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ORIGINAL ARTICLE

ANALYSIS OF SELECTED CHEMICAL PARAMETERS IN PIEMONTESE WINES

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ABSTRACT

Background. Piemontese wines are well known and valued all over the world. The most popular of them are *Barolo* and *Barbaresco* wines. However, in Poland, they are still little known and only now are being gradually introduced to a wider range of consumers.

Objective. The aim of this study was to evaluate the content of inorganic anions, minerals, sugars and glycerol of Piemontese wines from micro-region Langhe, classified as DOCG („Denominazione di Origine Controllata e Garantita", ie. controlled designation of origin guaranteed) and DOC („Denominazione di Origine Controllata", ie. controlled designation of origin) products.

Material and Methods. Seven types of red wines and one type of white wine were tested. High Performance Ion Chromatography with conductometric detection (HPLC-CD) was used to measure the content of inorganic anions, ie. fluorides, chlorides, sulfates and phosphates. Flame atomic absorption spectrometry (F-AAS) was used to measure the content of minerals, ie. magnesium, calcium, sodium, copper, potassium, zinc and iron, while High Performance Liquid Chromatography with charged aerosol detection (HPLC-CAD) was used to measure the content of glycerol and sugars, ie. fructose, glucose and sucrose.

Results. Our studies show that although Piemontese wines are characterized by a relatively low content of minerals in comparison with the wines from other regions, they contain a lot of ingredients that have beneficial effects for human health. Moreover, we observed that the studied wines contain particularly high concentration of inorganic ions – phosphates and fluorides. Furthermore, all tested red wines show far reaching similarities in their chemical properties, which is possibly a direct consequence of using in their production locally cultivated grape varieties.

Conclusions. Analysis of the wines from the Piemont region, classified as DOCG, DOC, confirmed that these are dry wines of a high quality.

Key words: *alcoholic beverages, wine, food analysis, food labelling*

STRESZCZENIE

Wprowadzenie. Wina piemonckie są znane i cenione na całym świecie. Wśród najpopularniejszych wymienia się wina *Barolo* i *Barbaresco*. W Polsce wina piemonckie są nadal mało znane i wprowadzane dopiero do dystrybucji.

Cel. Celem badań była ocena zawartości anionów nieorganicznych, składników mineralnych oraz cukrów i glicerolu w winach piemonckich, klasyfikowanych jako wina DOCG („Denominazione di Origine Controllata e Garantita", tj. o kontrolowanym oznaczeniu gwarantowanego pochodzenia) i DOC („Denominazione di Origine Controllata", tj. o kontrolowanym oznaczeniu pochodzenia) z mikroregionu Langhe.

Materiał i metody. W siedmiu czerwonych i jednym białym piemonckich winach oznaczano zawartość: składników mineralnych, tj. magnezu, wapnia, sodu, miedzi, potasu, cynku i żelaza z użyciem metody absorpcyjnej spektrometrii atomowej (ASA), anionów nieorganicznych, tj. fluorków, chlorków, siarczanów (VI) i fosforanów (V) z użyciem metody wysoko-

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sprawnej chromatografii jonowej z detekcją konduktometryczną (HPIC-CD) oraz glicerolu i cukrów, tj. fruktozy, glukozy i sacharozy z użyciem metody wysokosprawnej chromatografii cieczowej z detekcją wyładowań koronowych (HPLC-CAD).
Wyniki: Badania wykazały, że wina piemonckie mogą być korzystnym źródłem składników prozdrowotnych, choć w porównaniu z innymi winami z całego świata charakteryzują się niską zawartością składników mineralnych. Wykazują także specyfikę pod względem wysokiej zawartości anionów nieorganicznych, tj. fluorków i fosforanów. Odnotowano równocześnie duże podobieństwa w składzie chemicznym win czerwonych, co może być charakterystyczne dla danego rejonu upraw winorośli.
Wnioski. Analiza win piemonckich, klasyfikowanych jako DOCG i DOC potwierdziła, że są to wina wysokojakościowe i wytrawne.

Słowa kluczowe: *napoje alkoholowe, wino, analiza żywności, znakowanie żywności*

INTRODUCTION

Wine has been accompanying man since ancient times. For centuries it has been valued for its taste, used as a source of nutrients and played a role in many magical rituals. [5]. It is a complex water-based mixture of many compounds, such as ethanol, glycerol, sugars, organic acids, esters, aldehydes, and organic and inorganic ions [19, 26, 29, 33]. The composition of a wine – and hence, its taste and quality – depends on multiple parameters. These include, among others, variety of grapes, degree of their ripeness at the time of harvesting as well as a number of environmental factors, such as location of a plantation, type of soil and weather conditions [14, 24, 30, 33].

Quality of wine is determined by the concentration of glucose, fructose and glycerol, together with other components in grape juice [2, 4]. During alcoholic fermentation, more than 90% of sugars contained in grapes are being converted into ethanol and carbon dioxide, and the rest is subject to glycerol-pyruvate fermentation, which results in production of glycerol. Glycerol is one of the most abundant components present in wines; especially in dry ones [16]. Glycerol enhances taste characteristics, defines texture of a wine, and as such its bouquet, taste and smell [22, 32]. Glycerol formation during fermentation process is determined by many factors, including yeast strain, source of nitrogen, sulfuration, fermentation temperature, pH and aeration rate [16]. As glycerol content is used traditionally as a quality indicator, it can be added to a wine in order to mask its low quality [21].

Grape musts and grape pulp usually contain equal or very similar amounts of glucose and fructose [3]. The strains of *Saccharomyces cerevisiae* prefer mostly glucose, whereas other species, such as *Candida* or *Stellata, Zygosaccharomyces balii* prefer fructose [2, 3]. The amount of sucrose found in wines is normally insignificant. Its concentration depends on a variety of grapes from which the wine is made, age of a plantation and environmental conditions [15].

Based on a concentration of so-called residual sugars (which are the sugars remaining after fermentation of grape juice), wines are classified according to their degree of sweetness. They are divided into dry, semi-dry, semi-sweet, sweet and very sweet [2]. In some countries, it is allowed to add sugar to unfermented grape musts in order to increase the alcohol content after fermentation (chaptalization process) [33].

Wine quality is significantly affected by inorganic ion content, which may determine how harmful or even toxic these alcoholic beverages can be. Inorganic ion content is usually characteristic for a region of origin of a wine [8, 10]. Mineral content of a wine is determined by the climate and the type of soil in the area where the grapes were cultivated, as well as the type of the fertilizer used [5, 31]. The most abundant minerals in wines are calcium, potassium, sodium and magnesium with the highest concentration reached usually by potassium [14, 28]. Besides the grapes themselves, the materials remaining in contact with the fruits during processing can become a source of the minerals present in wines. In this way, copper, iron and zinc find their way into a wine [14, 27].

Qualitative and quantitative analysis of mineral content can be very helpful in identifying wines and providing information regarding their quality. Furthermore, the concentration of different minerals affects organoleptic properties of wine, such as its taste, freshness, flavor and color [13, 27]. When consumed in moderation, wine can be a rich source of macro- or microelements, which have beneficial effects for human health [29]. Therefore, content of certain minerals in wine requires continuous monitoring, both during the fermentation process, as well as in the final product. It has to comply with the national or professional norms such as the ones provided by OVI (International Organization of Vine and Wine) [27].

In the European Union wines are classified into table and regional ones based on their quality. Moreover, the certificates of PDO (Protected Designations of Origin) and PGI (Protected Geographical Indications) were introduced in EU in 2009. Furthermore some additional divisions within the above mentioned categories are used in certain EU member countries. For example, Italian quality wines (appellation) are divided into

two classes: „Denominazione di origine Garantita e Controllata" (DOCG) and „Denominazione di Origine Controllata" (DOC). On the other hand, table wines are divided into „Indicazione Geografica Tipica" (IGT) and „Vini da Tavola" in accordance with EU directives [1, 17]. Yet further divisions are implemented locally [6].

EU introduced legal regulations of determining the origin of wines in order to achieve a constant improvement in their quality. Similar regulations apply to balsamic vinegar. „Denominazione di origine controllata" (DOC) criteria define, among others, the maximum yield allowed per hectare, the area of production, the minimal alcohol content, the grape varieties, which may be used and sometimes also a minimal period of aging. DOCG wines have to meet all the criteria specified for DOC wines, then they are tested and tasted by the authorized administration employees before bottling [5, 6].

Piemont is one of the most important wine regions in Italy. Piemontese wines are usually created from indigenous grape varieties. The noble *Nebbiolo variety* is used to make red wines, such as *Barolo*, *Barbaresco*, *Barbera*, *Dolcetto* While the local *Cortese* variety is used to produce a white wine, *Gavi* [6, 17].

The aim of this study was to evaluate the content of glucose, fructose, sucrose and glycerol, as well as selected minerals and inorganic anions in the appellate (DOC, DOCG) wines of Piemont from the micro-region Langhe, Italy.

MATERIAL AND METHODS

Eight wines of Piemont, with a DOC or DOCG appellation, derived from Villadoria winery in Italy were analysed. Immediately after uncorking a bottle during the tasting the wine was collected into sterile, hermetically sealed plastic containers, which were then stored in a freezer at -21°C. Just before analysis, wines were thawed at room temperature. We analysed red dry wines, *Barolo* (DOCG, 2005), *Dolcetto Langhe* (DOC, 2011), *Tempra Nebbiolo d'Alba* (DOC, 2011), *Barolo Riserva* (DOCG, 2006), *Fagiano Rosso* (DOC, 2012), *Furet Dolcetto d'Alba* (DOC, 2012), *Barbera Piemonte* (DOC, 2011), as well as a white wine: *Gavi* (DOCG, 2011).

High Performance Ion Chromatography (HPIC), High Performance Liquid Chromatography (HPLC) and Flame Atomic Absorption Spectrometry (F-AAS) methods have been used to determine the chemical parameters in the wines to assess their quality as a DOC and DOCG products.

The content of sugars, glycerol, inorganic anions and minerals was analyzed in the collected wine samples according to previously published methods [4].

RESULTS

Glycerol, fructose, glucose and sucrose in the wines were analyzed using HPLC method (Table 1).

All tested wines contain less than 10 g/L of residual sugars. The smallest concentration of sugars was found in the white wine *Gavi* (3.06 g/L), and the highest one (4.41 g/L) in *Barbera Piemonte* wine. The glucose--fructose ratio (GFR) did not exceed a value of 1 in any case. The largest quantitative GFR was found in *Gavi* white wine, while the smallest value was observed in *Barbera Piemonte* and *Barolo*.

HPIC method allowed us to determine the concentration of fluorides, chlorides, sulphates and phosphates in the tested wines (Table 2). We have measured a comparable and high content of fluorides and chlorides in the studied red wines, whereas the content of these anions (especially fluoride) was significantly lower in the white *Gavi* wine.

Analysis of minerals using the F-AAS method allowed us to determine the concentration of magnesium, calcium, sodium, potassium, copper, zinc, manganese, and iron (Table 3). As was observed with inorganic anions, our analysis showed high level of similarity between all red wines that were tested. Concentration of certain minerals, especially magnesium, sodium and zinc, in the tested red wines was significantly higher than in the white wine. However, the concentration of potassium was the highest in the white *Gavi* wine

Table 1. The content of mono- and disaccharides, and glycerol in Piemontese wines

Wine	Glycerol [g/L] ± SD (n=3)	Fructose [g/L] ± SD (n=3)	Glucose [g/L] ± SD (n=3)	Sucrose [g/L] ± SD (n=3)	F+G+S [g/L]	GFR
Barolo	16.9±1.26	1.89±0.11	0.11±0.01	1.16±0.07	3.16	0.06
Dolcetto Lange	16.45±1.38	2.2±0.13	n.d.	1.08±0.03	3.28	-
Temprà Nebbiolo d'Alba	18.9±1.61	1.62±0.08	0.25±0.01	1.36±0.05	3.23	0.15
Barolo Riserva	18.72±1.47	2.26±0.17	0.3±0.02	1.2±0.04	3.76	0.13
Fagiano Rosso	18.13±0.81	2.9±0.23	n.d.	0.96±0.03	3.86	-
Furet Dolcetto d'Alba	18.74±0.18	2.72±0.26	n.d.	0.88±0.04	3.6	-
Gavi	11.83±0.59	2.11±0.37	0.36±0.01	0.59±0.01	3.06	0.17
Barbera Piemonte	21.8±1.78	2.76±0.01	0.19±0.03	1.46±0.01	4.41	0.07

F+G+S – glucose + fructose + sucrose; GFR glucose/fructose ratio; n.d. – not determined

Table 2. The content of inorganic anions in Piemontese wines

Wine	Fluorides [mg/L]± SD (n=3)	Chlorides [mg/L]± SD (n=3)	Sulfates [mg/L]± SD (n=3)	Phosphates [mg/L]± SD (n=3)
Barolo	122±0.34	52.13±3.43	574.96±2.31	776.96±2.31
Dolcetto Langhe	149.66±1.58	41.63±1.79	496.49±2.25	740.43±2.33
Temprà Nebbiolo d'Alba	124.3±1.4	29.29±2.44	503.57±2.2	575.71±2.29
Barolo Riserva	122.95±1.62	32.86±1.76	465.85±2.34	636.01±2.66
Fagiano Rosso	173.32±1.36	33.83±1.41	467.03±2.58	544.53±2.33
Furet Dolcetto d'Alba	212.73±1.44	95.46±1.61	482.23±1.62	485.98±2.81
Gavi	40.82±0.12	21.07±0.37	436.11±2.16	n.d.
Barbera Piemonte	147.88±2.2	50.62±2.34	497.1±2.32	630.26±0.65

n.d. – not determined

(268.12 mg/L), whereas in the red wines it ranged between 172.38 and 187.99 mg/L.

DISCUSSION

Sweetness of a wine is determined by the interplay of several factors, primarily sugar, alcohol, acids, and tannins [10, 26, 31]. According to the classification of the European Union, dry wines have to contain up to 10 grams of sugar per liter, medium dry 10-30 g/L, semisweet 30-60 g/L, sweet-dessert wines above 60 g/L, and very sweet above 100 g/L [2, 18]. Piemontese wines, tested in accordance to their residual sugars that equal up to 4.41 g/L (Table 1), can be classified as dry wines, regardless of grape variety, which is consistent with the information provided on their labels. Another important indicator used to evaluate wine quality is GFR. It is determined, among other factors, by the time of grape harvesting. Therefore GFR is an indicator of how ripe the harvested fruits were. Specifically, unripe grapes or their pulp have GFR value around 1.5, while fully ripe grapes – around 1 [2, 29].

While fructose is the main sugar in dry wines, it is glucose that is being fermented by the vast majority of yeast. As a consequence, GFR value decreases with the time of fermentation [2, 3]. When GFR value falls below 0.1 and the fructose content becomes ten times higher than the one of glucose, it is a sign of decreasing rate or completion of fermentation process. This indeed was observed in the analyzed wines. Interestingly, the level of sucrose in the studied wines remained at a relatively high level, as compared with the previously published results [2, 3, 15, 16].

All examined red wines were characterized by a relatively high concentration of glycerol, ranging between 16.45 and 21.8 g/L, while we measured only 11.83 g/L of glycerol in the white wine *Gavi* (Table 1). Concentration of glycerol, a secondary product in wine fermentation, usually ranges from 1 to 15 g/L [2]. Yet, in case of fine, mature grapes of a late harvest (autochthonous grape varieties from Piemont), glycerol content often ranges between 20 and 25 g/L [16, 21, 23, 29]. In winemaking, glycerol is usually considered to be positively affecting wine quality. In particular, it determines its taste characteristics [2, 21]. Although it is possible

Table 3. The content of minerals in Piemontese wines

Wine	**Mg** [mg/L] ±SD (n=3)	**Ca** [mg/L] ± SD (n=3)	**Na** [mg/L] ± SD (n=3)	**Cu** [mg/L] ± SD (n=3)	**K** [mg/L] ± SD (n=3)	**Zn** [mg/L] ± SD (n=3)	**Fe** [mg/L] ± SD (n=3)
Barolo	81.42 ±0.28	50.7 ±0.2	228.37 ±0.75	0.577 ±0	178.4 ±2.6	0.329 ±0.02	1.67 ±0.02
Dolcetto Langhe	100.45 ±0.03	41.7 ±0.23	198.1 ±1.61	0.613 ±0	187.99 ±3.87	0.338 ±0	2.19 ±0.01
Temprà Nebbiolo d'Alba	88.49 ±0.33	47.2 ±0.15	146.47 ±0.92	0.415 ±0	174 ±1.19	0.371 ±0	2.39 ±0.02
Barolo Riserva	84.45 ±0.58	53.3 ±0.26	164.4 ±1.39	0.883 ±0	172.38 ±2.46	0.338 ±0	1.33 ±0.01
Fagiano Rosso	76.26 ±0.66	51.1 ±0.31	177.53 ±1.85	0.334 ±0	183.59 ±0.99	0.325 ±	1.81 ±0.02
Furet Dolcetto d'Alba	71.56 ±0.6	61.4 ±0.05	207.27 ±1.22	0.546 ±0	179.64 ±1.68	0.307 ±0	0.82 ±0.01
Gavi	59.52 ±0.03	41.4 ±0.1	122.77 ±0.46	0.474 ±0.002	268.12 ±3.49	0.232 ±0	1.28 ±0.01
Barbera Piemonte	81.86 ±0.74	44.6 ±0.02	220.47 ±0.75	0.543 ±0	182.46 ±1.89	0.341 ±0	2.72 ±0.01

to increase the concentration of glycerol in wines by modifying fermentation conditions, by breeding new strains of wine yeast or by adding *Candida stellata* to fermentation starter cultures, the characteristics of the wines produced with the above mentioned techniques are not satisfactory [21]. Therefore high glycerol content in the tested wines proves their high quality, late harvesting and the use of noble varieties of grapes in their production.

Potassium is the predominant mineral of grape juice. In comparison with the published data regarding wines from different parts of the world [20, 24, 27, 30], we observed relatively low levels of this mineral in the Piemontese wines (Table 3). However the concentration of magnesium that we measured in these wines remained in the range reported previously for wines from other regions [24, 27, 30]. Typically, concentration of calcium and magnesium in a wine is similar [14, 27, 28]. Yet, in our samples, the concentration of calcium was repeatedly measured lower than the concentration of magnesium. Higher concentration of magnesium in a wine may be caused by substances which were used for wine clarification, spraying, and even by the method of grape storage [28].

The sodium concentration in wines is determined primarily by the composition of a soil where vines were grown (with soil salinity depending on the proximity of the sea) and water used for their watering [27, 28]. High sodium concentration is observed in wines originating from Australia, Macedonia and Spain [24, 27]. There is a limit for sodium content in wines, introduced in some countries due to health risk concerns. Specifically, Office International de la Vinge et du Vin recommends that the maximal concentration may not exceed of 60 mg/L [28]. Although zinc is a mineral which is essential for a proper growth of grapes, as well as for enzymatic processes in cells [28], its high content in wines is undesirable. Therefore OVI calls for introducing a limit of 5 mg/L for zinc in wines [20]. In all tested wines we found the concentration of zinc to be lower than the one reported for dry wines from other regions.

The presence of copper in a wine may be a result of soil fertilization, pesticide treatments [17] or the utilization of copper sulfate after fermentation in order to neutralize hydrogen sulfide [29]. In the tested wines, copper concentration was ranging from 0.334 to 0.883 mg/L. It remained within the limits determined by OVI (1 mg/L) [20]. The concentration of copper, as measured in other studies, was found to vary considerably between different wines. The lowest copper concentration was reported for Argentinian wines - 0.1 mg/L. Whereas the concentration of copper exceeding 1 mg/L was found in wines originating from Spain, Macedonia and Jordan [24, 27].

Iron is essential for a proper plant growth, particularly for synthesis of chlorophyll. This element gets into a wine directly from grapes [17], as well as from some materials used in the process of wine production, such as steep vindication equipment [29]. Our analysis shows that Piemontese wines have lower concentration of iron in comparison with the wines from Australia (0.06-11.49 mg/L), Greece (1.1-5.6 mg/L), Macedonia (0.1-4.0 mg/L), Serbia (2.7-12.2 mg/L) [27] or Spain (up to 12 mg/L) [24].

Sulfitation is sometimes necessary during vinification process to prevent oxidation and to inhibit bacterial growth. Yet it could be a source of residual sulfates in a final product. In the studied Piemontese wines, we measured relatively high concentration of sulfates (436.11-574.96 mg/L) in comparison with other analyzed wines [18, 30, 31]. Piemontese wines are characterized by the contents of chlorides ranging from 21.07 to 95.46 mg/L. In comparison, chloride concentration in Bulgarian, French and Spanish wines was higher [18]. There are various sources of chloride in a wine. For example chloride content in a wine is higher when a vineyard is located closer to seaside. Moreover, chlorides can be used to adjust organoleptic properties of wine or may get into a wine from barrels which were disinfected with a sea or salty water [8].

There are many known sources of fluorides in wines. Among them the most significant is fluoride-rich soil, which was exposed to minerals, weathering rocks, dust and rains. Analysis of fluorides content in the tested wines showed that these alcoholic beverages of the Langhe region are characterized by high content of fluorides, as compared to the wines from other regions. It should be noted that the use of HPIC in fluoride markings on beverages is still under debate, with the fluoride ion-selective electrode (ISE) being currently the method of choice. Results obtained with ISE are usually much lower than those obtained using HPIC [12]. Phosphates concentration in wines largely depends on a soil quality, abundance of minerals in it, as well as on chemical pesticides and fertilizers used. We found a rather high concentration of phosphates in Piemontese wines (485.98 to 776.96 mg/L). In comparison *Michalski* [18] reports the concentration of phosphates to be 32.0 mg/L for pink Spanish wines, and up to 123 mg/L for white wines of Hungary. Only in semisweet red Italian wine, phosphates concentration was found to be high (682 mg/L). This may indicate that Italian wines are rich in phosphates in general.

CONCLUSIONS

1. Analyzed wines, belonging to the DOGC and DOC classes, are dry and of a high quality. As a rich source

of various minerals, they can benefit the health of the consumers.
2. Chemical differences between the red wines and the white wine can be explained by differences in the processes of vinification and a use of different strains of grapes.

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Conflict of interest

The authors declare no conflict of interest.

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