CHEMICAL AND VOLATILE COMPOSITION OF THREE ITALIAN SWEET WHITE PASSITO WINES

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Abstract

Aims: This study was designed to gain knowledge of three Controlled Denomination of Origin (DOC) Italian sweet white Passito wines (Caluso Passito DOC, Cinque Terre Sciacchetrà DOC and Passito di Pantelleria DOC) produced in several areas of Italy from grapes dried with different systems and vinification techniques.

Methods and results: Physico-chemical and chromatic characteristics, sodium, potassium, gluconic acid, glucono-γ-lactone, acetaldehyde, sorbitol, laccase, organic acids and semi-quantitative free volatile profile were determined on these wines.
Caluso Passito DOC wines presented higher contents of organic acids (above all, malic acid), main metabolites from noble *Botrytis cinerea* (laccase, glycerol, gluconic acid and benzaldehyde) and low contents of total polyphenols. Among the volatile components, normal fatty acid ethyl esters (ethyl hexanoate and ethyl octanoate), branched-chain esters (ethyl 2-methylpropanoate, ethyl 2-methylbutyrate, ethyl 3-methylbutyrate) and benzaldeyde characterized this Passito wine. Cinque Terre Sciacchetrà DOC wines showed the lowest total acidity with a lower amount of malic acid and a higher content of polyphenols. This wine was characterized by some predominant acetates (isoamyl acetate), alcohols, benzaldehyde and an isoprenoid, ß-damascenone.
Passito di Pantelleria DOC wines presented higher amounts of ashes resulting in higher pH values compared to the other two Passito typologies. Due to its production from aromatic grapes, it showed several varietal components such as terpenes, while ethyl esters/acetates and alcohols were less represented.

Conclusion: This survey provides information allowing the characterisation of three Passito dessert wines at high commercial value.

Significance and impact of the study: This study provides oenological information to be utilised to protect and valorise the Controlled Denomination of Origin sweet wine production and contributes to the preservation of traditional and terroir productions and their commercialization.

Keywords: sweet wine, Caluso Passito, Cinque Terre Sciacchetrà, Passito Pantelleria, *Botrytis cinerea*

Résumé

Objectifs : Cette étude a pour but d'améliorer la connaissance de trois vins doux italiens *Passito* d'Appellation d'Origine Contrôlée (DOC) : Caluso Passito DOC, Cinque Terre Sciacchetrà DOC et Passito di Pantelleria DOC. Ces vins produits dans trois régions italiennes sont issus de raisins séchés par différentes techniques de passerillage et de vinification.

Méthodes et résultats : Les caractéristiques physico-chimiques et chromatiques, la teneur en sodium, en potassium, en acide gluconique, en gluco-γ-lactone, en acétaldéhyde, en sorbitol, en laccase, en acides organiques ainsi que le profil semi-quantitatif des composés volatils libres ont été déterminés dans les vins étudiés.
Les vins de l'appellation Caluso Passito ont présenté la plus forte teneur en acides organiques (en particulier en acide malique), des métabolites principaux dus à la pourriture noble (laccase, glycérol, acide gluconique et benzaldéhyde) et des teneurs faibles en polyphénols totaux. Parmi les composés volatils, les esters éthyliques d'acides gras à chaîne linéaire (hexanoate et octanoate), les esters à chaîne ramifiée (éthyle 2-méthylpropanoate, éthyle 2-méthylbutyrate, éthyle 3-méthylbutyrate) et la benzaldéhyde ont caractérisé ce vin passerillé.
Les vins de l'appellation Cinque terre Sciacchetrà ont présenté l'acidité totale la plus basse, une teneur plus faible en acide malique et une teneur en polyphénols plus élevée. Ce vin est caractérisé par quelques acétates prédominants (acétate d'isoamyle), alcools, benzaldéhyde et un isoprénoïde, la ß-damascénone.
Les vins de l'appellation Passito di Pantelleria ont présenté des plus forts taux de cendres, donc un pH plus élevé par rapport aux deux autres appellations. Ces vins, obtenus à partir de raisins aromatiques, contiennent plusieurs composés variétaux tels que les terpènes, tandis que les acétates d'éthyle et les alcools ont été moins présents.

Conclusion : Cette étude fournit des informations permettant la caractérisation de ces trois vins doux naturels (Passito) à forte valeur commerciale.

Signification et impact de l'étude : Ces données œnologiques permettent de mieux protéger et de valoriser les vins de ces appellations d'origine contrôlée et contribuent à la sauvegarde de ces produits traditionnels du terroir et à leur commercialisation.

Mots clés : vins doux, Caluso Passito DOC, Cinque Terre Sciacchetrà DOC, Passito Pantelleria DOC, *Botrytis cinerea*

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INTRODUCTION

Many typologies of wine are produced by partial or total use of over ripe and/or dehydrated grapes. Among these, « sweet » or « dessert wines » are generally well-known and commercially important. In fact, there are among these ones, the *Passito* wines, the ice wines, the Sauternes, the Tockaj as well as many others, that are produced in almost all viticultural areas in the world (BAILLY *et al*., 2006; MIKLÓSY *et al*., 2000, SOLEAS and PICKERING, 2007).

The *Passito* wines are produced in some grape-growing and producing areas both with sizeable production and with a limited but renowned one. In Europe, this production is disciplined by Council Regulation (EC's legislation 1493/1999) and its successive modifications (EC's legislation 2165/2005) and, in Italy, by the recent law (G.U. No. 82/2006).

Among the large patrimony of *Passito* wines produced in Italy, the production comprises of about one hundred Denomination of Origin *Passito* wines (SCIENZA, 2006). Generally, off-vine grape drying processes, with or without artificial environmental conditions are used for *Passito* wine production (BARBANTI *et al*., 2008; BELLINCONTRO *et al*., 2004; EBERLE *et al*., 2007; FRANGIPANE *et al*., 2007). Addition of ethanol for correction of the natural grape alcohol content is not allowed.

Differences in the grape varieties used, the post-harvest drying step of the grapes, fermentation conditions, the winemaking method and time of aging characterise the Italian *Passito* wines (D'AGOSTINO *et al*., 1998a and 1998b; D'AGOSTINO *et al*., 1999; DI STEFANO *et al*., 2002; GENOVESE *et al*., 2007; GUARRERA *et al*., 2005; VERSINI *et al*., 1999). All these aspects influence the physico-chemical and sensory characteristics of these products. Moreover, depending on grape drying conditions, a beneficial development of the noble rot, *Botrytis cinerea*, can influence the final characteristics of these wines (DIVOL and LONVAUD-FUNEL, 2004; DONÈCHE, 1993).

In recent years, interest in sweet *Passito*-wines has increased enormously and efforts of the grape and wine producers have been focused on improving the wine quality. Therefore, extensive information about all chemical oenological parameters and volatile composition would be of great interest to the winemakers.

Previous studies on Muscat grape variety and its vinification to make *Passito* di Pantelleria Controlled Denomination of Origin (DOC) wine, the major Southern production of *Passito* wine in Italy, were intense and reported in the literature (PAPUCCI *et al*., 1998; D'AGOSTINO *et al*., 1998a and 1998b), while no data have been reported on Caluso *Passito* DOC and Cinque Terre DOC wines, two representative sweet *Passito* wines from Northwest Italy.

The appellation Caluso *Passito* DOC belongs to wines produced in Northwest Italy (Piedmont region), from white Erbaluce grape variety. The grapes are usually harvested in September and placed on mats, or in perforated plastic boxes in one layer, or hung vertically in crates and let to dry for about six months in open rooms without environmental systems control (temperature, relative humidity, air flow).

Cinque Terre Sciacchetrà DOC is produced in the Cinque Terre area, a small viticultural area of mountain, near the sea, in Liguria, from a blend of Vermentino, Bosco and Albarola grapes in different percentages. Grapes are generally harvested in October then dried for about two months, using the methods mentioned above, but the external environmental conditions are quite different.

Passito di Pantelleria DOC wine is produced in the homonymous Mediterranean island, exclusively from the aromatic Muscat of Alessandria (Zibibbo) grapes. These are harvested generally in August-September and than sun-dried for about thirty days. Different typologies of dry-grapes can be obtained until a final sugar concentration of 600 g/L. In particular, the grape sugar content utilized in this *Passito* wine can vary from 20 up to over 34 °Brix, due to use of different ratios of mixed grapes of different ripeness that can be classified in ripe (20-25 °Brix), overripe (25-32 °Brix) and withered or « passolata » (32-35 °Brix) grape typologies. Thus, the initial production of the basic wines is interrupted up to 8-10 % vol. (v/v) of ethanol by refrigeration at low temperatures. After, overripe « malaga » grapes, totally withered with 15-20 % water residual, are added to this product and a refermentation of the product occurs in order to sweeten the final product (ASTORINO and DI STEFANO, 2003; D'AGOSTINO *et al*., 1999; D'AGOSTINO *et al*., 2000; D'AGOSTINO *et al*., 2002; D'AGOSTINO and PARRINELLO, 2002).

The Italian Production Disciplinaries determine at least 17 % the total title alcoholometric volumeter minimum for Caluso *Passito* DOC and Cinque Terre Sciacchetrà DOC, and 20 % for *Passito* di Pantelleria DOC. These wines can be commercialised only after four, two and one years of aging, respectively.

The work described here was carried out on several commercial wines of these three typologies, with the aim to evaluate the relative chemical and volatile composition. The overall goal was to characterise, valorise and preserve these traditional oenological products.

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MATERIALS AND METHODS

1. Wine samples

The survey was carried out on Caluso *Passito* DOC wine (CP) (23 samples) aged from 4 to 28 years, on Cinque Terre Sciacchetrà DOC wine (CTS) (20 samples) aged from 1 to 5 years, on *Passito* di Pantelleria DOC wines (PP) (18 samples) aged from 1 to 4 years. All samples were acquired directly on the market and/or from wineries.

2. Physico-chemical analyses

Physico-chemical parameters (pH, total acidity, volatile acidity, reducing sugars, alcoholic percentage, ash, ash alkalinity, dry extract, potassium and sodium) were determined according to European Official Methods (ECC, 1990). Gluconic acid, glucono-γ-lactone, acetaldehyde, and sorbitol concentrations were determined using enzymatic test kits from R-Biopharm Italia (Cerro al Lambro, MI, Italy) using a UV-1601 Spectrophotometer (Shimadzu, Japan). Laccase activity was evaluated according to FORTINA *et al*., 1996. Organic acids (citric, tartaric, malic, succinic and lactic), sugars (glucose and fructose) and glycerol were quantified by HPLC (Thermo Electron Corporation, Waltham, MA, USA) equipped with a UV detector (UV100) set to 210 nm and a refractive index detector (RI-150). The analyses were performed isocratically at 0.8 mL/min and 65 °C with a 300 x 7.8 mm i.d. cation exchange column (Aminex HPX-87H) and a Cation H^+ Microguard cartridge (Bio-Rad Laboratories, Hercules, CA, USA), using 0.0026N H_2SO_4 as mobile phase (SCHNEIDER *et al*., 1987; GERBI and TORTIA, 1991).

In wine samples, total phenolic content, evaluated using Folin-Ciocalteu reactive, flavan-3-ols reactive to vanillin (Flavanols Vanillin Assay), flavan-3-ols reactive to *p*-dimethylaminocinnamaldehyde (Flavanols reactive to DMACA), all expressed as mg/L (+) catechin, and proanthocyanidins, expressed as mg/L cyanidin chloride, were determined spectrophotometrically according to DI STEFANO *et al*. (1989).

Colour was measured using the CIELab color space values (CIE, 1986), which include the L* parameter (lightness), a* (red-green component), b* (blue-yellow component), with an algorithm proposed by PIRACCI (1994), using 40 measurements between 380 and 780 nm at 10 nm intervals with 10-mm quartz cells. Colour intensity (O.D.) was measured at 450 nm (BUCCELLI *et al*., 1999).

3. Volatile fraction analysis

Four mL of each wine sample were placed in a 10 mL pre-assembled clear glass vial (38 mm high and 22 mm in diameter) purchased from Supelco (Bellefonte, PA, USA). 10 μL of an ethanolic solution of 1-heptanol internal standard (10.8 μg/mL) and 1.2 g of NaCl (30 % w/v) were spiked to the sample wine, and mixed using a magnetic stirrer. The vials were capped with a 20 mm PTFE/silicone septum (Supelco, Bellefonte, PA, USA). The vials were placed in a water bath at 35 °C and stirred for 30 min until reaching equilibrium. The SPME fiber used was a StableFlex 2 cm — 50/30 μm divinylbenzene–carboxen–polydimethylsiloxane (DVB–CAR–PDMS) (Supelco, Bellefonte, PA, USA) according to FERREIRA and DE PINHO (2003). After exposure, the fiber was retracted and transferred to the injector operating in splitless mode at 270 °C for 4 min.

Compound identification was achieved with a Shimadzu GC-17A gas chromatograph (GC) coupled with a Shimadzu QP-5000 quadrupole mass spectrometer (Shimadzu Corporation, Kyoto, Japan). The GC was equipped with a DB-WAX fused silica capillary column, 30 m x 0.25 mm i.d., 0.25 μm film thickness (J&W Scientific Inc., Folsom, CA, USA) and a split–splitless injector. The carrier gas was ultrahigh pure helium with a flow rate of 1.0 mL/min. The following column temperature programming sequence was used: an initial temperature of 35 °C for 5 min, increased to 185 °C at 2 °C/min for an additional 2 min then raised to 210 °C at 10 °C/ min and maintained for 3 min. Mass spectra were recorded in the electron ionization mode of 70 eV in the 33–300 amu mass range. The ion source and the interface were maintained at 230 °C. The scan rate was 500 amu/s and the start time was 5.7 min to avoid overloading the mass spectrometer with ethanol.

Identification of peaks was performed by comparison of their retention time and mass spectra with those of pure reference standards. In addition the NIST12, NIST62 (National Institute of Standards and Technology, Gaithersburg MD, USA) and ADAMS (2001) mass spectral databases were consulted for the peak identification. Retention indices (RIs), calculated by linear interpolation relative to retention times of C_5-C_{25} *n*-alkanes as external references, were compared with those of literature data. Each compound was expressed as semiquantitative data (μg/L) with respect to the relative concentration of the internal standard by using the peak areas from the total ion current (TIC) without using correction factors.

4. Statistical analysis

All statistical analyses were performed using Statistica software package (ver.6.0; Statsoft Inc., Tulsa, OK, USA). A one-way Analysis of Variance (ANOVA) was carried out to highlight differences among the three typologies of wines with Duncan's test.

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RESULTS AND DISCUSSION

1. Physico-chemical parameters

Mean values and standard deviations for each chemical parameter determined on all samples of CP, CTS and PP wines are reported in table 1. For all Controlled Denominations of Origin wines studied, a high variability in each wine family on almost all different compositional parameters was observed. For CP and CTS wine samples, this variability could partially result from the artisanal winemaking technique performed without control of alcoholic and malolactic fermentation and by indigenous microflora (CONTERNO and GANDINI, 1998). Moreover the different aging of the wines may be responsible. Instead, the variability of PP wines, whose production is larger than the previous ones, is probably caused by differences in the initial sugar content, due to the rather diversified winery technologies of production using different degrees of grape raisin and winemaking. It is known that the different degrees of withering of the grape in the sun involves different transformations of sugars, malic acid, nitrogenous substances, polyphenol, besides aroma compounds such as terpenes (D'AGOSTINO *et al*., 2000).

In spite of the internal variability, each *Passito* wine typology could be well characterized and some chemical parameters allowed us to establish a significant difference ($p< 0.01$) except for ethanol, glucose, reducing sugars, volatile acidity and acetaldehyde (Table 1).

In particular, the total acidity, succinic and malic acid allowed us to recognize significant differences among the three product families. Malic acid resulted with a higher average content (≥ 2 g/L) in CP wines probably related to a higher initial content of malic acid in fresh Erbaluce grapes (BOVIO and NOVELLO, 1998) compared to CTS and PP wines that have low levels of this organic acid.

Ashes, alkalinity of ashes, potassium and sodium were other important chemical parameters allowing differentiation, as was also noticed by FRIÀS *et al*. (2003), for the sweet wines of the Canary Islands. PP wines were particularly rich in potassium (≥ 2 g/L) and sodium (100 mg/L). The higher content of sodium correlates with

Table 1 - Physico-chemical parameters (mean ± standard deviation) of 23, 20 and 18 Passito wine samples, respectively of Caluso Passito DOC, Cinque Terre Schiacchetrà DOC and Passito di Pantelleria DOC.

Values followed by different letters significantly differ for $p \leq 0.05$ (ns = not significant; *$p<0.05$; **$p<0.01$).

	Caluso Passito DOC	Cinque Terre Sciacchetrà DOC	Passito di Pantelleria DOC	Significance
Ethanol (% vol)	13.6 ± 0.6 a	13.7 ± 1.6 a	13.9 ± 0.9 a	ns
Glucose (g/L)	42.7 ± 12 a	54.9 ± 32.6 a	51.9 ± 17.1 a	ns
Fructose (g/L)	84.6 ± 15.6 a	101.5 ± 30.8 b	78.5 ± 26.7 a	**
Reducing sugars (g/L)	127.3 ± 25.7 a	156.4 ± 56.5 b	130.4 ± 41.6 ab	ns
Glucose/Fructose	0.5 ± 0.1a	0.5 ± 0.2 a	0.7 ± 0.2 b	**
Ashes (g/L)	3.6 ± 0.8 b	3.1 ± 0.6 a	4.7 ± 0.8 c	**
Alkalinity of ash (meq/L)	30.1 ± 6.9 a	52.4 ± 16.4 c	41.9 ± 7.2 b	**
Potassium (mg/L)	895.4 ± 308.9 a	1195.4 ± 267.8 b	2210.9 ± 597.9 c	**
Sodium (mg/L)	31.1 ± 86.9 a	53.8 ± 23.4 a	124.3 ± 41.4 b	**
Total Acidity (g/L as tartaric acid)	7.3 ± 0.8 c	5.4 ± 0.9 a	6.3 ± 0.9 b	**
pH	3.6 ± 0.1 a	3.6 ± 0.2 a	3.9 ± 0.1 b	**
Volatile Acidity (g/L as acetic acid)	1.0 ± 0.3 a	1.2 ± 0.2 b	1.1 ± 0.4 ab	ns
Citric Acid (g/L)	0.4 ± 0.1c	0.3 ± 0.1 b	0.2 ± 0.1a	**
Tartaric acid (g/L)	1.4 ± 0.4 a	2.0 ± 0.4 b	1.5 ± 0.4 a	**
Malic acid (g/L)	2.3 ± 0.7 b	1.0 ± 0.5 a	1.3 ± 0.6 a	**
Lactic acid (g/L)	1.1 ± 0.7 b	0.5 ± 0.5 a	1.7 ± 1.1 c	**
Succinic acid (g/L)	0.6 ± 0.2 b	0.7 ± 0.3 c	0.4 ± 0.1 a	**
Glycerol (g/L)	12.3 ± 1.6 b	12.7 ± 2.2 b	9.4 ± 1.1 a	**
Gluconic acid (g/L)	2.3 ± 1.0 b	0.8 ± 0.8 a	0.9 ± 0.7 a	**
Glucono-d-lactone (mg/L)	268.2 ± 184.0 b	98.5 ± 98.1 a	112.4 ± 97.9 a	**
Acetaldehyde (mg/L)	46.2 ± 13.4 a	37.2 ± 26.0 a	39.5 ± 25.1a	ns
Sorbitol (mg/L)	165.4 ± 50.1 a	262.4 ± 113.5 b	428.2 ± 123.1c	**
Laccase (U/mL)	4.3 ± 3.3 b	2.6 ± 1.9 a	1.6 ± 1.4 a	**
Total polyphenols (mg/L as (+) catechin)	251.3 ± 52.1a	605.8 ± 177.9 c	400.4 ± 97.3 b	**
Flavanols reactive to DMACA (mg/L as (+) catechin)	10.3 ± 4.9 a	73.8 ± 40.9 b	65.6 ± 25.3 b	**
Flavanols Vanillin Assay (mg/L as (+) catechin)	42.1 ± 8.3 a	70.1 ± 95.8 ab	105.8 ± 40 b	**
Proanthocyanidins (mg/L as cyanidin chloride)	36.3 ± 43.5 a	417.9 ± 228.6 c	203.9 ± 83.2 b	**
O.D. at 450 nm (10 mm O.P.)	0.9 ± 0.4 b	0.8 ± 0.3 b	0.6 ± 0.2 a	**
L* - lightness	81.1 ± 7.4 a	81.9 ± 6.7 a	87.1 ± 5.9 b	**
a* - red/green	3.8 ± 5.5 b	5.8 ± 5.9 b	-0.8 ± 3.5a	**
b* - yellow/blue	54.5 ± 13.5 b	55.8 ± 12.6 b	44 ± 13.2 a	**

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the exposure of the grapes to winds saturated with sea water, that characterize Pantelleria Island (D'AGOSTINO *et al*., 1998a).

The high concentration of laccase, gluconic acid, glucono-δ-lactone and glycerol principally in CP wine confirmed a prolonged action of the noble gray rots (*Botrytis cinerea*) on the grapes during of the withering phase (CALDERONE *et al*., 2004; DONÈCHE, 1993; GERBI *et al*., 2001; RIBÈREAU-GAYON *et al*., 1979) as compared to other wine families. Thus, the action of *B. cinerea* is different in the three products, due to climatic differences and the withering techniques.

Both different maceration techniques and different modalities of fermentation and aging (with or without wood barrels) have resulted in, with all three wine types, a marked difference in the content of total polyphenols in each wine family. Up to 606 mg/L as (+)catechin were observed in CTS wine families, characterized by a total maceration of destemmed and pressed grapes, to 400 mg/L in PP wines obtained generally with partial maceration of overripe grapes, to 251 mg/L in CP produced by only a must winemaking with, in some wineries, short maceration (6 - 24 hours) of pressed grapes that are made to increase the yield in the extracted must (ZEPPA *et al*., 2001).

Passito di Pantelleria wines generally presented the lower values of O. D. at 450 nm. Nevertheless, it does not seem that the difference of the phenolic composition and aging had a direct effect on the colour of the different wine typologies. The non-enzymatic browning of these wines could be due to formation of melanoidins, compounds that contribute the characteristic dark colour, derived from Maillard's reaction between reducing sugars and amino acids, considered as causes of browning of white sweet wines (DE ROSA, 1978; MORENO *et al*., 2007).

2. Volatile compounds

A total of 103, 110 and 105 volatile compounds are detected in the volatile fraction of CP, CTS and PP wines, respectively (table 2). The volatile components are grouped in the fermentative, varietal and aging/bouquet aroma classes. In the present work, the relative composition of every volatile compound for each wine type was calculated as relative concentration to the internal standard, such as a semi-quantitative content expressed in μg/L (mean concentration ± standard deviation) of each volatile compound respect to the internal standard.

An objective comparison among different sweet wine typologies was established in terms of qualitative and semi-quantitative differences on volatile compounds.

Six volatile compounds, probably some acetals, were detected, overall in CP wines, but it was not possible to identify them since their mass spectra were not confirmed in mass spectral databases and no standards were available.

There were statistically significant differences among the three types of wine regarding many aroma volatile compounds (64 components compared to 135 total volatiles detected in all wines). Variations in the semi-quantitative content of each volatile compound for all three sweet *Passito* wines is ascribable to the differences in production technology used and the aging time of the wines too.

Among the fermentation compounds, medium-chain fatty acid ethyl esters (ethyl butyrate, ethyl hexanoate, ethyl octanoate and ethyl decanoate), diethyl succinate, acetates of higher alcohols (isoamyl acetate) and fusel alcohols, formed both by enzymatic pathways from yeasts during the fermentation process and by chemical esterification during long-term aging (RIBÉREAU-GAYON *et al*., 2000), were the dominant analytical signals in all three *Passito* wines. Ethyl esters are known to have very pleasant fruity, honey and sweet scents which contribute to the aromatic finesse of wines (UGLIANO and HENSCHKE, 2009). Some of these volatile components, such as ethyl hexanoate and ethyl octanoate, were significantly different in the semi-quantitative content among wines ($p \leq 0.001$). These compounds, as the total ethyl and acetate esters, constitute an important part of volatile compounds in the CP wines in comparison with the other two types. Semi-quantitative levels of these ethyl esters in this wine type were similar as reported in the sweet white Fiano wine (GENOVESE *et al*., 2007) even if, in this study, a major number of identified ester compounds was found.

In CP wines, relatively significant higher signals of ethyl 2-methylpropanoate, ethyl 2-methylbutyrate and ethyl 3-methylbutyrate were found ($p \leq 0.001$), followed by the CTS family of wines. These volatile compounds are examples of important odour esters characterized by low odour thresholds 15, 18 and 3 μg/L, respectively (FERREIRA *et al*., 2000), with strawberry-like aromas (GUTH, 1997; PIOMBINO *et al*., 2004;), indicating that these compounds are among the most powerful odorants in the class of esters. In PP wines, the semi-quantitative level of the total sum of ethyl ester class was, on average, low compared to the other wine types. It is known that the effect of storage may or may not decrease the ester composition. This is related to their different hydrolysis/esterification equilibria, since these are influenced by wine pH and storage temperature (RAMEY and OUGH, 1980; CÂMARA *et al*., 2006). In all the wine types studied, ethyl esters were observed to decrease during the aging of the samples (data not shown) (DÍAZ-

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Table 2 - Qualitative and semi-quantitative profile of volatile compounds (µg/L) (mean ± standard deviation) detected in Caluso *Passito* DOC, Cinque Terre Sciacchetrà DOC and *Passito* di Pantelleria DOC wine typologies (1/3).

Compound (µg/L)[a]	Caluso Passito DOC	Cinque Terre Sciacchetrà DOC	Passito di Pantelleria DOC	Signif.[b]
Fermentative volatile compounds				
Esters				
Ethyl 2-methylpropanoate	86 ± 60 b	22 ± 32 a	4 ± 7a	***
Propyl acetate	7 ± 15	3 ± 11	-[e]	ns
Isobutyl acetate	51 ± 28 b	64 ± 55 b	25 ± 14 a	**
Ethyl butyrate	227 ± 71b	184 ± 181 ab	123 ± 82 a	*
Ethyl 2-methylbutyrate	67 ± 45c	29 ± 21 b	3 ± 2 a	***
Ethyl 3-methylbutyrate	104 ± 63c	56 ± 39 b	8 ± 3a	***
Butyl acetate	1 ± 1	1 ± 2	2 ± 2	ns
Isoamyl acetate	481 ± 126b	614 ± 315c	311 ± 140 a	***
Ethyl pentanoate	7 ± 9	9 ± 22	5 ± 3	ns
Ethyl crotonate	1 ± 1a	1 ± 1a	2 ± 4 b	***
Methyl hexanoate	-	1 ± 2	-	ns
Ethyl hexanoate	1531 ± 491c	979 ± 814 b	451 ± 234 a	***
Isopentyl isobutyrate	1 ± 1	1 ± 1	1 ± 1	ns
Ethyl pyruvate	9 ± 6 b	5 ± 9 a	2 ± 4 a	**
Pentyl butanoate	1 ± 1	1 ± 2	-	ns
Hexyl acetate + monoterpene [c]	-	-	13 ± 12	ns
Hexyl acetate	16 ± 7	24 ± 24	-	***
Ethyl 2-ethylhexanoate	1 ± 2	-	-	ns
Ethyl (*E*)-3-hexenoate	-	6 ± 24	-	ns
Ethyl (*Z*)-3-hexenoate	1 ± 1	1 ± 2	-	ns
Ethyl heptanoate	12 ± 11	1 ± 21	22 ± 14	ns
Ethyl lactate	301 ± 209	210 ± 187	226 ± 178	ns
Methyl octanoate	4 ± 6	6 ± 12	2 ± 4	ns
Ethyl octanoate	2828 ± 908 b	1606 ± 1950 a	843 ± 800 a	***
3-Methylbutyl hexanoate	-	1 ± 3	-	ns
Ethyl 4-octenoate	-	-	4 ± 5	ns
Ethyl nonanoate	4 ± 4 a	17 ± 12 b	6 ± 5 a	***
Diethyl malonate	-	1 ± 2	-	ns
Methyl ethyl succinate	-	2 ± 3	1 ± 2	*
Ethyl decanoate	136 ± 102	223 ± 309	84 ± 88	ns
Diethyl methylsuccinate	2 ± 4	1 ± 1	-	*
Ethyl benzoate	22 ± 11	288 ± 1187	3 ± 2	ns
3-Methylbutyl octanoate	1 ± 1	1 ± 2	-	ns
Diethyl succinate	1283 ± 449 c	966 ± 561b	442 ± 183 a	***
Ethyl 9-decenoate	80 ± 246	7 ± 14	21 ± 25	ns
Methyl salicylate	1 ± 1	1 ± 3	1 ± 1	ns
Ethyl phenyl acetate	33 ± 12 c	17 ± 12 b	7 ± 4a	***
2-Phenylethyl acetate	27 ± 13	27 ± 10	39 ± 42	ns
Diethyl malate	5 ± 5	3 ± 5	-	**
∑ *Ethyl and acetate esters*	7238	5352	2638	
Fusel alcohols				
1-Propanol	164 ± 54 b	128 ± 99 ab	88 ± 43 a	***
2-Methyl-1-propanol	768 ± 207 b	953 ± 620 b	492 ± 105 a	**
2-Pentanol	2 ± 3	1 ± 1	1 ± 2	ns
1-Butanol	38 ± 16	47 ± 29	41 ± 19	ns
2-Methyl-1-butanol	5368 ± 1201 b	6369 ± 2713 b	3501 ± 745 a	***
1-Pentanol	6 ± 4 a	5 ± 3 ab	11 ± 2 b	***
∑ *Higher major alcohols*	6346	7502	4134	
Higher minor alcohols				
3-Ethoxy-1-propanol	1 ± 1	1 ± 2	1 ± 2	ns
4-Methyl-1-pentanol	2 ± 2 b	5 ± 4 c	1 ± 1a	***
2-Heptanol	8 ± 8 b	2 ± 4 a	4 ± 5 ab	*
3-Methyl-1-pentanol	11 ± 10 b	15 ± 13 b	6 ± 9 a	**
3-Octanol	4 ± 5	-	4 ± 3	ns
2-Octanol	-	1±1	10 ± 8	***
1-Octen-3-ol	-	-	10 ± 17	ns
6-Methyl-5-hepten-2-ol	-	-	3 ± 5	ns
2-Ethyl-1-hexanol	36 ± 35 b	28 ± 31 b	2 ± 7 a	***
2-Nonanol	11 ± 32	4 ± 11	3 ± 6	ns
d,l 2,3-Butanediol	287 ± 155 b	190 ± 114 a	186 ± 113 a	*
1-Octanol	12 ± 13 a	15 ± 9 a	52 ± 86 b	*
1-Octanol + 5-Methyl-2-furfural [c]	-	-	7 ± 17	ns
meso 2,3-Butanediol	39 ± 24 a	54 ± 42 ab	73 ± 38 b	**
1,2-Propanediol	-	2 ± 6	4 ± 8	*
1,2-Propanediol + 1-ethyl-1H-Pyrrole-2-carboxaldehyde [c]	-	-	1 ± 3	ns
1-Nonanol	-	2 ± 6	19 ± 7	***
Benzyl alcohol	6 ± 9	6 ± 16	4 ± 6	ns
2-Phenylethyl alcohol	570 ± 368	1107 ± 2395	555 ± 370	ns
∑ *Higher minor alcohols*	980	1431	943	

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Table 2 - Qualitative and semi-quantitative profile of volatile compounds (µg/L) (mean ± standard deviation) detected in Caluso *Passito* DOC, Cinque Terre Sciacchetrà DOC and *Passito* di Pantelleria DOC wine typologies (2/3).

Compound (µg/L)[a]	Caluso Passito DOC	Cinque Terre Sciacchetrà DOC	Passito di Pantelleria DOC	Signif.[b]
Aldehydes				
Hexanal	3 ± 6	1 ± 4	2 ± 3	ns
Octanal	1 ± 2	1 ± 2	-	ns
Nonanal	3 ± 10	2 ± 6	-	ns
Decanal	-	4 ± 13	-	ns
Benzaldehyde	553 ± 349 b	327 ± 757 b	17 ± 17 a	**
∑ *Aldehydes*	560	334	19	
Ketones				
3-Penten-2-one	1 ± 2	1 ± 2	1 ± 2	ns
2-Heptanone	7 ± 9 b	2 ± 3 a	1 ± 1 a	**
3-Octanone	-	-	1 ± 2	ns
3-Hydroxy-2-butanone	18 ± 13	19 ± 24	10 ± 10	ns
2-Octanone	-	-	2 ± 5	ns
3-Nonanone	1 ± 1	1 ± 1	-	ns
2-Nonanone	15 ± 16 b	8 ± 9 a	2 ± 3 a	**
Acetophenone	6 ± 15	-	-	ns
∑ *Ketones*	46	30	15	
Acids				
Acetic acid	698 ± 243	797 ± 275	735 ± 421	ns
Isobutyric acid	13 ± 33	19 ± 19	3 ± 7	ns
Butanoic acid	3 ± 7 a	5 ± 6 ab	14 ± 11 b	***
3- + 2-Methyl-butanoic acid [c]	9 ± 12 ab	30 ± 25 a	19 ± 14 b	*
Hexanoic acid	75 ± 65	62 ± 35	67 ± 40	ns
Octanoic acid	87 ± 87	47 ± 36	86 ± 136	ns
Decanoic acid	-	-	2 ± 5	ns
Benzoic acid	-	1 ± 1	-	ns
∑ *Acids*	885	960	926	
Azotate compounds				
1-Ethyl-1H-pyrrole-2-carboxaldehyde	-	1 ± 4	1 ± 5	ns
N-(3-Methylbutyl)acetamide	-	1 ± 4	-	ns
∑ A*zotate compounds*	-	2	1	
Sulphur compound				
3-(Methylthio)-1-propanol	-	-	2 ± 4	ns
Hydrocarbons				
Linear alkane (C11)	9 ± 9	36 ± 32	-	***
Ethyl benzene	3 ± 13	3 ± 8	-	ns
Xylene isomers [d]	11 ± 43	2 ± 9	1 ± 1	ns
Indane	1 ± 4	2 ± 7	-	ns
Dehydro-*p*-cymene	9 ± 12	1 ± 3	-	ns
∑ *Hydrocarbons*	33	44	1	
Phenols				
4-Ethyl-guaiacol	1 ± 3	18 ± 58	1 ± 4	ns
4-Ethyl-phenol	1± 1	4 ± 11	1 ± 2	ns
∑ *Phenols*	1	22	2	
Varietal volatile compounds				
C6 compounds				
1-Hexanol	487 ± 141 b	416 ± 190 b	218 ± 41 a	***
trans 3-Exen-1-ol	6 ± 2 b	5 ± 4 b	1 ± 2 a	***
cis 3-Exen-1-ol	3 ± 3 a	6 ± 15 a	14 ± 7 b	**
∑ *C6 compounds*	496	427	233	
Terpenoids				
2,2,6-Trimethyl-6-vinyltetrahydropyran	5 ± 5 a	6 ± 7 a	13 ± 8 b	**
β-Myrcene	-	-	2 ± 3	ns
1,4-Cineole	82 ± 49	3 ± 8	-	***
Limonene	-	8 ± 30	7 ± 4	ns
p-Cymene	5 ± 6	1 ± 3	-	**
Cis-Rose oxide	1 ± 2	-	9 ± 7	***
trans-Furan linalool oxide	-	6 ± 15	20 ± 29	**
Nerol oxide	2 ± 8 a	8 ± 16 a	40 ± 23 b	***
cis-Furan linalool oxide	1 ± 1a	1 ± 2 a	8 ± 3 b	***
Terpineol acetate	-	1 ± 2	11 ± 8	***
Linalool	2 ± 8 a	87 ± 89 b	842 ± 231 c	***
p-Menth-3-en-1-ol	1 ± 2	-	-	ns
4-Terpineol	334 ± 331 a	19 ± 22 a	2 ± 3 b	***
ho-Trienol	1 ± 1a	1 ± 1 a	63 ± 36 b	***
2,6-Dimethyl-5,7-octadien-2-ol	-	-	1 ± 2	ns
Geranial	1 ± 4	-	-	ns
α-Terpineol	2 ± 7 a	35 ± 24 a	200 ± 100 b	***
trans-Pyran linalool oxide	-	-	6 ± 4	ns
cis-Pyran linalool oxide	-	-	1 ± 1	ns
Citronellol	-	3 ± 10	36 ± 15	***
Nerol	-	-	20 ± 12	ns
cis-Geranylacetone	1 ± 4	1 ± 3	-	ns
Geraniol	-	1 ± 3	40 ± 24	***
∑ *Terpenoids*	435	180	1321	

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Table 2 - Qualitative and semi-quantitative profile of volatile compounds (µg/L) (mean ± standard deviation) detected in Caluso *Passito* DOC, Cinque Terre Sciacchetrà DOC and *Passito* di Pantelleria DOC wine typologies (3/3)

Compound (µg/L)[a]	Caluso Passito DOC	Cinque Terre Sciacchetrà DOC	Passito di Pantelleria DOC	Signif.[b]
Bouquet/Ageing volatile compounds				
Furanic derivatives				
2-Pentylfuran	-	-	3 ± 7	ns
Furfuryl formate	3 ± 14	-	2 ± 4	ns
Ethyl 2-furoate	24 ± 8 b	9 ± 8 a	1 ± 1 c	***
2-Furanmethanol	-	1 ± 1	7 ± 15	**
2-Furfural	688 ± 241 b	193 ± 211 a	152 ± 133 a	***
5-Methyl-2-furfural	26 ± 21 b	3 ± 10 a	2 ± 8 a	***
∑ *Furanic derivatives*	741	205	166	
Lactones				
γ-Butyrolactone	22 ± 9 b	32 ± 19 c	13 ± 6 a	***
cis-β-methyl-γ-octalactone	1 ± 2	-	1 ± 1	*
γ-Nonalactone	-	-	4 ± 3	ns
∑ *Lactones*	23	32	18	
Acetals				
2,4-Dimethyl-1,3-dioxane	4 ± 11	-	-	ns
1,1-Diethoxy-pentane	3 ± 7	1 ± 4	1 ± 1	ns
1,1-Diethoxy-3-methylbutane	18 ± 16 b	6 ± 13 a	3 ± 3 a	***
2-Ethoxytetrahydro-2H-pyran	1 ± 3	-	-	ns
∑ *Acetals*	26	7	4	
C13-norisoprenoids				
cis-Vitispirane	33 ± 12	70 ± 33	-	***
trans-Vitispirane	19 ± 8	35 ± 19	-	***
Actinidol ethil ether	10 ± 9	-	-	ns
β-Damascénone	3 ± 2 a	8 ± 8 b	4 ± 1 a	*
∑ *norisoprenoids*	65	113	4	

[a] Semi-quantitative contents calculated from peak area/IS concentration
[b] Values followed by different letters significantly differ for $p<0.05$ (ns = not significant; *$p<0.05$; **$p<0.01$; *** $p<0.001$)
[c] Coeluition ; [d] ∑ Isomer; [e] Compound not detected

MAROTO *et al.,* 2005) thus stable preservation of these esters was observed in CP DOC white *Passito* wine.

Among higher alcohol acetates, isoamyl acetate was present in all wines. It is one of most powerful odorants of wines with a distinctly banana-like fragrance, also a key compound in the aroma of a red wine such as Pinotage (VAN WYCK *et al.*, 1979). A higher average values of this volatile component was detected in CTS wines with significant differences among the other two *Passito* wines ($p\leq0.001$). Isoamyl acetate was observed in other white aged wines too (LAMABROPOULOS and ROUSSIS, 2007). In each *Passito* wine family, isoamyl acetate showed highest values among acetates, derived from yeast amino acid metabolism. This compound has been reported having low odour threshold value of 30 µg/L in 10 % ethanol (FERREIRA *et al.*, 2002).

Similarly to the ethyl esters, we observed that the signals of higher alcohol acetates decreased during aging in all wines (data not shown). This trend could indicate that these compounds, produced by enzymatic reactions in excess of their equilibrium concentrations, hydrolyse during storage until equilibrium with their corresponding acids and alcohols is reached. Moreover, the difference in total level of semi-quantitative content of ethyl and acetate esters in CP and CTS wines compared to PP wines can be explained by the condition of fermentation of these wines at lower temperatures that, according to the literature (CLARKE and BAKKER, 2004), favour the formation of acetates and esters.

Among dicarboxylic acid ethyl esters, diethyl succinate was significantly in relative different levels in CP wines compared to the other *Passito* wines. Contrarily, as generally reported in white wines (KARAGIANNIS *et al.*, 2000; FRANCIOLI *et al.*, 2003), the relative amount of diethyl succinate decreased in all *Passito* wines during aging.

Higher alcohols are formed during yeast fermentation by two different pathways, in particular, from carbohydrates and amino acids. These compounds were the second most abundant group of volatile components in all *Passito* wines, in particular 2-methyl-1-propanol and 2-methyl-1-butanol. CP and CTS wines exhibited the highest levels of these two alcohols with significant differentiation ($p\leq0.01$ and $p\leq0.001$, respectively) compared to PP wines. Factors causing higher alcohols in these northern *Passito* wines can be explained by a faster fermentation rate (RIBÉREAU-GAYON *et al.*, 2000) or since the must, obtained from more overripe grape such as PP wines, is richer in glutamic acids, from which yeasts produce lower levels of fusel alcohols and other volatile by-products (KLIEWER, 1968). Among normal fatty acids in all wines it was found from acetic

acid to octanoic acid, which production depends on the composition of the must and fermentation conditions. By comparing the semi-quantitative data expressed as μg/L of internal standard concentration with those reported in bibliography, an underestimation of major fermentation compounds such as higher alcohols and organic acids is evident.

2,3-Butanediol isomers, detected in this study, could be produced both from reduction of diacetyl (BARTOWSKY and HENSCHKE, 2004) and by exposure of the grapes to the sunlight used in these naturally sweet wines in comparison to fortified sweet wines (VERSINI *et al*., 1999). 2,3-Butanediol meso, was found in relatively high content in PP. This compound was positively correlated with honey/caramel and sherry-like/oxidized characteristics in Canadian ice wines (CLIFF *et al*., 2002). 2-Ethyl-1-hexanol was present overall in CTS and CP wines, while 1-octen-3-ol was only present in PP wines.

Among aldehydes, benzaldehyde has a bitter almond odour. CP and CTS wines showed significantly dominant levels of benzaldehyde ($p \leq 0.01$) than PP wines. Benzaldehyde could be related to the desirable noble rot attack on grapes and consequently a contemporaneous accumulation of 2-furfural (GOETGHEBEUR *et al*., 1992), as found particularly in CP wines. Benzaldehyde is related to an important key aroma contribution of cherry-jam flavour, as reported by VERSINI *et al*. (1999), in Amarone DOC wines with level up to 200 μg/L.

Among varietal aroma components, terpenoidic composition was detected principally in PP wines, derived from Muscat of Alessandria aromatic cultivar. Linalool is the floral representative monoterpene alcohol of this special sweet wine, followed by a high content of α-terpineol, ho-trienol, geraniol, linalool oxides, citronellol and nerol, as already reported in the literature (DI STEFANO, 1986; RIBÉREAU-GAYON *et al*., 2000).

Moreover, *cis*-rose oxide, was also identified for the first time in this study in PP and CP wines, considered a key floral aroma with a low threshold, typical of Gewurztraminer wines (GUTH, 1997). The other two sweet white *Passito* wines are made from non-aromatic grapes. Thus, a limited number and low abundance of monoterpenes were generally present in these wines. Nevertheless, the volatile profile of CP is characterized by 4-terpineol and 1,4-cineol. In the present work, it was observed an decreasing of the level of the 4-terpineol during the aging (data not shown). This component is considered with a spicy-balsamic and resinous/floral scent at high amount such as 400-500 μg/L in wines, detected also in *Passito* Amarone DOC wines (VERSINI *et al*., 1999).

Finally, 2,2,6-trimethyl-6-vinyltetrahydropyran, another monoterpene, was detected in all *Passito* wines. It is known as a decomposition product of linalool, formed at low pH and present even in some distilled Gewurztraminer grapes (DEMYTTENAERE *et al*., 2003).

Norisoprenoids are volatile compounds formed during maturation and conservation of the wines because of oxidative degradation of carotenoids that are unstable in the presence of oxygen, elevated temperatures and exposure to sunlight. Among odorant norisoprenoids, ß-damascenone was identified in all types of wines studied ($p \leq 0.05$) and found even in other wines, such as sweet Fiano wines (GENOVESE *et al*., 2007) and in some aromatic wines such as Gewurztraminer and Riesling wines. This molecule is described having a flower-like odour like that of exotic flowers (DEMYTTENAERE *et al*., 2003) with a low threshold, 0.05 ppb, in wine (FERREIRA *et al*., 2002).

In CTS and CP wines, an isoprenoid, vitispirane is considered as a volatile compound with a camphor-like odour. Its isomers were found only in CP and CTS wines. It was also identified in sparkling wines (FRANCIOLI *et al*., 2003) and in many varieties of grapes and wines, generated during aging by acid chemical hydrolysis from multiple precursors such as carotenoids (SCHNEIDER *et al*., 2001; FERREIRA and DE PINHO, 2003). These components were, in fact, absent in the free volatile fraction of younger PP wines.

Another bouquet class, the furanic compounds, also useful as a wine age indicator, is important in sweet wines since they arise from the degradation of sugars, above all in CP wines. As already mentioned, 2-furfural was observed in all three typologies of sweet wine even if relatively high levels were found in CP wines compared to other *Passito* wines ($p \leq 0.001$), confirming the increase of this compound in wines with longer storage. This component seems to have an important contribution to the aroma, if its level is higher with respect to its odour threshold (5 μg/L) (FERREIRA *et al*., 2002). 3-Methyl-γ-octalactones are most important to the sensory characteristics of wines aged in oak wood. Some γ-lactones are formed by cyclisation of the corresponding γ-hydroxy-carboxylic acids. The predominant one was the C4-lactone (γ-butyrolactone) in all sweet wines, and probably arises from glutamic acid or related compounds (GENOVESE *et al*., 2007), whose sensory role seems to contribute to the aroma of Sherry wines (UGLIANO and HENSCHKE, 2009).

Moreover, four heterocyclic and non- heterocyclic acetals, such as 1,1-diethoxy-3-methyl-butane, that was always the predominant detected in all typologies, were found in CP wines. Also in grappa and liquors these

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molecules as recently identified (MASINO *et al*., 2009; FAN and QIAN, 2006).

CONCLUSIONS

This study was conducted as an exploratory effort to define a typical profile for some Italian sweet white *Passito* wines. In addition we attempt to define the physico-chemical characteristics, color and volatile components of the three types of dessert wines. It was possible to define distinctive elements and peculiarities that link each family of products to its own territory of origin, to the raw material and to the technology employed during production. Moreover, all these aspects were quite well retained even during storage and bottle aging in each of the sweet wine types, thus creating elements able to valorise the commercial aspects of these relatively highly priced wines.

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