.[14]

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However, for its use to be possible, the waste produced needs to meet the existing legislation, such as Normative Instructions n° 27/2006[15] and 25/2009[16] of the Ministry of Agriculture, Livestock, and Supply (MAPA) and Resolutions n° 481/2017[17] and 375/2006[18] of the National Environment Council (CONAMA). All this current legislation allows the use of organic waste in agriculture as a biofertilizer, soil conditioner, or substrate as long as it observes the maximum concentrations of heavy metals, pathogens harmful to human, animal, and plant health, and also it must not be a vector of plagues or contain weed seeds.

Composting is a biotechnological process through which the polymeric substances present in organic wastes are degraded by the hydrolytic enzymes released by mesophilic and thermophilic organisms during mineralization in aerobic conditions.[19] The products of the composting process are carbon dioxide and stable carbon forms, resulting in the decomposition and mineralization of organic matter and the production of humic substances.[19,20]

The process follows the following phases: the first is mesophilic, which is characterized by the accelerated growth of fungi and bacteria due to the quick absorption of starch and soluble sugars; the second phase classified as thermophilic or thermophilic, is characterized by microbial enzymes that degrade proteins, hemicellulose, and cellulose; the third phase being considered the second mesophilic phase (cooling phase) occurs when the activity of the thermophilic microorganisms ceases with the exhaustion of the substrates; finally, the maturation phase is characterized by the formation of humic substances.[21,22]

With that, the biocompost obtained at the end of the process must present high quality being rich in nutrients and beneficial microorganisms, besides being safe to use. The properties of this biocompost can still be improved if, during the process, other types of residues are added, such as manures, green residues, and even urban wastes, increasing the nutrient content and biological activity.[14,23]

Furthermore, the biocompost tends to present high contents of organic matter, phosphorus, and potassium.[24] It was also reported that the phytotoxicity of composting products is reduced when co-composted with animal manure.[25,26]

However, the production of data on organic wastes from winemaking is quite variable, and the proportion in which each component makes up the bagasse depends on several factors, such as soil type, grape variety, climate, harvest,[12] processing methods and production site.[27] Thus, the biocompost produced will present specific characteristics of the region.

Therefore, the work aimed to evaluate the composting process of grape marc, sheep manure and mango leaves, evaluating in the final compost its physical-chemical, nutritional and microbiological characteristics for use as a biofertilizer/soil conditioner in accordance with Brazilian legislation.

Materials and methods

The composting process was performed in the experimental area of the Federal University of São Francisco Valley (UNIVASF), Campus Juazeiro-BA, located at latitude 09°24′ S, longitude 40°31′ W, and an elevation of 371 m. The climate is classified as BShw, dry steppe climate of low latitude with summer rains – semiarid climate, according to the Köppen-Geiger Köppen Climate Classification, with a mean temperature of 24.8 °C and mean annual rainfall of 422 mm. The duration of composting process was 120 days (March to July 2019), as suggested by the Ministry of the Environment[28] for the biocompost to reach humification (completion of the process).

The composted wastes were: grape marc and sheep manure, from the 'Quintas de São Braz' winery, located in Petrolina-PE, being the grape marc obtained by the process of grape pressing for the production of red wine, and constituted by the mixture of peel, seeds, stalks, and moisture. Sheep manure was used as a nitrogen-rich material, also employing mango leaves as a straw material.

The grape marc was subjected to a physical pretreatment with sun exposure drying for 10 days in an open yard, for total volatilization of the alcoholic content. Afterward, the grape marc was crushed to reduce the particle size of the material (varying from 1 to 5 centimeters), and the mango leaves was used in its normal size (varying from 15 to 22 cm of length). The composting pile was assembled according to Pereira Neto,[29] in an open yard with concrete floor, observing a proportion of 70% of carbon-rich material (divided in 50% of grape marc and 20% of mango leaves) for 30% of sheep manure (rich in nitrogen). The height of the pile was 70 cm, and during this period, the pile was regularly irrigated to maintain moisture and the material was manually turned every 15 days to maintain aeration.

To perform the material analyses, samples were collected in the middle part of the pile, so that it was subdivided into four equidistant sections (Fig. 1). For the collection of 4 subsamples that formed one compound sample for physical, chemical and microbiological characterization. The removal of the subsamples was performed with the aid of a PVC tube with 2.5 cm diameter, which was inserted into each section at a depth of 30 cm from the surface.[21]

The temperature of the pile was daily monitored using a chemical thermometer, with measurements in the following positions: top (55 cm), middle (35 cm), and base (15 cm), besides performing the thermographic imaging using a FLIR® thermal camera, model T420.

The analyses were performed at the Environmental Engineering Laboratory-LEA of Federal University of São Francisco Valley (UNIVASF) and the Agro-environmental Laboratory of Brazilian Agricultural Research Corporation (Embrapa-Semiarid).

The parameters were carried out in triplicate to calculate averages and standard deviation, and the data were compared to the values defined by the Normative Instruction (NI) n° 25/2009[16] and 27/2006[15] of the Agricultural Defense Secretariat (SDA) of the Ministry of Agriculture, Livestock and Supply (MAPA), and the Resolution n° 375/2006 of the National Environment Council (CONAMA)[18] (Table 1).

Results and discussion

The evolution of temperature and moisture are related to microbial activities and biological reactions, as well as, associated with the capacity of the process to reduce pathogens.[35] These were the main physical parameters monitored during the composting process.

The compost pile consisted of a mixture of grape marc, sheep manure and hose leaves did not present any problems with the temperature evolution, as the values in the pile increased rapidly (Fig. 2) and reached the thermophilic phase (>40 °C) on the 3rd day of the process and lasted for 10 days. This behavior is associated with the initial Carbon/Nitrogen ratio, of 33:1 (Table 3), range within the cited by Kiehl[36] as being a good C/N ratio (25:1 to 35:1). During the thermophilic phase, the highest temperature (57 °C) was noted at the top of the pile on the 5th day of the process. This occurred due to the high content of decomposable organic matter, favoring the activity of aerobic microbes.[37]

At the end of the thermophilic phase (13th day), the temperature dropped gradually for the environmental values as the biological activity ceased. This temperature decrease meant that most organic components were consumed during the mesophilic and thermophilic phases. Similar conditions were verified by Asses et al.,[38] when composting a mixture of sewage sludge and oil production wastes, and, according to Meng et al.,[39] few easily degraded compounds remained in the last phase or in the cooling phase.

In the present study, grape waste composting process presented an increase in temperature on the 30th day, to 37 °C. According to Bernal et al.,[40] this temperature oscillation (30–45 °C) during the cooling phase occurs due to the permanence of a reduced bacterial population together with more complex molecules to be degraded.

According to the Resolution nº 481/2017 of the National Environment Council (CONAMA),[17] for open composting systems the temperatures must be above 55 °C for at least 14 days in order for the sanitation of the compost to occur, that is, for the reduction/elimination of pathogenic organisms. Zhang and Sun,[41] cite that the temperature in the range of 50–60 °C for at least 3 days indicates that the biocompost is free of pathogenic organisms, and this condition was achieved in this study from the 5th to 10th day of process (Fig. 2).

According to Bustamante et al.[42] and Paradelo et al.,[43] the addition of grape marc without residual alcohol (like the one used in this study) can raise the temperature of composting, especially in the thermophilic phase, guaranteeing the removal of pathogens and undesirable organisms. Therefore, if only the temperature parameter was considered, the biocompost produced would already be able to be used in agriculture because it reaches the necessary values for the removal of pathogens, there is no risk to man and soil.

The effect of the addition of mango leaves in the mixture and also of the presence of stalks, which present a lignocellulosic nature, may have affected the bacteriological activity during composting, leading to a shorter thermophilic phase and a longer cooling phase (Fig. 2), revealing a more dominant fungal activity. Corroborating to this, Figure 3 presents

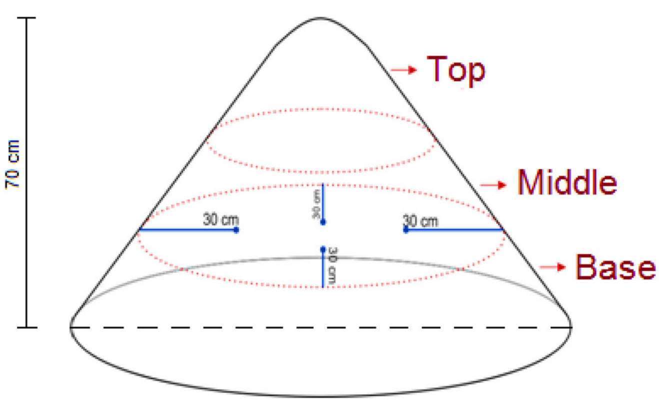

Figure 1. Sample collect in the compost pile.

Table 1. Parameters analyzed, sampling frequency and methodology of the analyses performed during the composting process.

Parameters	Sampling frequency	Methodology
	Physical-chemicals	
Temperature (°C)	Daily for the first two weeks, then every 15 days until the 120th day	APHA[30]
Moisture (%); pH; electrical conductivity (mS/cm)	Monthly	Teixeira et al.[31]
Organic matter (%)	Monthly	Goldin[32]
Volatile solids (g L^{-1})	Monthly	APHA[30]
Organic carbon (g kg^{-1})	Monthly	Teixeira et al.[31]
Total nitrogen, ammoniacal nitrogen and nitrate (mg kg^{-1}); organic nitrogen (%)	Monthly	APHA[30]
Particle distribution (%); Density (g cm^{-3})	Monthly	Teixeira et al.[31]
Volume (%)	Final	Geometric calculus[a]
	Nutricional	
Calcium (Ca), phosphorus (P), potassium (K), sodium (Na), magnesium (Mg) and exchangeable aluminum (Al) (g kg^{-1})	Monthly	Teixeira et al.[31]
Iron (Fe), copper (Cu), zinc (Zn), chrome (Cr), lead (Pb) and nickel (Ni) (g kg^{-1})	Initial and final	USEPA[33]
	Microbiological	
Total coliforms and *Escherichia coli* (NMP g^{-1} de ST)	Final	APHA[30]
	Other variables	
Smells, insects, spiders, fungi, ants and flies	Monthly	Rodrigues et al.[34]

[a]Geometric calculus of the collectors used, these, made of high density polyethylene (HDPE) and with the following internal dimensions: 51.5 cm wide, 32 cm long and 28 cm high, totaling a volume of 45.78 cm^3.

thermal images during the process, where at 13th day (Fig. 3c) it is possible to note the beginning of the cooling phase lasting until 120th day. Bustamante et al.[44] reported the same result on the effect of the lignocellulosic fraction of grapevine branches during the composting with sludge of anaerobic digestors, such as reported by Asses et al.[38] when composting sludge with olive and pruning wastes.

With that, when observing the evolution of temperature (Fig. 2), it is possible to conclude that the biocompost already presented the characteristics of the finalized biocompost from the 30th day of the composting process, which would result in a biofertilizer with C/N ratio of approximately 18:1 (Table 3), a value close to that recommended by the legislation (20:1). In the Sub-Middle São Francisco Valley, a region of semiarid climate, with climatic conditions characterized by high temperatures, low rainfall, and air humidity, but that presents water via irrigated perimeters, this condition potentializes the degradation process of soil organic matter. In this manner, the sandy soils of the region, which constantly need organic matter, can be favored by the composting process in less time for greater maintenance of organic matter in the soil.

Figure 4 exhibits the evolution of the moisture content, which began with 60% and presented later reduction to 40% at the end of the process. With that, the moisture content was maintained within the range considered adequate, within 40–60%.[45] In case the moisture content in the composting pile was below 40%, it would lead to a reduction in the microbial activity, and if above 60%, there would occur the obstruction of the porous spaces between the particles, damaging aeration.[46]

For Asses et al.,[38] among the chemical properties that indicate the adequate progress of the composting process are pH and electrical conductivity (EC), which are represented in Figure 5.

The characteristic profile of the pH (Fig. 5) in the composting process began in 6.51, increasing to values above 8.0 in the thermophilic phase, and closing the cycle in 7.0. This result is related to the bioconversion of organic acids in aerobic conditions and to the mineralization of proteins, peptides, and amino acids, leading to ammonification followed by pH increase.[47] The values verified during the process kept within the adequate range, which must be equal to or above 6, following the NI n° 25/2009 (MAPA).

Table 2. Evolution of reduction of volatile solids, organic carbon and organic matter during composting.

Time Days	Volatile solids g L^{-1}	Organic carbon g kg^{-1}	Organic matter
1	508.28 ± 10.06	410.25 ± 23.50	896 ± 22.00
30	130.40 ± 15.16	228.00 ± 3.39	648 ± 19.01
60	120.45 ± 28.63	113.12 ± 8.15	571 ± 8.50
90	119.12 ± 32.11	95.61 ± 3.35	570 ± 31.15
120	54.13 ± 14.05	119.31 ± 12.50	522 ± 27.05

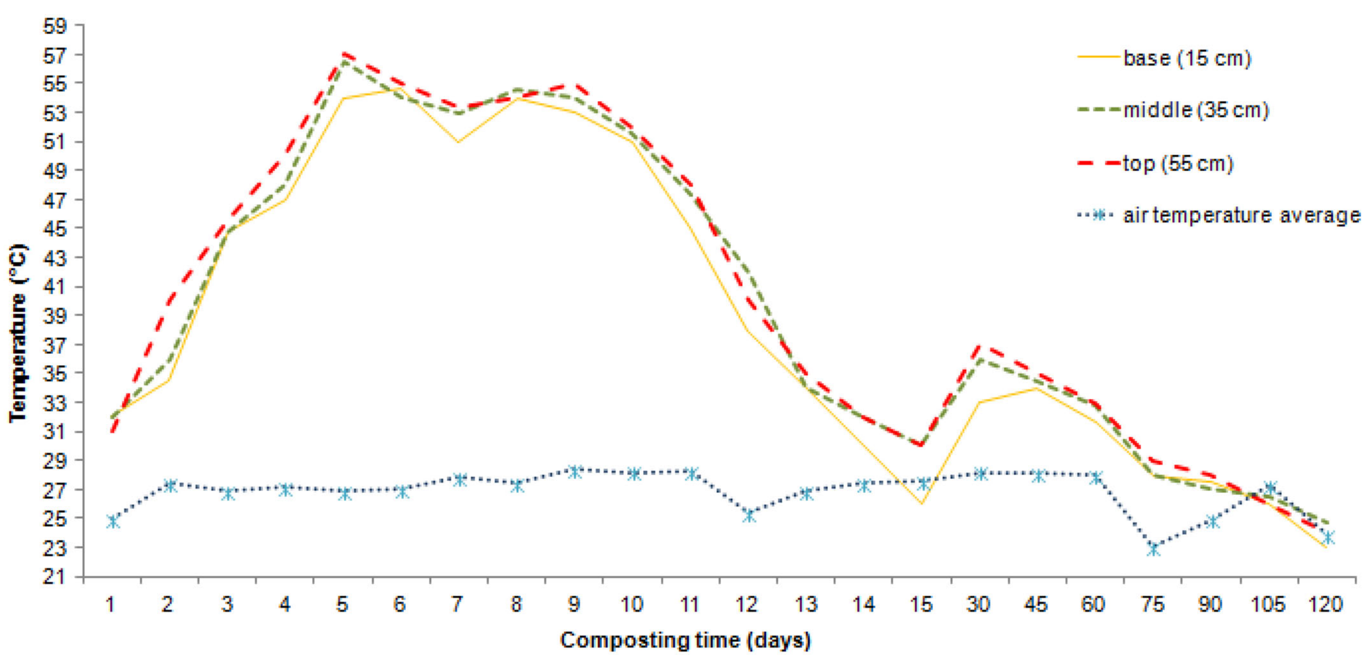

Figure 2. Temperature evolution in the compost pile.

Table 3. Nitrogen and C/N ratio during composting.

Time days	Total nitrogen g kg^{-1}	C/N –	Ammoniacal nitrogen N-NH_4^+ mg kg^{-1}	Nitrate	Organic N % of total N
1	12.17 ± 0.50	33.00:1	9.36 ± 0.36	206.67 ± 7.15	99.91
30	12.56 ± 0.50	18.14:1	1.25 ± 0.16	0.62 ± 0.10	99.98
60	13.10 ± 0.40	8.63:1	0.90 ± 0.18	0.90 ± 0.18	99.92
90	14.30 ± 0.20	6.68:1	0.28 ± 0.09	0.24 ± 0.10	99.99
120	20.37 ± 1.51	5.85:1	1.80 ± 0.02	2.34 ± 0.48	99.99

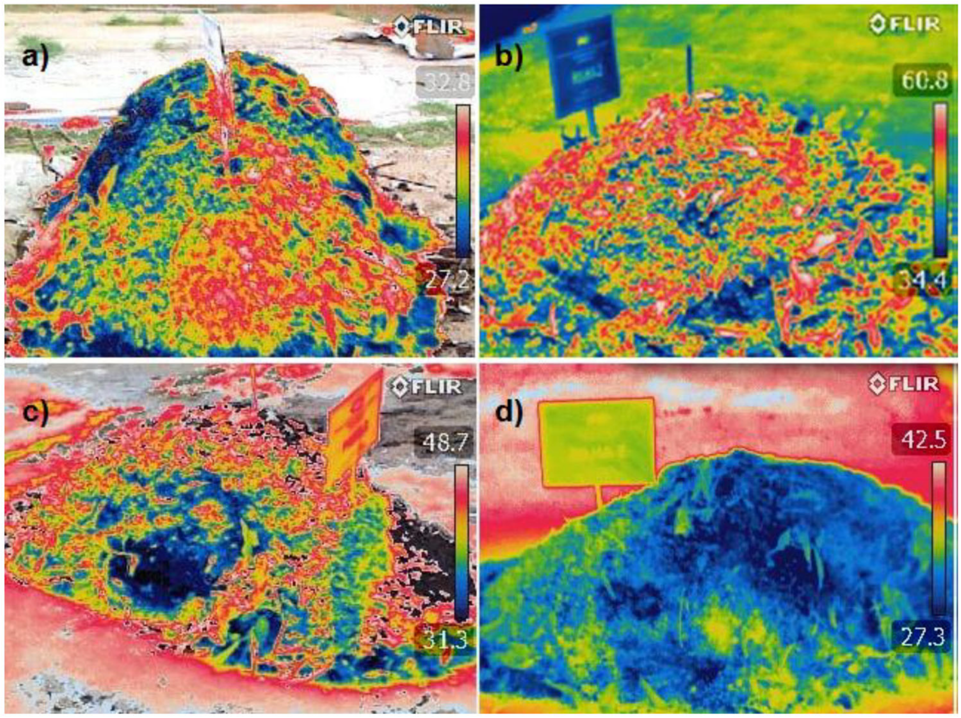

Figure 3. Thermographic image representing the phases: (a) mesophilic, on the 2th day process; (b) thermophilic, on the 4th day; (c) second mesophilic phase, 13th day and (d) humification, at 90 days.

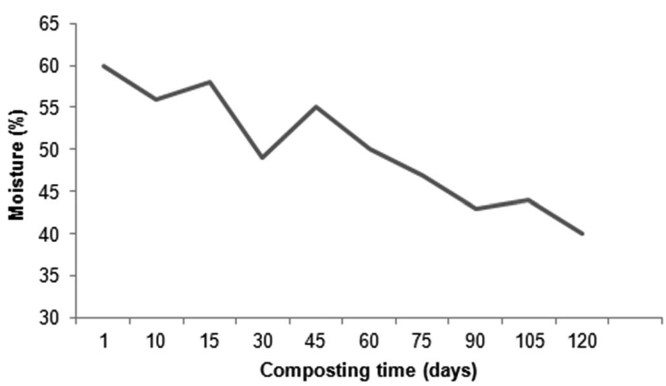

Figure 4. Moisture evolution during composting process.

The electrical conductivity (EC), used as an indirect measurement of the amount of salts present in the material, serves as a parameter to estimate the salinity of the biocompost[48] Figure 5 presents the values of electrical conductivity, which had its greatest reduction (from 1.69 to 0.24 mS cm^{-1}) during the first month of the composting process, that is, in the first mesophilic phase and in the thermophilic stage, with final values representing a reduction of almost 86% of the initial. This result is related to the transformation of organic matter into humic substances, generating a cation exchange capacity (CEC) that retains cations, causing a reduction of the electrical conductivity since it quantifies the non-adsorbed and soluble salts[49] However, the same did not occur to Paradelo et al.[43] during the composting of vinification lees mixed to grape pomace, in which the EC remained with high values since the lees hindered drainage in the composting process.

The results verified regarding a pH of 7.0 and the EC of 0.24 mS cm^{-1} indicate a biocompost with a corrective effect in soil acidity and that does not present a risk of salinization, can be used in the reduction or elimination of expenses in the correction of the acidity of the soil as cited by Andrade and Abreu.[50]

Furthermore, regarding the chemical properties, Table 2 presents the evolution of organic matter degradation with the reduction of the organic fractions throughout the process.

Regarding volatile solids (VS) (Table 2), Varma et al.[51] observed a drastic reduction due to the high temperatures, and as the composting process advanced, the microbial activity acting in the organic content provided that more VS were removed. The same happened in this study, in which at 30 days of the composting process the biocompost presented higher removal (74.34% of the concentration), and, at the end of the process (120 days) the total removal was 89.35%. With that, the result obtained in the composting process complies with CONAMA Resolution n° 375/2006, which cites that the concentration of VS must be reduced to a minimum of 38% of the concentration. This demonstrates that in the present study, performed in a region of semiarid climate, the biocompost with 30 days of composting process reached almost the double of the minimal VS removal suggested by the legislation, presenting several advantages related to cost and time of the process.

Regarding the organic carbon (Table 2), the highest removal occurred at 30 days of composting, with 44.4%. However, other authors obtained much lower values, such as Varma et al.,[52] when composting a mixture of plant residues, bovine manure, sawdust, and dry leaves (proportion of 5:4:1:0.9) for a period of 20 days, removing 11.4% of organic carbon, and Varma et al.[51] with the same residues (proportion of 9:1:1:0.9) and same composting time,

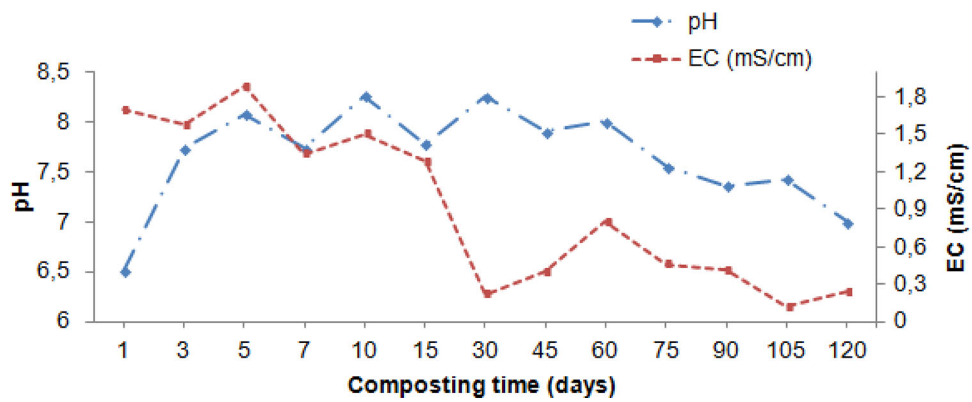

Figure 5. pH and electrical conductivity evolution during the composting process.

obtained a higher removal of 4.89%. According to NI n° 25/2009, the minimum concentration of organic carbon established for use as fertilizer is 15%, a value attended to at 30 days of the process (22.8%). Furthermore, throughout the composting process, it was verified that the removal of the organic carbon presented stabilization of concentrations from 60 days onwards. This is explained by the possible reduction in microbial activity due to the scarcity of carbon and nutrients already consumed in the first mesophilic phase and in the thermophilic phase. This behavior is in agreement with the third phase of the composting process, maturation.[21,22]

The highest organic matter removal occurred during the first month of the composting process, with about 27.6%, and, at the end of the process, the total removal was 41.68%. These values are below the interval from 55 to 68% of the normally registered removal for the composting with sewage sludge, animal manure, and olive oil processing wastes with different lignocellulosic materials.[53] According to Cayuela et al.,[54] the incorporation of grape stalks in the mixture may have contributed to a reduction in the mineralization of organic matter since the stalks present greater variability as to shape, reducing the available surface for microorganisms.

The evaluation of the C/N ratio, as well as the variations in the total nitrogen content and its fractions throughout the composting time are described in Table 3.

During the composting process, the total nitrogen (Table 3) presented an increase in the final value of 20.37 g kg^{-1} and, according to De Bertoldi et al.,[55] this increase can be explained by the mineralization of organic matter and the consequent loss of H_2O and CO_2, causing a reduction in the C/N ratio. It is noted that from 30 days of the process onwards the biocompost was already stabilized, presenting N values of 12.56 g kg^{-1} and C/N ratio of 18.14, close values to those obtained by Albuquerque et at.,[56] who, when composting a mixture of grape stalk, grape pomace, and goat manure (proportion of 1:1:7:1), after 50 days of the process presented results of 13.92 g kg^{-1} of N and 13.9 of C/N, demonstrating that the biocompost was ready to be used.

The C/N ratio at the end of the 120 days of the composting process was 5.85 (Table 3), a value considered low. However, several studies present wide ranges of C/N ratio,[57] and according to Salgado et al.,[14] a low C/N ratio allied to a low population of bacteria, can promote higher contents of humic substances, resulting in a good soil conditioning.[14] This ratio represents a strong connection within the microbial activity since fungi or bacteria use 30 parts of cellulose for one part of N in the process of biodegradation. Thus, C works as an energy source and an essential component for microorganisms, whereas N is employed in the formation of amino acids, proteins, and nucleic acids.[14] Furthermore, an increase in the availability of total N by the process of biological degradation is verified in Table 3, as well as a decrease in the C/N ratio in relation to the composting time. The reduction of this ratio has been indicated by several authors as an indicator of stability in the composting process although, given the great variation in the raw-materials employed in the process, not only use this ration as indicator.[47]

The result obtained in the C/N ratio (5.85:1) is considered adequate by the CONAMA Resolution n° 481/2017, which cites that the composting process should guarantee a carbon/nitrogen ratio in the final biocompost lower or equal to 20:1.

Regarding the amoniacal nitrogen (N-NH_4^+) loss (Table 3), it is related to the alkaline pH range, which raises the volatilization rates of this gas.[58,59] Gigliotti et al.[47] cite that no increase of N-NH_4^+ at the beginning of the composting process was expected due to the use of cellulose, hemicellulose and lignin-rich wastes, which present slower degradation.

During the composting process, organic nitrogen was the main constituent of total nitrogen (Table 3). This can be proved by analyzing the inorganic or mineral fraction through the concentrations of ammoniacal nitrogen (N-NH_4^+), initially corresponding to 9.33 mg kg^{-1} (0.08% of total N) and at 120th days with 1.80 mg kg^{-1} (0.009% of total N), both values considered very low in relation to organic N. Such results are inferior to those by Gigliotti et al.[47] when composting a mixture of olive wastes, olive pruning, and cereal straws, obtaining 2.2 and 5.3% of mineral N in relation to the total N at the beginning and at the end of the process, respectively.

Regarding the particle size of the fertilizers, the smaller the particle, the greater the contact surface of the product in the soil, affecting the dissolution rate and also the absorption of moisture. Furthermore, another important factor is

Table 4. Particle distribution and density during composting.

Time	Sieve mesh (mm) (>4)	(4–2)	(2–1)	(1–0.5)	(0.5–0.3)	(0.3–0.125)	(<0.125)	Density
days	Particle distribution (%)							$g\ cm^{-3}$
1	45.60	34.88	6.92	1.61	0.72	8.56	1.70	0.051
30	13.74	38.46	11.74	5.917	5.44	20.52	4.18	0.052
60	29.69	24.01	10.34	7.09	5.69	16.98	6.18	0.075
90	26.41	38.37	9.51	3.24	3.13	16.79	2.53	0.08
120	14.01	33.00	16.99	11.0	6.42	15.46	3.12	0.09

Table 5. Evolution of nutrients, beneficial elements, heavy metals and microbiological characteristics during composting of winery waste.

Time	Macronutrients			
	Calcium	Phosphorus	Potassium	Magnesium
days	$g\ kg^{-1}$			
1	1.74 ± 0.20	4.94 ± 0.47	74.91 ± 3.84	17.14 ± 3.55
30	2.80 ± 0.80	2.12 ± 0.40	52.00 ± 7.55	1.48 ± 0.12
60	3.72 ± 0.38	2.10 ± 0.09	42.73 ± 4.00	1.66 ± 0.28
90	4.25 ± 0.15	1.23 ± 0.02	35.70 ± 5.30	1.86 ± 0.26
120	5.17 ± 0.65	1.92 ± 0.35	21.31 ± 3.40	0.75 ± 0.14
Time	Micronutrients			
	Copper	Iron	Manganese	Zinc
days	$g\ kg^{-1}$			
1	3.92 ± 0.11	66.40 ± 5.21	0.60 ± 0.20	4.16 ± 0.81
120	0.50 ± 0.50	9.04 ± 0.04	0.25 ± 0.27	0.55 ± 0.08
Time	Critical elements			
	Aluminum		Sodium	
days	$g\ kg^{-1}$			
1	0.02 ± 0.004		1.90 ± 0.40	
30	0.016 ± 0.003		2.53 ± 0.51	
60	0.015 ± 0.004		1.75 ± 0.27	
90	0.013 ± 0.003		1.38 ± 0.14	
120	0.013 ± 0.004		1.50 ± 0.50	
Time	Heavy metals			
	Chrome	Lead	Nickel	
days	$g\ kg^{-1}$			
1	0.09 ± 0.01	0.21 ± 0.09	1.0 ± 0.10	
120	0.09 ± 0.06	0.09 ± 0.01	0.009 ± 0.001	
Time	Microbiological			
	Total coliforms		*E. coli*	
days	NMP/g de ST			
1	–		–	
120	900		150	

there is some uniformity between the particles so that the application is not uneven.[60]

The final biocompost differed in its physical properties. Table 4 presents the variation of the density and distribution of particles throughout the composting process. During the process, there was a reduction in the proportion of particles with larger sizes and an increment in the particles of smaller particle size. Such a transformation is confirmed when analyzing the evolution of density during the process, which began in 0.051 g cm^{-3}, and at the end of the process the value was 0.09 g cm^{-3}. This increase in density occurs due to the occupation of porous spaces by particles with smaller sizes.

According to the NI n° 25/2009 (MAPA), the biocompost can be classified according to their particle size as thick bran, bran, powder, and granulated. As for the particle size specification established in this study, the biocompost was classified as bran, presenting a maximum of 25% of the material passed through a 0.5 mm sieve, and a minimum of 75% of material retained above 0.5 mm.

If considering that in the semiarid, with irrigated fruit farming, the decomposition of organic material in the soil occurs in an accelerated manner, it is necessary that the produced biocompost presents particles of different sizes, but specifically with larger sizes so that they are not rapidly degraded, acting in the maintenance of OM in the soil. With that, this class of biofertilizer can collaborate in the promotion of a better aggregation in the soil and stabilization o aggregates.

Lastly, the volume of the pile was calculated at the end of the composting process, presenting a reduction of 51.21%. This value is much higher than that obtained by Reis,[61] who, when composting different organic residues, obtained at 112 composting days for grape pomace a reduction of 17% in volume and, when composting olive pomace, carob pulp and eucalyptus bark obtained 16, 41 and 52% (the bigger reduction and which came closest to the present study).

The value obtained in this study is close to the range obtained by Sbizarro et al.[62] in their T1 (30:1 – sheep manure + sugarcane straw), T2 (19:1 – sheep manure + sugarcane straw), T3 (30:1 – bovine manure + sugarcane straw), T4 (19:1 – bovine manure + sugarcane straw), and T5 treatments (30:1 – sheep manure + bovine

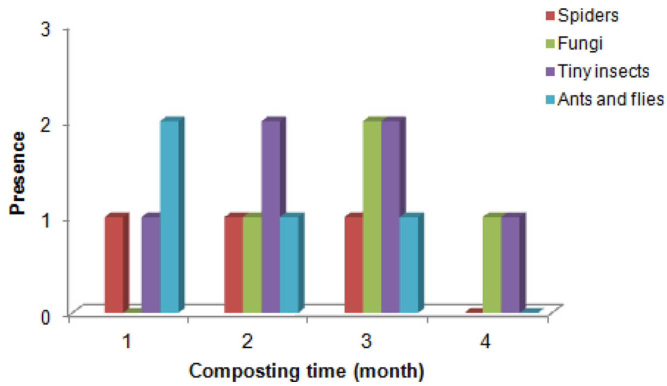

Figure 6. Other parameters evaluated during the composting process.

manure + sugarcane straw) with volume reductions of 60.95%, 59.45%, 54.20%, 47.60%, and 48.16%. According to Petric et al.,[63] this volume reduction is the result of the degradation of organic matter during the composting process.

The main characteristics related to nutritional and microbiological parameters are exhibited in Table 5. Except for calcium (Ca), the remaining macronutrients presented a content reduction during the composting process: phosphorus (P), with a reduction of 61.13%; potassium (K), with a reduction of 71.55%, and magnesium (Mg), with a reduction of 95.62%.

As for the chemical composition (Table 5), only the Ca element presented an increase in its content throughout the composting process. This result needs to be carefully analyzed since an increase in the content of any nutrient does not mean an addition during the process, and it must be considered that during the reductions of total solids there may occur increases in the concentration of some nutrients in the final biocompost.[64]

According to the NI n° 25/2009 (MAPA), for soil application the biocompost must present a minimum content of 1% (10 g kg^{-1}) of Ca, therefore, the biocompost produced does not meet this condition. Rodrigues et al.[34] studies the composting of floated sludge in a dairy effluent treatment plant and observed that, after the maturation of the biocompost, the concentrations of Ca were 0.7%, close value to that obtained in this work, which was 5.17 g kg^{-1}, corresponding to 0.51% of Ca at the end of the process.

Regarding P, the removal occurs by the mineralization of this component linked to organic matter, that is, through the microbial digestion that transformation of organic phosphorus to an inorganic form is made.[65] The final value for P was 1.92 g kg^{-1}, with no reference value determined by MAPA.

The element K, the most abundant nutrient in biocomposts derived from winery wastes,[66] presented initial and final contents of 74.91 and 21.31 g kg^{-1} and, similar to P, it also does not present a reference value determined by MAPA. The higher value can be explained by the fact that it occurs in the form of the K^+ ion within the plants. This causes the nutrient to be released, in a moist environment, even before the mineralization of the organic residue. With that, the periodic irrigation in the composting pile probably displaced the ion through preferential water flows, resulting in more reduced values.

The final value of Mg (approximately 4.4% of the initial value) (Table 5) was higher than the values found by Rodrigues et al.,[34] who obtained concentrations of 0.24% at the end of the process, and Sanes et al.[67] found Mg concentrations varying from 0.10 to 0.20% at the end of the composting process. However, the initial material used in the study by Sanes et al.[67] presented low Mg concentrations, differently from the present work. According to the NI n° 25/2009 (MAPA), for soil application, the biocompost must have a minimal content of 1% of Mg and, therefore, the result does not meet the normative.

The micronutrients presented variable values, copper (Cu) presented a final value in the established minimum limit (0.5 g kg^{-1}) for organic fertilizers according to NI n° 25,[16] iron (Fe) presented a final value of 9.04 g kg^{-1}, higher than the minimum established (2 g kg^{-1}), manganese (Mn) (0.25 g kg^{-1}) and zinc (Zn) (0.55 g kg^{-1}) presented values below the minimum limit (0.5 and 1 g kg^{-1}).

Aluminum (Al) and sodium (Na) are some of the elements considered critical, as studies demonstrate that the quickest visible symptom caused by Al toxicity is inhibition of root growth, resulting in reduced root size and absorption of water and nutrients.[68] High sodium contents can affect the germination and initial development of plants since they increase the electrical conductivity and, in the most serious cases, they can cause severe economic problems due to widespread plant death since the soil becomes inapt for agriculture.[69–71]

Considering the region of semiarid climate, the ideal is that the concentration of these elements is close to nullity since due to the low rainfall and soils with poor drainage there is a strong salinization trend. In this study, Al and Na presented removals of 35% and 21.01% at the end of the process. Rodrigues et al.[34] obtained Na concentrations of 0.02%, a value below that found in this study, which was 0.15%, although still being a low value according to the authors. The NI n° 25/2009 (MAPA) does not establish a maximum level for sodium in the organic fertilizers. Al was the element that presented the highest removal, even with its initial value already presenting a low concentration. According to Costa,[72] organic compounds present the property to bind to metallic ions of iron, zinc, and copper, in this case promoting the bio-oxidation and removal of the toxic effect of Al.

Regarding heavy metals (Table 5), the final values for chromium (Cr), lead (Pb) and nickel (Ni) (0.09; 0.09 and 0.009 g kg^{-1}) were in accordance with the maximum value (0.2; 0.15 and 0.07 g kg^{-1}) allowed for use as organic fertilizer by NI n° 27/2006,[15] and did not present a risk of contamination by these elements.

As for the sanitary aspect, the values for total coliforms and *Escherichia coli* (*E. coli*) found in the final biocompost were 900 and 150 (NMP/g of MS). The NI n° 27/2006 does not establish a reference value for total coliforms, but for thermotolerant coliforms (*E. coli*), the value admitted for use as a substrate for plants or as a soil conditioner is 1000 NMP/g of MS.[15] This result confirms the use of the temperature parameter as directly related to the removal of coliforms, given the limits defined for the microbiological parameters, since the removal of these organisms occurs during the thermophilic phase.

During the composting process, eight parameters were monitored, such as the presence of smells, insects, spiders, and fungi (Fig. 6). This monitoring aimed to find a relationship between the parameters and the temperature of the composting pile.

Such as described by Rodrigues et al.,[34] the smell had a low intensity and only in the first month of the process. Bartelt et al.,[73] when composting fruit waste, verified that the smells released by the composting pile attracted insects. The presence of microorganisms in the process is normal since the biocompost presents considerable amounts of organic matter and several microbial factors perform an important role in the production of the biocompost.

The presence of fungi was noted from the second month of the composting process, in the cooling phase, when most of the microorganisms die. According to Pelczar et al.,[74] fungi are heterotrophic organisms that feed on inanimate organic matter, decomposing complex residues of animals and plants and transforming them into simpler chemical forms.

The number of organisms in the composting pile is variable among regions. Rodrigues et al.[34] noted the presence of flies (Diptera), insect larvae (Insecta), ants (Hymenoptera), and earthworms (Haplotaxida) in all stages, even with low intensity. On the other hand, the variable that presented the highest intensity throughout the process was that of small insets.

During the composting process, the alteration in the coloration of the composting pile was also observed, initially red, ending in a dark brown hue, a coloring considered adequate by Lima et al.[75]

Conclusion

The biocompost produced by mixing grape marc, sheep manure and mango leaves can be used as organic fertilizer or as soil conditioner, since it proves to be safe in terms of pathogens and phytotoxic elements, according to NI n° 27 (MAPA).[15] And by observing the C/N, organic carbon and VS ratio values at 30th days of process, the biocompost was already stabilized according to the Normative Instruction n° 25 (MAPA)[16] and CONAMA Resolution n° 375.[18]

This result represents a reduction of 90 days in the composting process, which can motivate the greater use of the winery waste, giving an environmentally appropriate destination and generating savings with their reinsertion in the supply chain.

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