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Aroma Composition of Wines Produced from Grapes Treated with Organic Amendments

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Abstract: The application to agriculture of wheat-straw spent mushroom substrate amendments (compost/vermicompost) used to grow *Pleurotus ostreatus* has been analyzed. The study was conducted in a vineyard where the effect on (1) the physicochemical properties of the soil and the leaf and (2) the analytical characteristics and the aromatic composition of the wine were analyzed. The application of the amendments resulted in an increase in organic matter and macronutrients (NO_3^-, P_2O_5 and K_2O) in the soil. With regard to the leaves, the NO_3^- and K_2O contents of those vines fertilized with vermicompost were higher, and the metallic content was the same regardless of the treatment applied. The analysis of the colorimetric parameters showed that there was a higher content of compounds with red and violet colorations in the case of wine obtained after treatment with vermicompost. In addition, for this type of wine, a higher concentration of volatile compounds was obtained. Thus, after grouping the aroma compounds into aroma series, the greatest differences among vermicompost wines and the rest were obtained in the fruit, floral, herbaceous, and green fruit series. The principal component analysis showed that the vermicompost treatment clearly differentiated the wine from the rest of the wines, in addition to its effects on the aromatic series, the values in the total polyphenol index, and the compounds responsible for brown tones.

Keywords: compost; vermicompost; wine; aroma compounds; volatilome; aroma series

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1. Introduction

At present, winegrowing soils cover a surface of approximately 7.3×10^6 hectares across the world, mainly concentrated amongst the Mediterranean regions (Spain, France, Italy, Turkey) and China, the USA, Argentina, and Chile [1]. In most of these wine-growing areas, agriculture is intensive, which implies a significant environmental impact and, consequently, an important loss of fertility in the soils [2].

In recent years, sustainable agricultural practices in accordance with circular bioeconomy, based on the organic and biodynamic management of the vineyards, have been fostered in order to (1) minimize the adverse effects caused by intensive agriculture and heavy machinery and (2) reduce dependence upon chemical fertilizers and upon plague and disease control techniques relying on components of synthesis [3–6]. In this perspective, the application of compost and vermicompost in agriculture can be a sustainable alternative for vineyards, considering their impact on viral and bacterial communities as well as the organic matter and nutrients they provide, such as nitrogen, phosphorous, and potassium [7–10].

In the literature, we find numerous studies analyzing the impact of the application of organic amendments. However, these present dissimilar results, depending on the agricultural practices and the origin, composition, and dose of the compost/vermicompost applied to the vineyard. Evans et al., in 2013 [11], analyzed the application of compost tea made from cattle manure, hen droppings, vegetable waste, and salmon fish farming waste, and confirmed that their use can be regarded as a strategy for the comprehensive management of grapevine illnesses. Döring et al., in 2015 [12], compared the conventional comprehensive management (compost + chemical fertilizers + herbicides + fungicides) with organic management (compost) and biodynamic (natural treatments + copper + sulfur) management and observed that these sustainable amendments, despite not having an adverse impact on the quality of the wine, reduced the growth and the yield of the grapevine. The study of Gaiotti et al., in 2017 [13], demonstrated that the addition of cattle manure compost and compost from waste from pruning grapevines has a positive impact on the growth of the roots of the grapevine and on its vegetative growth and improves the yield and the composition of the grape. Martínez et al., in 2018 [14], stated that the application of compost and vermicompost from hen droppings and sawdust enhanced the assimilation of nutrients and the yield of the grapevine.

In view of this data, it is clear the application of organic amendments (compost/ vermicompost) needs to be further studied, and particularly the impact of these amendments on the analytical and sensory quality of the wines. This is a field that is still practically unexplored but for a few studies, namely Korboulewky et al., who, in 2004 [15], analyzed the impact of the application of sewage sludge on the concentration of 13 volatile organic compounds of the wine, and González et al., who, in 2018 [16], studied the effects of the application of grape pomace compost on *Vitis vinifera* cv. Chelva grapevines, analyzing the concentration of 81 volatile compounds of the wine.

The present study analyzed the application of two amendments to agriculture, compost and vermicompost, obtained from the treatment of lignocellulosic waste (wheat-straw spent mushroom substrate) from the cultivation of *Pleurotus ostreatus*. In fact, although there are studies on co-composting [17] and co-vermicomposting [18] with this type of waste, there is no literature delving into their individual treatment to obtain organic amendments with a potential use in agriculture. In particular, these amendments were applied to a vineyard in order to analyze the nutritional state of the soil and the levels of heavy metals in it, as well as the effects of the amendments on the aromatic components of the wine.

2. Materials and Methods

2.1. Vineyard, Climate, Soil and Leaf

The vineyard is located in Puente Genil, Córdoba Spain (37°24′51′′ N, −4°42′0′′ W) in southwestern Spain, in a calcarean soil. In this area, the annual average rainfall is 580 mm, and the average temperature is 16.2 °C. The grape variety used was *Vitis vinifera* cv. Syrah.

Representative samples of soil and leaves were taken for each replica of the treatment. The samples of the soil were obtained from different points at a depth from 0 to 0.30 m [13,19], and the samples of leaves following the method proposed by Failla et al., 1993 [20].

2.2. Composting and Vermicomposting

Two treatments, composting and vermicomposting, were applied to the wheat straw waste used as a substrate in the cultivation of *Pleuratus ostreatus* at Huertos de Hytasal S.L. (Seville, Spain). Both processes were performed over a period of 120 days in a covered plant to avoid the impact of climatological conditions.

The composting was carried out following the open trapezoidal pile system (2.0 m height, 2.5 m width and 6 m length), using a pile that was irrigated and mechanically turned at five specific moments during the process. With these working conditions, the four phases that characterize this type of process were completed (mesophilic, thermophilic, cooling, and maturation phases), and the temperature (never above 65 °C) and the moisture content (never less than 40%) were controlled.

Regarding vermicomposting, this was performed through the addition of 3500 worms of the *Eisenia fetida* species at the bed (0.40 m height, 1.5 m width, 4.0 m length), which was irrigated weekly, thus preserving the temperature at <25 °C and hence avoiding jeopardizing the development of the worms.

Once the bioprocesses were completed, the products were mechanically sieved with a rotary sieve of a 1 cm mesh. From the total mass following each sifting, representative samples of compost and vermicompost were built, from subsamples taken from different points and at different depths (center > 0.50 m; intermediate, 0.25–0.40 m; and superficial, 0–0.15 m).

2.3. Treatments in Experimental Plots

The fertilizer was applied at the end of the month of February, before the sprouting period. Nine plots were required for the different treatments: three unfertilized (Control), three with compost (T_1), and three with vermicompost (T_2). The application of the different amendments was estimated to provide a total of 50 kg of nitrogen per hectare of soil, in this manner respecting the established limit for areas vulnerable to nitrate pollution of agricultural origin (170 kgN/ha), according to Directive CEE/676/91 [21]. The organic amendments were added to the strip of land at a distance of between 0.20–0.50 m with respect to the stem and at a depth of 0–0.30 m, complying with the regulation for sustainable nutrition in agricultural soils (Spanish RD 1051/2022) [22]. The end of the study was considered as the time of harvest (last days of August).

2.4. Physicochemical Characterization of Organic Amendments, Soil, and Leaf

The solid samples were dried at 60 °C to a constant weight and then crushed with a blade mill (ORTO-ALRESA) and stored at −20 °C until subsequent analysis. The pH and electrical conductivity (EC) were measured in the aqueous extract (1:25 ratio). The solution and solid phase were then separated via centrifugation at 3000 rpm for 20 min and filtered through a 0.45 μm membrane. The pH and EC values were measured with a pH meter and conductivity meter, respectively. The organic matter content was measured by loss on ignition with samples oven dried for 4 h at 550 °C. The total content of carbon and nitrogen were determined with a LECO CHNS-932 Elemental Analyzer (Leco Corporation, St. Joseph, MI, USA). The quantity of phosphorus was quantified with UV–visible spectrophotometry as per the Olsen method. The content of nitrogen (NO_3^-) and the granulometric analysis were performed following the methods specified by official regulations (Spanish RD 999/2017, RD 1110/1991) [23,24].

The evolution of the maturity of the material was evaluated on dry samples taken at the beginning and at the end of each process, finely ground in Agate mortar, and placed on the sample compartment of a Spectrum TWO-FTIR-ATR (Perkin Elmer, Waltham, MA, USA) at a working range of 4000–400 cm^{-1}. The methodology of Zucconi et al., 1981 [25] was used to evaluate the phytotoxicity. The germination index (GI) was determined by combining the measurements of seed germination and root elongation of cress seeds (*Lepidium sativum* L.) (Figure 1).

Lastly, the analysis of K, Cr, Cu, Ni, Pb, and Zn was performed with the absorption technique with a flame atomic absorption spectrometer (A Analyst 300, Pelkin Elmer, Waltham, MA, USA) following the acid digestion of the dry samples, as per the method described by Rosal, 2007 [26].

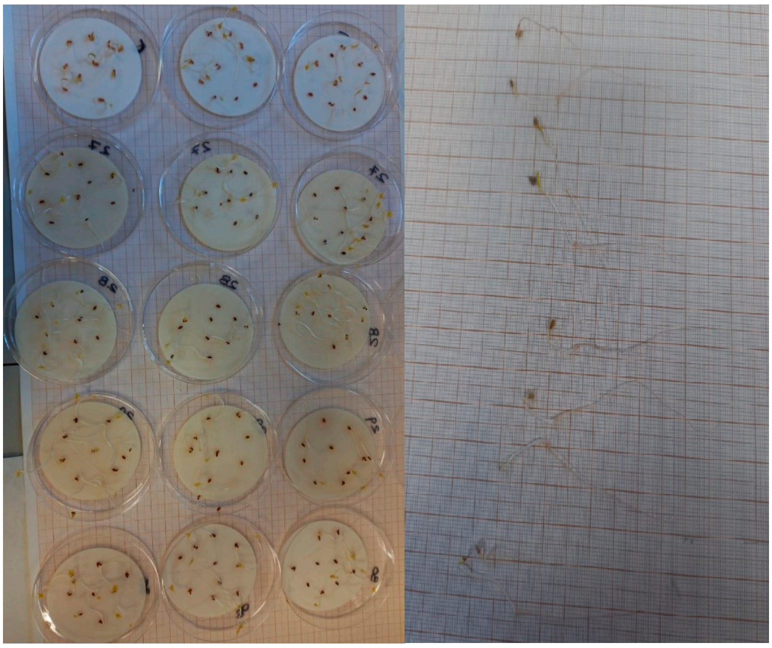

Figure 1. Images of the germinations and the roots of cress seeds.

2.5. Chemical Standards

The identification and quantification of aroma compounds were carried out with standard solutions of pure compounds of analytical grade, purchased from Sigma-Aldrich (St. Louis, MO, USA), Merck (Rahway, NJ, USA), and Fluka (Buchs, Switzerland). Pure water was obtained from a Milli-Q purification system (Millipore, Burlington, MA, USA).

2.6. Winemaking Conditions

Nine batches of five kilograms of red grapes of the Syrah variety were hand crushed and the resulting must and grape skin were placed in five-liter Erlenmeyer flask. The first three batches contained grapes from vines treated with compost. The second three batches contained grapes from vines treated with vermicompost. The last three batches contained grapes from untreated vines.

Each batch was inoculated with the commercial yeast strain Uvaferm VN from Lallemand (Grenaa, Denmark) according to the instructions of the supplier. Fermentation was performed at 18 °C and was considered complete when the density was below 995 g per liter.

2.7. Determination of Oenological Parameters

Oenological parameters such as pH, titratable acidity, volatile acidity, ethanol, and residual sugar content were determined according to the official EEC methods [27]. Three biological replicates were used to undertake the analysis.

We measured the total phenol index (TPI) as a spectrophotometric measurement at 280 nm. The absorbance at 420, 520, and 620 nm were also determined in a Perkin-Elmer Lambda 25 spectrophotometer (Waltham, MA, USA) after filtering the samples through a HA-0.45 µm paper (Millipore, Milford, MA, USA).

2.8. Volatile Compounds Determination

Volatile compounds in must and wines can be classified according to their contents of major volatile compounds (≥10 mg/L) and minor volatile compounds (<10 mg/L). Three biological replicates were used to undertake the analysis.

2.8.1. Major Volatile Compounds

These were identified and quantified in a gas HP 6890 Series II chromatograph equipped with the capillary column CP-WAX 57 CB (50 m in length, 0.25 mm in internal diameter and 0.4 μm in coating thickness) and an FID, according to the conditions described by Peinado et al., 2004 [28]. To identify and quantify the analyzed compounds, standards were injected under the same conditions as the samples. Additional information about LRI used to identify volatile compounds is detailed in Table S1.

2.8.2. Minor Volatile Compounds

These compounds were identified and quantified in a two-step process, both described previously in detail by López de Lerma et al., 2018 [29]. The first one consists of an extraction procedure using stir bars (film thickness 0.5 mm, 10 mm length, Gerstel GmbH, Mülheim an der Ruhr, Germany). These are placed in a vial containing 10 mL of 1:10 diluted sample and 0.1 mL of ethyl nonanoate (0.4464 mg/L) as an internal standard. After 100 min of stirring at 1500 rpm, the stir bars were removed and put into a desorption tube for chromatographic analysis.

The second phase consists of the determination of the volatile compounds in a GC-MS equipped with a Gerstel TDS 2 thermal desorption system. Desorption tubes, containing the stir bars, are heated at 280 °C with the aim of releasing the volatile compounds in a CIS 4 PTV cooling system programmed at 25 °C, which contains a Tenax adsorption tube. Lastly, the CIS is heated to release the volatiles in the GC-MS equipped with an Agilent-19091S capillary column (30 m × 0.25 mm i.d., 0.25 μm film thickness). The mass detector works in scan mode at 1850 V and checks the mass from 39 to 300 amu.

To identify the volatile compounds, the retention times of standards injected under the same chromatographic conditions as the samples were used. Also, the NIST and Wiley spectral libraries were used. Quantification was made using calibration curves of the standard. Additional information about the LRI used to identify volatile compounds is detailed in Table S1.

2.9. Calculation of Aroma Series

The odor activity values (OAV) of the volatile compounds were determined as per the ratio between concentration and the odor perception threshold. Aromatic series were obtained as the sum of the OAVs of the volatile compounds with similar aroma descriptors. In this way, 10 aroma series, namely chemistry, fruity, green fruit, creamy, caramel, floral, green, citrus, fatty, and waxy, were obtained. The same compound can be included in one or several aroma series based on its aromatic descriptors.

2.10. Statistical Analysis

Except for FT-IR, the analyses were carried out in triplicate. The statistical study of the significant differences between the average of the physicochemical parameters of the soil and the leaf was conducted with the Student t-test for independent samples with the software package IBM SPSS Statistics 25.

ANOVA analysis was carried out to test the differences among the physicochemical parameters analyzed in the organic amendment, the soil, and the leaf. In addition, homogeneous group analyses were carried out to analyze the differences in wine compositions resulting from the different treatments. A footprint of the wines was obtained with multivariate analysis using the aroma series. Lastly, the aroma series were used to perform a cluster and principal component analysis. To this end, the statistical software Statgraphics Centurion XVI of StatPoint Technologies Inc. (Warrenton, VA, USA) was used.

3. Results and Discussion

3.1. Temperature in Composting and Vermicomposting Processes

The evolution of temperature during composting is one of the main parameters characterizing the quality of the process, since it is correlated with the degradation of the organic matter and the generation of microbiota. Figure 2 shows the evolution of internal and ambient temperatures throughout the treatment of the waste.

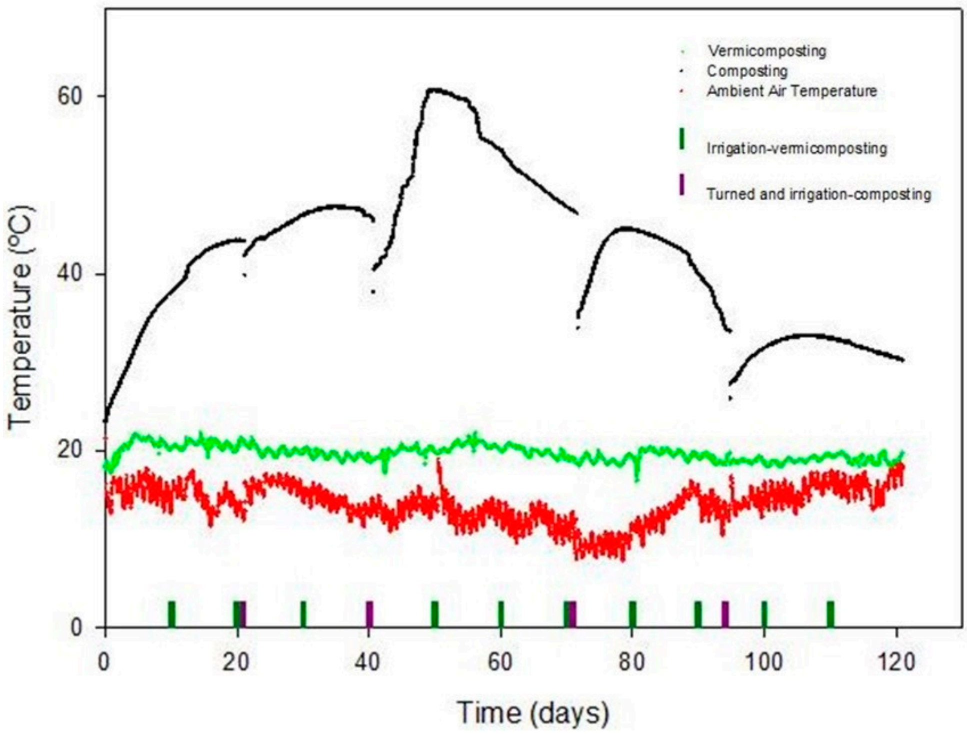

Figure 2. Changes in the temperature and frequency of irrigation and turning in both processes.

The increase in the internal temperature is observed from the first day of the composting process, reaching thermophilic values from day 14. During the most intense bioxidative phase, temperatures were above 55 °C for more than 14 days, which favors sanitation according to Regulation (UE) 1009/2019 [30]. After the fourth week of composting, temperature decreased, remaining within levels above 45 °C until day 70. These results are similar to those obtained by other authors who used spent fungi substrate for co-composting processes [31].

With regard to vermicomposting, internal temperatures at the base oscillated between 17 °C and 24 °C during the process (Figure 2), thus not having an adverse impact on the evolution of the *Eisenia fetida* species [32,33].

3.2. Physicochemical Characterization of Compost and Vermicompost

Table 1 shows the physicochemical properties of the organic amendments applied to the experimental plots. The increase in pH and the decrease in the C/N ratio during the treatment of the waste can be used as indicators of the evolution of its maturity [34]. In this regard, when comparing both parameters in the amendments studied, at the final stage, results show evidence of the greater maturity of vermicompost. In any case, both amendments present optimal levels for their use in agriculture as per the parameters found in the literature. Hogg et al., 2002 [35], explain that pH values between 6.0–8.5 in the amendments are appropriate for agricultural use, and Bernal et al., 2009 [36], suggest that C/N ratio values < 20 indicate maturity.

Table 1. Physicochemical characteristics of the compost and vermicompost used.

Property	Compost	Vermicompost
pH	6.8 ± 0.1	7.84 ± 0.02
EC (dS·m^{-1})	4.0 ± 0.5	1.90 ± 0.02
OM (%)	64 ± 2	60.6 ± 0.4
N (%)	2.1 ± 0.4	2.2 ± 0.3
P (%)	0.30 ± 0.01	0.20 ± 0.01
K (%)	2.6 ± 0.4	3.8 ± 0.2
C/N	13.4	12.9
GI (%)	74.8	84.3
Cr (mg/kg)	26.7 ± 0.6	24.7 ± 0.5
Cu (mg/kg)	6.1 ± 0.1	10.5 ± 0.3
Ni (mg/kg)	11.9 ± 0.5	12.3 ± 0.5
Pb (mg/kg)	16.3 ± 0.6	15.5 ± 0.4
Zn (mg/kg)	34 ± 5	27 ± 2
Granulometry (mm)	15.0 *	15.0 *

EC: electrical conductivity; OM: organic matter; GI: germination index. Data expressed in dry weight basis. * >90% particles passed through a 15 mm sieve.

With regard to electrical conductivity, the final amendment must present values < 4 dS/m for safe use in agriculture [37,38]. The mean value of compost was remarkably higher than that of vermicompost. This may be due to the different conditions of humidity of each process, which, in the case of vermicomposting, may have caused a leaching effect (saline lixiviation), given the higher frequency of irrigation. Both electrical conductivity values were similar to, and even lower than in some instances, the ones revealed by other studies for amendments (compost/vermicompost) from urban wastes [39,40], livestock wastes (hen droppings/manure) [34,41], and even agricultural wastes such as olive tree pruning byproduct [42]. The macronutrient (NPK) concentrations were higher or similar to those found by other authors in studies of composting and vermicomposting using urban wastes [43], vegetable wastes [44], and agro-industrial wastes [31]. Phytotoxicity is a very useful decisive criterion for assessing whether organic materials are appropriate for agricultural use. Both amendments presented percentages above the minimum value (GI < 50%) established by Zucconi et al., 1981 [25], to indicate the absence of phytotoxicity.

In both the compost and the vermicompost, the values observed in organic matter (>35%), C/N ratio (<20), and granulometry (<25 mm) were within the limits established by the legislation for agricultural applications and, as per the heavy metals, the total concentrations were under the limits established for the maximum quality category, Class A, pursuant Spanish RD 506/2013 [45].

Finally, the graph in Figure 3 shows the FT-IR spectra of the initial lignocellulosic waste and of the products obtained in each of the processes. As the composting and vermicomposting processes evolve, the matter is transformed due to biodegradation and to polymerization reactions, hence increasing the concentration of organic humic substances. FT-IR can be used to assess the stabilization of organic matter and the quality of the final product [46].

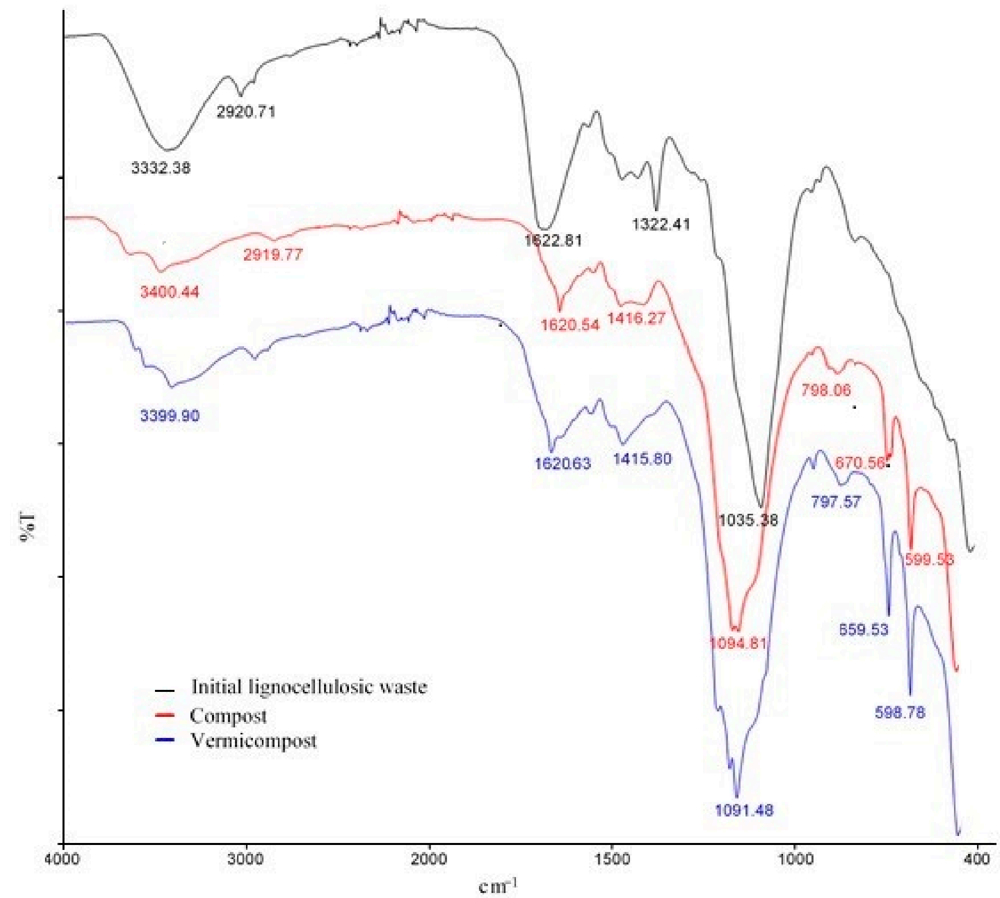

Figure 3. Fourier transform infrared (FT-IR) spectra to initial matter, compost and vermicompost.

FTIR was applied to reveal how the functional groups changed, comparing the bands in the initial substrate to the ones identified in the organic amendments. In this study, a broad band of 3400 cm^{-1} was observed, corresponding to –NH inter and intramolecular bonds and –OH elongations of carboxylic, phenolic, or alcoholic nature and whose intensity significantly decreased in the compost and the vermicompost. At approximately 2930 cm^{-1}, C–H stretching vibrations appear, characteristic of saturated carbons pertaining to aliphatic chains, which progressively decrease when the maturation of the material progresses. At 1620, 1509, and 1416 cm^{-1}, C=C stretching vibrations contribute characteristics of unsaturated carbons of aromatic structures and C=O vibrations of conjugated quinones and ketones. The broad band appearing at 1094 cm^{-1} corresponds to C–O vibrations of esters, significantly present as the process evolves and, in this case, more intense in vermicompost. With regard to the band appearing at 1035 cm^{-1}, it corresponds to C–O vibrations associated with structures of polysaccharide remnants, whose intensity significantly decreases in the spectra of the product, probably due to the degradation of the carbohydrates as the process evolves. The bands in the areas between 450 and 800 cm^{-1} correspond to C–H vibrations of substituted aromatic rings, which appear at the final stages of the processes and are related to the humic transformation of the material. Evidently, the FT-IR spectra shows an increase in the intensity of the bands related to the aromatic groups in the products compared to the control, especially in the case of vermicompost. Interpretations of the IR spectra are based on work found in the literature [47–52] and are consistent with the analysis that compared pH and C/N ratio in compost (6.8, 12.4) and vermicompost (7.8, 10.9), whose values indicated greater maturity in the material that underwent vermicomposting.

3.3. Physicochemical Characterization of Soil and Leaf

Table 2 shows the results obtained in the analysis of the soils treated with organic amendments and of the soil without treatment (control) at the end of the experiment. In the cases studied, pH mean values were determined at 8.10–8.35, which reduce the availability of nutrients for the plant to absorb [53,54], and mean conductivity values at 0.17–0.80 $dS \cdot m^{-1}$, levels remarkably under the threshold of salinity (1.50 $dS \cdot m^{-1}$) referred to in literature for the cultivation of grapevines [55]. It is observed that the treatments with these kinds of organic amendments remarkably increased the presence of organic matter and macronutrients (NO_3^-, P_2O_5, K_2O) in the soils at the end of the experiment. As per heavy metals, the treatments with the amendments increased the total concentration (Cr, Cu, Ni, Pb) in the soil, although the levels reached (7.7–36.1 $mg \cdot kg^{-1}$) were significantly under the maximum content allowed in agricultural soils with pH $\geq$ 7 (70.0 $mg \cdot kg^{-1}$–200 $mg \cdot kg^{-1}$), as per Spanish legislation (RD 1051/2022) [22].

Table 2. Soil properties at the end of the experiment.

Property	Treatment			F Test Sig.
	Control	Compost	Vermicompost	
pH	8.32 a ± 0.04	8.10 b ± 0.01	8.10 b ± 0.05	**
EC ($dS \cdot m^{-1}$)	0.170 b ± 0.002	0.80 a ± 0.02	0.74 a ± 0.01	***
OM (%)	1.86 c ± 0.04	2.0 b ± 0.2	2.7 a ± 0.1	*
NO_3^- (%)	0.027 b ± 0.001	0.161 a ± 0.005	0.17 a ± 0.02	*
P_2O_5 (%)	0.09 b ± 0.01	0.30 a ± 0.02	0.30 a ± 0.01	*
K_2O (%)	0.005 c ± 0.001	0.119 b ± 0.007	0.181 a ± 0.007	*
Cr (mg/kg)	2.2 c ± 0.2	11.5 a ± 0.3	7.7 b ± 0.1	**
Cu (mg/kg)	2.3 c ± 0.1	17.0 b ± 0.8	22 a ± 1	*
Ni (mg/kg)	6.1 c ± 0.4	26 b ± 1	36 a ± 2	**
Pb (mg/kg)	11.6 c ± 0.3	33.1 a ± 0.9	20 b ± 1	*
Zn (mg/kg)	<LOQ(20)	<LOQ(20)	<LOQ(20)	-

Data expressed in dry weight basis. EC: electrical conductivity; OM: organic matter; LOQ: limit of quantification. Different letters indicate significant differences (* $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$), ns = non-significant. (–) No test was performed.

With regard to the analysis of the leaves of the grapevine, Table 3 shows the results obtained at the end of the experiment. The physicochemical analysis performed on the leaves showed that the contents of NO_3^- and K_2O were significantly higher with vermicompost compared to the control and compost treatments. These results provide evidence of the relation of the availability of the nutrients in the soil with the application of vermicompost and the variation of the nutritional conditions of the plant, and are consistent with the conclusions of other studies [56].

Table 3. Leaf properties at the end of the experiment.

Property	Treatment			F Test Sig.
	Control	Compost	Vermicompost	
OM (%)	77 ± 2	75 ± 6	79 ± 4	ns
NO_3^- (%)	1.49 b ± 0.17	1.13 c ± 0.07	2.2 a ± 0.2	*
P_2O_5 (%)	0.38 ± 0.03	0.45 ± 0.01	0.45 ± 0.01	ns
K_2O (%)	0.374 c ± 0.009	0.401 b ± 0.005	0.436 a ± 0.014	*
Cr (mg/kg)	1.2 ± 0.1	1.49 ± 0.02	1.74 ± 0.03	ns
Cu (mg/kg)	13.7 a ± 0.3	10.8 b ± 1	12.6 b ± 0.7	*
Ni (mg/kg)	3.0 ± 0.1	2.3 ± 0.1	2.91 ± 0.04	ns
Pb (mg/kg)	3.0 ± 0.1	2.7 ± 0.1	2.4 ± 0.2	ns
Zn (mg/kg)	<LOQ (20)	<LOQ (20)	26 ± 2	-

Data expressed in dry weight basis. OM: organic matter; LOQ: limit of quantification. Different letters indicate significant differences (* $p < 0.05$), ns = non-significant. (–) No test was performed.

Regarding the content of organic matter and assimilable phosphorous, no significant differences were found between the treatments. And, as per the content of heavy metals, results showed that the application of these types of amendments to the soil, despite the slight increase in the total concentration of metals in the soil treated compared to the control, caused no increase in the content of metals in the leaves. These results can be explained based on two phenomena that reduce the mobility of the metals and, therefore, their availability for the plant. On the one hand, the formation of insoluble metal chelates with the humic fraction present in the organic amendment [57,58] and, on the other hand, the capacity of the worms to reduce the bioavailability of the heavy metals [59,60]. These results are similar to the ones provided by the studies of other authors on organic amendments with different origins that were also applied to vineyards [61,62].

3.4. Enological Parameters

The climatic conditions of the year were characterized by long episodes of extreme heat in the winemaking region in which the research was carried out. High temperatures, above 40 °C, caused a sharp decline in photosynthesis due to the disruption of the functional integrity of the photosynthetic machinery in the chloroplasts [63]. As a consequence of the decrease in photosynthesis, fruit ripening was suppressed. In addition, high temperatures shift carbon partitioning to favor vegetative growth at the expense of fruit growth and ripening [64,65]. Due to this, low levels of ethanol, around 13% (*v*/*v*), were obtained in all wines. On the other hand, high temperatures involve a high malic acid degradation, which affects pH values (Table 4). As can be seen, no significant differences are observed in common enological variables except for those related to color parameters. Vermicompost wines were those with the highest total polyphenol index and showed more red and blue pigments than the rest of the wines.

Table 4. Enological variables and color parameters in the wine obtained after different vine treatments.

Property	Treatment		
	Control	**Compost**	**Vermicompost**
pH	4.03 a ± 0.04	3.95 a ± 0.06	4.04 a ± 0.05
Titratable acidity (g tartaric acid/L)	5.3 a ± 0.1	5.5 a ± 0.1	5.5 a ± 0.1
Volatile acidity (g acetic acid/L)	0.51 a ± 0.02	0.50 a ± 0.03	0.52 a ± 0.02
Ethanol % (*v*/*v*)	13.2 a ± 0.2	13.3 a ± 0.2	13.1 a ± 0.2
Reducing sugars (g/L)	2.2 a ± 0.3	2.4 a ± 0.4	2.6 a ± 0.3
TPI	49 b ± 2	49 b ± 1	60 a ± 1
Absorbance 420 nm	6.2 a ± 0.1	5.2 b ± 0.1	3.4 c ± 0.2
Absorbance 520 nm	19.2 c ± 0.1	23.8 b ± 0.2	25.8 a ± 0.1
Absorbance 620 nm	1.18 c ± 0.08	1.95 b ± 0.06	3.48 a ± 0.07

TPI: total polyphenol index; different letters indicate significant differences at 95% confidence level.

3.5. Effects of the Organic Amendment in the Wine Volatile Composition

Table 5 lists the volatile aroma compounds determined in the wines. Among them, the concentration of 2,3-butanediol and isoamyl alcohols stand out. Other compounds with amounts above the mg/L are isobutanol, 2-phenylethanol, ethyl acetate, diethyl succinate, γ-butyrolactone, and γ-crotonolactone.

Alcohols are related to nitrogen metabolism by yeast. Usually, high levels of these compounds indicate a low content of available nitrogen [66]. Only 2-phenyethanol shows significant differences between the treatments, with the highest value being that of the control sample of the wine. However, a high concentration of a volatile compound does not always imply a greater impact on the wine aroma. In this regard, the odor threshold and the odor activity value (OAV) must be considered.

Table 5. Concentration (µg/L, except where indicated), odor perception threshold (OPT) and aroma series (AS) assigned to the volatile aroma compounds determined in wines obtained after different vine treatments.

Volatile Aroma	Treatment			OPT	AS
	Control	Compost	Vermicompost		
ALCOHOLS					
Isobutanol (mg/L)	17 a ± 1	17 a ± 1	18 a ± 1	40	1
Isoamyl alcohols (mg/L)	253 a ± 13	235 a ± 22	255 a ± 21	30	1
2,3-butanediol (mg/L)	423 a ± 27	281 b ± 11	239 c ± 21	668	4
Furfuryl alcohol	985 a ± 64	1035 a ± 66	1073 a ± 96	8000	5
Hexanol-1	718 b ± 33	514 c ± 31	881 a ± 58	8000	6
2-ethyl-1-hexanol	731 a ± 35	241 c ± 23	330 b ± 24	8000	7, 8
2-phenylethanol (mg/L)	39 a ± 1	24 c ± 2	33 b ± 3	10	9
Guaiacol	84 ab ± 6	88 a ± 8	71 b ± 6	75	1, 5
4-vinylphenol	273 b ± 22	397 a ± 28	249 b ± 22	180	1
ACETATES					
Methyl acetate	26 a ± 2	22 ab ± 1	21 b ± 2	470	1
Ethyl acetate (mg/L)	30 a ± 3	17 b ± 2	32 a ± 3	7.5	1
Isoamyl acetate	91 b ± 4	58 c ± 2	102 a ± 4	30	2
β-Phenylethyl acetate	329 b ± 7	284 c ± 22	503 a ± 28	250	5, 9
ETHYL ESTERS					
Ethyl propanoate	54 a ± 5	43 b ± 4	43 b ± 4	10	2
Ethyl butanoate	443 a ± 10	412 a ± 31	397 a ± 26	20	2
Ethyl 3-methylbutanoate	3.8 a ± 0.1	4.0 a ± 0.2	4.0 a ± 0.3	3	2, 3
Ethyl hexanoate	113 b ± 10	127 ab ± 12	143 a ± 11	14	2, 3
Ethyl 4-hydroxybutanoate	24 a ± 1	19 b ± 1	18 b ± 1	1000	5
Ethyl octanoate	229 a ± 10	222 a ± 15	348 b ± 12	5	2, 10
Ethyl decanoate	199 a ± 5	76 c ± 1	140 b ± 2	200	2, 10
Ethyl dodecanoate	24.7 a ± 0.4	4.6 c ± 0.2	7.6 b ± 0.3	500	10
Ethyl tetradecanoate	4.7 a ± 0.1	2.7 b ± 0.1	2.9 b ± 0.1	4000	10
Ethyl hexadecanoate	23 a ± 3	16 b ± 1	19 b ± 1	2000	10
Diethyl succinate (mg/L)	11 b ± 1	11 b ± 1	17 a ± 1	200	2
Ethyl vainillate	91 a ± 7	102 a ± 7	90 a ± 5	990	1, 4
LACTONES					
γ-butyrolactone (mg/L)	12.3 a ± 0.6	10.2 b ± 0.3	11.0 b ± 0.8	35	4
γ-crotonolactone (mg/L)	35.3 a ± 0.3	31 a ± 1	32 a ± 3	35	4
γ-nonalactone	22 b ± 1	38 a ± 3	21 b ± 2	30	2, 4
ALDEHYDES					
Heptanal	1.2 b ± 0.1	2.6 a ± 0.2	2.5 a ± 0.2	3	6
Octanal	0.86 b ± 0.08	2.5 a ± 0.2	2.5 a ± 0.2	2.5	7
Nonanal	1.3 c ± 0.1	3.1 a ± 0.2	2.7 b ± 0.2	2.5	7
Decanal	0.9 b ± 0.1	2.0 a ± 0.1	2.0 a ± 0.1	1.25	7, 10
Benzaldehyde	21 b ± 2	38 a ± 3	41 a ± 3	350	2
Furfural	1088 a ± 63	776 c ± 59	970 b ± 43	150,000	5
5-methylfurfural	397 a ± 38	358 a ± 12	390 a ± 30	20,000	5
5-hydroxymethylfurfural	430 a ± 32	232 b ± 18	272 b ± 22	100,000	5
KETONES					
3-heptanone	5.3 a ± 0.1	4.8 b ± 0.2	5.1 ab ± 0.3	7.5	6
6-methyl-5-hepten-2-one	106 b ± 11	164 a ± 12	123 b ± 10	50	6, 7
TERPENOIDS					
Limonene	19 a ± 2	21 a ± 1	19 a ± 2	10	1, 7
β-Damascenone	276 b ± 7	241 c ± 22	385 a ± 16	7	9
Geranyl acetone	13.0 a ± 0.2	13.1 a ± 0.5	13.2 a ± 0.2	60	9

Table 5. *Cont.*

Volatile Aroma	Treatment			OPT	AS
	Control	Compost	Vermicompost		
β-farnesene	11.5 a ± 0.2	11.2 ab ± 0.2	11.1 b ± 0.2	20	6
E-Nerolidol	11 a ± 1	10.9 a ± 0.2	11 a ± 1	700	6, 9
E-methyljasmonate	5.3 b ± 0.4	5.2 b ± 0.3	6.4 a ± 0.4	70	9
Z-dihydrofarnesol	12.1 b ± 0.1	14 a ± 1	13 ab ± 1	20	9
(Z,E)-farnesol	26 a ± 1	18 b ± 1	18 b ± 1	20	9
FATTY ACIDS					
Octanoic acid	3124 b ± 277	2183 c ± 198	3937 a ± 316	500	8
Decanoic acid	889 a ± 36	318 c ± 27	521 b ± 25	1000	8
Dodecanoic acid	180 a ± 14	138 b ± 6	173 a ± 16	2100	8
Hexadecanoic acid	62 a ± 3	62 a ± 1	61 a ± 5	100,000	8

Odor perception threshold (OPT) and aromatic series (AS) assigned to the volatile aroma compound (1: chemistry; 2: fruity; 3: green fruit; 4: creamy; 5: caramel; 6: green; 7: citrus; 8: fatty; 9: floral; 10 waxy); different letters indicate significant differences ($p < 0.05$).

Fifteen compounds show odor activity values above the unity. Ethyl octanoate and β-damascenone reach the highest values. Both compounds show the highest OAV in wines obtained from grapes treated with vermicompost. Some of these compounds do not depend on the treatment (isoamyl alcohols, ethyl butanoate, ethyl 3-methylbutanoate and limonene). 2-phenylethanol and ethyl propanoate show the highest OAV in control wines, whereas isoamyl acetate, β-phenylethyl acetate, ethyl hexanoate, and octanoic acid reach the highest values in vermicompost wine. Lastly, 4-vinylphenol and 6-methyl-5-hepten-2-one show the highest OAV in compost wine. Ethyl acetate shows no significant differences between control and vermicompost wine. Adding up the OAV of the volatile compounds in each wine, the aromas mentioned represent at least 50% of the sum. Dumitriu et al., 2020 [67], provide similar results, describing many of these compounds as responsible for the aroma of red wine treated with different oak pieces.

3.6. Aroma Series

As pointed out by Hein et al., 2009 [68], the aroma is not only determined by the volatile compounds of the wine, but also the interactions amongst them, including synergistic and antagonistic effects. Evaluating these effects is especially complex, and impossible in most cases due to the high quantity of aroma compounds, although the odor activity value can help to obtain information about the impact of a given compound on a wine aroma [69]. It is widely accepted that an OAV above the unity in volatile compounds indicates a potential contribution to wine aroma.

One way to observe all the volatile compounds is to group them based on their odor descriptor [70–72]. In this way, an analytical volatilome fingerprint is obtained, reducing the number of variables to consider when analyzing differences due to a given oenological treatment. In this study, chemistry, fruity, green fruit, creamy, caramel, floral, green, citrus, fatty, and waxy aroma series were obtained (Table 6).

Table 6. Aroma series calculated in wines obtained after different vine treatments.

Aroma	Treatment		
	Control	Compost	Vermicompost
Chemistry	17.5 a ± 0.6	16.1 a ± 0.7	17.5 a ± 0.8
Fruity	87 b ± 2	83 b ± 6	110 a ± 5
Green fruit	9.3 b ± 0.7	10.4 ab ± 0.9	11.5 a ± 0.8
Creamy	2.8 a ± 0.1	3.0 a ± 0.1	2.4 b ± 0.1
Green	3.9 c ± 0.2	5.4 a ± 0.3	4.6 b ± 0.3
Citrus	5.7 c ± 0.3	9.2 a ± 0.2	8.1 b ± 0.1
Fatty	7.3 a ± 0.6	4.7 b ± 0.4	8.5 a ± 0.7
Herbal	42 b ± 1	39 c ± 3	57 a ± 2
Floral	47 b ± 1	40 b ± 3	62 a ± 3
Waxy	47 b ± 2	46 b ± 3	72 a ± 2

Different letters indicate significant differences ($p < 0.05$).

Regarding vermicompost, fruity, herbal, floral, and waxy wine series show the highest values, whereas the creamy series shows the lowest, especially the values of the fruity series, which is mainly influenced by the chemical family, namely ester, which involves acetates, ethyl esters, and lactones. Green and citrus series show the highest values in wine with compost and the lowest is that of the fatty series. The chemistry series does not depend on the treatment.

The multivariate analysis of the series consists of the standardization of a given aroma series, with the aim of analyzing the influence of the different treatments on the variability of such series. Using this method, a spider chart is obtained, the unity being the medium value of a given series, taking all wines into consideration (Figure 4). In this sense, although the fruity series shows the highest value in all wines, control and compost wines show values below the average. In vermicompost wine, the variability compared to the average is around 17%, whereas fatty, floral, and herbal series show a variability of around 25%, the waxy series being the one with the highest variability (30%). In compost wine, the citrus series is 20% higher compared to the average value. Lastly, except for the chemistry (2%), creamy (3%), and fatty series (6%) in the control wine, lower values compared to the average were observed.

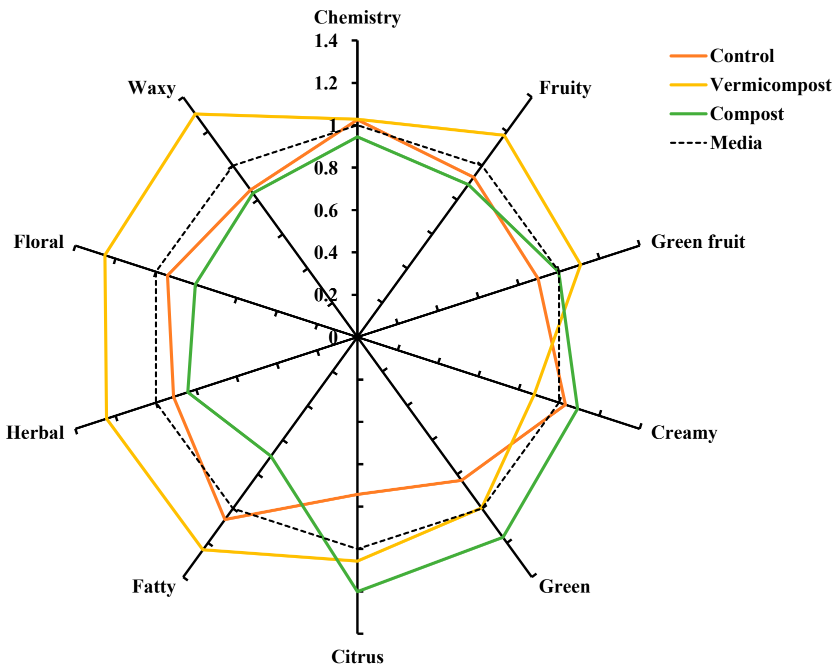

Figure 4. Multivariate analysis of the aroma series.

3.7. Cluster Analysis and Principal Components Analysis

Cluster analysis is a statistical technique aimed at classifying wines based on their similarities. To this end, the parameters for such classification must be established. This study used aroma series and colorimetric parameters. As can be observed in Figure 5, vermicompost wines are clearly differentiated from compost and control wines. As per these two, the distance in between seems to indicate that there are no remarkable differences. This type of analysis is only descriptive and provides evidence of the variations among the different treatments.

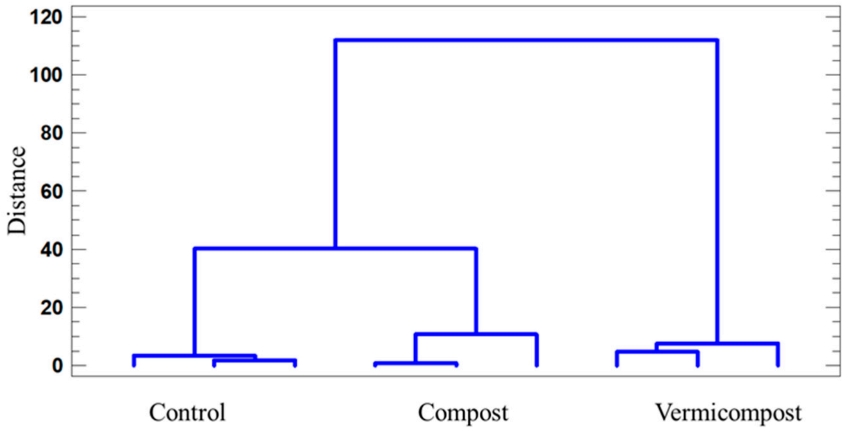

Figure 5. Cluster analysis according to the Ward method using the aroma series and the colorimetric parameters of the wines as classifying variables.

To delve into the variables responsible for the differentiation of the wines, a principal component analysis was carried out. Two main components were obtained that explained more than 94% of the variability observed (Figure 6).

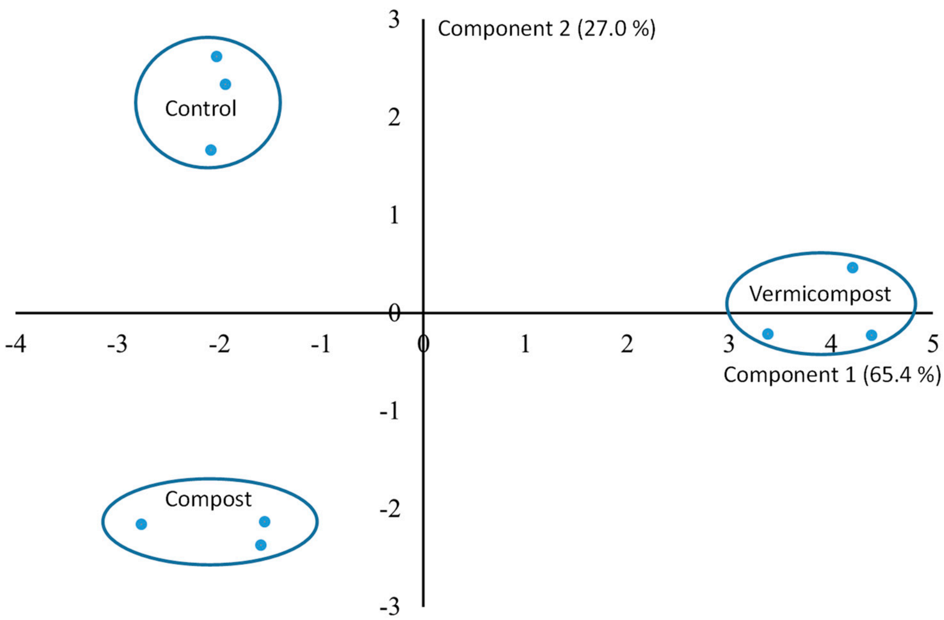

Figure 6. Principal component analysis using the aroma series and the colorimetric parameters of the wines as the classifying variables.

The first accounts for 65.4% and relates to the differentiation of vermicompost wine and the rest of the wines. The second relates to the differentiation between compost and control wines and accounts for 27% of the observed variability. In this sense, total polyphenol index, fruity, herbal, and waxy series significantly contribute to positive values of component 1, whereas absorbance 420 nm and creamy series contribute to the negative values of this component (Table 7). Regarding component 2, the series that contributed to control wine the most were chemistry and fatty, whereas citrus and green, in addition to absorbance at 520 nm, contributed to differentiating compost wine (Table 7).

Table 7. Weight of the selected variables to perform the principal component analysis. In bold the main variables of each component.

Variables	Component 1	Component 2
TPI	**0.3220**	0.0195
Absorbance 420 nm	**−0.3038**	0.1682
Absorbance 520 nm	0.2410	**−0.3440**
Absorbance 620 nm	0.3089	−0.1604
Chemistry	0.1522	**0.3435**
Fruity	**0.3224**	0.0635
Green fruit	0.2533	−0.2187
Creamy	**−0.2952**	−0.1184
Green	0.0086	**−0.4929**
Citrus	0.0714	**−0.4986**
Fatty	0.2384	**0.3460**
Herbal	**0.3228**	0.0898
Floral	0.3132	0.1546
Waxy	**0.3285**	0.0151

TPI: total polyphenol index.

4. Conclusions

The results obtained have shown that the agricultural application of compost and vermicompost from the spent substrate of mushroom cultivation does not cause adverse effects on the physicochemical properties of the vineyard soil, but results in a positive increase in its content in organic matter and macronutrients. Although the total concentration of each metal slightly increased in the soil of the treated plots, with respect to the control soil, in no case was metal contamination of the aerial parts of the vine observed. On the other hand, among the oenological parameters usually analyzed, the highest content of compounds responsible for the red and violet coloration in wines being obtained from grapes treated with vermicompost stands out. The control wine had high absorbance values at 420 nm, which is related to brown tones. Regarding the aroma composition, the vermicompost wine was the one with the highest aroma concentration, especially fruity, floral, herbal, green fruit, fatty, and waxy aromas. Lastly, considering the sum of all values of the aroma series, compost wine showed the lowest levels; however, its citrus, green, and fatty series excelled compared to the rest of the wines. Therefore, it can be assumed that organic amendments have an impact on the composition of the grape and, consequently, on wines. It can be concluded that the use of these types of amendments can be an alternative source of nutrients for the sustainable management of the vineyard, although it would be interesting to analyze the effects in the longer term and at the same study doses.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/app13148001/s1, Table S1: Major and minor aroma compounds identified in the wines.

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