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# Romanian white wine authentication based on mineral content

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***Abstract***

The aim of this study is to evaluate the usefulness of the mineral analysis of the Romanian white wines for the variety authentication using an ICP-MS method. For this reason were analysed three different varieties of white wines (*Chardonnay, Riesling* and *Feteasca alba)* from the same vineyard. The mineral analysis was conducted on 27 elements (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, K, Li, Mg, Mn, Na, Ni, Pb, Rb, Se, Sr, Tl, U, V and Zn). It seems that the mineral in higher concentration were Li, Zn, Cu, Be and Ni, so we can conclude that these metals are assimilated different by the vine varieties. The Principal Component Analysis was conducted for achieving the influence of each mineral of the wine type.

***Keywords***: authentication, white wine, ICP MS, PCA

## 1. Introduction

The wine quality, according to Marini et al. [1], is based on three factors, which are globally referred to as the "quality triangle": the type of grape (the species of vine – varietals); the climate and the soil (which affect the quality of the grape); the human factor, which includes cultivation techniques, production, preservation and ageing methods. The wine label should be accurate and not misleading the consumer. This geographical origin and variety based wine classification is important in terms of quality and economic reasons. Wines from different regions may differ in quality and process. Reliable assessment of grape variety is necessary to protect consumer from adulteration and false labelling [2,3].

The wine variety can be assessed using some approaches as: sensorial analysis (is keeping the privilege of the most used method for the biological properties of the wine varieties), mineral profile analysis (the vines varieties are accumulating selective some metals, so their authentication can be made using some elements as Li, Ni, Ca and Rb), aminoacid profile (although pedological factors, agrotechnical and the technological causes large variations, the aminoacid nature is dependent on the biological peculiarities of the plant and therefore may contribute to the recognition of the variety of vines) and poliphenolic compounds analysis (Phenolic compounds were identified as relevant markers for the chemotaxonomic differentiation of wines. They are naturally present in the wine due to the content of the grapes as plant secondary metabolites and therefore constituents of wine obtained. Polyphenols are believed to play an important role in recognizing and predicting of wine quality) and volatile profile (Volatile composition is an important factor influencing the quality attributes conclusion aromatic and wine. Some volatile compounds in grape berry are original which are synthesized, but most of them are formed during fermentation of wort and then

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during aging. Parameters that influence wine aroma composition are: composition grape varietal characteristics of the grapes, light intensity, temperature, soil, climate, degree of maturation, cultivation practices [4,5].

The aim of this study is to evaluate the usefulness of the mineral analysis of the Romanian white wines for the variety authentication using an ICP-MS method.

## 2. Material and methods

***Materials.*** 9 samples of white wines – Chardonnay, Riesling and Feteasca albă were purchased from the local market of Suceava, Romania. The samples were from the same vineyard, but from different lots.

***Sample preparation.*** Approximately 1 ml of each wine sample was weighted into PTFE vessels and dissolved in 9 mL 65% $HNO_3$ and 1 mL 30% $H_2O_2$. The digestion procedures were carried out in a micro-wave oven (Spped wave MWS-2, Berghof Products + Instrument Gmbh, Germany) according to instrumental parameters and settings reported previously (in a part Apparatus). Blank solutions were prepared in the same way.

***Reagents and solutions.*** All reagents were of analytical grade. Double deionized water (18 MΩ cm resistivity) produced by a water purification system (Thermofisher, Germany) was used in all solutions. The elements standard solutions were prepared by diluting stock solution of 1000 mg/L of Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, K, Li, Mg, Mn, Na, Ni, Pb, Rb, Se, Sr, Tl, U, V and Zn. Honey samples were digested with concentrated nitric acid (65% $HNO_3$, Sigma Aldrich, Germany) and hydrogen peroxide (30% $H_2O_2$ pure p.a, Sigma Aldrich, Germany).

***Apparatus.*** The mineral elements analysis was performed using an Agilent Technologies 7500 Series (Agilent, USA) system coupled plasma-mass spectrometer. The ICP-MS parameters were: nebulizer 0.9 mL/min, RF power 1500 W, carrier gas 0.92 L/min, makeup gas 0.17 L/min, mass range 7-205 uma, integration time 0.1 s, acquisition 22.76 s. Detector parameters: discriminator 8 mV, analogue HV 1770 V and pulse HV 1070 V.

***Statistical Analysis.*** Statistical analysis was performed using the version 5.1 of the Statgraphics Plus software system. The data corresponding to each variable were analyzed by one-factor analysis of variance (ANOVA). Multiple comparisons were performed using the least significant difference test (LSD) and Fisher ratio (F), and statistical significance was set at $\alpha = 0.05$.

The Principal Component Analysis (PCA) was performed using Unscrambler X 10.1 (CAMO Process AS, Oslo, Norway), all the multi elements were weighted and normalized to perform the cluster analysis. The Principal Components Analysis (PCA) was applied to describe the relations among the multi elements composition.

## 3. Results and Discussion

The ICP-MS was used as a spectrometric method for the simultaneously determination of the 27 elements, after a prior acid mineralization. The determination of the detection limits of each element was used for achieving the capability of the method as a routine analysis method. The limits of detection (LOD) and limits and quantification (LOQ), were calculated with three and ten timed the standard deviation of the blank divided by the slope of the analytical curve, respectively [6,7]. In the table 1 are presented the limit of detection, limit of quantification, precision and recovery for each element studied. It can be observed that the LOD values are ranging between 0.251 – 18.321, while the LOQs are ranging between 0.761 and 385.313 ppt. The coefficient of variation for the 27 elements analysed ranged between 1.21 and 4.89%, fulfilling the required criteria of 5%.The analytical quality control was also verified by the recovery experiments for the 27 selected elements, spiking at two selected concentration levels, 10 and 100 ppm. The recoveries, depicted in Table 1, were in the range of 94–105%.

In the table 2 is presented the multi element composition for the three wine varieties studied (*Riesling*, *Chardonnay* and *Fetească albă*). According to the ANOVA, it seems that eight elements studied do not have a significant variance according to the wine type (Ga, As, Rb, Sr, Cd, Cs, Ba and Tl), the rest of nineteen elements have a significant variance according to the wine type.

The highest value of *Fisher* ratio was observed in the case of Li, Zn, Cu, Be and Ni (*Fisher ratio* > 100) content, so we can conclude that these metals are assimilated different by the vine varieties.

For the all three wine types, it was observed that Na, Ca, Mg and Al were in the biggest concentration (higher than 10 mg/l).

The lowest concentration were observed in the case of As, Ga, Sr, Cd and Tl (lower than 0.1 mg/l). In terms of heavy metals (As, Cd, Cr. Cu, Hg, Fe, Mn, Ni, Pb and Zn) there were not observed concentration higher than those established by European Food Safety Authority [8].

***Table 1.*** LOD, LOQ, precision, recovery for the 27 elements analyzed using ICP-MS

| Analyte | LOD (ppt) | LOQ (ppt) | Precision (CV %) | Recovery (%) |
|---|---|---|---|---|
| Ag | 19.512 | 59.121 | 1.29 | 99 |
| Al | 3.812 | 11.55 | 3.21 | 97 |
| As | 0.751 | 2.276 | 2.75 | 98 |
| Ba | 0.915 | 2.772 | 1.21 | 94 |
| Be | 0.351 | 1.064 | 2.54 | 96 |
| Ca | 3.156 | 9.563 | 4.87 | 96 |
| Cd | 62.624 | 189.751 | 1.95 | 104 |
| Co | 86.254 | 261.35 | 4.09 | 103 |
| Cr | 0.592 | 1.794 | 2.93 | 97 |
| Cs | 0.51 | 1.545 | 1.29 | 105 |
| Cu | 0.346 | 1.048 | 4.21 | 98 |
| Fe | 0.829 | 2.512 | 4.87 | 99 |
| Ga | 0.325 | 0.985 | 2.41 | 99 |
| In | 36.214 | 109.728 | 2.21 | 105 |
| K | 118.321 | 358.513 | 4.89 | 101 |
| Li | 0.271 | 0.821 | 4.21 | 98 |
| Mg | 1.212 | 3.672 | 4.05 | 99 |
| Mn | 0.456 | 1.382 | 4.21 | 99 |
| Na | 115.125 | 348.829 | 3.89 | 98 |
| Ni | 0.261 | 0.791 | 3.26 | 95 |
| Pb | 1.598 | 4.842 | 1.35 | 103 |
| Rb | 0.251 | 0.761 | 2.65 | 97 |
| Se | 1.61 | 4.878 | 1.92 | 104 |
| Sr | 87.916 | 266.385 | 2.98 | 101 |
| Tl | 5.104 | 15.465 | 1.89 | 102 |
| U | 0.924 | 2.8 | 1.98 | 102 |
| V | 0.271 | 0.821 | 1.51 | 99 |
| Zn | 22.659 | 68.657 | 2.98 | 98 |

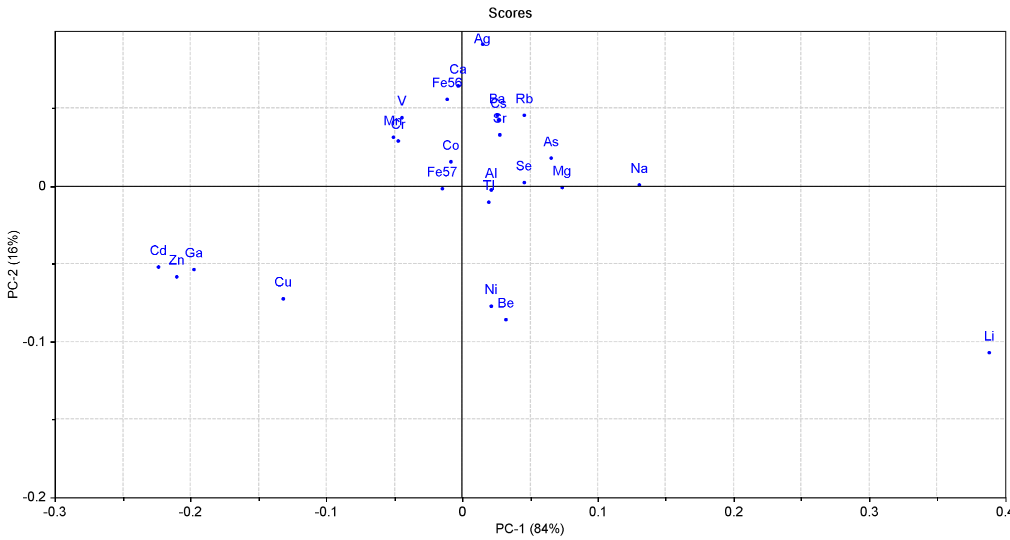


***Figure 1.*** Principal component analysis – scores

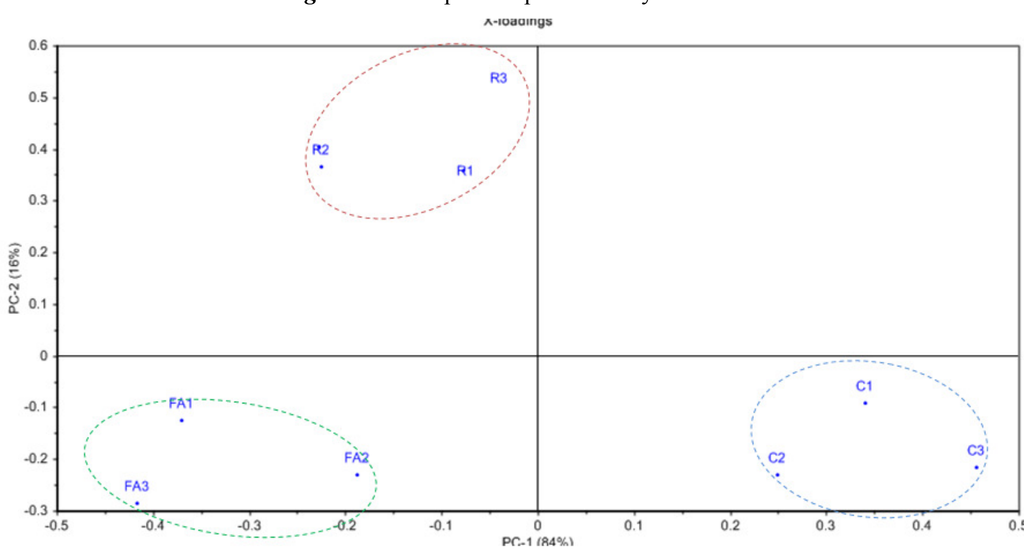


***Figure 2.*** Principal component analysis – loadings

The principal component analysis was conducted to evaluate the global effect of the multi-element composition on the wine varieties, from a descriptive point of view.

In the figures 1 and 2 are presented the scores and compound loadings of PCA analysis performed. It was found that the two principal components (PCs) explained 100% of the variations in the data set. The PC1 explains 86% of the variability and the PC2 explains 14%.

It can be observed that the samples are grouped into three main groups according to their variety (*Chardonnay, Riesling* and *Feteasca alba).* The elements which influence the *Chardonnay* type wine projection are Li, Ni and Be, Cu, Cd, Ga and Zn influence the *Feteasca alba* type wine projection and V, Mn and Cr influence the *Riesling* type wine projection. The element Fe57, Al, Co, Tl and Se, which are very closed to the origins, do no influence the projection.

## 4. Conclusions

The mineral analysis of the *Riesling, Chardonnay* and *Feteasca alba* is a useful tool for the authentication of these wines. The Principal Component Analysis revealed that the mineral analysis is proper for variety type authentication of white wines (each type of wine was placed in a different group). The heavy metals concentration in the three wine have not exceed the EFSA regulations.

**Compliance with Ethics Requirements.** Authors declare that they respect the journal's ethics requirements. Authors declare that they have no conflict of interest and all procedures involving human / or animal subjects (if exist) respect the specific regulation and standards.

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