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Article

Aroma Identity of Red Wines from Old-Vine Genotypes Cultivated on Mount Etna

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Abstract

The slopes of Mount Etna (Sicily, Italy) are home to an extraordinary viticultural heritage, comprising several vine genotypes at risk of extinction, whose oenological potential remains largely unexplored. This study aims to provide an aromatic characterization of red wines produced from five old-vine genotypes (Barbarossa Etna, Madama Nera, Moscatella Nera, Minnella Nera, and Terribile), comparing them with local and international cultivars. The wines were produced using standardized microvinification techniques; physicochemical parameters were measured according to OIV methods, volatile aromatic compounds were analyzed using HS-SPME/GC-MS, and sensory analysis was carried out using quantitative descriptive analysis. The wines from old-vine genotypes exhibited physicochemical characteristics in line with Etna DOC standards. The profiling of volatile organic compounds revealed clear cultivar-specific aromatic characteristics, with Moscatella Nera exhibiting the most complex and terpene-rich content. The sensory analysis confirmed a distinctive aroma for each old-vine genotype wine, with substantial differences from traditional and international wines. These results support the oenological significance of these old-vine genotypes and their potential reintroduction into production to enhance the biodiversity and identity of Etna wines.

Keywords: Etna DOC red wine; old-vine genotypes; aroma volatile compounds; aroma sensory analysis

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1. Introduction

The slopes of Mount Etna (Sicily, Italy) represent one of the most extraordinary wine-growing environments in the Mediterranean basin. The combination of complex volcanic soils, diverse microclimates, marked diurnal temperature fluctuations, and elevations ranging from approximately 400 to over 1000 m above sea level has driven genetic selection, resulting in a highly diverse and locally adapted grapevine germplasm pool [1,2]. Viticulture in this area has ancient origins, as documented by archaeological evidence and historical records dating back to the 5th–3rd centuries B.C. [3]. Early twentieth-century studies reported the presence of nearly 50 ancient cultivars in the Etna region, many of which are now extinct or preserved only in isolated old vineyards [4]. Recently, some of these cultivars have been rediscovered and described in various studies that have assessed their agronomic characteristics and oenological potential [5,6].

Nowadays, the main red grape cultivars employed for Etna DOC red wine production are Nerello Mascalese and Nerello Cappuccio, which dominate the current vineyard landscape and have been the focus of extensive scientific research [7–11]. In contrast, several old minor indigenous genotypes, commonly referred to as "relic jewels", remain poorly characterized despite their historical relevance and potential oenological value [12]. The conservation and valorization of these genetic resources are of major strategic relevance in the context of sustainable viticulture and local biodiversity preservation.

Indeed, the wine sector is currently facing one of its most critical challenges: climate change. Rising temperatures, shifts in precipitation patterns, and an increased frequency of extreme weather events are directly affecting grapevine cultivation, with consequences for yield stability, fruit composition, and overall wine quality [13]. Alongside these environmental pressures, the sector is also experiencing a clear shift in consumer preferences, with growing demand for wines that express a strong and authentic link to terroir identity. In this context of climatic uncertainty and evolving market expectations, the wine sector is required to adapt its production strategies, while researchers are called upon to develop solutions that ensure both sustainability and wine quality [10,14,15]. For this reason, in different areas of the world [16–21], minor autochthonous vine genotypes are attracting renewed scientific interest due to their potential adaptability to marginal environments and abiotic stress conditions. Beyond their agronomic resilience, these genotypes may also provide distinctive compositional and sensory traits capable of enhancing wine typicity and regional identity.

Among the factors influencing wine quality and consumer perception, the volatile aromatic profile plays a key role [22,23]. Wine aroma derives from a complex mixture and interaction of volatile organic compounds influenced by grape genotype, environmental conditions, viticultural practices, and winemaking techniques [24–26]. The characterization of volatile compounds is therefore particularly relevant for underexplored grapevine resources, as it may reveal unique chemical and sensory fingerprints useful for varietal differentiation and product valorization [27–29]. However, limited information is currently available regarding the aromatic composition and sensory characteristics of wines produced from Etna old-vine genotypes.

Therefore, the present study investigated the oenological and aromatic characteristics of red wines produced from five old-vine genotypes from the Etna area (Barbarossa Etna, Madama Nera, Moscatella Nera, Minnella Nera, and Terribile) through volatile profile analysis and sensory evaluation. The experimental wines were compared with those from two traditional Sicilian cultivars (Nerello Mascalese and Nerello Cappuccio) and one international cultivar (Cabernet Sauvignon) to assess the oenological potential, aromatic distinctiveness, and potential contribution of these genotypes to the diversification and identity of Etna wines.

2. Materials and Methods

2.1. Plant Material, Field Conditions, and Experimental Design

The study was conducted in 2024 and included five minor red-berried *Vitis vinifera* L. genotypes (Barbarossa Etna, Madama Nera, Terribile, Moscatella Nera, and Minnella Nera), two traditional Etna cultivars (Nerello Mascalese and Nerello Cappuccio), and one international cultivar (Cabernet Sauvignon). The five minor red-berried genotypes were selected within a broader agronomic and oenological recovery and valorization program

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started in the early 2000s [6]. Nerello Mascalese and Nerello Cappuccio were included as reference traditional red grapevine cultivars of the Etna DOC wine, as they constitute the ampelographic basis of Etna Rosso and Etna Rosato production [7]. Cabernet Sauvignon was included as a reference international red cultivar and is reported as one of the most widely cultivated international red grapevine varieties in the world and also in the Etna area, according to the latest Sicilia DOC Consortium elaboration based on SIAN data [30].

The grapevines were cultivated in the same experimental vineyard located on the northern slope of Mount Etna, in the Randazzo district (Catania, Sicily, Italy; 37.879° N, 14.948° E) under identical environmental and agronomic conditions. The Nerello Mascalese, Nerello Cappuccio, and Cabernet Sauvignon vineyards were approximately 7 years old, whereas the vineyards containing the five minor accessions, established more recently, were approximately 5 years old, at their third production year. All vines were grafted onto 775 Paulsen rootstock, trained to a vertical shoot positioning (VSP) system, and spur-pruned according to a unilateral cordon training system. Vine spacing was 1.2 m between rows and 1.0 m within rows. Vines were grown under rainfed conditions on volcanic soil, described as well-drained and moderately fertile, with a loamy-sandy texture and sub-acid pH. The disease control program during the season followed the Integrated Pest Management (IPM) guidelines of the Sicily Region [31].

The experimental design consisted of three randomized blocks for each genotype, with each block including 50 vines, resulting in a total of 150 vines per genotype. Grapes harvested from these blocks were merged to form the final lot used for the microvinifications in order to reduce the influence of intra-vineyard variability and to obtain a sample more representative of the overall varietal expression.

A weather station located near the experimental vineyard provided monthly minimum, mean, and maximum air temperature (T), rainfall, and relative humidity data (Figure S1).

2.2. Microvinifications

The harvest date was defined according to the achievement of optimal commercial ripening, titratable acidity of 5.5–6.0 g/L, and a soluble solids content (SSC) of at least 20–23 °Brix. The grapes were manually harvested and vinified in the experimental winery of the Regional Institute of Wine and Oil (IRVO) in Marsala (Sicily, Italy). The same protocol of vinification was adopted for each sample.

Microvinifications were performed in 50 L stainless steel vessels. The musts of destemmed and crushed grapes were fermented for 9 days at 26–28 °C in contact with the skins after the addition of dry yeast NDA21 (0.3 g/L), followed by sulfiting at 0.05 g/L. Malolactic fermentation was induced by the addition of biomass (0.005 kg/L dregs). Standard winemaking practices were applied throughout the process, including regular cap management and monitoring of fermentation kinetics until the completion of alcoholic fermentation. After setting, the wines were placed in dark bottles and stored in a conditioned room at 18–20 °C and 70–80% relative humidity until analyses.

2.3. Wine Chemical Analysis

The key physicochemical parameters of the wines, including volatile alcohol content, pH, total and volatile acidity, reducing sugars, dry extract, total polyphenols, glycerol, and malic, lactic, and tartaric acids, were measured by applying the official analytical methods set out in the International Organization of Vine and Wine (OIV) [32].

2.4. Extraction and Analysis of Volatile Compounds

The volatile aroma profile of the red wines was characterized using a well-established procedure based on headspace solid-phase microextraction (HS-SPME) coupled with gas chromatography–mass spectrometry (GC-MS) [33]. Specifically, a 40 mL vial was loaded with 20 mL of wine sample; extraction was carried out in the headspace using a DVB/CAR/PDMS (divinylbenzene/carboxen/polydimethylsiloxane) fiber with a 50/30 µm film thickness, fitted in its manual holder (Supelco, Bellefonte, PA, USA). The vial was kept at 30 °C with continuous stirring of the sample; following a 15 min equilibration step, extraction lasted 20 min. Afterwards, the SPME fiber was inserted into the splitless injector of the gas chromatograph, held at 260 °C, to allow thermal desorption of the analytes over 3 min. Volatile compound analysis was performed using a Shimadzu GC 2010 Plus gas chromatograph coupled in-line with a TQMS 8040 triple quadrupole mass spectrometer (Shimadzu, Milan, Italy). The instrumental conditions applied were as follows: VF-WAXms capillary column, 60 m, 0.25 mm i.d., film thickness 0.25 µm, polar column (Agilent Technologies Italia S.p.A., Milan, Italy); temperature program: 45 °C for 5 min, ramp to 80 °C at 10 °C/min, then to 240 °C at 2 °C/min; carrier gas helium at a constant flow of 1 mL/min; transfer line temperature 250 °C; acquisition range 40–200 m/z; scan speed 1250 amu/s. As reported in previous studies [26], each compound was identified by combining mass spectral data with the NIST'24 library (NIST/EPA/NIH, Wiley, Gaithersburg, MD, USA), the FFNSC 3.0 database, linear retention indices, literature data, and, where available, injection of reference standards. Quantification of the volatile compounds was carried out using the standard addition method [34]; standards were sourced from Sigma–Aldrich s.r.l. (Milan, Italy) at the highest purity grade available. Each wine sample was analyzed in triplicate.

2.5. Quantitative Descriptive Analysis

The quantitative descriptive analysis (QDA) of the wine samples was conducted in accordance with the protocol adapted from Scacco et al. (2010) [35]. A panel of 8 (4 males and 4 females) judges was selected from among students and staff of the Department of Veterinary Sciences, University of Messina. All participants signed an informed consent form in agreement with the ethical principles of the Declaration of Helsinki. Before inclusion, candidates underwent preliminary screening tests to assess their sensory ability with respect to wine-related aromas. In accordance with ISO standards [36], the sensory profiles were developed using three groups of selected tasters, each of whom was trained during a series of sessions. The panel identified the aroma's descriptive attributes based on the frequency with which assessors used each term across the various sessions, retaining only those cited by more than 60% of the assessors. The final set consisted of 9 aroma descriptors (floral, fruity, red fruits, citrus, rose/violet, apple/pear, herbaceous, woody, and spicy). Sensory intensities were evaluated on a nine-point scale, ranging from 1 (absent sensation) to 9 (extreme intensity). Each wine was evaluated in triplicate. All sessions took place between 10:00 and 12:00 in individual tasting booths [37]. Assessors received 50 mL of wine at 22 °C ± 1 °C (room temperature), served in standardized glasses [38] labeled with three-digit codes and covered to minimize volatile loss. The order of sample presentation was randomized across both assessors and sessions. Water was available between samples for palate cleansing. Data were recorded using a computerized direct-entry system (FIZZ Byosistemes, version 2.00 M, Couternon, France).

2.6. Statistical Analysis

Data were analyzed using one-way ANOVA, followed by Tukey's HSD post hoc test ($p < 0.05$). Multivariate analyses were applied to volatile compound datasets to explore relationships among samples by using the R environment (v 4.5.3) [39]. The total volatile organic compounds (VOCs) matrix was analyzed using principal component analysis (PCA) by applying the singular value decomposition method to assess the covariances/correlations between individuals. The libraries used for PCA analyses were "factoextra" and "FactoMineR" [40]. For the PCA analyses, k-means clustering was conducted. The optimal number of clusters was estimated by the "average silhouette" method. Moreover, hierarchical clustering was performed on the total VOCs matrix using Euclidean distance and Ward's method (ward.D2), and results were visualized as a heatmap generated with the pheatmap package [41].

Bubble plots were generated with the ggplot2 package [42] exclusively on varietal VOC abundance data (expressed on a log10 scale).

3. Results and Discussions

3.1. Physicochemical and Chemical Parameters of Wine

The physicochemical analyses carried out on the wines showed significant differences, as reported in Table 1.

Table 1. Physicochemical parameters (mean ± SD, $n = 3$) of old-vine genotype and reference wines.

Parameter	Barbarossa Etna	Madama Nera	Minnella Nera	Moscatella Nera	Terribile	Cabernet Sauvignon	Nerello Cappuccio	Nerello Mascalese
Alcohol (%vol)	11.78 ± 1.05 c	10.18 ± 0.95 d	11.04 ± 0.97 c	12.50 ± 1.03 b	12.50 ± 1.01 b	13.10 ± 1.02 a	11.60 ± 1.04 c	13.35 ± 1.12 a
pH	3.49 ± 0.12	3.50 ± 0.21	3.63 ± 0.06	3.88 ± 0.11	3.56 ± 0.08	3.97 ± 0.12	3.66 ± 0.10	3.80 ± 0.14
Total acidity (g/L)	6.95 ± 0.42 a	6.81 ± 0.48 a	6.77 ± 0.47 a	6.18 ± 0.34 a	6.69 ± 0.38 a	5.35 ± 0.24 b	5.19 ± 0.28 b	5.10 ± 0.35 b
Volatile acidity (g/L)	0.17 ± 0.02 c	0.30 ± 0.02 b	0.19 ± 0.02 c	0.14 ± 0.02 c	0.30 ± 0.01 b	0.62 ± 0.04 a	0.59 ± 0.03 a	0.39 ± 0.02 b
Reducing sugars (g/L)	1.54 ± 0.13 c	1.91 ± 0.18 b	2.25 ± 0.19 a	2.51 ± 0.21 a	1.59 ± 0.08 c	1.88 ± 0.11 b	2.55 ± 0.19 a	1.87 ± 0.15 b
Dry extract (g)	29.40 ± 1.3 b	29.54 ± 2.1 b	29.30 ± 1.9 b	33.40 ± 2.2 a	27.80 ± 1.6 c	33.36 ± 3.12 a	26.50 ± 2.24 c	31.90 ± 2.78 a
Total polyphenols (mg/L)	1529 ± 112.7 c	2048 ± 167.8 a	1633 ± 98.3 bc	2085 ± 155.4 a	1927 ± 105.6 ab	1848 ± 145.3 b	1277 ± 104.6 d	1854 ± 167.9 b
Glycerol (g/L)	11.73 ± 0.8 ab	9.90 ± 0.56 c	10.82 ± 0.88 b	12.47 ± 0.88 a	10.89 ± 0.75 b	9.75 ± 0.78 c	9.43 ± 0.82 c	12.15 ± 1.02 a
Malic acid (g/L)	1.80 ± 0.03 a	1.38 ± 0.06 b	1.92 ± 0.07 a	1.98 ± 0.11 a	1.18 ± 0.98 b	0.34 ± 0.01 c	0.02 ± 0.01 d	0.02 ± 0.01 d
Lactic acid (g/L)	0.42 ± 0.01 d	0.76 ± 0.06 c	0.61 ± 0.04 cd	0.57 ± 0.02 cd	0.73 ± 0.05 c	1.78 ± 0.13 a	1.56 ± 0.12 ab	1.31 ± 0.11 b
Tartaric acid (g/L)	3.34 ± 0.12 a	3.82 ± 0.18 a	3.40 ± 0.16 a	2.41 ± 0.07 b	3.51 ± 0.18 a	2.24 ± 0.19 b	2.89 ± 0.23 b	3.47 ± 0.24 a

Different lowercase letters within the same row indicate significant differences (Tukey's HSD, $p < 0.05$). Rows without letters indicate no significant differences. Old-vine genotype wines: Barbarossa Etna, Madama Nera, Minnella Nera, Moscatella Nera, and Terribile. Reference wines: Cabernet Sauvignon, Nerello Cappuccio, Nerello Mascalese.

The wines produced from old-vine genotypes generally showed lower alcohol contents than those obtained from Cabernet Sauvignon and Nerello Mascalese. This behavior may reflect a lower sugar accumulation capacity under the pedoclimatic conditions of the Etna area and could represent a valuable trait in the context of climate change, where wines with moderate alcohol levels are increasingly appreciated both technologically and commercially. In particular, Madama Nera showed the lowest alcohol concentration, whereas Moscatella Nera and Terribile reached values closer to those of the reference cultivars.

The acidity profile differed among the samples. Total acidity was consistently higher in wines obtained from old-vine genotypes than in Cabernet Sauvignon, Nerello Cappuccio, and Nerello Mascalese wines, while volatile acidity was significantly lower. Higher total acidity may indicate a greater capacity for acid retention during berry ripening, contributing to fresher and potentially more balanced wines. Conversely, the relatively low volatile acidity observed in most old-vine genotype wines could suggest regular fermentation dynamics and limited microbial alteration during vinification.

Reducing sugar content varied among the samples, with the highest values detected in Minnella Nera and Moscatella Nera wines, comparable to Nerello Cappuccio, whereas Barbarossa Etna and Terribile showed the lowest concentrations. Although all wines remained within the range typical of dry red wines, these differences reflect differences in fermentation kinetics.

The total polyphenol content was generally higher in wines produced from old-vine genotypes, particularly in Madama Nera and Moscatella Nera. This result is of considerable oenological interest, as phenolic compounds are strongly associated with wine color, mouthfeel, antioxidant properties, and overall sensory quality. Moreover, polyphenolic richness may positively influence color stability and aging aptitude in red wines. Previous studies highlighted that grape genotype and winemaking conditions strongly affect phenolic composition and its contribution to wine typicity and quality [43]. Such characteristics may therefore contribute positively to the distinctive identity and technological potential of wines produced from these underutilized genotypes.

The highest glycerol concentration was detected in Moscatella Nera, with values comparable to Nerello Mascalese. Since glycerol contributes to mouthfeel, smoothness, and perceived body, this parameter may partially influence the sensory complexity and balance of these wines [44].

Regarding organic acids, old-vine genotype wines consistently showed higher malic acid concentrations, particularly in Moscatella Nera, Minnella Nera, and Barbarossa Etna, whereas lactic acid content remained markedly lower than in the reference wines. This trend may indicate limited malolactic fermentation in old-vine genotype wines, potentially linked to varietal characteristics or differences in fermentation behavior. Malolactic fermentation is known to convert the sharper L-malic acid into softer L-lactic acid, thereby reducing wine acidity and modifying sensory perception and microbial stability [45,46]. Consequently, the higher malic acid levels in old-vine genotype wines may contribute to a greater acidity sensory perception.

Tartaric acid concentrations were often comparable to those observed in Nerello Mascalese wines. The only exception was Moscatella Nera, which showed lower values, similar to Cabernet Sauvignon and Nerello Cappuccio.

Overall, although the physicochemical profiles of the old-vine genotype wines remained within the compositional range typical of Etna red wines, several distinctive traits emerged, including higher acidity retention, lower alcohol content, and, in some genotypes, enhanced phenolic richness.

3.2. Volatile Aroma Compounds

The volatile profiles of wines obtained from old-vine genotypes showed qualitative and quantitative differences when compared with both the international variety Cabernet Sauvignon and the Etna DOP cultivars Nerello Mascalese and Nerello Cappuccio, as reported in Table S1.

A total of 87 volatile aroma compounds were identified and quantified by GC-MS, belonging to seven chemical classes: esters, alcohols, acids, C_{13}-norisoprenoids, terpenes, aldehydes, and hydrocarbons. Alcohols constituted the quantitatively dominant fraction, followed by esters, acids, aldehydes, terpenes, hydrocarbons, and C_{13}-norisoprenoids. Significant inter-varietal differences were also observed across all chemical classes ($p < 0.05$), underscoring the influence of genotype on the volatile composition of these wines. Figure 1 reports the total concentration of each chemical class per genotype, which clearly illustrates the predominance of esters and alcohols, as well as the exceptional terpene content in Moscatella Nera. These findings are consistent with the extensive chemical diversity documented in minority and autochthonous *Vitis vinifera* cultivars, in which metabolomic differentiation at the level of volatile classes has been proposed as a reliable tool for chemotaxonomic characterization [16–19,27,47].

Esters represented the most qualitatively represented chemical class across all samples, with total concentrations ranging from 3228 μg/L (Terribile) to 5292 μg/L (Nerello Mascalese). Ethyl octanoate was the most abundant ester detected, reaching up to 2794 μg/L in Nerello Mascalese and 2680 μg/L in Barbarossa Etna, concentrations well above the odor threshold of 580 μg/L, thereby confirming its contribution to the fruity and candy-like aromatic character of these wines. Ethyl decanoate was the second most concentrated ester, with particularly high content in Moscatella Nera (1134 μg/L), Cabernet Sauvignon (956 μg/L), and Nerello Mascalese (935 μg/L), all exceeding its odor threshold of 200 μg/L. Ethyl hexanoate was markedly high in Minnella Nera (1333 μg/L), substantially surpassing its threshold of 420 µg/L, conferring this variety with distinctive green-apple and fruity notes. These medium-chain fatty acid ethyl esters are biosynthesized intracellularly by *Saccharomyces cerevisiae* through the action of ester synthases, and their production is modulated by fermentation temperature, nitrogen availability, and unsaturated fatty acid content of the must [48]. Isoamyl acetate, responsible for the characteristic banana-like odor, was most abundant in Barbarossa Etna (400 µg/L), well above its low odor threshold of 30 µg/L, indicating a significant olfactory contribution specific to this old-vine genotype. 2-Phenethyl acetate was more abundant in Madama Nera (75.93 µg/L) compared with all other genotypes, imparting rose-like, floral-green, and fruity notes; this compound is a direct metabolic product of 2-phenylethyl alcohol via alcohol acetyltransferase activity, and both it and its precursor are associated with floral and rose-like aromas in wine [49]. Ethyl 2-hydroxy-4-methylpentanoate was detected exclusively in appreciable quantities in Terribile (10.25 µg/L) and Cabernet Sauvignon (3.30 µg/L), conferring a distinctive blackberry aroma. Methyl salicylate, associated with wintergreen and mint descriptors, was detected only in Minnella Nera and Moscatella Nera.

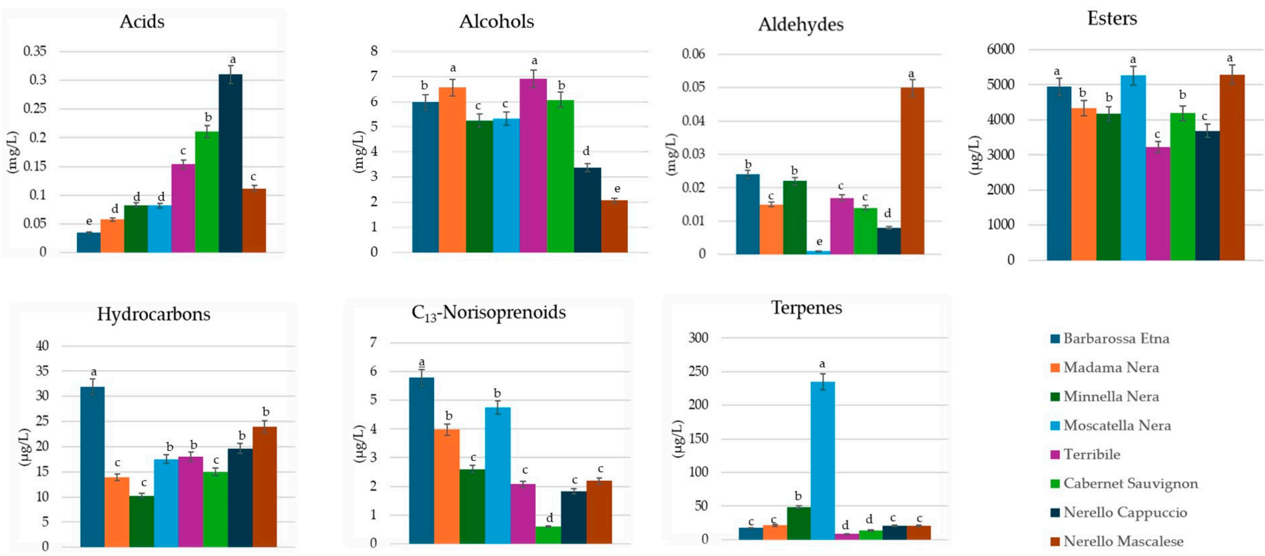

Figure 1. Quantitative volatile aroma composition for each class of substances in old-vine genotypes (Barbarossa Etna, Madama Nera, Minnella Nera, Moscatella Nera, and Terribile) and reference (Cabernet Sauvignon, Nerello Cappuccio, and Nerello Mascalese) wine samples. All values are expressed as mean ± standard deviation ($n = 3$). Different letters within each panel indicate significant differences among genotypes (Tukey's HSD, $p < 0.05$).

Higher alcohols constituted the second most qualitatively abundant chemical class, with total concentrations ranging from 2.07 mg/L (Nerello Mascalese) to 6.92 mg/L (Terribile) (Figure 1). 2-Phenylethyl alcohol was by far the most abundant alcohol across all genotypes (1.02–5.47 mg/L), with Terribile and Madama Nera showing the highest concentrations (5.47 and 5.22 mg/L, respectively) and Nerello Mascalese the lowest (1.02 mg/L). 2-Phenylethyl alcohol is biosynthesized via the Ehrlich pathway from L-phenylalanine, a reaction catalyzed in *S. cerevisiae* by the transaminases [49]. The markedly higher 2-phenylethyl alcohol in the old-vine genotype wines compared with Nerello Cappuccio and Nerello Mascalese supports a cultivar-mediated influence on grape-derived phenylalanine availability or on the metabolic flux through the Ehrlich pathway, rather than a purely environmental or microbiological effect. Hexanol, with herbaceous and fatty descriptors, was close to the odor threshold (110 µg/L) in Barbarossa Etna and Cabernet Sauvignon. This C_6 alcohol originates from the lipoxygenase–hydroperoxide lyase (LOX–HPL) pathway acting on grape berry polyunsaturated fatty acids (linoleic and linolenic acids) during crushing [50]. Dodecanol, tetradecanol, and hexadecanol, long-chain fatty alcohols with floral and waxy notes, were detected exclusively in Terribile, Cabernet Sauvignon, and Nerello Mascalese, suggesting variety-specific differences in the extent of lipid catabolism during fermentation.

Volatile acids had a total content ranging from 0.035 mg/L (Barbarossa Etna) to 0.310 mg/L (Nerello Cappuccio) (Figure 1). Acetic acid was dominant in all samples, with the lowest concentrations in Barbarossa Etna (0.035 mg/L) and the highest in Nerello Cappuccio (0.274 mg/L). All values were well below the statutory limit of 1.2 g/L for red wines, indicating a controlled and reproducible fermentation process across all samples. Medium- to long-chain fatty acids (octanoic, decanoic, dodecanoic, and tetradecanoic) were detected predominantly in Terribile and Cabernet Sauvignon. Figure 1 highlights the higher acid content in Nerello Cappuccio, with a total acid content approximately an order of magnitude higher than Barbarossa Etna and the other genotypes. The consistently low acid profile of the four old-vine genotype wines (Barbarossa Etna, Madama Nera, Minnella Nera, and Moscatella Nera) is an oenologically favorable feature, suggesting cleaner fermentation profiles and a lower burden of volatile acidity, as also shown by physicochemical parameters (Table 1) [48].

Three C_{13}-norisoprenoids were identified: 6-methyl-5-hepten-2-one, α-ionone, and β-ionone. Total norisoprenoid contents ranged from 0.59 μg/L (Cabernet Sauvignon) to 5.79 μg/L (Barbarossa Etna) (Figure 1). α-Ionone (rose, fruity) was most abundant in Barbarossa Etna (3.25 μg/L) and absent in Cabernet Sauvignon. β-Ionone (rose, floral, and violet) was absent in Terribile and Cabernet Sauvignon while particularly elevated in Moscatella Nera (1.70 μg/L) and Barbarossa Etna (1.54 μg/L). 6-Methyl-5-hepten-2-one, a carotenoid degradation product associated with green and citrus notes, showed the highest concentrations in Barbarossa Etna (1.00 μg/L) and Moscatella Nera (0.95 μg/L). C_{13}-norisoprenoids are derived from the oxidative cleavage of carotenoids (principally β-carotene and lutein) catalyzed by carotenoid cleavage dioxygenases (CCDs) [51–53]. β-Ionone, formed directly from β-carotene, is recognized as one of the most sensorially significant apocarotenoids in wine, with odor thresholds reported as low as 0.007–0.09 μg/L [52], thus strongly impacting the sensory feature of wines.

The terpene fraction showed the most evident inter-genotype differences among all compound classes (Figure 1). Total terpene contents ranged from 8.73 μg/L (Terribile) to 235.04 μg/L (Moscatella Nera), with the latter showing more than ten-fold greater terpene content than any other variety and more than twenty-five-fold greater than the terpene-poorest cultivars (Terribile and Cabernet Sauvignon). Moscatella Nera was characterized by exceptionally elevated concentrations of linalool ethyl ether (93.31 μg/L), citronellol (30.63 μg/L), limonene (23.02 μg/L), terpinolene (21.39 μg/L), α-terpineol (14.57 μg/L), and nerol (7.34 μg/L). Linalool, with a low odor threshold of 6 μg/L, exceeded this threshold in Moscatella Nera (17.30 μg/L), confirming its direct olfactory contribution of fruity and citrus notes. Myrcene and (E)-β-ocimene were detected exclusively in Moscatella Nera. This monoterpene profile, encompassing linalool, citronellol, α-terpineol, nerol, limonene, and terpinolene, is qualitatively and quantitatively reminiscent of the muscat aromatic profile documented in Muscat-related cultivars, in which linalool, geraniol, α-terpineol, citronellol, and nerol constitute the core terpene fingerprint [54]. Minnella Nera also showed a noteworthy intermediate terpene profile, with elevated citronellyl acetate (8.12 μg/L) and the exclusive presence of geranyl acetate (0.73 μg/L), consistent with an intermediate aromatic status between true aromatic and fully non-aromatic varieties [55]. In contrast, Barbarossa Etna, Terribile, and Cabernet Sauvignon exhibited notably poor terpene fractions, which is consistent with the non-aromatic status.

Aldehydes were present at minor concentrations (0.001–0.050 mg/L), with acetaldehyde as the dominant compound. Nerello Mascalese was the richest wine in aldehydes, attributable exclusively to acetaldehyde (0.050 mg/L), more than twice the level of any other variety. This may reflect a higher oxidative potential during fermentation or post-fermentation or cultivar-specific differences in alcohol dehydrogenase (ADH) activity. Aliphatic aldehydes such as nonanal and decanal, both with odor thresholds near 1 μg/L and associated with green and orange-peel character, were detected exclusively in Terribile and Cabernet Sauvignon.

Statistical Elaboration of VOC Data

The results of the PCA highlighted a high level of variability, with the first two principal components accounting for 48.3% of the total variance (30.2% for PC1 and 18.1% for PC2). Regarding the analysis of individuals (Figure 2A), k-means clustering enabled the identification of four clusters. The first cluster was represented by Moscatella Nera wine, which exhibited the highest level of differentiation from the other genotypes. The second cluster included Barbarossa Etna and Madama Nera; the third cluster comprised Minnella Nera, Nerello Mascalese, and Nerello Cappuccio; and the fourth cluster includes Cabernet Sauvignon and Terribile. In addition, the analysis of variables provided further insight into the compounds driving the observed differentiation. The PC contribution analyses (Figure 2B) indicated that several volatile compounds strongly contributed to the separation along the first two principal components, highlighting their key role in discriminating among genotypes. The VOCs that contributed most to sample discrimination were β-ionone, heptyl acetate, and ethyl 9-decenoate, which were primarily responsible for separation along the first principal component. Terpinolene, myrcene, (E)-β-ocimene, nerol oxide, linalool ethyl ether, δ-2-carene, nerol, citronellol, ethyl dodecanoate, limonene, ethyl heptanoate, isopentyl octanoate, and nonanal contributed most strongly to the second principal component (PC2).

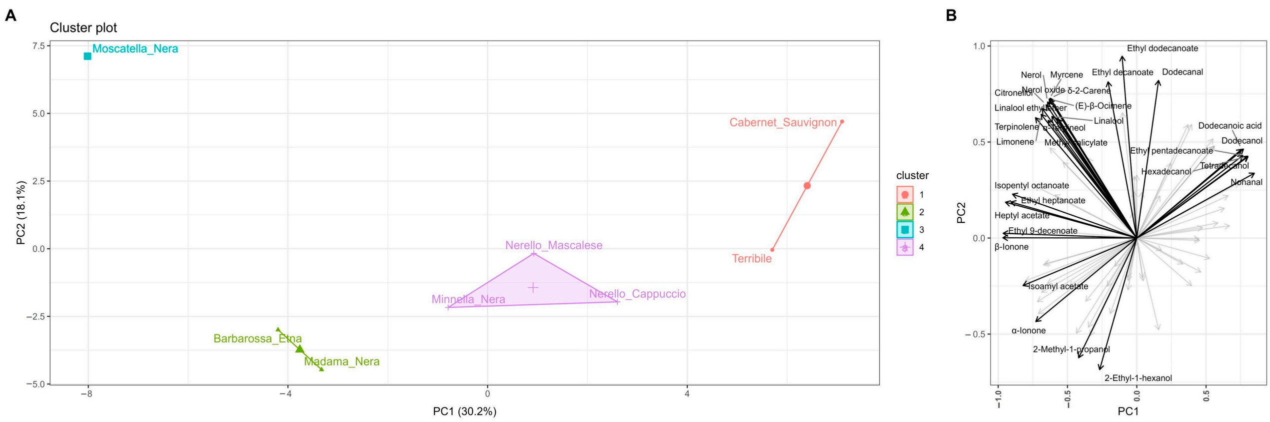

Figure 2. Principal Component Analysis (PCA) of VOCs identified on the wine grape clusters under study. Panel (**A**) displays the distribution of distinct individuals clustered using the K-means methodology. Panel (**B**) displays the contribution of all VOCs to the total variance; only the top 30 compounds contributing most to the variability have been labeled.

The results of the hierarchical clustering (Figure 3) confirmed those obtained from the k-means clustering. Cabernet Sauvignon and Terribile formed a cluster, while the Moscatella Nera wine clustered alone. Finally, Barbarossa Etna and Madama Nera formed another cluster that was very close to Minnella Nera, with Nerello Cappuccio and Nerello Mascalese grouping together. Clear differentiation among the grapevine genotypes was driven by distinct groups of VOCs contributing to their chemical characterization. In particular, Moscatella Nera exhibited markedly higher concentrations of linalool, α-terpineol, δ-2-carene, (*E*)-β-ocimene, myrcene, nerol oxide, methyl salicylate, limonene, terpinolene, citronellol, linalool ethyl ether, and nerol. Terribile was instead characterized by increased levels of 2-heptanol, (E)-β-bisabolene, and ethyl 2-hydroxy-4-methylpentanoate. Barbarossa Etna showed a particularly rich content in (E)-3-hexen-1-ol, whereas Madama Nera was mainly distinguished by higher abundances of (Z)-3-hexen-1-ol, α-terpinene, and 2-phenethyl acetate. Finally, the cluster including Minnella Nera, Nerello Cappuccio, and Nerello Mascalese displayed variety-specific metabolic signatures: Minnella Nera was mainly characterized by geranyl acetate, ethyl (E)-4-heptenoate, and ethyl hexanoate; Nerello Cappuccio by α-muurolene and acetic acid; and Nerello Mascalese by propyl octanoate and acetaldehyde.

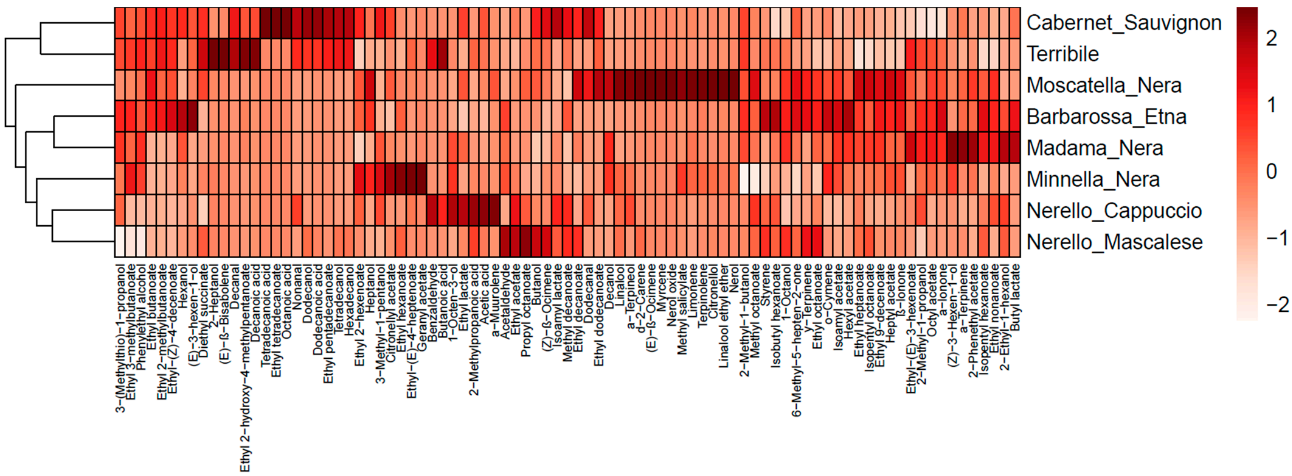

Figure 3. Heatmap with hierarchical clustering of metabolomic variables across the grapevine genotypes under study. The color scale represents normalized values of the variables, ranging from dark red (high values, >2) to white/light pink (low values, <−2). The row dendrogram clusters the genotypes based on the similarity of their VOC profiles.

Finally, concerning the varietal VOC abundance characterizing the red berry genotypes (Figure 4), linalool and linalool ethyl ether were the most abundant compounds across nearly all genotypes, with Moscatella Nera showing the highest levels (7.65 and 7.38, respectively), followed by Nerello Cappuccio (7.1 and 6.1, respectively) and Nerello Mascalese (7.01 and 6.1, respectively). Limonene was also widely detected, particularly in Moscatella Nera (6.77) and Minnella Nera (6.28). Geranyl acetate and citronellyl acetate showed notable abundance exclusively in Moscatella Nera (5.27 and 6.89, respectively), suggesting a distinctive volatile profile for this variety.

Terribile and Barbarossa Etna displayed an overall lower abundance across most compounds, with a few exceptions. Cabernet Sauvignon showed a relatively uniform and moderate distribution of varietal volatile aroma compounds, without any particularly dominant compound. Terpene-related compounds such as α-terpineol, α-terpinene, γ-terpinene, and *p*-cymene were detected at low-to-moderate levels across all genotypes, while terpenes such as (E)-β-ocimene and (Z)-β-ocimene were more selectively distributed. Overall, the results highlight Moscatella Nera as the variety with the richest and most abundant varietal volatile profile, while genotypes such as Terribile and Barbarossa Etna exhibited a more limited volatile composition, confirming the previously observed results.

3.3. Sensory Analysis

The results of the qualitative descriptive sensory analysis of the wine aroma profiles are presented in Figure 5. The radar plot highlighted a clear differentiation in the sensory aroma profiles among the old-vine genotypes and the three reference wine samples, revealing distinct characteristics associated with each wine.

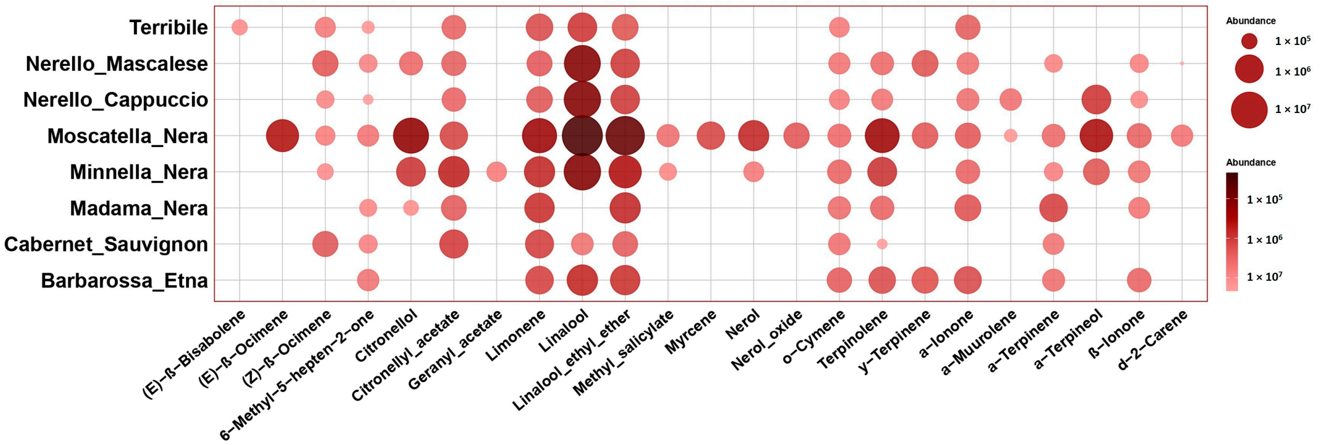

Figure 4. Bubble plot showing the abundance of varietal volatile compounds characterizing the red berry genotypes across the grapevine genotypes under study. Each bubble represents the abundance of a given compound in a specific variety, with both size and color intensity proportional to abundance on a log10 scale, ranging from 1×10^5 (light pink, small bubbles) to 1×10^7 (dark red, large bubbles). Absent or non-detected compounds are represented by the absence of a bubble.

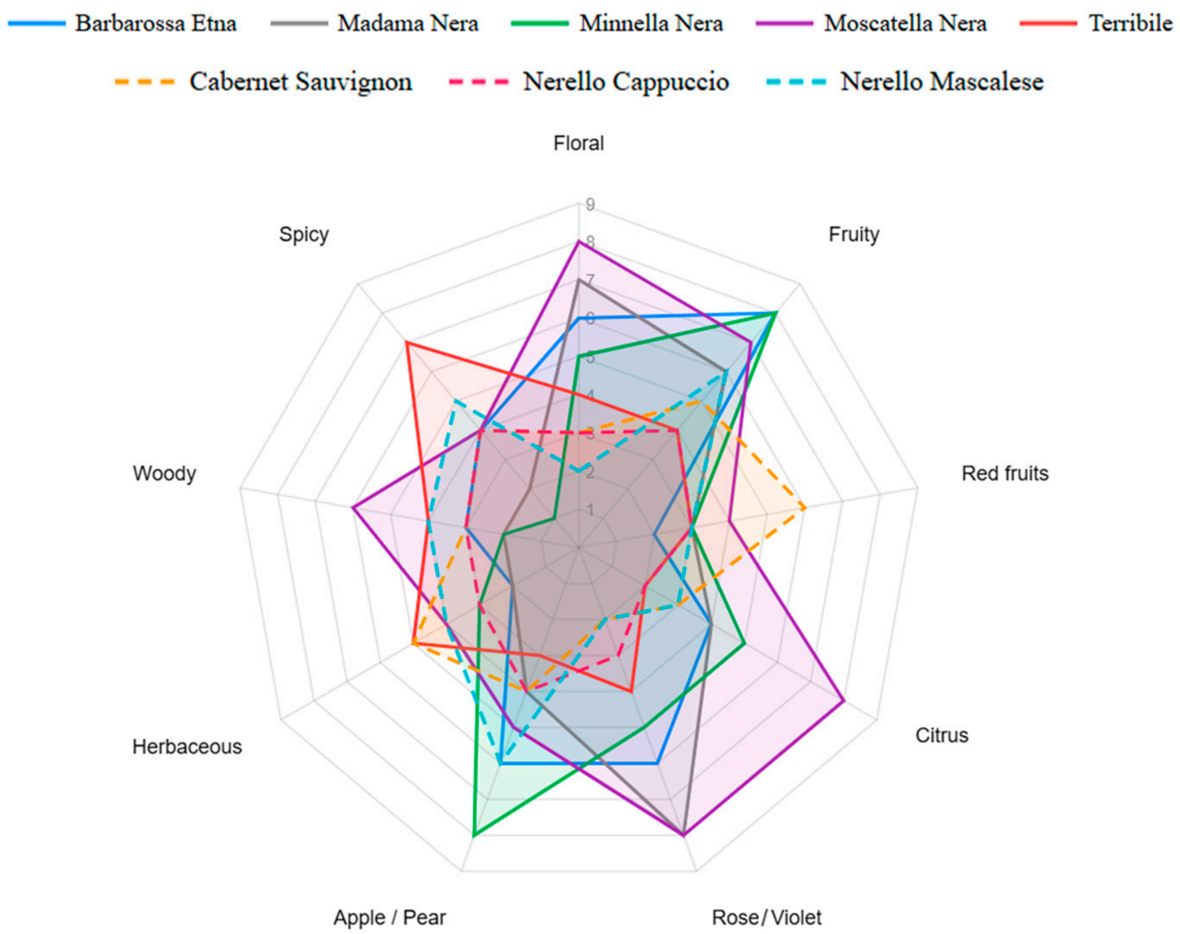

Figure 5. Graphical representation of the sensory aroma profile of wine samples from old-vine genotypes (Barbarossa Etna, Madama Nera, Minnella Nera, Moscatella Nera, and Terribile) and reference wine samples (Cabernet Sauvignon, Nerello Cappuccio, and Nerello Mascalese).

Moscatella Nera showed a well-defined and distinct aromatic profile, achieving the highest scores for floral, rose/violet, citrus, and red fruit odor descriptors. This finding is directly supported by the GC-MS data, which showed a total terpene content ten-fold greater than any other variety. In fact, in Moscatella Nera, the terpene fraction was dominated by linalool ethyl ether (93.31 μg/L), linalool (17.30 μg/L), citronellol (30.63 μg/L), limonene (23.02 μg/L), α-terpineol (14.57 μg/L), and nerol (7.34 μg/L); this monoterpene fingerprint is qualitatively and quantitatively consistent with the muscat aromatic profile and constitutes the core terpene signature responsible for floral and citrus sensory character [55,56], while β-ionone (1.70 μg/L) with an extraordinarily low odor threshold strongly contributes to the high rose/violet odor [57]. This wine was also characterized by a red fruit aroma potentially related to the higher presence of ethyl butanoate and ethyl 2-methylbutanoate.

Barbarossa Etna received the highest fruity score, a finding directly attributable to its isoamyl acetate concentration (400 μg/L), which far exceeds the reported wine odor threshold of 30 μg/L and confirms the decisive olfactory role of this acetate ester in shaping the aromatic character of this genotype; the elevated fruity score of Barbarossa Etna is further supported by high ethyl octanoate (2680 μg/L, threshold ~5 μg/L) and the highest total C_{13}-norisoprenoids content across all samples (5.79 μg/L), with α-ionone (3.25 μg/L, rose and fruity) and β-ionone (1.54 μg/L, violet and floral) together accounting for its distinctive rose/violet odor.

Madama Nera, which exhibited the highest rose/violet sensory score, was characterized by the highest concentration of 2-phenylethyl acetate (75.93 μg/L), a compound widely recognized as a major contributor to rose-like and floral-honey aroma notes in wine [58,59].

Minnella Nera's high apple/pear and fruity scores are explained by its exceptionally elevated ethyl hexanoate content (1333 μg/L), well above the 14–420 μg/L wine odor threshold of this medium-chain fatty acid ethyl ester.

In contrast, Terribile exhibited the least intense fruity profile, aligning with its GC-MS profile, which revealed the lowest total ester concentration (3228 μg/L). Conversely, its prominent spicy aroma can be attributed to the exclusive presence of (E)-β-bisabolene, a sesquiterpene responsible for the balsamic, woody, and spicy odor.

The woody aroma descriptor received generally low sensory scores across all wines, except for Moscatella Nera and Terribile, which exhibited a more pronounced woody character. This sensory perception appears to be associated with the higher concentration of specific terpenes, particularly myrcene, α-terpinene, and β-ocimene, compounds known to contribute to woody aroma.

Compared with the reference wines (Nerello Cappuccio, Nerello Mascalese, and Cabernet Sauvignon), the sensory profile of the old-vine genotype wines was characterized by a pronounced expression of floral and fruity notes. The only exception was red fruit attribute, which was stronger in Cabernet Sauvignon.

Taken together, the integration of trained sensory panel data with GC–MS volatile compound analysis demonstrates that the observed sensory differences clearly define the enological identity of these old-vine genotypes. These findings further support their potential as valuable sources of sensory typicity and aromatic diversity within Sicilian viticulture.

4. Limitations

The study successfully characterized the aromatic profile of wines produced from old-vine genotypes grown in the Etna area. However, the study has some limitations, which will be the subject of future research.

First, the experimental observations were obtained from a single harvest season; therefore, climatic variability may have influenced grape composition and volatile expression.

Multi-year studies will be necessary to evaluate the stability of the identified varietal fingerprints.

Moreover, although standardized microvinification protocols were adopted to minimize technological variability, small-scale fermentations may not completely reproduce commercial winemaking conditions. Consequently, the observed aromatic profiles should be interpreted as representative of the intrinsic varietal potential rather than definitive commercial wine styles.

In addition, all cultivars were grown within the same viticultural area, which allowed a controlled comparison under shared pedoclimatic conditions but limited the possibility of disentangling genotype and terroir effects.

Finally, while the sensory analysis performed by a trained panel provided useful analytical information, further consumer-oriented studies would be valuable to assess the market perception and acceptance of wines produced from these old-vine genotypes.

Despite these limitations, the present work provides one of the first integrated physicochemical, volatile, and sensory characterizations of several underexplored Etna grapevine genotypes and highlights their potential contribution to viticultural biodiversity and terroir-driven wine differentiation.

5. Conclusions

This study provided the first comprehensive oenological characterization of five old-vine genotype wines from the Mount Etna area, comparing them with traditional (Nerello Mascalese and Nerello Cappuccio) and international (Cabernet Sauvignon) cultivars.

Our findings demonstrate that these minor genotypes possess a distinct and well-defined chemical and sensory identity. From a physicochemical perspective, they align with the standards of Etna DOC production, showing suitable alcohol potential and acidity levels, with particularly high polyphenol content in genotypes such as Madama Nera and Moscatella Nera.

The volatile profile analysis revealed that these old genotypes are not merely alternatives to dominant cultivars but represent unique aromatic fingerprints. In particular, Minnella Nera and Moscatella Nera showed a significant abundance of terpenes and norisoprenoids (such as α-ionone and β-ionone), contributing floral and citrus notes that are less pronounced in the reference cultivars. Their elevated terpene and norisoprenoid contents, together with a rich ester profile, suggest a strong potential for sensory complexity and niche market valorization.

Sensory analysis confirmed the chemical data, highlighting specific descriptors, such as floral, red berry, and spicy notes, that allow these wines to be clearly distinguished from both Cabernet Sauvignon and the local cultivars Nerello Mascalese and Nerello Cappuccio.

From a broader perspective, these results reinforce the importance of conserving viticultural biodiversity and integrating old cultivars into quality production chains, particularly within high-value appellations such as Etna.

The data presented here provide a robust chemical foundation supporting the oenological relevance of these genetic resources, in line with recent calls for terroir-driven authenticity in contemporary wine markets.

In conclusion, the recovery and promotion of these old-vine genotypes offer a strategic opportunity for the Sicilian wine industry. Their reintroduction could enhance biodiversity and preserve a unique genetic heritage at risk of extinction, strengthening the terroir-driven marketing strategies and providing consumers with distinctive and identifiable products. Moreover, these autochthonous genotypes have historically adapted to the specific semi-arid and volcanic conditions of Mount Etna, supporting climate change adaptation.

Future research should focus on multi-vintage trials to assess the stability of these aromatic profiles and explore the potential of blending these minor genotypes with traditional cultivars to further innovate the identity of Etna DOC wines.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/beverages12070079/s1, Figure S1: Weather data (temperatures, rainfall, and daily vapor pressure deficit (VPD)) relating to the area where the experimental vineyard is located and the period under study. Table S1: Aroma volatile compound composition for each class of substances in wines from old-vine genotypes and reference wines from Mount Etna. Table S2: QDA aroma sensory data of old-vine genotype and reference wines from Mount Etna.

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Institutional Review Board Statement: This manuscript reports on a sensory analysis conducted on wine samples with the participation of students and staff from the Department of Veterinary Sciences, University of Messina, following the requirements for sensory testing as described in ISO 8586:2023 [34]. In line with current regulations on ethics in food science research, informed consent was obtained from all participants, with full assurance of data protection and privacy. The activity did not involve any novel foods, health claims, or human experimentation. Therefore, in line with applicable national regulations on data protection and the ethical guidance for food-related research issued by the European Commission and in accordance with the Code of Ethics of the World Medical Association (Declaration of Helsinki) for research involving human subjects, ethical approval by an institutional review board or ethics committee was not required.

Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

Data Availability Statement: All relevant data will be shared upon request.

Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

DOC	Controlled Designation of Origin
VSP	Vertical Shoot Positioning
IPM	Integrated Pest Management
IRVO	Regional Institute of Wine and Oil
OIV	International Organization of Vine and Wine
HS-SPME	Headspace Solid-Phase Microextraction
GC-MS	Gas Chromatography–Mass Spectrometry
DVB/CAR/PDMS	Divinylbenzene/Carboxen/Polydimethylsiloxane
QDA	Quantitative Descriptive Analysis
ISO	International Organization for Standardization
VOCs	Volatile Organic Compounds
PCA	Principal Component Analysis

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