Comparison of fortified, sfursat, and passito wines produced from fresh and dehydrated grapes of aromatic black cv. Moscato nero (Vitis vinifera L.)

Comparison of *fortified*, *sfursat,* and *passito* wines produced from fresh and dehydrated grapes of aromatic black cv. Moscato nero (*Vitis vinifera* L.)

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ABSTRACT

Moscato nero d'Acqui is an Italian aromatic black winegrape variety characterized by a low content of anthocyanins (mostly tri-substituted), a satisfactory content of high molecular mass tannins, and a fair amount of terpenes. The grapes were subjected to a postharvest dehydration process under controlled thermohygrometric conditions (16-18 °C, 55-70 RH%, 0.6 m/s air speed). with the aim to produce three different special wine types (*fortified, sfursat,* and *passito*) from fresh, partially dehydrated (27 °Brix), and withered (36 °Brix) grapes, respectively. Chemical traits of produced grapes and wines were then evaluated through spectrophotometric, HPLC, and GC-MS methods. Increased contents of skin phenolic compounds and reduced extractable contents of seed phenolic compounds were observed as dehydration progressed. Few significant differences were found in the anthocyanin profile of grapes, although the relative abundance of coumaroylated anthocyanins was higher in dehydrated grapes. The predominant free volatile compound found in grapes was geraniol, which decreased with increasing water loss, whereas the contents of major glycosylated volatile compounds increased even above the concentration effect. The changes in the phenolic composition among wines agreed with those among grape skins. *Fortified* wines were chromatically unsatisfactory probably due to the low content of total anthocyanins, whereas *sfursat* and *passito* wines meet good chromatic characteristics as a result of the concentration effect during grape dehydration. *Fortified* and *sfursat* wines had free aroma profiles richer in 2-phenylethanol and citronellol, whereas *passito* wines were mainly composed of 2-phenylethanol and 2-phenylethyl acetate, citronellol being the predominant terpenol in all the wine types studied.

Keywords: Moscato nero d'Acqui; Phenolic compounds; Free and bound volatile compounds; Dehydrated grapes; Special red wines.

1. INTRODUCTION

Nowadays, one of the key factors of the economical development of the viticulture and wine industry in specific limited geographical areas is the exploitation of ancient, local grape varieties for the development of oenological products with special features. In recent years, the growing interest to rediscover ancient and minor varieties has promoted many studies in order to retrain these realities (Maul et al., 2015; Urcan et al., 2016). This is particularly true in Italy, a country with a rich varietal endowment due to a centuries-old wine-growing tradition.

Vitis vinifera L. cv. Moscato nero d'Acqui is an ancient and local aromatic red grape variety, which could be found sporadically in old vineyards in the provinces of Asti and Alessandria, and takes its name from the town of Acqui, located at the center of this production zone (North-West Italy). The Moscato nero d'Acqui cultivar was described for the first time in 1875 (De Maria & Leardi 1875), while in 1971 it was included in the National register of vine varieties (Mipaaf). Nowadays, the spread of this variety is limited, but new vineyards are being planted in Tortona (South-East Piedmont; Raimondi, Valota, & Schneider, 2009) and the nursery production is active (Pecile, Zavaglia, & Ciardi, 2016). The Moscato nero d'Acqui cultivar can be called simply Moscato nero. It is not a synonym of Moscato d'Amburgo (Muscat Hamburg, one of the most known table grape varieties), even though the two cultivars belong to the large family of Muscat vines that have in common the characteristic Muscat flavor. Monoterpenes are responsible for the varietal aroma of Muscat cultivars (Selli, Canbas, Cabaroglu, Erten, & Gunata, 2006). In the past, Moscato nero d'Acqui grapes were consumed as table grapes, but the oenological potential was noted (Mannini et al. 2012). From the comparison among 34 *Vitis vinifera* genotypes emerges that the studied cultivar is characterized by low content of total anthocyanins, although the accumulation of these compounds is significantly year-dependent (Ferrandino, Carra, Rolle, Schneider & Schubert, 2012). The vine is known by low productivity and good vigor. The bunches are loose and usually resistant to rot, which makes them suitable to the dehydration process used for special (sweet) wines production.

During postharvest grape dehydration, important metabolic changes occur due to water loss and, in specific conditions, to the development of *Botrytis cinerea* (as noble rot). Water stress induces an active metabolism that affects the chemical composition and physical properties of grape berries (Rolle et al., 2013, Toffali et al. 2011), while *Botrytis cinerea*, when developing as noble rot, favors the release of aroma compounds from the berry skin (Genovese, Gambuti, Piombino, & Moio, 2007) and other skin modifications (Carbajal-Ida, Maury, Salas, Siret, & Mehinagic, 2016; Rolle et al., 2012). Therefore, the dehydration process promotes the release, concentration, synthesis, and oxidation of phenolic and volatile compounds (Bellincontro, De Santis, Botondi, Villa, & Mencarelli, 2004; Costantini, Bellincontro, De Santis, Botondi, & Mencarelli, 2006; Mencarelli et al., 2010), although the varietal aroma of winegrapes is particularly influenced in botrytized berries because of the mould-induced oxidation of monoterpenes (Câmara, Herbert, Marques, & Alves, 2004). In fact, the interaction between the level of *B. cinerea* infection and the degree of grape withering is of great relevance for modulating the aroma of *passito* wines (Tosi et al., 2013). Furthermore, the increased production of glycerol enhances the wine mouthfeel (Rolle et al., 2012; Vincenzi et al., 2012).

To gain knowledge on the enological potential of the Moscato nero d'Acqui variety, the two main aims of this work were: *i)* to investigate the phenolic and aromatic composition of fresh grapes and dehydrated grapes using two different postharvest treatment times, *ii)* to evaluate the aptitude to the production of special wines, particularly *fortified* (sweet wine from fresh grapes), *sfursat* (dry wine from partially dehydrated grapes), and *passito* (sweet wine from dehydrated grapes). *Fortified* wine belongs to the category of liqueur wines that are made from grape musts (including partially fermented grape musts) and/or wine, to which distillates, spirits and alcohol of vitivinicultural origin are added alone or in a mixture (OIV, ECO 2/2007). *Sfursat* and *passito* wines are produced

by using partially dehydrated and withered grapes, respectively. The knowledge of the enological potential and special features of Moscato nero d'Acqui would allow the better exploitation of this particular red aromatic variety enhancing the distinctive character of special wines.

2. MATERIALS AND METHODS

2.1. Grape and wine samples

Red grapes of *Vitis vinifera* L. cv. Moscato nero d'Acqui were harvested at ripeness (about 200 g/L must sugars concentration) in a vineyard located in Santo Stefano Belbo (Cuneo province, Piedmont, North-West Italy) in September 2013. Three sets of small clusters were selected (3 replicates for fresh grape sample). Six batches of 125 kg of grapes (3 replicates for each of the two withered grape samples) were placed in a single layer in plastic boxes (60 cm x 40 cm x 15 cm, with bottom holes of about 1.5 cm x 5 cm to improve air flow), in quantities of about 1.5 kg grapes for each box, and dehydrated in a thermohygrometrically controlled chamber (16-18 °C, 55-70% relative humidity, 0.6 m/s air speed). The dehydration process was carried out for 23 days for the production of *sfursat* wine (until reaching 27 °Brix, "partially dehydrated" grapes sample) and for 52 days for the production of *passito* wine (until reaching 36 °Brix, "withered" grapes sample).

Before the analysis of each sample, the randomly-taken berries were manually separated from the stalk and then used as follows: three replicates of 100 berries for the determination of technological ripeness parameters, three replicates of 10 berries for phenolic compound determination, and three replicates of 150 berries for volatile compound determination.

Micro-scale vinifications were made at the experimental winery of the University of Torino. For each type of wine (*fortified*, *sfursat*, and *passito)*, two replicates of about 100 kg of grapes each were processed. The vinification protocol was the same for all the six trials (2 replicates × 3 wines) but using fresh grapes for *fortified* wine, partially dehydrated grapes for *sfursat* wine, and withered grapes for *passito* wine. The grapes were destemmed and crushed, the mash was then placed into a fermenter saturated with CO_2, and 20 mg/L of sulfur dioxide were added. After about 6 hours, 20 g/hL of yeast (LSA ES181 previously activated, Esseco, Trecate, IT), and 8 g/hL of yeast nutrients (ammonium sulfate and ammonium phosphate) were added. Alcoholic fermentation was carried out at controlled temperature (22±2 °C) until reaching 5% v/v of ethanol. The pomace was pressed and the resulting must-wine continued the fermentation without the solid parts. In the case of *fortified* wine, the fermentation was stopped, when the residual sugar content in the fermenting must was about 100 g/L, with the addition of 100 mg/L of sulfur dioxide and 95% v/v food-grade ethanol up to a total alcohol content of about 13% v/v, and the resulting wine was stored at 10 °C for one month. In the case of *sfursat* wine, the fermentation progressed until less than 5 g/L of sugars were present and 50 mg/L of sulfur dioxide were added, while in *passito* wine the fermentation was stopped when the residual sugar content was about 110 g/L by adding 100 mg/L of sulfur dioxide. *Fortified*, *sfursat* and *passito* wines were stored at 0 ºC for 2 weeks, filtered (Seitz K300 grade filter sheets, Pall Corporation, Port Washington, NY, USA), and finally bottled.

2.2. Chemical analysis of grapes and wines

2.2.1 Reagents and standards

Solvents of HPLC-gradient grade and all other chemicals of analytical-reagent grade were purchased from Sigma-Aldrich (Milan, Italy). The solutions were prepared in deionized water produced by a Milli-Q system (Merck Millipore, Darmstadt, DE). Chemical standards of delphinidin-3-O-glucoside chloride, cyanidin-3-O-glucoside chloride, petunidin chloride, peonidin-3-O-glucoside chloride, malvidin-3-O-glucoside chloride, and cyanidin chloride were supplied by Extrasynthèse (Genay, France), whereas those of (+)-catechin, linalool oxide (mixture of isomers), linalool, α-terpineol, citral, citronellol, nerol, geraniol, rose oxide (mixture of isomers), geranyl acetate, geranic acid, benzyl alcohol, 2-phenylethanol, and 2-phenylethyl acetate were purchased from Sigma-Aldrich.

2.2.2 Standard parameters

In the grape musts resulting from manual grape crushing and centrifugation, total soluble solids content (°Brix) was measured using an Atago °Brix temperature compensating refractometer (Atago Corporation, Tokyo, Japan). In the grape musts and in the wines obtained after five months from bottling, reducing sugars (glucose and fructose, g/L) and organic acids (tartaric acid and malic acid, g/L) were determined by high performance liquid chromatography (HPLC) (Agilent Technologies, Santa Clara, CA, USA) using a refractive index detector and a diode array detector (DAD) set to 210 nm (Giordano, Rolle, Zeppa, & Gerbi, 2009). Citric acid (g/L) in grape musts, and glycerol (g/L) and ethanol (% v/v) in wines were determined following the same HPLC methodology. pH was determined by potentiometry using an InoLab 730 pH meter (WTW, Weilheim, Germany), and titratable acidity (g/L tartaric acid) and volatile acidity (g/L acetic acid) were estimated according to the International Organization of Vine and Wine methods (OIV, 2008). Gluconic acid (g/L) in grape musts was determined using an enzymatic kit (R-Biopharm Italia, Cerro al Lambro, MI, Italy) and a UV-1800 spectrophotometer (Shimazdu Corporation, Kyoto, Japan).

2.2.3 Extraction and determination of phenolic compounds

The grape berries were weighed, and the skins and seeds were manually separated from the pulp using a laboratory spatula. The berry skins were quickly put into 25 mL of a hydroalcoholic buffer solution of pH 3.2 composed of 5 g/L of tartaric acid, 2 g/L of sodium metabisulfite, and 12% v/v of ethanol (Torchio, Cagnasso, Gerbi, & Rolle, 2010). The skins were homogenized for 1 minute at 8000 rpm with an Ultra-Turrax T25 high-speed homogenizer (IKA Labortechnik, Staufen, Germany) and subsequently centrifuged in a PK 131 centrifuge (ALC International, MI, Italy) for 15 minutes at 3000 x g and 20 °C. The supernatant was then used for skin analysis. The berry seeds were put into 10 mL of the same buffer solution used for the skins and then placed in an oven at 30 °C for 1 week (Torchio et al., 2010). Afterwards, the seeds were discarded and the solution was then used for seed analysis.

Spectrophotometric methods were used to determine total anthocyanins (mg malvidin-3-O-glucoside chloride/kg grape or L wine, as TA), absorbance at 280 nm (as A_{280}/kg grape or L wine), total flavonoids [mg (+)-catechin/kg grape or L wine, as TF], flavanols reactive to vanillin [mg (+)-catechin/kg grape or L wine, as FRV], and proanthocyanidins (mg cyanidin chloride/kg grape or L wine, as PRO) (Torchio et al., 2010). A UV-1800 spectrophotometer (Shimazdu Corporation) was used.

The anthocyanin profile of berry skins and wines was determined by HPLC-DAD following the protocol described by Rolle, Torchio, Giacosa, & Río Segade (2015). The skin extracts or the wines were previously diluted with 0.05 mol/L of sulfuric acid to have ethanol contents less than 4% v/v and submitted to reverse-phase solid-phase extraction using a 1 g Sep-Pak C18 cartridge (Waters Corporation, Milford, MA, USA). Anthocyanin compounds were recovered with methanol. The HPLC-DAD system and chromatographic conditions were those reported by Rolle et al. (2015). The chromatographic separation was performed on a LiChroCART column (250 mm × 4 mm i.d.) (Merck, Darmstadt, Germany), which was packed with LiChrospher 100 RP-18 (5 μm) particles (Alltech, Deerfield, IL, USA), using two mobile phases (A, formic acid/water, 10:90 v/v; B, formic acid/methanol/water, 10:50:40 v/v/v) at a flow rate of 1 mL/min. After identification at 520 nm, the amounts of individual anthocyanins were expressed as percentages.

2.2.4 Wine color parameters

The wine color was evaluated by the color intensity, color hue, and CIELab parameters including lightness (L*), red/green color coordinate (a*), and yellow/blue color coordinate (b*) according to the methods proposed by OIV (2008). A UV-1800 spectrophotometer (Shimazdu Corporation) was used with a 2-mm path length cuvette.

2.2.5 Extraction and determination of volatile compounds

The grape berries were treated following the method reported by Rolle et al. (2015). The berries were crushed under a nitrogen atmosphere with a laboratory blender (Waring Laboratory, Torrington, USA) (1 min for fresh berries, 2 min for partially dehydrated and withered berries) and then centrifuged (7000 x g, 15 min, 4°C). For the determination of free volatile compounds, a 5 mL-aliquot of the supernatant was diluted with 5 mL of deionized water, adjusted at pH 5, and placed into a 20 mL glass headspace sampling vial containing 2 g of sodium chloride and 200 µL of a 1-heptanol solution (1.55 mg/L in 10% v/v ethanol) as internal standard (Rolle et al., 2015). In the case of wines, the same treatment was carried out replacing the supernatant by 5 mL of the wine sample previously diluted twice with a 0.2 mol/L citrate-phosphate buffer solution of pH 5.

For the determination of glycosylated volatile compounds, the method reported by Wang, Kang, Xu, & Li (2011) was slightly modified. Briefly, 10 mL of the supernatant or 10 mL of the wine were diluted with 10 mL of deionized water and then submitted to reverse-phase solid phase extraction using a 1 g Sep-Pak C18 cartridge (Waters Corporation). The free volatile compounds were released with 10 mL of dichloromethane, while the glycosylated volatile compounds were recovered with 10 mL of methanol and transferred to an evaporating flask. Subsequently, methanol was evaporated using a vacuum rotavapor (BÜCHI R-210, BÜCHI Labortechnik AG, Flawil, CH) at 35 °C and the residue obtained was re-dissolved in 10 mL of the buffer solution of pH 5. The enzymatic hydrolysis was carried out by adding 50 mg of an AR-2000 commercial preparation with β-glycosidase activity (DSM Oenology, Heerlen, NL) and 0.1 g of polyvinylpolypyrrolidone (PVPP) to avoid the inhibitory effect of tannins, heating at 40 °C for 24 h. Finally, the extract was placed into a 20 mL glass headspace sampling vial containing 2 g of sodium chloride and 200 µL of 1-heptanol internal standard solution.

The determination of free and glycosylated volatile compounds was separately carried out using head space solid phase microextraction coupled with gas chromatography-mass spectrometry (HS-SPME-GC-MS). A 50/30 µm DVB/CAR/PDMS (divinylbenzene-carboxen-polydimethylsiloxane) fibre from Supelco (Bellefonte, PA, USA) was exposed to the headspace of the capped vial for 20 min at 40 ºC (Sánchez-Palomo, Díaz-Maroto, & Pérez-Coello, 2005) and the thermal desorption was performed at 250 ºC for 5 min. An Agilent 7890C gas chromatograph (Little Falls, DE, USA) equipped with a DB-WAXETR capillary column (30 m × 0.25 mm, 0.25 µm, J&W Scientific Inc., Folsom, CA, USA) and coupled to an Agilent 5975 mass selective detector was used. The chromatographic and MS conditions were previously reported (Sánchez-Palomo et al., 2005). Identification was done according to retention indices previously reported by Urcan et al. (2017) as well as to mass spectra of pure standards and/or to the NIST database (http://webbook.nist.gov/chemistry/). Semi-quantitative determinations (µg/kg berries or µg/L wine) were performed using the internal standard method (Englezos et al., 2016).

2.3. Statistical analysis

Statistical analyses were carried out using the SPSS Statistics software package, version 19.0 (IBM Corporation, Armonk, NY, USA). The Tukey-b test for $p<0.05$ was used to assess significant differences by one-way analysis of variance (ANOVA).

3. RESULTS AND DISCUSSION

3.1. Standard parameters of fresh and dehydrated grapes

The chemical parameters for Moscato nero d'Acqui fresh grapes, partially dehydrated grapes (27 °Brix), and withered grapes (36 °Brix) are shown in Table 1. During the postharvest dehydration process, the berry weight decreased due to water loss and the grape must components,

such as reducing sugars and organic acids, were concentrated with the exception of tartaric acid. In fact, the previously reported decrease of malic acid as a consequence of gluconeogenesis and increased respiration (Centioni, Tiberi, Pietromarchi, Bellincontro, & Mencarelli, 2014) was compensated in partially dehydrated berries and even overcome in withered grapes by the concentration effect. Nevertheless, the berries are metabolically reactive to water stress and so chemical modifications also occur. In addition, it is important to consider that the presence of mycelial mass (*Botrytis cinerea* and other moulds) affects the content of different grape metabolites such as organic acids and sugars (Lorenzini, Azzolini, Tosi, & Zapparoli, 2012). In botrytized grapes, glucose oxidase enzymes oxidize glucose to gluconic acid. Furthermore, the hydrogen peroxide released in the above reaction forms, via a Fenton reaction, a scavenger able to oxidize tartaric acid to glyoxylic acid (Vivas et al., 2010). Therefore, in withered grapes, the glucose/fructose ratio and tartaric acid content decreased significantly, whereas the gluconic acid content and pH value increased in relation to fresh grapes. During dehydration, the trend of titratable acidity was quite similar to that of tartaric acid content in agreement with the findings of Rolle et al. (2013), although the differences were less significant for titratable acidity.

3.2. Grapes phenolic composition

The grape phenolic composition gives important information on the potential of Moscato nero d'Acqui cultivar (Table 2). The grapes are characterized by a satisfactory concentration of tannins, whereas the anthocyanin content is somewhat low. Both skins and seeds showed a quite low FRV/PRO ratio, which means that the grape tannins of Moscato nero d'Acqui are highly polymerized, and therefore low bitter and more astringent wines could be obtained (Cheynier et al., 2006; Peleg, Gacon, Schlich, & Noble, 1999).

The evolution of phenolic compounds for the skins and seeds during the grape dehydration process was different (Table 2). For the skins, an increase of the A_{280} value, which is usually used as a fast index for total phenolic compounds, was observed with increasing water loss. Furthermore, TA, TF, FRV, and PRO were more abundant in withered grapes, followed by partially dehydrated grapes, in relation to fresh grapes as a consequence of the concentration effect (Centioni et al., 2014; Moreno et al., 2008). A production factor (PF; Ruiz, Zea, Moyano, & Medina, 2010) was calculated by dividing the average content of phenolic compounds in partially dehydrated and withered grapes between their content in fresh grapes (1.3 and 1.7, respectively). Considering a 20% of error for PF resulting from the concentration effect by water evaporation (Noguerol-Pato, González-Álvarez, González-Barreiro, Cancho-Grande, & Simal-Gándara, 2013), synthesis or degradation reactions did not occur during the postharvest dehydration process of Moscato nero d'Acqui grapes. Mencarelli et al. (2010) have demonstrated that the temperature is of great importance to avoid anthocyanin oxidation induced by the activity of polyphenol oxidase and peroxidase enzymes during grape dehydration. Temperatures between 10 and 20 ºC were required for reaching higher anthocyanin contents in Aleatico grapes dehydrated from 10 to 30% weight loss (WL) when compared with fresh grapes. The dehydration temperature used in the present study (16-18 ºC) reduced the risk of anthocyanin oxidation in Moscato nero d'Acqui grapes because the increase underwent in the anthocyanin content corresponds to the concentration effect by water loss. Nevertheless, other researchers found no significant change in Raboso Piave grapes dehydrated from 10 to 30% WL and in Nebbiolo grapes dehydrated at 20% WL even at 20 ºC (Bonghi et al., 2012; Nicoletti et al., 2013), but an increase was observed in Nebbiolo grapes dehydrated at 10 ºC (Nicoletti et al., 2013) in relation to fresh berries. Using different dehydration rates, Bonghi et al. (2012) showed that key genes involved in anthocyanin biosynthesis were unaffected or down-regulated, whereas Mencarelli et al. (2010) reported that they were up-regulated at 10 and 20 ºC.

Regarding skin flavanols, contradictory results were found for different cultivars. Increased catechin content was observed during the dehydration of Aleatico grapes at 10 and 20 ºC (Mencarelli et al., 2010), whereas some authors pointed out a low molecular mass flavanol decrease

in Raboso Piave and Corvina berries (Bonghi et al., 2012; Rolle et al., 2013) probably related to oxidative reactions by increased activity of polyphenol oxidase and laccase enzymes. However, Moreno et al. (2008) observed no significant effect on skin proanthocyanidin content in Pinot noir grapes. These discrepancies seem to be mainly associated with a genotype effect on the content and profile of skin phenolic compounds (Torchio et al., 2016). In the present study, the higher FRV/PRO ratio obtained at the greater dehydration degree showed that the increase of skin low polymerized flavanols (FRV) was higher than that of high molecular mass flavanols (PRO) during grape dehydration. This agreed with the decrease of the skin proanthocyanidin average degree of polymerization previously reported by other researchers (Moreno et al., 2008).

For the seeds, the A_{280} index and the TF, FRV, and PRO contents decreased as dehydration progressed. This trend differed from that reported for Pinot noir grapes (Moreno et al., 2008) where the PRO content in the seeds increased significantly during dehydration, and for Corvina (Rolle et al., 2013) where TF, FRV, and PRO contents also increased using similar thermohygrometric conditions to the present study. The PRO content also increased progressively during the dehydration of Cesanese grapes (Centioni et al., 2014). Nevertheless, Torchio et al. (2016) reported a significantly lower PRO content in Avanà seeds from grapes dehydrated using slow and fast processes than that found in fresh grapes, although the FRV content was similar. Río Segade et al. (2016) also pointed out that the lowest extractable FRV and PRO contents of seeds from Nebbiolo grapes were found at 30 and 45% WL when the results were expressed on a seed weight basis. This decrease could be justified by the consistence of the changes underwent in the seeds during grape dehydration with those observed from extended ripening, such as intensive lignification of the medium integument and the dehydration of the outer integument, which could prevent increasingly flavanols from being extracted (Bautista-Ortín et al., 2012; Cadot, Miñana-Castelló, & Chevalier, 2006). Furthermore, the degradation of low molecular mass flavanols and the hydrolysis of larger oligomers could occur depending on genotype and dehydration conditions, these reactions overcoming the concentration effect due to water loss (Rolle et al., 2013). The FRV/PRO ratio of the seeds decreased when dehydration progressed, as previously observed in Corvina grapes (Rolle et al., 2013), but in disagreement with lowering seed proanthocyanidin average degree of polymerization reported for other cultivars (Moreno et al., 2008; Río Segade et al., 2016). It is important to take into account that the contribution of seed flavanols decreased with increasing the dehydration level, which might affect the sensorial properties of resulting wines, particularly astringency and bitterness.

The anthocyanin profile of Moscato nero d'Acqui skins from fresh and dehydrated grapes is characterized by a prevalence of tri-substituted anthocyanins (Table 3), particularly malvidin-3-O-glucoside and its derivatives accounted for about 43-47% of total anthocyanin forms. A high percentage of peonidin-3-O-glucoside derivatives (about 28% of total forms) was also found in all grape samples. On the other hand, the relative abundance of acylated anthocyanins was low (6.6-8.0%). Some significant differences were found in the percentages of coumaroylated anthocyanins among fresh, partially dehydrated, and withered grapes, which increased as dehydration progressed in agreement with a previous study performed at 10 and 20 ºC up to 30% WL (Mencarelli et al., 2010). Nicoletti et al. (2013) hypothesized that anthocyanin acylation increases in response to berry thermal stress to stabilize these red pigments.

3.3. Grapes aroma composition

In Moscato nero d'Acqui grapes, fifteen free and sixteen glycosylated volatile compounds were identified and quantified (Table 4). The aroma composition of fresh, partially dehydrated, and withered grapes is essentially characterized by a high prevalence of geraniol in both free and glycosidically-bound forms (80.8-88.0% of total free volatile compounds and 77.8-79.4% of total glycosylated volatile compounds). Regarding free volatile compounds, the second most important contribution in fresh grapes is given by nerol representing 6.8%, followed by citral and linalool.

Terpenes are responsible for the characteristic Muscat aroma (Selli et al., 2006), linalool, geraniol, and nerol being also the major free monoterpenes in Muscat cultivars, such as Muscat Hamburg, Moscatuel, and Bimeijia (Fenoll, Martinez, Hellin, & Flores, 2012; Rolle et al., 2015; Yang, Wang, Wu, Fang, & Li, 2011). The contents found of free geranic acid, *cis*-furan-linalool oxide, and *cis*-pyran-linalool oxide increased significantly as dehydration progressed, whereas geraniol decreased. Furthermore, a PF of 3 for geranic acid and *cis*-furan-linalool oxide in withered grapes, and 3.9 and 6.9 for *cis*-pyran-linalool oxide in partially dehydrated and withered grapes, respectively, which were greater than those expected by the effect of water evaporation ($1.0 < PF < 1.6$ for partially dehydrated grapes, $1.4 < PF < 2.1$ for withered grapes), suggest that synthesis reactions occurred during dehydration. Linalool oxides are originated from the oxidation of linalool and also from the metabolic activity of *Botrytis cinerea* (Bock, Benda, & Schreier, 1986; Rapp & Marais, 1993). A study performed on non-aromatic Erbaluce white cultivar showed a significant decrease of the geraniol content in botrytized grapes (Rolle et al., 2012). Thereby the second most abundant free volatile compound in partially dehydrated grapes was nerol accounting for 4.0%, followed by *cis*-pyran-linalool oxide (3.7%), whereas the content of this free linalool oxide (7.2%) was higher than that of nerol (5.6%) in withered grapes. The total content of free volatile compounds in partially dehydrated and withered grapes (260 and 239 µg/kg, respectively) was lower than that of fresh berries (430 µg/kg) mainly due to the decrease of geraniol.

The total content of glycosidically-bound volatile compounds was higher than that of free forms and increased during the postharvest dehydration process from 2452 µg/kg in fresh grapes to 4019 µg/kg and 4761 µg/kg in partially dehydrated and withered grapes, respectively. Various glycosylated volatile compounds increased progressively during dehydration, such as linalool, citral, citronellol, nerol, geraniol, and benzyl alcohol, whereas a significantly higher content of geranic acid was only found in partially dehydrated grapes in relation to fresh berries. The increase of citronellol and geranic acid contents exceeded the expected by water loss in partially dehydrated ($PF = 2.4$ and 9.1, respectively) and withered ($PF = 4.3$ and 3.2, respectively) grapes, as well as that of nerol only in partially dehydrated grapes ($PF = 1.7$), showing synthesis reactions or easier release of these compounds from the berry skin in relation to fresh berries. On the other hand, the content of glycosylated hotrienol decreased ($PF < 0.3$) indicating that it was degraded or transformed into other compounds during postharvest dehydration. As occurred for free volatile compounds, the second most important contribution of glycosylated volatiles in fresh grapes corresponded to nerol representing 12.2%, followed by citral and linalool. Nerol continued to be the second most abundant glycosylated volatile compound in partially dehydrated and withered grapes (12.9 and 11.7%, respectively), followed by geranic acid in partially dehydrated grapes but citronellol and citral in withered grapes.

3.4. Standard parameters of fortified, sfursat, and passito wines

The compositional differences among fresh, partially dehydrated, and withered grapes, and winemaking affected wine standard characteristics (Table 5). Residual sugar and ethanol contents, as well as the glucose/fructose ratio, were directly influenced by the vinification protocol because fermentation was almost complete only in *sfursat* wines (Table 5). Also, a significantly higher content of glycerol was found in *sfursat* and *passito* wines probably due to the development of *Botrytis cinerea* during postharvest grape dehydration (Rolle et al., 2012).

3.5. Wines phenolic composition and chromatic characteristics

Significant differences were found in the phenolic composition of the three types of Moscato nero d'Acqui wines (Table 5). The A_{280} value and TA, TF, FRV, and PRO contents were lower in *fortified* wines than in *sfursat* and *passito* wines and, in turn, *sfursat* wines showed a lower richness in phenolic compounds than *passito* wines. It is important to take into account that this trend agreed

with that observed in skins of fresh, partially dehydrated, and withered grapes as consequence of the concentration effect by water loss. Other researchers also pointed out an increased content of phenolic compounds in red naturally sweet wines in relation to red sweet fortified wines made from Garnacha tintorera grapes as consequence of water evaporation during dehydration (Figueiredo-González, Cancho-Grande, & Simal-Gándara, 2013). Furthermore, skin phenolic compounds diffuse to the pulp during postharvest dehydration because of cell wall degradation and cells disruption (Marquez, Serratosa, Lopez-Toledano, & Merida, 2012), facilitating their extraction and leading to more red-colored wines. According to the FRV/PRO ratio, *fortified* wines contained a significantly higher proportion of highly polymerized proanthocyanidins, whereas *sfursat* wines were richer in monomeric and oligomeric flavanols. This disagrees with the slightly lower value of proanthocyanidin average degree of polymerization found for red sweet fortified wines than for red naturally sweet wines (Figueiredo-González, Regueiro, Cancho-Grande, & Simal-Gándara, 2014). Regarding the anthocyanin profile of Moscato nero d'Acqui wines (Table 3), if compared to that of the grapes there are some differences. Indeed, during the first days of fermentation, the diffusion in the grape-juice of di-substituted anthocyanins is higher than the diffusion of tri-substituted forms (González-Neves, Gil, & Barreiro, 2008), cyanidin being particularly oxidable and its concentration decreases rapidly. Instead the wines showed high percentages of malvidin, which is less prone to oxidation (Cheynier, Souquet, Kontek, & Moutounet, 1994). Malvidin-3-O-glucoside was the predominant anthocyanin form in all the wines obtained with percentages ranging from 70.6 to 74.7% of total anthocyanins. Among all of the three wines studied, significant differences were observed for non-acylated forms. The lower percentage of malvidin-3-O-glucoside corresponded to *passito* wines. Instead, the lower relative abundances of peonidin-3-O-glucoside and cyanidin-3-O-glucoside were found for *fortified* wines, which suggest that lower losses of di-substituted anthocyanins occurred during winemaking of dehydrated grapes in relation to fresh grapes.

As can be seen from the chromatic characteristics (Table 5), *sfursat* and *passito* wines were darker according to the significantly lower values of L* and higher color intensity, when compared with *fortified* wines. Furthermore, the two first wines showed significantly lower values of color hue and higher values of a* and b* coordinates, which indicate that they exhibited more reddish hue with higher red and yellow color components. In fact, the chromatic characteristics of *fortified* wines were not satisfactory. The overall colorimetric differences among the wines made from fresh grapes (*fortified* wines) and those made from dehydrated grapes (*sfursat* and *passito* wines) were over the perceptibility threshold because of the high values obtained of ΔE* parameter (45.0 and 49.0). This parameter was calculated from the average values of L*, a*, and b* coordinates (OIV, 2008). However, the difference among *sfursat* and *passito* wines was hardly perceptible by the human eye (ΔE* parameter = 4.6; Gonnet, 2001; Torchio, Río Segade, Gerbi, Cagnasso, & Rolle, 2011).

3.6. Wines aroma composition

The aroma profile of the three wines studied is shown in Table 6. A total of 18 free and 16 glycosylated volatile compounds were identified and quantified because geranyl acetate and 2-phenylethyl acetate were not detected in glycosylated form. Regarding free volatile compounds, 2-phenyl ethanol was the predominant compound in *fortified*, *sfursat*, and *passito* wines, representing 75.5, 81.3, and 68.0% of total free volatile compounds, respectively. This aromatic alcohol is mainly formed from 2-phenylalanine during alcoholic fermentation. Its significantly higher content in *sfursat* and *passito* wines than in *fortified* wines suggested that the formation of 2-phenyl ethanol is related to the accumulation of amino acids during grape postharvest dehydration (Noguerol-Pato et al., 2013). Nevertheless, free 2-phenyl ethanol cannot contribute actively to the aroma of the wines as a consequence of its high olfactory threshold (14'000 μg/L) as also occurred in Garnacha tintorera naturally sweet wine and sweet fortified wine (Noguerol-Pato, González-Álvarez,

González-Barreiro, Cancho-Grande, & Simal-Gándara, 2012). Furthermore, 2-phenyl ethanol prevailed also in dry red Amarone wines produced from withered grapes (Tosi et al., 2012).

Other major free volatile compounds in all the three Moscato nero d'Acqui wines were citronellol (the second most abundant compound in *fortified* and *sfursat* wines), linalool, and 2-phenylethyl acetate (the second most abundant compound in *passito* wines), the two terpenols being significantly more abundant in *sfursat* wines but the acetate was in *passito* wines. Free citronellol and linalool contents were above their odor threshold (40 and 6 µg/L, respectively) and they were active odorants providing pleasant nuances of fruit and flowers. Instead, free 2-phenylethyl acetate was a key odorant (odor threshold of 250 µg/L) only in *passito* wines, giving floral notes.

Another free volatile compound found in concentrations above its odor threshold (0.5 µg/L) was rose oxide. *Passito* wines were significantly richer in both *trans* and *cis* isomers, and in turn *sfursat* wines showed *trans*-rose oxide contents higher than *fortified* wines. This monoterpenoid might be considered a marker of characteristic Muscat aroma (Ruiz-García, Hellín, Flores, & Fenoll, 2014), but its abundance in wines is influenced by the yeast metabolism (Koslitz, Renaud, Kohler, & Wüst, 2008). Free geranyl acetate also contributed to the final aroma of *fortified* wines (odor threshold of 9 µg/L) with floral nuances, although significant differences were not found in its content among the three wines studied. Contrarily, *trans*-furan-linalool oxide was significantly more abundant in *fortified* wines and benzyl alcohol was in *passito* wines, but the two free volatile compounds did not contribute actively to the wine aroma. Tosi et al. (2012) reported a significant increase of benzyl alcohol in wines made from botrytized grapes probably due to fungal enzymatic activity. It is important to take into account that higher total contents of terpenes were obtained for the wines made from dehydrated grapes (*sfursat* and *passito* wines). The enzymatic hydrolysis of aroma precursors by ß-glucosidases and chemical oxidation reactions, which are favored by mould grape infection, can modify the terpene content (Tosi et al., 2012).

The predominant glycosylated volatile compound in the three Moscato nero d'Acqui wines was geraniol (75.2-76.2%), followed by nerol (14.8-15.6%). The profile of glycosylated volatile compounds in *fortified*, *sfursat*, and *passito* wines was quite similar to that found in fresh, partially dehydrated, and withered grapes, respectively. Nevertheless, many significant differences were observed. The contents of linalool, citronellol, nerol, and geraniol increased progressively and significantly from *fortified* wines to *sfursat* and *passito* wines according to the dehydration level of the grapes used for their production and in agreement with the trend already observed in grapes (Table 4). *Fortified* wines showed citral contents significantly lower than *sfursat* and *passito* wines but higher contents of geranic acid.

4. CONCLUSIONS

On the basis of the results obtained in the present study, it can be stated that the Moscato nero d'Acqui variety has a good potential for the production of *sfursat* and *passito* wines. The positive relation between grape dehydration and wine composition (by means of phenolic and aroma traits) showed the impact of water evaporation on the quality of Moscato nero d'Acqui wine obtained. In particular, the highest concentration of phenolic compounds in dehydrated grapes, due to concentration effects, is matched by the highest concentration of these compounds in *sfursat* and *passito* wines compared to *fortified* wines. In turn, *sfursat* and *passito* wines had better chromatic characteristics than *fortified* wines, which showed unsatisfactory color characteristics. Regarding the aroma profile of wines, a concentration effect was also observed with grape dehydration, but water stress could have also promoted synthesis reactions and/or facilitated the release of glycosylated terpenes (citronellol, geranic acid, and nerol) in the grapes. Therefore, *sfursat* and *passito* wines showed higher aroma richness than *fortified* wines. For the production of a sweet red wine from Moscato nero d'Acqui fresh grapes, additional techniques may be tested as the

implementation of cold pre-fermentative maceration, which could be useful to promote the extraction of phenolic and aroma compounds.

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Table 1. Standard physico-chemical parameters of Moscato nero d'Acqui fresh and dehydrated grapes.

Parameter	Fresh grapes	Partially dehydrated grapes (~27°Brix)	Withered grapes (~36°Brix)	Sign.[a]
Berry weight (g)[b]	3.27 ± 0.50 c	2.53 ± 1.20 b	1.88 ± 0.70 a	***
Reducing sugars (g/L)	202 ± 1 a	283 ± 1 b	392 ± 5 c	***
Glucose/Fructose ratio	0.92 ± 0.01 b	0.89 ± 0.01 ab	0.86 ± 0.01 a	*
pH	3.20 ± 0.01 a	3.40 ± 0.03 ab	3.60 ± 0.12 b	*
Titratable acidity (g/L as tartaric acid)	5.8 ± 0.0 b	6.1 ± 0.1 b	5.5 ± 0.1 a	**
Malic acid (g/L)	1.8 ± 0.1 a	1.8 ± 0.1 a	3.0 ± 0.5 b	*
Tartaric acid (g/L)	6.1 ± 0.1 b	7.2 ± 0.1 c	5.1 ± 0.1 a	***
Citric acid (g/L)	0.21 ± 0.01 a	0.30 ± 0.04 a	0.56 ± 0.06 b	**
Gluconic acid (g/L)	0.10 ± 0.07 a	0.33 ± 0.04 ab	0.54 ± 0.11 b	*

Values are expressed as average ± standard deviation (n = 3). [a]Sign: *, **, and *** indicate significance at $p < 0.05$, 0.01, and 0.001, respectively. Different Latin letters within the same row indicate significant differences (Tukey-b test; $p < 0.05$). [b]Calculated on sets of 10 berries.

Table 2. Skin and seed phenolic composition of Moscato nero d'Acqui fresh and dehydrated grapes.

Parameter	Skins				Seeds			
	Fresh grapes	Partially dehydrated grapes (~27°Brix)	Withered grapes (~36°Brix)	Sign.[a]	Fresh grapes	Partially dehydrated grapes (~27°Brix)	Withered grapes (~36°Brix)	Sign.[a]
TA (mg malvidin-3-O-glucoside chloride/kg)	314 ± 49 a	497 ± 28 b	585 ± 93 b	***	-	-	-	-
A_{280} (1/kg)	25.4 ± 2.7 a	37.1 ± 0.4 b	47.5 ± 5.8 c	***	12.8 ± 2.5 b	8.5 ± 1.3 a	6.8 ± 1.6 a	**
TF (mg (+)-catechin/kg)	1542 ± 113 a	2210 ± 108 b	2926 ± 278 c	***	847 ± 157 b	667 ± 90 a	699 ± 59 ab	*
FRV (mg (+)-catechin/kg)	515 ± 61 a	648 ± 63 a	1012 ± 157 b	***	527 ± 113 c	341 ± 56 b	214 ± 59 a	***
PRO (mg cyanidin chloride/kg)	1628 ± 138 a	2116 ± 55 b	2636 ± 295 c	***	888 ± 157 b	618 ± 100 a	494 ± 99 a	**
FRV/PRO ratio	0.31 ± 0.01 a	0.32 ± 0.03 ab	0.38 ± 0.02 b	**	0.59 ± 0.03 b	0.55 ± 0.01 b	0.43 ± 0.04 a	*

Values are expressed as average ± standard deviation (n = 3). [a]Sign: *, **, and *** indicate significance at $p < 0.05$, 0.01, and 0.001, respectively. Different Latin letters within the same row indicate significant differences (Tukey-b test; $p < 0.05$). TA = total anthocyanins, A_{280} = absorbance measured at 280 nm, TF = total flavonoids, FRV = flavanols reactive to vanillin, PRO = proanthocyanidins.

Table 3. Anthocyanin profile (as percentage of total forms) of Moscato nero d'Acqui fresh and dehydrated grapes and of the wines produced from them.

Anthocyanin form	Grapes				Wines			
	Fresh grapes	Partially dehydrated grapes (~27°Brix)	Withered grapes (~36°Brix)	Sign.[a]	*Fortified* wines	*Sfursat* wines	*Passito* wines	Sign.[a]
Delphinidin-G	7.5 ± 1.0	9.2 ± 1.5	7.7 ± 1.0	ns	3.6 ± 0.3	3.1 ± 0.3	4.3 ± 0.3	ns
Cyanidin-G	7.5 ± 0.6	9.2 ± 0.9	8.0 ± 1.6	ns	1.9 ± 0.1 a	3.1 ± 0.1 b	3.2 ± 0.1 b	***
Petunidin-G	7.6 ± 1.0	8.6 ± 1.0	8.0 ± 0.9	ns	8.2 ± 0.1 a	8.0 ± 0.1 a	9.0 ± 0.2 b	*
Peonidin-G	26.9 ± 3.1	26.5 ± 2.5	25.4 ± 4.1	ns	7.3 ± 0.2 a	8.2 ± 1.1 ab	10.7 ± 0.2 b	*
Malvidin-G	44.0 ± 2.0	39.8 ± 2.0	42.9 ± 4.3	ns	74.7 ± 1.2 b	74.3 ± 0.3 b	70.6 ± 0.8 a	*
Delphinidin acetylG	0.1 ± 0.1 a	0.1 ± 0.1 b	0.1 ± 0.1 b	*	0.1 ± 0.1 a	0.4 ± 0.1 b	0.2 ± 0.1 ab	*
Cyanidin acetylG	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	ns	nd	nd	nd	ns
Petunidin acetylG	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	ns	nd	0.7 ± 0.4	0.2 ± 0.1	ns
Peonidin acetylG	0.3 ± 0.1	0.3 ± 0.1	0.3 ± 0.1	ns	0.2 ± 0.1	0.2 ± 0.1	0.2 ± 0.1	ns
Malvidin acetylG	0.7 ± 0.1	0.6 ± 0.1	0.7 ± 0.1	ns	0.6 ± 0.1	0.5 ± 0.1	0.4 ± 0.1	ns
Delphinidin-p-coumaroylG	0.4 ± 0.1	0.4 ± 0.1	0.4 ± 0.1	ns	0.2 ± 0.1	nd	nd	ns
Cyanidin-p-coumaroylG	0.7 ± 0.1 a	0.8 ± 0.1 b	0.9 ± 0.1 b	**	0.4 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	ns
Petunidin-p-coumaroylG	0.4 ± 0.1 a	0.4 ± 0.1 ab	0.5 ± 0.1 b	**	0.2 ± 0.1	0.3 ± 0.2	0.1 ± 0.1	ns
Peonidin-p-coumaroylG	1.2 ± 0.3 a	1.4 ± 0.2 ab	1.7 ± 0.2 b	**	0.8 ± 0.1	0.3 ± 0.3	0.3 ± 0.3	ns
Malvidin-p-coumaroylG	2.3 ± 0.3 ab	2.2 ± 0.3 a	2.8 ± 0.4 b	*	1.8 ± 0.3	0.6 ± 0.7	0.8 ± 0.4	ns
Peonidin-caffeoylG	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	ns	nd	nd	nd	ns
Malvidin-caffeoylG	0.3 ± 0.1 b	0.2 ± 0.1 a	0.2 ± 0.1 b	**	nd	nd	nd	ns

Percentage values are expressed as average ± standard deviation (n = 3 for grapes; n = 2 for wines). [a]Sign: *, **, ***, and "ns" indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively. Different Latin letters within the same row indicate significant differences (Tukey-b test; $p < 0.05$). G = 3-O-glucoside. nd = not detectable.

Table 4. Free and glycosidically-bound aroma compounds of Moscato nero d'Acqui fresh and dehydrated grapes.

Compound (μg/kg berries)	Free volatile compounds				Glycosidically-bound volatile compounds			
	Fresh grapes	Partially dehydrated grapes (~27°Brix)	Withered grapes (~36°Brix)	Sign.[a]	Fresh grapes	Partially dehydrated grapes (~27°Brix)	Withered grapes (~36°Brix)	Sign.[a]
t-Rose oxide	0.5 ± 0.3	0.6 ± 0.3	0.5 ± 0.7	ns	25.3 ± 1.9	21.5 ± 5	26.9 ± 11.8	ns
c-Rose oxide	nd	nd	nd	ns	6.5 ± 3.3	6.3 ± 2.5	4.9 ± 1.6	ns
t-furan-linalool oxide	0.04 ± 0.01	0.04 ± 0.02	0.04 ± 0.02	ns	3.4 ± 2.4	4.7 ± 0.1	3.5 ± 1.9	ns
c-furan-linalool oxide	0.02 ± 0.02 a	0.02 ± 0.02 a	0.06 ± 0.02 b	*	0.9 ± 0.2 b	0.4 ± 0.2 a	1.1 ± 0.2 b	**
Linalool	7.8 ± 1.3	3.9 ± 2.4	3.8 ± 1.5	ns	46.1 ± 0.1 a	65.8 ± 9.8 b	84.1 ± 4.5 c	**
Hotrienol	0.12 ± 0.04	0.04 ± 0.02	0.06 ± 0.02	ns	3.3 ± 2.3 b	0.9 ± 0.3 a	0.3 ± 0.2 a	*
α-terpineol	0.02 ± 0.01	0.04 ± 0.06	0.02 ± 0.02	ns	12.1 ± 0.7	14.3 ± 1.8	13.6 ± 4.1	ns
Citral	13.6 ± 6.4	3.7 ± 0.4	5.1 ± 0.9	ns	51.7 ± 2.3 a	66.0 ± 23.1 a	102.1 ± 4.8 b	*
t-pyran-linalool oxide	0.1 ± 0.1	0.1 ± 0.1	0.2 ± 0.1	ns	2.1 ± 0.4	2.1 ± 0.6	2.4 ± 1.2	ns
c-pyran-linalool oxide	2.5 ± 2.2 a	9.7 ± 1.4 b	17.2 ± 1.5 c	**	0.7 ± 0.7	0.3 ± 0.4	0.6 ± 0.5	ns
Citronellol	7.1 ± 4.5	2.5 ± 0.4	5.3 ± 2.3	ns	25.3 ± 13.0 a	61.4 ± 17.1 a	109.2 ± 20.3 b	**
Nerol	29.2 ± 0.7	10.4 ± 3.9	13.4 ± 7.8	ns	300.2 ± 12.3 a	518.4 ± 50.8 b	557.6 ± 46.1 b	***
Geraniol	368.2 ± 9.3 c	228.4 ± 15.4 b	193.2 ± 3.6 a	***	1938.5 ± 99.8 a	3127.2 ± 370.2 b	3781.0 ± 190.4 c	***
Benzyl alcohol	0.8 ± 0.6	0.1 ± 0.1	0.2 ± 0.1	ns	16.7 ± 1.4 a	17.9 ± 1.6 a	22.9 ± 2.6 b	*
2-phenylethanol	0.04 ± 0.01	0.04 ± 0.02	0.04 ± 0.02	ns	8.4 ± 7.9	16.2 ± 3.5	17.8 ± 2.4	ns
Geranic acid	0.02 ± 0.02 a	0.02 ± 0.02 a	0.06 ± 0.02 b	*	10.5 ± 1.8 a	95.8 ± 24.0 b	33.4 ± 15.8 a	**

Values are expressed as average ± standard deviation (n = 3). [a]Sign: *, **, ***, and "ns" indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively. Different Latin letters within the same row indicate significant differences (Tukey-b test; $p < 0.05$). nd = not detectable.

Table 5. Standard parameters, phenolic composition, and chromatic characteristics of Moscato nero d'Acqui wines.

Parameter	*Fortified* wines	*Sfursat* wines	*Passito* wines	Sign.[a]
Ethanol (% v/v)	13.3 ± 0.1 a	15.9 ± 0.1 c	13.7 ± 0.1 b	***
Residual sugars (g/L)	94 ± 5 b	5 ± 1 a	114 ± 2 c	***
Glucose/Fructose ratio	0.6 ± 0.1 a	4.6 ± 1.2 b	0.3 ± 0.1 a	*
pH	3.30 ± 0.01 a	3.60 ± 0.01 b	3.70 ± 0.01 c	***
Titratable acidity (g/L as tartaric acid)	3.9 ± 0.1 a	5.8 ± 0.1 c	5.4 ± 0.1 b	***
Volatile acidity (g/L as acetic acid)	0.2 ± 0.1 a	0.3 ± 0.1 b	0.6 ± 0.1 c	***
Malic acid (g/L)	1.3 ± 0.1	1.4 ± 0.1	1.6 ± 0.1	ns
Tartaric acid (g/L)	1.6 ± 0.1	1.4 ± 0.1	1.4 ± 0.3	ns
Glycerol (g/L)	4.6 ± 0.2 a	10.0 ± 0.4 b	12.3 ± 0.1 c	***
TA (mg malvidin-3-O-glucoside chloride/L)	72 ± 2 a	101 ± 3 b	165 ± 3 c	***
A_{280} (1/L)	16.2 ± 0.3 a	30.1 ± 0.5 b	45.2 ± 0.2 c	***
TF (mg (+)-catechin/L)	540 ± 17 a	979 ± 15 b	1750 ± 57 c	***
FRV (mg (+)-catechin/L)	190 ± 21 a	485 ± 42 b	1034 ± 80 c	**
PRO (mg cyanidin chloride/L)	494 ± 58 a	665 ± 46 a	1926 ± 82 b	***
FRV/PRO ratio	0.38 ± 0.01 a	0.73 ± 0.01 c	0.54 ± 0.02 b	***
L*	93.6 ± 0.1 b	63.8 ± 3.8 a	61.1 ± 1.0 a	**
a*	6.2 ± 0.9 a	36.3 ± 2.1 b	37.8 ± 0.8 b	***
b*	6.4 ± 0.3 a	21.7 ± 1.8 b	25.1 ± 0.5 b	***
Color hue	1.42 ± 0.08 b	0.98 ± 0.07 a	1.11 ± 0.02 a	*
Color intensity (10 mm optical path)	0.28 ± 0.01 a	1.70 ± 0.17 b	1.92 ± 0.05 b	**

Values are expressed as average ± standard deviation (n = 2). [a]Sign: *, **, ***, and "ns" indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively. Different Latin letters within the same row indicate significant differences (Tukey-b test; $p < 0.05$). TA = total anthocyanins, A_{280} = absorbance measured at 280 nm, TF = total flavonoids, FRV = flavanols reactive to vanillin, PRO = proanthocyanidins. L* = lightness, a* = red/green color coordinate, b* = yellow/blue color coordinate.

Table 6. Free and glycosidically-bound aroma compounds of Moscato nero d'Acqui wines.

Compound (μg/L wine)	Free volatile compounds				Glycosidically-bound volatile compounds			
	Fortified wines	*Sfursat* wines	*Passito* wines	Sign.[a]	*Fortified* wines	*Sfursat* wines	*Passito* wines	Sign.[a]
t-Rose oxide	8.2 ± 0.5 a	48.8 ± 16.7 b	95.5 ± 4.6 c	**	9.0 ± 5.2	14.4 ± 0.8	20.7 ± 2.1	ns
c-Rose oxide	0.2 ± 0.1 a	0.1 ± 0.1 a	10.9 ± 0.1 b	***	0.8 ± 1.1	2.3 ± 0.7	5.2 ± 2.8	ns
t-furan-linalool oxide	7.2 ± 0.9 b	0.1 ± 0.1 a	0.1 ± 0.1 a	**	4.7 ± 0.6	6.8 ± 3.2	9.1 ± 0.2	ns
c-furan-linalool oxide	0.1 ± 0.1	0.2 ± 0.1	0.3 ± 0.1	ns	1.6 ± 0.6	1.9 ± 0.1	1.0 ± 0.7	ns
Linalool	89.5 ± 0.8 a	110.5 ± 1.6 b	89.7 ± 6.6 a	*	27.4 ± 1.8 a	47.6 ± 0.9 b	56.6 ± 1.2 c	***
Ho-trienol	4.1 ± 2.9	3.2 ± 0.3	11.4 ± 5.3	ns	0.5 ± 0.1	0.5 ± 0.6	1.1 ± 0.5	ns
α-terpineol	0.2 ± 0.1	15.6 ± 9.2	11.5 ± 5.2	ns	11.3 ± 1.0	17.8 ± 0.1	13.8 ± 7.4	ns
Citral	0.2 ± 0.1	0.2 ± 0.1	0.2 ± 0.1	ns	52.2 ± 3.6 a	67.8 ± 3.9 b	78.1 ± 2.1 b	**
t-pyran-linalool oxide	0.1 ± 0.1	0.3 ± 0.1	0.4 ± 0.2	ns	4.9 ± 1.7	4.7 ± 2.3	4.4 ± 2.6	ns
Geranyl acetate	15.4 ± 6.8	6.4 ± 8.1	8.4 ± 0.4	ns	nd	nd	nd	-
c-pyran-linalool oxide	2.1 ± 1.1	2.6 ± 0.7	1.8 ± 1.4	ns	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	ns
Citronellol	107.1 ± 0.9 a	536.9 ± 58.4 c	302.5 ± 24.2 b	**	26.2 ± 2.1 a	49.7 ± 0.8 b	64.9 ± 0.9 c	***
Nerol	2.4 ± 0.8	22.7 ± 9.4	35.6 ± 20.2	ns	281.6 ± 18.8 a	444.2 ± 5.8 b	501 ± 6.1 c	***
2-phenylethyl acetate	47.2 ± 29.8 a	177.7 ± 93.6 a	526 ± 12.3 b	**	nd	nd	nd	-
Geraniol	21.4 ± 9.9	19.9 ± 3.6	27.4 ± 16.6	ns	1434.4 ± 164.9 a	2167.6 ± 9.2 b	2567.6 ± 22.5 c	**
Benzyl alcohol	0.1 ± 0.1 a	0.7 ± 0.4 ab	1.6 ± 0.4 b	*	5.2 ± 2.3	3.0 ± 0.9	2.7 ± 0.7	ns
2-phenylethanol	947.9 ± 66.0 a	4151.2 ± 194.6 c	2423.2 ± 124.7 b	***	23.7 ± 1.9	16.8 ± 10.9	25.2 ± 1.2	ns
Geranic acid	2.3 ± 1.6	9.4 ± 9.0	16.6 ± 6.1	ns	24.7 ± 0.7 c	7.1 ± 1.8 a	17.5 ± 1.9 b	**

Values are expressed as average ± standard deviation (n = 2). [a]Sign: *, **, ***, and "ns" indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively. Different Latin letters within the same row indicate significant differences (Tukey-b test; $p < 0.05$). nd = not detectable.

Highlights

- Suitability of Moscato nero d'Acqui grapes to dehydration and winemaking was tested
- Grape dehydration increased phenolic contents with similar anthocyanin forms ratio
- Free terpene concentration decreased mainly in the first dehydration phase
- *Sfursat* and *passito* wine had higher aroma richness and better color characteristics