THE MIGRATION OF ELEMENTS IN WINE TREATED WITH BENTONITES

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ABSTRACT

Background. In the present study, the migration of elements from and to bentonites after processing Chardonnay white wine from the Danube Plain region, Bulgaria, was investigated.

Materials and methods. Sixteen of the most common brands and types (natural, activated, sodium, calcium, sodium-calcium) of bentonites available on the Bulgarian market were selected. Inductively coupled plasma-optical emission spectrometry (ICP-OES), inductively coupled plasma with mass spectrometry (ICP-MS), flame atomic emission spectrometry (FAES) and flame atomic absorption spectrometry (FAAS) were used for element content determination.

Results. For some elements (Al, Ca, Cr, Fe, Hg, P, Pb and Si), an increase in their concentration compared to the untreated wine was reported in most cases. For others (B, Cu, Li, Mg, Mn, Ni and Zn), the changes in concentration were more often not significant. Only the K content decreased as a result of treatment with all studied bentonites.

Conclusion. It was possible to conclude that bentonite treatment of wines leads to different changes in the analyzed wine. It is quite possible that with a different dose of bentonite application, results that deviate from those described above would be obtained. This is to be the subject of further research.

Keywords: wine, bentonite, elements, ICP-techniques, FAES, FAAS

INTRODUCTION

Bentonite was introduced into oenological practice about 85–86 years ago for the purpose of processing wines. It is preferred over kaolin. Bentonite has been shown to have specific properties that allow it to be applied at lower doses to clarify and stabilize wines against colloidal turbidity (Ribereau-Gayon et al., 1981).

One of these properties – strong sorption capacity – is directly related to its structure. It is a type of clay made up of multiple layers of repeated crystal structures of Si and Al, around which tetrahedral and octahedral structures are built from the accompanying oxygen atoms and hydroxyl groups, respectively. Water molecules and ions are located between the individual layers, and can be exchanged with the medium. The interlayer spacing in bentonite is around twice or even more than twice as great (about 12–18 Å) as that in kaolin (about 7 Å). It can increase as a result of the sorption of additional amounts of water (Madsen and Müller-Vonmoos, 1989).

From an oenological point of view, another valuable property is the presence of bentonite charge. Under wine conditions, bentonite has a negative electrostatic

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charge (Alexandre et al., 2009). This enables it to flocculate on the basis of mutual neutralization of opposite charges with the cations or other positively charged colloids in the wine. All the properties of bentonite mentioned above contribute to its application in wine practice in order to achieve colloidal stability of white, rosé and red wines (Boulton et al., 1996; Chobanova, 2016; Jackson, 2020; Ribereau-Gayon et al., 2021; Kemp et al., 2022; Bakardzhiyski, 2022; Bakardzhiyski, 2023a, Bakardzhiyski, 2023b).

One result of bentonite's properties is the presence of a strong ion-exchange capacity, due to which it has the opportunity to exchange ions with the environment, and specifically with wine. Some of the wine's ions are sorbed from the bentonite, reducing their concentration. Other ions from the bentonite can be released into the wine, as a result of which their content increases. A study by Nicolini et al. (2004) on this topic only showed a significant decrease in the concentrations of Cu, K, Rb and Zn. The concentrations of all the other elements which they studied increased compared to the untreated wine. Catarino et al. (2008) confirmed the results of the above study. According to them, the content of Cu, K, Rb and Zn significantly decreased, and the authors added boron to this group of elements. The concentrations of Al, As, Ba, Be, Bi, Ca, Cd, Co, Fe, Ga, Ge, Li, Mg, Mn, Mo, Na, Nb, Ni, Sb, Sc, Sn, Sr, Tl, Y, V, W and Zr increased. The works of Shimizu et al. (2022) and Temerdashev et al. (2022) also report significant changes in the elemental composition of wine after bentonite treatment.

According to our previous study (Bakardzhiyski et al., 2023) on the ability to exchange elements in a model environment, prepared according to Resolution OENO 11/2003 (OIV, 2022), almost all 16 studied bentonites showed the ability to release Al, B, Ca, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, P, Pb, Si and Zn. And some of them also released Ag, As, Cd, Hg, La and Ni. Some of the elements for which an increase in concentration was reported in the model environment (as a result of migration from bentonite) are of great oenological importance and have a role in provoking the development of metallic turbidity. Other elements are important for consumer health.

These were the main reasons to continue the research to establish the possibility of migration of elements from the same bentonites to real wine. Such a large-scale study, covering some of the most widely applied commercial brands of bentonites on the Bulgarian market and analysing their effect on Bulgarian wine, has not previously been conducted in Bulgaria.

MATERIALS AND METHODS

Materials

For the purposes of this study, Chardonnay white wine from Bulgaria, Danube Plain region (2021) vintage was used. It was in the storage stage after alcoholic fermentation and no treatment had been applied. Sixteen of the most common bentonites on the Bulgarian market – those most often used in wine cellars in Bulgaria – were selected. Samples were provided by their official distributors in Bulgaria. Their nature, composition and mode of activation are different and are listed in Table 1. The authors' previous research (Bakardzhiyski

Table 1. Bentonites used and sample codes

Bentonite, code	Main composition	Nature	Sample, code
B1	Ca	a	WB1
B2	Ca	a	WB2
B3	Ca	a	WB3
B4	Ca	n+a	WB4
B5	Na	a	WB5
B6	Na	a	WB6
B7	Mixture	a	WB7
B8	Na-Ca	no data	WB8
B9	no data	a	WB9
B10	Na	n	WB10
B11	Na-Ca	no data	WB11
B12	Na	n	WB12
B13	Na	n	WB13
B14	Ca-Na	no data	WB14
B15	Na	n	WB15
B16	Na	n	WB16

Note: Na – sodium bentonite; a – activated; Ca – calcium bentonite; n – natural.

et al., 2023) shows the elemental desorption capabilities for each of investigated bentonites in a model environment. The present study is mainly focused on the ability of each of the bentonites to exchange elements under real wine processing conditions.

Bentonites have strong hygroscopicity due to their clay structure. In order to eliminate the absorbed water and to prepare suspensions with equal amounts of bentonite, portions of all 16 bentonite samples were dried under the conditions described in Resolution OENO 11/2003 (OIV, 2022). From each sample, the dried bentonite was accurately weighed in a beaker using an analytical balance, and the bentonite samples were soaked with deionized water for 12 hours. The porridges obtained were quantitatively transferred in a measuring flask and brought to the exact volume required in order to prepare 5% bentonite suspensions. Each of the investigated bentonite suspension samples was added to 300 mL of wine under constant stirring on a magnetic stirrer for 5 min in an amount providing a dose of 2 g of bentonite per 1 L of wine. The bentonite dose was selected based on the author's previous research (Bakardzhiyski, 2023a; 2023b) on the protein stabilization of Bulgarian white wines. The doses for treatment depend both on the wine variety and the bentonite type, and can vary in a wide interval (from 0.4 to up 5 g/L). The most likely applied doses are from 1.0 to 3.5 g/L. The authors selected the dose of 2.0 g/L as a middle value without aiming to achieve colloidal stability but only to assess elements' sorption from wine and desorption from bentonites when applying different bentonites at a fixed dose. The bentonite-loaded experimental wines were left to rest for 3 days to settle the resulting flocs and compact the sediment. Liquid phase was decanted from the sediment and filtered through a "blue band" filter. The test samples obtained were marked WX, where the "X" codes the corresponding bentonite. Three parallel samples from each wine were mineralized using nitric acid before instrumental measurement. In order to establish the degree of migration of elements from the bentonites to the wine ICP-OES, ICP-MS, FAAS and FAES were used to measure the concentration of the desired elements.

Methods

The content of the elements As, B, Cd, Co, Cu, Fe, La, Li, Mn, Ni, Pb and Zn in the analysed samples was determined by the instrumental technique of inductively-coupled plasma-optical emission spectrometry (ICP-OES) by Varian Vista-MPX CCD Simultaneous. The instrument is equipped with a glass concentric nebulizer and a glass cyclonic spray chamber. The selected element content was measured with reference to the following wavelengths [nm]: As 188.980; B 249.772; Cd 226.502; Co 228.615; Cu 324.754; Fe 259.940; La 333.749; Li 670.783; Mn 257.610; Ni 231.604; Pb 220.353; Zn 213.856.

The content of the elements Ag, Al, Cr, Hg, P and Si was determined using inductively-coupled plasma with mass spectrometry (ICP-MS – Agilent 7900). The mass spectrometer was optimized to obtain minimal values of spectral interference and maximum intensity of the analyte signals. The isotopes of selected elements ([107]Ag, [27]Al, [52]Cr, [201]Hg, ^{31}P, [28]Si) were measured without He-cell.

The content of the macroelements Ca and Mg was determined by the instrumental technique of flame atomic absorption spectrometry (FAAS). The content of the macroelements K and Na was determined by flame atomic-emission spectrometry (FAES). In both cases, a Perkin Elmer Analyst 400 was used. The optimal instrumental parameters recommended by the manufacturer were used under an air-acetylene flame.

Reagents

Concentrated HNO_3, ≥69.0%, Trace SELECT (Fluka, Germany) was used for acid mineralization of the wine samples treated with bentonite.

In order to prepare ICP-OES calibration standard solutions, a multielement standard solution (Certified Reference Material, CPA Chem) containing 100 mg/L of the elements Al, Ag, As, B, Ba, Be, Bi, Ca, Cd, Cs, Co, Cr, Cu, Fe, In, K, La, Li, Mg, Mn, Mo, Na, Ni, Nb, Pb, Rb, Sb, Se, Sr, Ti, Tl, V, U and Zn was used. Single element standard solutions with initial concentration of 10 mg/L of the elements Ag, Al, Cr, Hg, P and Si (Fluka, Germany) were mixed. After appropriate dilution, calibration standard solutions for ICP-MS measurements were obtained. Single element standard solutions of the elements Ca, K, Mg and Na (Fluka, Germany) with an initial concentration of 1,000 mg/L were used after appropriate dilution to prepare calibration standard solutions for FAAS/FAES measurements.

All solutions were prepared with double deionized water (Millipore purification system Synergy, France).

Statistics

Excel software was used for data processing. Concentration data for the elements were obtained as the average of three parallel measurements of each of the three parallel wine samples mineralized.

RESULTS AND DISCUSSION

Macroelements migration

The concentrations of K, Mg and P in the control (untreated) wine W0 and their change as a result of the addition of different bentonites are shown in Figure 1. The concentration of potassium in the control was 474±20 mg/L. The addition of bentonites, regardless of their nature and method of activation, led to a decrease in the concentration of K in all investigated cases. The decrease varied between 14% and 23%. It was most pronounced in the sample with imported bentonite B10, and weakest in the sample treated with bentonite B7. Potassium is the element with the highest concentration in wine. The decrease in the samples was probably due to mutual flocculation between

opposite charges and precipitation of part of the potassium with the bentonite particles, which are negatively charged in the wine.

The concentration of Mg was almost unaffected by the addition of bentonite (compared to the control). Phosphorus did not behave identically in all the experimental samples. In most of them, it was preserved in almost the same concentration as in the starting wine. A more significant deviation was observed in samples WB8, WB10 and WB11, where there was a clear increase in the concentration of this element of about 5 to 8 times compared to the untreated wine sample.

An increase in concentration after bentonite treatment was also observed for the elements Ca and Na (Fig. 2). The Ca concentration found in the control was 12±1 mg/L. It is noteworthy that the activated calcium bentonites (B1, B2, B3) do not release this element into the wine (or at least not to a significant extent). Treatment with them did not lead to a significant change in the concentration of Ca in the wine. This was probably due to the replacement of a large part of the calcium ions with sodium ions in the process of activation.

One piece of indirect evidence for this view is the increase of Na in the wine samples with imported
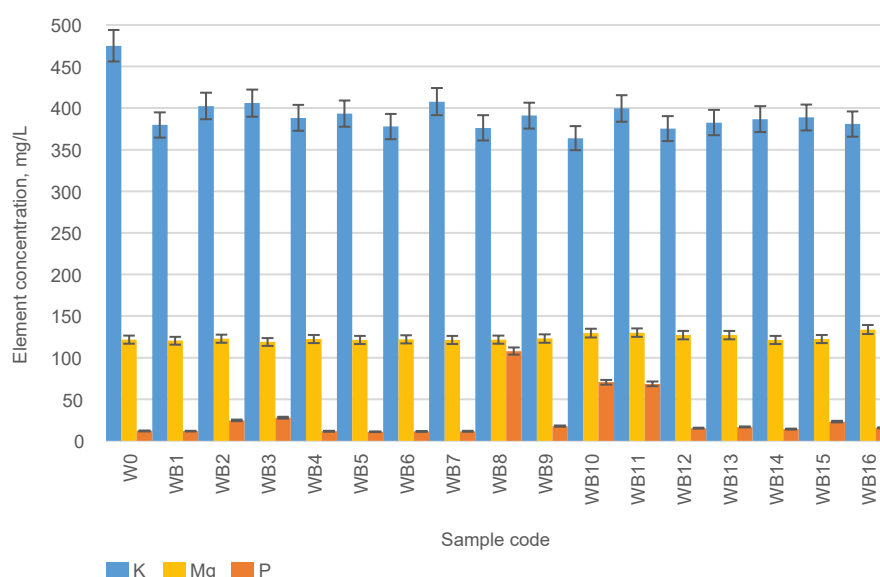
Fig. 1. Concentration of K, Mg and P in control (untreated) wine W0 and wine after the addition of different bentonites (WB1 – WB16). RSD = 2–5%
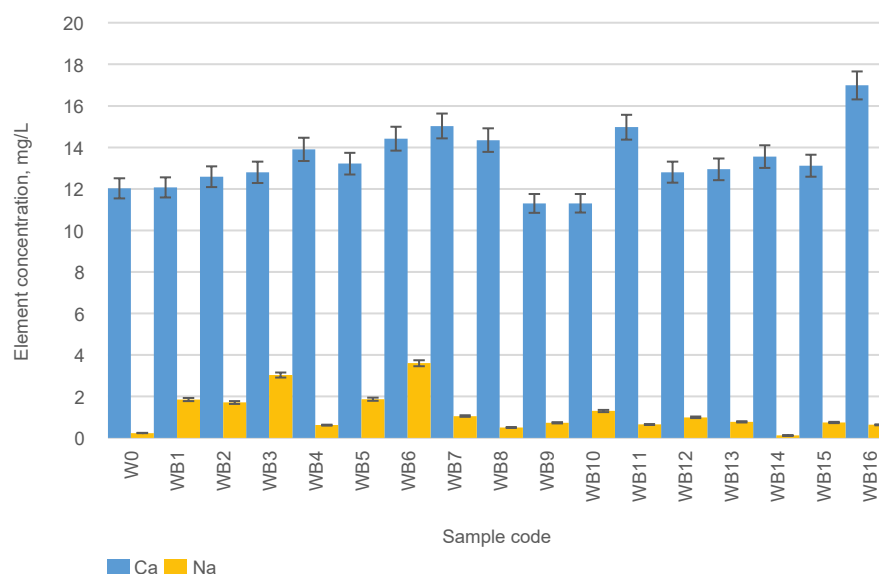
Fig. 2. Concentration of Ca and Na in control (untreated) wine W0 and wine after the addition of different bentonites. RSD = 2–5%

activated bentonites (B1, B2, B3 and B6). As a result of treating the wine with natural calcium or mixed sodium-calcium bentonites, the concentration of Ca increased compared to the control wine. In these samples (WB4, WB6, WB7, WB8 and WB11) the reported increase was about 16–25%. Sample WB16 had the largest increase in Ca concentration (over 40%), This is illogical from the point of view that this bentonite is described by the manufacturer as natural sodium bentonite. Probably, parallel to the sodium cations in its composition, there is also a significant amount of calcium cations, and the bentonite has the ability to actively exchange them with the environment. A decrease in calcium content (by about 6%) was only reported in wines treated with the bentonites B9 and B10.

Almost all activated bentonites, whether sodium or calcium (B1, B2, B3, B5 and B6), enriched the treated wine samples with Na. The concentrations of this element in the treated samples were about 7 to 14 times higher (highest for bentonite B6) than in the control wine (0.24±0.02 mg/L). This change is probably related to the bentonites' activation process. It is important to say that none of the wine sampless treated with these bentonites exceeded the maximum permissible

concentration for Na of 60 mg/L, according to OIV (2022). However, bentonite's ability to release Na into wine should not be overlooked. This would be of particular importance when processing wines from areas located near large bodies of water, which are naturally rich in this element.

Oligo- and microelement migration
The content of B and Si in the analyzed wine samples is shown in Figure 3. The content of B in the untreated wine was 6.5±0.5 mg/L. None of the studied bentonites had any effect on this element. Thus, the boron concentration in analyzed wines remained almost unchanged.

The element Si in the control wine sample was below the detection limit of instrumental measurement (0.76 mg/L). Most of the bentonites released this element into the medium (wine). The most significant enrichment with Si was reported in experimental sample WB8, where its concentration reached 5.7±0.5 mg/L. Significantly lower concentrations were found in samples WB2, WB3, WB10 and WB11, ranging from 1.5 mg/L to 1.9 mg/L. It should be noted that in addition to an increase in the concentration of Si, an increase in the content of Al was also reported in
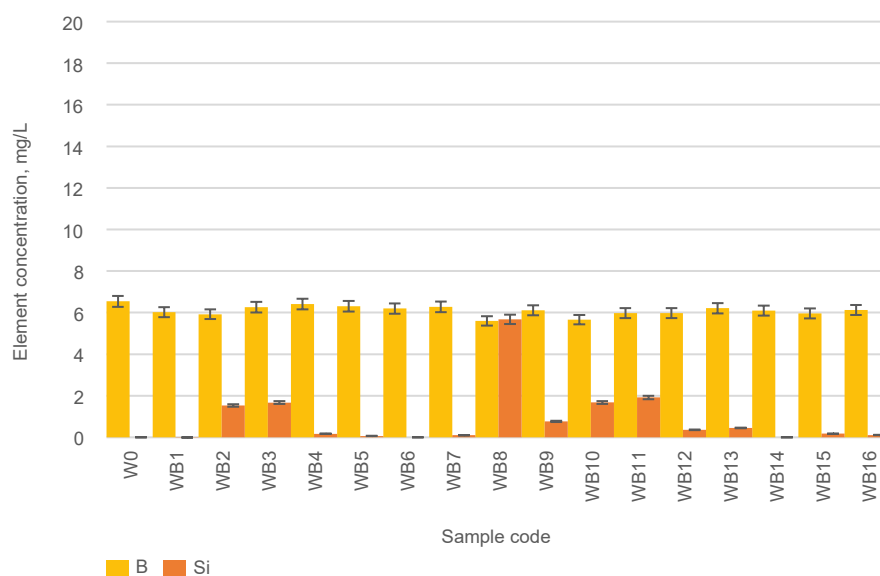
Fig. 3. Concentration of B and Si in control (untreated) wine W0 and wine after the addition of different bentonites. RSD 2–5%

these experimental samples (Fig. 5). This is not surprising given that bentonites are generally known to be hydrated aluminosilicates (Ribereau-Gayon et al., 2021; Chobanova, 2016; Jackson, 2020; Boulton et al., 1996).

The concentrations of some oligoelements in the untreated wine and experimental samples are shown in Figures 4 and 5. These elements are also of great importance for the quality of the wine. They can cause various turbidities from the group of so-called metal haze. In addition, the elements Cu, Fe and Zn are related to consumer health, which is why the content of these elements is subject to strict control and OIV has decreed maximum permissible values for their concentrations in wine (OIV, 2022).

The content of Fe in the untreated wine was found to be 1.10 ± 0.12 mg/L. As a result of treatment with bentonites B1 and B16, the content of this element in the experimental samples increased to the greatest extent – by about 2–2.5 times. In experimental samples WB2, WB9, WB11 and WB15, the addition of the respective bentonites did not significantly change the Fe content. Application of the remaining bentonites resulted in a significant increase in the concentration of this element, by about 25–68%. No test sample (even

WB1) exceeded the maximum permissible value of 8 mg/L Fe, but these results indicate that as a result of colloidal wine stabilization with some bentonites, it is quite realistic to suppose that this limit may be exceeded when the Fe presents at a higher concentration in the untreated wine. This could lead to the development of ferriphosphate or ferritanate turbidity. Taking into account the health effects of increased iron content, it may be necessary to carry out an additional demetallizing treatment in order to decrease its concentration. The described results confirm a preliminary study (Bakardzhiyski et al., 2023) of the possibilities of element transfer between these same bentonites and a model environment, according to Resolution OENO 11/2003 (OIV, 2022). In the cited study, bentonites B1, B16, B4 and B8 showed the greatest ability to release Fe in the model environment.

The Cu content in the control wine was found to be 0.17 ± 0.02 mg/L, which is below the maximum permissible level for this element (1 mg/L) according to OIV (2022). Regarding all the bentonites tested, only B9 showed a significant ability to release Cu into the wine. The Cu content in this test sample was 0.26 ± 0.02 mg/L. The value is also far below the maximum permissible level. Like Fe content, these results
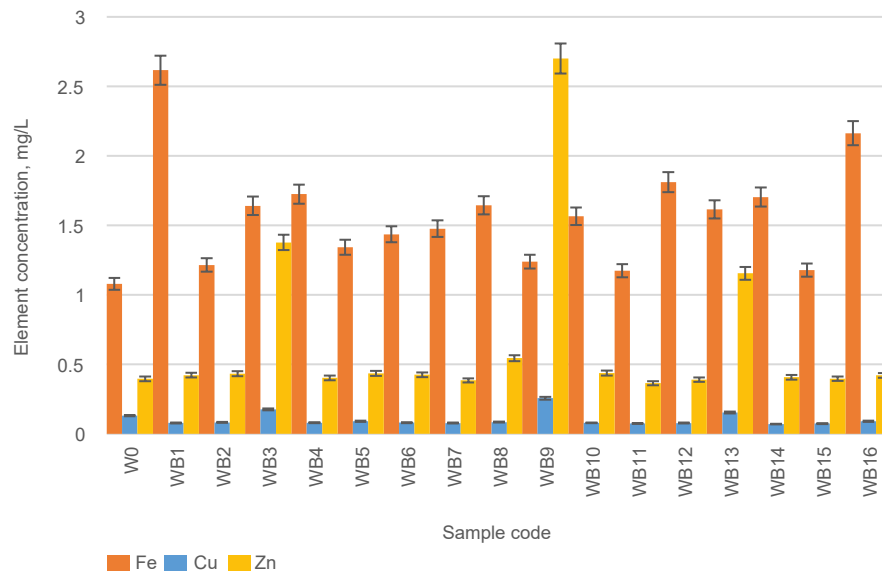
Fig. 4. Concentration of Cu, Fe and Zn in control (untreated) wine W0 and wine after the addition of different bentonites. RSD 2–5%
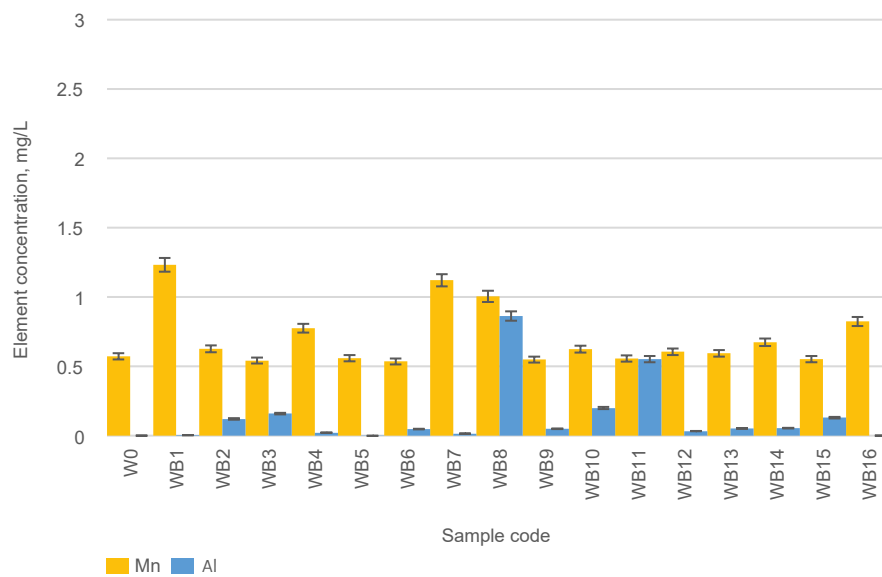
Fig. 5. Concentration of Al and Mn in control (untreated) wine W0 and wine after the addition of different bentonites. RSD 2–5%

should be taken into account when conducting wine processing with this bentonite.

The bentonite B9 showed a very strong ability to release Zn in the wine as well. As a result of treatment with this bentonite, the concentration of this element in the experimental variant WB9 increased by nearly 7 times compared to that in the control (0.40±0.03 mg/L). Despite this strong increase in Zn concentration, the

maximum limit of 5 mg/L according to OIV (2022) was not exceeded. Bentonites B3 and B13 also released zinc into the wine. In the experimental varieties of wines treated with them, an increase in the concentration of Zn by about 3.0–3.5 times compared to the control was found.

The results obtained for Al and Mn transfer in wine following treatment with different bentonites are shown in Figure 5.

Introducing bentonites B1, B7 and B8 led to an approximately two-fold increase in Mn concentration in the experimental wine samples treated with them (WB1, WB7 and WB8) compared to the untreated wine (in W0, the Mn content was found to be 0.57±0.04 mg/L). In the experimental samples WB4 and WB16, increases of 36% and 44%, respectively, were reported compared to the control. The probable reason for this is the ability of the these bentonites to release Mn. In the remaining experimental samples, the concentration of manganese did not change significantly after treatment.

Aluminium was not detected in the control sample W0 (<0.070 mg/L). With the exception of bentonite B16, all other studied bentonites showed the ability to release this element into the wine in greater or lesser amounts. The concentrations were highest in test

samples WB8 and WB11, followed by WB10, WB2 and WB3. These were the same samples in which an increase in Si content was found. The probable reason for the increased Al concentration is the same and will not be commented on again. The maximal permissible concentration of Al in wine is 3 mg/L (OIV, 2022). No concentration exceeding this limit was found in any of the test samples. But it should be taken into account that its concentration in wine may increase after treatment with bentonite.

Trace elements migration
Figures 6, 7 and 8 present the behavior of some trace elements in the analyzed wine samples after their treatment with different bentonites.

Lead was not detected in the control wine sample (<0.31 µg/L). Highly elevated Pb concentrations were found in experimental samples WB3, WB8 and WB9, equal to 0.080±0.006 mg/L, 0.070±0.005 mg/L and 0.050±0.005 mg/L, respectively. Despite the elevated concentrations, none of them exceeded the maximum permissible concentration of 0.1 mg/L (OIV, 2022). In the above-cited study (Bakardzhiyski et al., 2023), bentonites B3 and B9 exhibited the strongest Pb-releasing ability in a model environment. Bentonites B1,
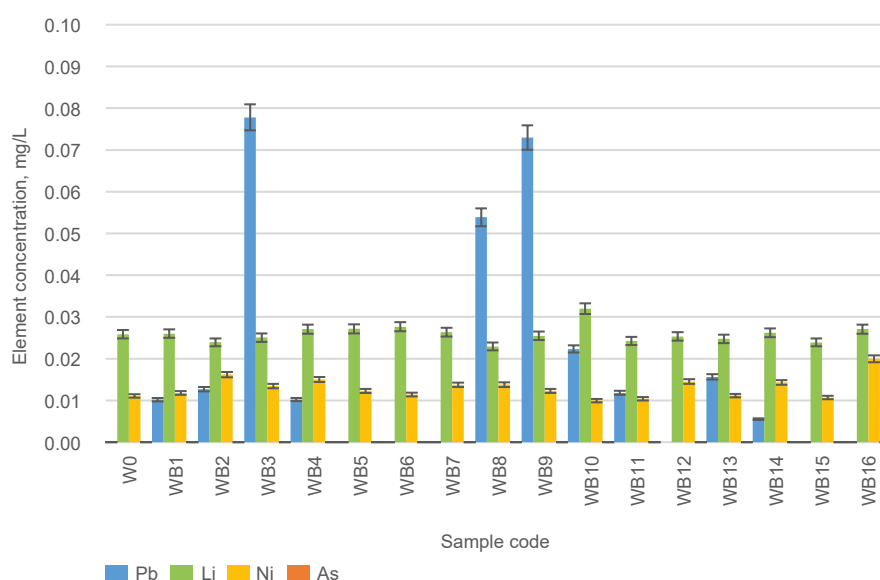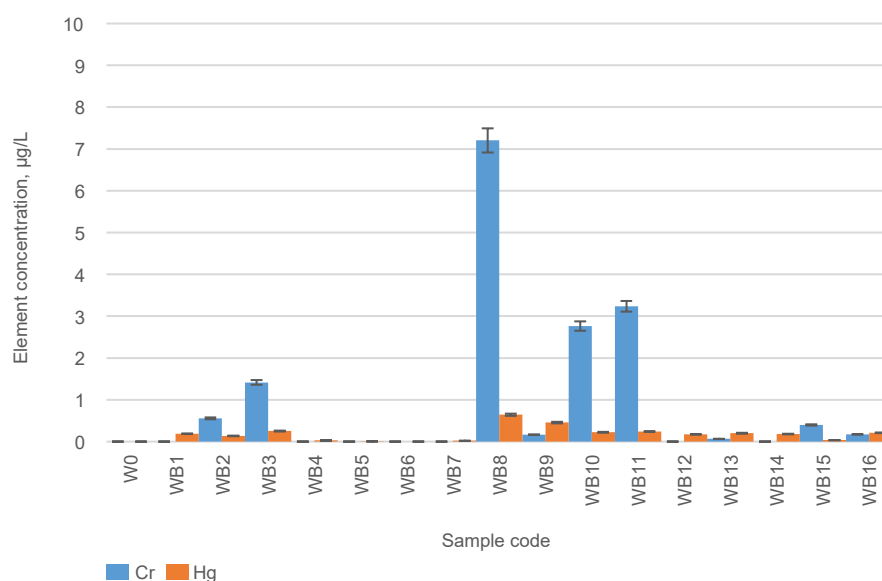

Fig. 6. Concentration of As, Li, Ni and Pb in control (untreated) wine W0 and wine after the addition of different bentonites. RSD 2–5%
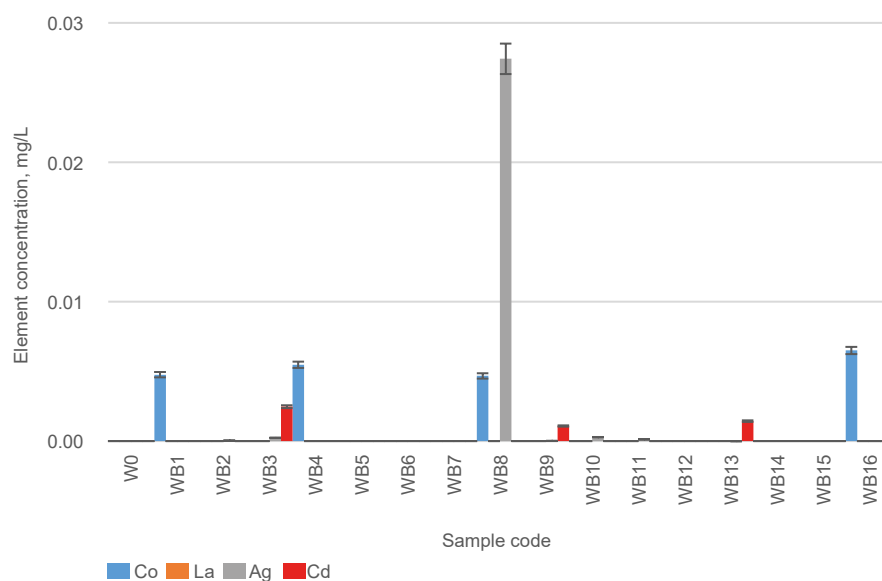
Fig. 7. Concentration of Ag, Cd, Co and La in control (untreated) wine W0 and wine after the addition of different bentonites. RSD 2–5%

Fig. 8. Concentration of Cr and Hg in control (untreated) wine W0 and wine after the addition of different bentonites. RSD 2–5%

B2, B4, B10, B11, B13 and B14 also released this element into the experimental wines, leading to concentrations between 0.010±0.001 and 0.020±0.001 mg/L. The remaining bentonites did not release this element into the wine into which they were introduced. However, it is important from an oenological point of view to consider the possibility of enriching the wine with lead after treatment with some bentonites.

The concentration of Li in the untreated wine was 0.026±0.002 mg/L. The investigated bentonites did not exhibit any ability either to release or to sorb this element from the wine. Their behaviour was approximately similar regarding Ni, which was detected in the wine before treatment with the different bentonites at a concentration level of 0.010±0.001 mg/L. Bentonites B16 and B2 showed the most significant ability to release this element into the wine. The concentration found in the test wines treated with them was about 60–100% higher than the concentration in the original wine. This behaviour was also observed in a model environment (Bakardzhiyski et al., 2023). Arsenic was not detected either in the initial control wine or after its treatment with any of the bentonites tested (<0.003 mg/L).

Cobalt was not detected in the control wine sample (<0.13 μg/L), but as a result of the introduction of bentonites B1, B4, B8 and B16, it was found in the corresponding experimental wines at concentrations in the range 0.005–0.007 mg/L (Fig. 7). The highest Co content was reported in test sample WB16. It should be noted that similar behaviour of the listed bentonites was also reported in a model environment (Bakardzhiyski et al., 2023). In the other experimental samples, the concentration of Co was below the detection limit of the instrumental technique used (<0.13 μg/L).

Lanthanum was not detected in either the control or the experimental samples (<3.61 μg/L). The only sample in which Ag was detected was WB8, at a concentration of 0.027±0.002 μg/L. This element was not present in the untreated wine. However, this concentration was far from the maximum limit of 0.1 mg/L (OIV, 2022). In the control, Cd was below the ICP-OES detection limit (<0.080 μg/L). But bentonite B3 showed the greatest ability to release this element into the wine. The reported concentration in the relevant experimental sample was 0.0025±0.0002 mg/L. Concentrations of approximately half this level were reported in the samples treated with bentonites B9 and B13. These values are far below the limit of 0.01 mg/L, according to OIV (2022).

The results obtained for Cr and Hg content in experimental wine samples are shown in Figure 8. These two elements were not present in the untreated wine (control sample). The introduction of bentonite led to their appearance in some experimental samples. The concentration of chromium was highest (7.2±0.4 μg/L) in the wine treated with bentonite B8. Its concentration was less than half this level in samples B10 and B11. In the remaining samples, it was either at even lower concentrations or below its ICP-MS detection limit (<0.79 μg/L).

The concentration levels obtained for Hg in the samples treated with bentonites B8, B9, B3, B11 and B16 exceeded the maximum permissible concentration of 0.2 μg/L (OIV, 2022). In the test samples WB13, WB1, WB14 and WB12, the concentration was at the limit, but still below the permissible level. The remaining bentonites also showed a capacity to release Hg into the wine, but to a much lesser extent. Only treatment with bentonite B6 did not result in Hg transfer to the treated wine. These results indicate that for some bentonites, at high doses (around and above 2 g/L) the danger of wine being enriched with Hg is real, and one must either look for another bentonite or apply one of the permitted alternative methods to achieve colloidal stability.

CONCLUSION

The treatment of wine with different bentonites leads to different changes in its elemental composition. For some elements (Al, Ca, Cr, Fe, Hg, P, Pb and Si), an increase in their concentrations compared to the untreated wine was reported in most cases. For other elements (B, Cu, Li, Mg, Mn, Ni and Zn) the changes in their concentration were more often not significant. For the latter, there were single exceptions where the opposite results were obtained after treatment with some bentonites. Some of them were close to and others exceeded the maximum permissible limits in wine. The bentonites WB8, WB9, WB10 and WB11 are distinguished by the highest desorption capacity and transfer elements such as Ag, Al, Cr, Cu, Fe, P, Pb, Si, and Zn into treated wines. The trend of decreasing K after treatment with all the studied bentonites was the most persistent. It is quite possible that with a different dose of bentonite, results that deviate from those described above could be obtained. This is to be the subject of further research.

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