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Classification of wines according to several factors by ICP-MS multi-element analysis

E.P. Pérez-Álvarez[a,*], R. Garcia[b,c], P. Barrulas[d], C. Dias[d], M.J. Cabrita[e,*], T. Garde-Cerdán[a]

[a] Instituto de Ciencias de la Vid y del Vino (Gobierno de La Rioja, CSIC, Universidad de La Rioja), Carretera de Burgos, km. 6, 26007 Logroño, Spain
[b] ICAAM - Instituto de Ciências Agrárias e Ambientais Mediterrânicas, IIFA, Universidade de Évora, Núcleo da Mitra, Ap. 94, 7006-554 Évora, Portugal
[c] LAQV, REQUIMTE, Departamento de Química, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, 2829-516 Caparica, Portugal
[d] Laboratório HERCULES, Universidade de Évora, Palácio do Vimioso, Largo Marquês de Marialva 8, 7000-809 Évora, Portugal
[e] Departamento de Fitotecnia, Escola de Ciências e Tecnologia, ICAAM - Instituto de Ciências Agrárias e Ambientais Mediterrânicas, Universidade de Évora, Núcleo da Mitra, Ap. 94, 7006-554 Évora, Portugal

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ABSTRACT

Wines from different grape varieties, geographical zones, soil types, foliar N application, SO_2 addition and oak ageing were analyzed by inductively coupled plasma mass spectrometry (ICP-MS). For this purpose, ICP-MS methodology was optimized. The elements which allowed differentiate wines from studied grape varieties were Sr, Ca, Mg and Mn. Geographical zones were classified according to Sr, Ba, Ni, and Cu. Cs and Pb were the main elements to discriminate the wines from the 3 soil types. Wines from several N foliar doses application were classified by Pb, Ni, Mn and Zn. The content of Cs, Mg, Cu and Pb in wines characterized the SO_2 addition. Finally, wines storage in barrels were differentiate by Na and Cs concentration. The discriminant functions classify 100% of the wines, with the exception of grape variety (97.0%) and oak ageing (95.8%). Consequently, ICP-MS can be applied to classified wines according to viticultural and oenological factors.

1. Introduction

Wine is a complex matrix and, it contains low level concentration of mineral elements (the level of the major elements, Ca, K, Mg, Na, and Mg usually ranged between 10 and 1000 mg/L, minor elements such as Al, Fe, Cu, Mn, Rb, Sr, and Zn are present in the range of 0.1 to 10 mg/L and trace elements, including Ba, Cd, Co, Cr, Li, Ni, Pb, and V are in the range of 0.1–1000 μg/L) (Pohl, 2007). Some of these trace elements, such as Fe, Zn, Cu, Cr, and Se, are essential for the human organism in that they form an integral part of one or more enzymes involved in a metabolic or biochemical process (Pan, Tang, Chen, Wu, & Han, 2013). Elements are also important for efficient alcoholic fermentation and for the prosthetic metallo-enzyme activation of yeast (Rodríguez Mozaz, García Sotro, Garrido Segovia, & Ancín Azpilicueta, 1999). In addition, minerals can contribute to stability and clarity in the wine and its color, and they may affect the organoleptic characteristics of the wine, mainly Zn and Fe (Iglesias, Besalú, & Anticó, 2007), or wine conservation, i.e. precipitation of K and Ca tartrates (McKinnon & Scollary, 1997). Wine minerals are useful because of the possibility of toxicological risk, such as Cd and Pb (Lara, Cerutti, Salonia, Olsina, & Martinez, 2005) and food regulations (Frías, Conde, Rodríguez, Dohnal, & Pérez-Trujillo, 2002).

Mineral content in wines depends on diverse factors: i) natural sources, which reflect the vineyard soil geochemistry and represent the uptake of minerals from soils (Kment et al., 2005). For instance, vineyards located near coastal areas may be influenced by wind from sea or ocean (González-Hernández, Hardisson de La Torre, & Arias Léon, 1996; Frías, Pérez Trujillo, Peña, & Conde, 2001; Sauvage, Frank, Stearne, & Millikan, 2002; Frías, Conde, Rodríguez-Bencomo, García-Montelongo, & Pérez-Trujillo, 2003); ii) external contamination of the grapevine during growth (from inorganic pesticides, herbicides, fungicides and fertilizers applications, plant protection practices or from environmental pollution (Álvarez, Moreno, Jos, Cameán, & González, 2007; Fiket, Mikac, & Kniewald, 2011; Kment et al., 2005; Pohl, 2007); iii) contamination during the winemaking process (prolonged contact with the materials from which pipes, casks and barrels are made, use of additives, fining and clarifying substances…) (Jakubowski, Brandt, Stuewer, Eschnauer, & Görtges, 1999; Fiket et al., 2011; Kment et al., 2005; Lara et al., 2005; Rusjan, Strlič, Pucko, Šelih, & Korošec-Koruza, 2006; Pohl, 2007; Kruzlicova, Fiket, & Kniewald, 2013); and iv) grape maturity and variety, and climatic conditions (González-Hernández et al., 1996). Therefore, knowledge of the mineral content in wines is a growing concern for both consumers and producers (Pohl, 2007).

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[*] Corresponding authors.
E-mail addresses: evapipeal@msn.com (E.P. Pérez-Álvarez), mjbc@uevora.pt (M.J. Cabrita).

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Usually, atomic absorption spectrometry (AAS), atomic emission spectrometry (AES), inductively coupled plasma mass spectrometry (ICP–MS) and inductively coupled plasma optical emission spectrometry (ICP–OES) are the analytical methods used for the determination of elements in foods (Orvini, Speziali, Salvini, & Herborg, 2000). Multielemental analysis in wines, ICP-MS is the most versatile technique, which provides high detection power (due to low detection limits) and high selectivity and sensitivity (Eschnauer, Jakob, Meierer, & Neeb, 1989; Thiel & Danzer, 1997; Murányi & Papp, 1998; Murányi & Kovács, 2000; Castiñeira Gómez, Brandt, Jakubowski, & Andersson, 2004; Jos, Moreno, González, Repetto, & Cameán, 2004; Šperková & Suchánek, 2005; Ivanova-Petropulos et al., 2013, 2015). This technique offers the advantage of analyzing several elements that are present in low concentration in wines at the same time thus considerably shortening the length of time taken for analysis (Williams, Jarvis, & Wills, 1992).

Taking into consideration that wine is a complex water-ethanol mixture, containing various inorganic and organic substances at different levels (Álvarez et al., 2007; Pyrzynska, 2007; Gonzálvez, Armenta, Pastor, & De La Guardia, 2008; Moreno et al., 2008; Voica, Dehelean, & Pamula, 2009; Rodrigues et al., 2011), the largest matrix effects of the samples must be eliminated before the ICP-MS analysis is carried out. For this reason, the stage of pre-treatment of samples in wine mineral analysis can be time-consuming, labor-intensive, and prone to potential contamination problems, as it is a very delicate stage and one that must be properly carried out. The preparation of wine samples for mineral elements determination includes microwave-assisted acid digestion, UV-assisted digestion, thermal digestion in an open reactor, sample dilution, dry and wet ashing, and also less common approaches such as de-alcoholization or analyte separation (Gonzálvez et al., 2008; Ivanova-Petropulos et al., 2016). Moreover, when choosing the method of wine sample preparation, one must consider which procedure provides the best results in the shortest time, with minimum losses and contamination risks, consumes the smallest quantities of reagents and samples and generates the smallest amount of residue and waste (Gonzálvez et al., 2008).

Several studies have looked at the use of spectroscopic methods, such as AAS or ICP-MS, for elemental characterization of wines or for testing wine authenticity, in the case of, for example investigating fraud or adulteration (Brescia et al., 2002). In this study, major, minor and trace elements of 34 AOC Rioja wines (red and white) were determined by ICP-MS. Different calibration methodologies (standard addition and external calibration) and two different approaches for the pre-treatment of samples (direct acidic dilution of the wines and digestion procedures) were compared. Moreover, the element content was used in order to classify the wines according to different grape varieties, geographical zones, soil types, foliar nitrogen application, with or without SO_2 addition and oak ageing.

2. Material and methods

2.1. Reagents and materials

All reagents used were of analytical grade. For sample dilution and preparation of standards, ultrapure water was used (18.2 mΩ, Milli-Q, Millipore-Merck, Darmstadt, Germany). Nitric acid Suprapur® grade (65.0%, Merck) was used for wine digestion, dilutions and for standard solution preparation. Multi-element certificate standard solution ICP-MS-68B-A (100 mg/L) was from High-Purity Standards (Charleston, SC, USA). Tuning solution (ICP-MS Tuning solution), containing 10 µg/L each of Ce, Co, Li, Tl, and Y in a matrix of 2% HNO_3 (Agilent Technologies, Palo Alto, CA, USA), was used for ICP-MS instrument optimization in order to achieve $CeO^+/Ce^+ < 2\%$ and $Ce^{2+}/Ce^+ < 3\%$. The signal was maximized while maintaining 0.9–1.1% CeO^+/Ce^+ during tuning. Ru, Rh, and Ir were used as internal standards for possible instrumental drifts and matrix effects corrections.

2.2. Wine samples

A total of 34 wine samples (8 whites and 26 reds) were collected from AOC Rioja, Spain. All the wines were elaborated in the same cellar following traditional procedures used in AOC Rioja (Spain) and came from the 2016 vintage, except those aged in barrels, which were elaborated in 2015. The white grape varieties were: Viura and Tempranillo blanco. Tempranillo blanco grapes came from five AOC Rioja grapevine growing zones with different edaphoclimatic conditions (La Grajera (2 wines), Valdegón (1 wine), Cenicero (1 wine), Corera (1 wine), and Alfaro (1 wine)), while the Viura grapes came from La Grajera (1 wine). Moreover, a Tempranillo blanco wine from La Grajera was aging in barrels. The red samples were elaborated with Tempranillo, Garnacha, Maturana, and Graciano grape varieties. Tempranillo red wines were from different soils, classified as *Fluventic haploxerepts* (FH), *Typic calcixerepts* (TC), and *Petrocalcic palexeroll* (PP), according to the Soil Survey Staff (2010) classification, and located in Uruñuela (La Rioja). Three wines were elaborated from each of the three soil types (a total of 9 wines). Moreover, urea foliar applications were carried out according to Pérez-Álvarez, Garde-Cerdán, García-Escudero, and Martínez-Vidaurre (2017), and the treatments were made in triplicate (9 wines in total). In addition, wines from Tempranillo, Graciano, Maturana, and Garnacha grape varieties were produced from La Grajera (4 wines). The wines of Tempranillo and Graciano were also aged in barrels (2 wines). Fermentations were carried out without the addition of SO_2 in two Tempranillo wines (2 wines), one of which was aged in barrels.

2.3. Sample preparation

Two different procedures were assayed over the 34 wine samples: acidic wine dilution and acidic digestion. The perfluoroalkoxy (PFA) beakers used for storing and treating the samples were cleaned to avoid contamination by any traces of metal. The containers were treated with 5% HNO_3 and then washed with Milli-Q water.

2.3.1. Dilution

Samples were diluted 10 fold for minor and trace elements (Al, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Cd, Cs, Ba, and Pb) and 100 fold for major elements (Na, Mg, P, K, and Ca) with 2% HNO_3, without any prior preparation.

2.3.2. Digestion

Wine digestion was carried out in the PFA beakers adding 5 mL of each wine and 2 mL of 65% HNO_3; then, the beakers were put on a hotplate between 50 and 70 °C until the samples became colorless because of the digestion of the acid. Once the digestion step had been completed, the solutions were cooled to room temperature. The tempered samples were transferred to PFA volumetric flasks and fulfilled with Milli-Q water up to 50 mL.

2.4. ICP-MS analysis

Multi-element determination was performed on an Agilent 8800 Triple Quadrupole ICP-MS, equiped with a Micromist nebulizer. In accordance with the analytes of interest, the collision/reaction cell was in “no-gas mode”, “He mode”, “O_2 mode” and “NH_3 mode”.

Plasma gas flow rate was 15 L/min and collision and reaction gases flow rates were 4 mL/min, 0.5 mL/min and 1.5 mL/min for He, O_2 and NH_3, respectively. Analyses were optimized at 1550 W forward power and 1.1 L/min carrier gas flow with no dilution or makeup gas. Sampling depth (10 mm) and lens parameters were optimized for highest signal and optimum peak shape while maintaining low oxides.

In “no-gas mode”, selected masses at Q1/Q2 were 23/23 (Na), 24/24 (Mg), 39/39 (K), 101/101 (Ru), 103/103 (Rh), 111/111 (Cd), 115/115 (In), 133/133 (Cs), 137/137 (Ba), 139/139 (La), 140/140 (Ce), 141/141 (Pr), 146/146 (Nd), 147/147 (Sm), 153/153 (Eu), 157/157

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