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THE IMPACT OF THE YEAR OF HARVESTING, DRYING AND LYOPHILIZATION ON THE MINERAL PROFILE OF GRAPE POMACE

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Abstract

Wine pomace is characterized by relatively high mineral content and provides an interesting alternative for foodstuff fortification. Therefore the aim of this study was to determine the mineral profile of dried and lyophilized grape pomace from three different cultivars of *Vitis vinifera* sp.

We investigated grape pomace from Merlot, Silvaner and Pinot Gris from Slovakia, harvested in years 2018 and 2019 as a possible sources for animal and human nutrition. Mineral profile analysis was performed by using the high resolution continuum source atomic absorption spectrometer contrAA 700 for: calcium, phosphorus, magnesium, sodium, potassium, zinc, copper, iron and manganese. In order to calculate basic statistic characteristics, determine significance of differences, and compare the results analysis of variance, one-way ANOVA was performed, using a $P < 0.05$.

The harvesting year strongly affected the mineral content of dried pomaces. Significant ($P < 0.05$) differences in magnesium, sodium, potassium, iron, and manganese concentration were detected in all cultivars. Despite several significant differences ($P < 0.05$) in the mineral profile of some cultivars, the effect of the harvest year was less pronounced in the case of lyophilized samples. There were only few differences ($P < 0.05$) in minerals concentration of dried and lyophilized pomaces, in relation to the method used, mainly in year 2019 and cultivar Merlot. However, the difference between the two drying methods was not confirmed ($P > 0.05$). The phosphorus content of the analyzed pomaces was not affected either by the year of harvesting or drying or lyophilization. These results indicate a significant impact of the harvesting year and grape variety on the mineral profile of grape by-products. Consequently, their composition should be determined on a case-by-case basis.

Overall, it can be concluded that the use of grape pomace as a source of minerals in food industry is promising, but further research is required.

***Key words**: Grape pomace, Wine by-products, Mineral profile, Nutrition, Additives, Fortification, Lyophilization.*

1. Introduction

Wine industry generates large quantities of by-products with problematical disposal, which can lead to serious environmental problems (Dwyer *et al.*, [1], Bekhit *et al.*, [2]). The use of these by-products in feed or food industry as substantial source of certain nutrients and biologically active compounds can contribute to lower production costs and to creating new feed mixtures and sources to improve the nutritive value of animal and human nutrition (Fontana *et al.*, [3]). Nutritional composition, including mineral profile, of grape pomace is variable and mainly influenced by environmental conditions, grape variety (Spanghero *et al.*, [4]) and drying methods (Basalan *et al.*, [5]) among other factors. According to Bennemann *et al.*, [6], wine pomace contains an interesting amount of copper, manganese and iron. Chikwanha *et al.*, [7], state that this by-product contains moderate amount of some minerals (potassium, calcium, phosphorus, copper, iron and sulfur), thus makes it a potentially good source of these nutrients. High concentration of macrominerals (calcium, phosphorus, potassium) and microminerals (copper, iron) in grape pomaces was reported by Hanušovský *et al.*, [8], and Kolláthová *et al.*, [9]. Mineral profile analysis of grape pomaces caried out by Zairati *et al.*, [10], revealed a high zinc, copper, potassium, calcium, magnesium, sodium, phosphorus and iron contents. Based on these results Zairati *et al.*, [10], also suggest to use grape pomace as a dietary supplement for people who need essential mineral elements. But fresh grape pomace is highly perishable due to its high moisture content and water activity (Xiao *et al.*, [11], Goula *et al.*, [12]). Therefore dehydration of wet pomace is a first step before developing further applications (Tseng and Zhao, [13]). Jordan, [14], reported that grape pomace should be dried immediately to prevent spoilage due to mold growth and ensure quality and safety during storage (Taseri *et al.*, [15]). Drying, as one of the most frequently used methods for food and bioproducts preservation, can remove moisture content to a very low level, markedly reduce microbial, enzymatic degradation or any moisture-mediated deteriorative reactions (Xiao *et al.*, [16] , Xiao, [17]). For fully benefit from grape pomace, it is critical to develop drying conditions that can maximize the retention of nutrients while remaining economically feasible (Wang *et al.*, [18]). Several studies have have been carried out to the effects of different drying methods on the biochemical changes of grape pomace (Yinqiang *et al.*, [19], Goula *et al.*, [12], Martynenko and Kudra, [20], Wang *et al.*, [18], Chikwanha *et al.*, [7]). Freeze-drying is considered too expensive and justified only for high-value biomaterials (Wang *et al.*, [18]). Hot air convective drying is still a commonly used industrial method of fruit pomace drying, even though the heat can negatively affect product quality (Chen and Martynenko, [21]).

This study aimed to investigate the impact of harvesting year on mineral composition of grape pomaces, which was confirmed. The second aim, comparison of two drying methods in the context of mineral profile of wine pomaces, resulted in no difference between them.

2. Material and Methods

Grape pomaces were obtained from the experimental farm of the Slovak University of Agriculture located in Kolíňany, farm Oponice, Slovakia (48° 28′ N, 18° 8′ E). The pomaces, as by-products of juice pressing in wine industry, mainly contained of grape skins and seeds, residuals of grape pulp and fragments of grape stalks. In total samples from 3 varieties (Merlot, Silvaner and Pinot Gris) harvested in years 2018 and 2019 were analysed. Laboratory samples were processed in the Laboratory of Quality and Nutritive Value of Feeds at the Department of Animal Nutrition at the Slovak Agricultural University in Nitra.

For the samples two drying methods were applied: hot-air pre-drying at 50 ± 55 ^{0}C and freeze-drying at -54 °C (Freeze Dryer Ilyshin, EU). After the drying was complete, samples were milled by laboratory mill (Fritsch, GER) to pass 1 mm sieve and the content of residual dry matter and ash was detrmined according to EC No 152/2009, [22]. The contents of mineral nutrients were analyzed by high resolution continuum source atomic absorption spectrometer contrAA 700 (Analytik Jena) and 6400 spectrophotometer. The determination of individual elements content was based on the absorptions measured at the following wavelengths: calcium (Ca) at 422.7 nm, phosphorus (P) at 666 nm, magnesium (Mg) at 285.2 nm, sodium (Na) at 589 nm, potassium (K) at 766.5 nm, zinc (Zn) at 213.9 nm, copper (Cu) at 324.7 nm, iron (Fe) at 248.3 nm, and manganese (Mn) at 279.5 nm.

To calculate basic statistic characteristics, to determine significance of differences and to compare results one-way ANOVA and t-test were performed at $P < 0.05$ level. The SAS statistical package was used (SAS Inc., New York City, USA).

3. Results and Discussion

According to Table 1. the year of harvesting strongly affected the mineral content of dried pomaces.

Significant ($P < 0.05$) differences in magnesium, sodium, potassium, iron, and manganese concentration were detected in all cultivars. The haversing year impact in the case of other analyzed minerals in dried pomaces was less evident. Significant ($P < 0.05$) differences were observed only for calcium content in variety Pinot Gris, copper concentration in cultivar Silvan and zinc content in Merlot and Silvan cultivars.

Effect of harvesting year on mineral profile of freez-dried grape pomaces is shown in Table 2.

Table 2 shows that, despite several significant differences ($P < 0.05$) in mineral profile of some cultivars, lyophilized samples were less affected by harvesting year in comparison with air-dried pomaces. Most of the minerals differed significantly ($P < 0.05$) by year only in one cultivar (calcium and copper in Merlot, potassium and zinc in Silvan, manganese in Pinot Gris) and maximum in two varieties (magnesium in Merlot and Pinot Gris, sodium in Silvan and Pinot Gris, iron in Merlot and Silvan). None of the minerals in freeze-dried grape pomaces was significantly affected by year of harvesting in all cultivars. Among all the samples analyzed, both dried and lyophilized, were no significant differences in the concentration of phosphorus by the year of harvest observed. A justification for the dissimilarities between mineral profiles of grape pomaces of the same variety harvested in years 2018 and 2019 could be the difference in the climatic conditions from year to year (weather factors, especially temperature, humidity and solar radiation). Bennemann *et al.*, [6], and Piazzolla *et al.*, [23], state that these extrinsic factors exert great influence on the development, production and quality of grapes and

Table 1. Effect of harvesting year on mineral profile of air-dried grape pomaces

Mineral	Unit	Year	Merlot	Silvaner	Pinot Gris
Calcium	g x kg^{-1} of DM	2018 2019	3.06 ± 0.01 2.73 ± 0.04	2.57 ± 0.09 3.07 ± 0.04	3.87 ± 0.04^a 3.42 ± 0.04^b
Phosphorus	g x kg^{-1} of DM	2018 2019	1.97 ± 0.00 4.81 ± 4.16	1.91 ± 0.08 2.29 ± 0.00	2.40 ± 0.00 2.84 ± 0.00
Magnesium	g x kg^{-1} of DM	2018 2019	0.92 ± 0.01^a 0.70 ± 0.00^b	0.80 ± 0.00^a 0.77 ± 0.00^b	1.09 ± 0.01^a 0.89 ± 0.00^b
Sodium	g x kg^{-1} of DM	2018 2019	0.23 ± 0.01^a 0.14 ± 0.00^b	0.15 ± 0.00^a 0.17 ± 0.00^b	0.22 ± 0.00^a 0.13 ± 0.00^b
Potassium	g x kg^{-1} of DM	2018 2019	10.55 ± 0.07^a 12.35 ± 0.07^b	9.16 ± 0.07^a 19.25 ± 0.21^b	13.15 ± 0.35^a 16.55 ± 0.49^b
Copper	mg x kg^{-1} of DM	2018 2019	7.96 ± 0.00 8.24 ± 0.08	7.95 ± 0.04^a 9.40 ± 0.08^b	11.04 ± 2.77 12.10 ± 0.14
Iron	mg x kg^{-1} of DM	2018 2019	53.80 ± 0.00^a 63.70 ± 0.42^b	47.10 ± 0.00^a 66.85 ± 0.64^b	75.90 ± 0.42^a 84.35 ± 0.35^b
Manganese	mg x kg^{-1} of DM	2018 2019	7.04 ± 0.05^a 7.76 ± 0.02^b	5.32 ± 0.00^a 6.92 ± 0.17^b	9.20 ± 0.37^a 7.66 ± 0.28^b
Zinc	mg x kg^{-1} of DM	2018 2019	40.10 ± 0.99^a 25.25 ± 0.35^b	20.20 ± 0.15^a 30.75 ± 0.07^b	28.60 ± 2.12 31.15 ± 0.35

Legend: DM - dry matter.

grape by-products. Overall the harvesting year was evaluated by several authors as an aspect that strongly influences the levels of nutraceutical compounds in grape pomace (Cecchini, [24], Özcan *et al.*, [25], Lucarini *et al.*, [26], and Sharma *et al.*, [27]).

The effect of drying method on mineral profile of studied grape pomaces is demonstrated in Table 3 (year 2018), and Table 4 (year 2019).

In samples from 2018 significant differences ($P < 0.05$) only in calcium (Merlot), sodium (Silvan, Pinot Gris), iron (Merlot, Pinot Gris) and zinc (Merlot) content were detected. The magnesium, sodium, potassium, copper and manganese concentrations of these pomaces showed no difference ($P > 0.05$), either air or freeze dried. On the other hand, pomaces from 2019 showed more diversity in their mineral profiles according to the method of drying, with Merlot as the most affected cultivar. From this point of view, samples from 2018 seem to be more consistent. However, despite these differences ($P < 0.05$) in mineral profile of dried and lyophilized pomaces, the difference between the two drying methods was not confirmed ($P > 0.05$). As in the case of harvesting year, the drying method also did not affect the phosphorus content of grape pomaces. The pre-treatments, drying method and drying conditions,

Table 2. Effect of harvesting year on mineral profile of freez-dried grape pomaces

Mineral	Unit	Year	Merlot	Silvaner	Pinot Gris
Calcium	g x kg^{-1} of DM	2018 2019	2.69 ± 0.03^a 3.16 ± 0.03^b	2.69 ± 0.01 3.03 ± 0.11	3.52 ± 0.01 3.47 ± 0.03
Phosphorus	g x kg^{-1} of DM	2018 2019	1.91 ± 0.08 1.52 ± 0.00	2.13 ± 0.07 2.26 ± 0.00	2.40 ± 0.00 2.32 ± 0.08
Magnesium	g x kg^{-1} of DM	2018 2019	0.92 ± 0.02^a 0.68 ± 0.01^b	0.85 ± 0.01 0.86 ± 0.00	1.05 ± 0.01^a 0.97 ± 0.01^b
Sodium	g x kg^{-1} of DM	2018 2019	0.26 ± 0.01 0.17 ± 0.00	0.29 ± 0.00^a 0.14 ± 0.00^b	0.20 ± 0.00^a 0.10 ± 0.00^b
Potassium	g x kg^{-1} of DM	2018 2019	11.90 ± 0.14 11.60 ± 0.00	10.40 ± 0.14^a 17.60 ± 0.14^b	13.70 ± 0.14 13.25 ± 0.21
Copper	mg x kg^{-1} of DM	2018 2019	7.64 ± 0.16^a 5.58 ± 0.06^b	8.89 ± 0.30 9.79 ± 0.14	10.90 ± 2.40 16.20 ± 0.14
Iron	mg x kg^{-1} of DM	2018 2019	51.90 ± 0.00^a 22.05 ± 0.07^b	57.10 ± 1.84^a 41.40 ± 2.83^b	96.00 ± 1.41 90.05 ± 0.64
Manganese	mg x kg^{-1} of DM	2018 2019	7.10 ± 0.33 6.65 ± 0.04	5.49 ± 0.38 6.44 ± 0.06	8.45 ± 0.07^a 7.00 ± 0.00^b
Zinc	mg x kg^{-1} of DM	2018 2019	24.75 ± 0.07 27.55 ± 0.78	15.20 ± 0.71^a 26.80 ± 0.85^b	26.50 ± 0.71 31.45 ± 0.35

Legend: DM - dry matter.

Table 3. Effect of drying method on mineral profile of grape pomaces harvested in year 2018

Mineral	Unit	Drying method	Merlot	Silvaner	Pinot Gris
Calcium	g x kg^{-1} of DM	AIR LYO	3.06 ± 0.01[a] 2.69 ± 0.03[b]	2.57 ± 0.09 2.69 ± 0.01	3.87 ± 0.04 3.52 ± 0.01
Phosphorus	g x kg^{-1} of DM	AIR LYO	1.97 ± 0.00 1.91 ± 0.08	1.91 ± 0.08 2.13 ± 0.07	2.40 ± 0.00 2.40 ± 0.00
Magnesium	g x kg^{-1} of DM	AIR LYO	0.92 ± 0.01 0.92 ± 0.02	0.80 ± 0.00 0.85 ± 0.01	1.09 ± 0.01 1.05 ± 0.01
Sodium	g x kg^{-1} of DM	AIR LYO	0.23 ± 0.01 0.26 ± 0.01	0.15 ± 0.00[a] 0.29 ± 0.00[b]	0.22 ± 0.00[a] 0.20 ± 0.00[b]
Potassium	g x kg^{-1} of DM	AIR LYO	10.55 ± 0.07 11.90 ± 0.14	9.16 ± 0.07 10.40 ± 0.14	13.15 ± 0.35 13.70 ± 0.14
Copper	mg x kg^{-1} of DM	AIR LYO	7.96 ± 0.00 7.64 ± 0.16	7.95 ± 0.04 8.89 ± 0.30	11.04 ± 2.77 10.90 ± 2.40
Iron	mg x kg^{-1} of DM	AIR LYO	53.80 ± 0.00[a] 51.90 ± 0.00[b]	47.10 ± 0.00 57.10 ± 1.84	75.90 ± 0.42[a] 96.00 ± 1.41[b]
Manganese	mg x kg^{-1} of DM	AIR LYO	7.04 ± 0.05 7.10 ± 0.33	5.32 ± 0.00 5.49 ± 0.38	9.20 ± 0.37 8.45 ± 0.07
Zinc	mg x kg^{-1} of DM	AIR LYO	40.10 ± 0.99[a] 24.75 ± 0.07[b]	20.20 ± 0.15 15.20 ± 0.71	28.60 ± 2.12 26.50 ± 0.71

Legend: DM - dry matter; AIR - hot-air drying; LYO - freez-drying.

Table 4. Effect of drying method on mineral profile of grape pomaces harvested in year 2019

Mineral	Unit	Drying method	Merlot	Silvaner	Pinot Gris
Calcium	g x kg^{-1} of DM	AIR LYO	2.73 ± 0.04[a] 3.16 ± 0.03[b]	3.07 ± 0.04 3.03 ± 0.11	3.42 ± 0.04 3.47 ± 0.03
Phosphorus	g x kg^{-1} of DM	AIR LYO	4.81 ± 4.16 1.52 ± 0.00	2.29 ± 0.00 2.26 ± 0.00	2.84 ± 0.00 2.32 ± 0.08
Magnesium	g x kg^{-1} of DM	AIR LYO	0.70 ± 0.00 0.68 ± 0.01	0.77 ± 0.00[a] 0.86 ± 0.00[b]	0.89 ± 0.00[a] 0.97 ± 0.01[b]
Sodium	g x kg^{-1} of DM	AIR LYO	0.14 ± 0.00[a] 0.17 ± 0.00[b]	0.17 ± 0.00 0.14 ± 0.00	0.13 ± 0.00[a] 0.10 ± 0.00[b]
Potassium	g x kg^{-1} of DM	AIR LYO	12.35 ± 0.07[a] 11.60 ± 0.00[b]	19.25 ± 0.21[a] 17.60 ± 0.14[b]	16.55 ± 0.49 13.25 ± 0.21
Copper	mg x kg^{-1} of DM	AIR LYO	8.24 ± 0.08[a] 5.58 ± 0.06[b]	9.40 ± 0.08 9.79 ± 0.14	12.10 ± 0.14[a] 16.20 ± 0.14[b]
Iron	mg x kg^{-1} of DM	AIR LYO	63.70 ± 0.42[a] 22.05 ± 0.07[b]	66.85 ± 0.64 41.40 ± 2.83	84.35 ± 0.35[a] 90.05 ± 0.64[b]
Manganese	mg x kg^{-1} of DM	AIR LYO	7.76 ± 0.02[a] 6.65 ± 0.04[b]	6.92 ± 0.17 6.44 ± 0.06	7.66 ± 0.28 7.00 ± 0.00
Zinc	mg x kg^{-1} of DM	AIR LYO	25.25 ± 0.35 27.55 ± 0.78	30.75 ± 0.07 26.80 ± 0.85	31.15 ± 0.35 31.45 ± 0.35

Legend: DM - dry matter; AIR - hot-air drying; LYO - freez-drying.

can significantly influence the quality of final products (Nowicka *et al.*, [28], Wang *et al.*, [18]). But it should be noted that the mineral content of grape pomace has wide variations than other chemical components also due to the strong influence of the edaphoclimatic conditions, viticultural practices and the winemaking process (García-Lomillo and González-SanJosé, [29]). Gunya *et al.*, [30], state that freeze-dried samples are usually known to retain nutrients better compared to high temperature preservation methods because of its minimal levels of oxidation. This is partially consistent with results of the presented study. Furthermore these authors also reported freeze-dried samples to have higher levels of minerals compared to oven drying. Chikwanha *et al.*, [7], studied the impact of preservation technique on the quality of Cape Wineland pomaces and came to a conclusion, that variety, drying methods and their interaction were significant for their mineral profiles. However, in the study of Chikwanha *et al.*, [7], the finding for mineral content of freeze-dried samples is in contrast to finding of Gunya *et al.*, [30].

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4. Conclusions

- Results of the presented study indicate a significant impact of harvesting year and grape variety on the mineral profile of grape pomaces. Consequently, their composition should be determined on a case-by-case basis.

- The selection of drying method should consider type of mineral to be preserved, variety of wine pomace and the cost of drying method.

- Overall, it can be concluded that the use of grape pomace as a source of minerals in food industry is promising, but further research is required.

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