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The Impact of Bentonite Fining on Apple Juice Clarity and Sodium Content

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ABSTRACT

In the experiment, four calcium bentonites (Ca bentonites) with different sodium contents in their activator were used for the pretreatment fining before the pasteurization of apple juice. The percentages of sodium carbonate in the activator of bentonites were 0% (B-0), 30% (B-30), 50% (B-50) and 70% (B-70). For the determination of correlation between bentonite dosage and juice turbidity after fining, the apple juice was treated with the following dosages: +40 g/hL, +50 g/hL, +60 g/hL, +70 g/hL and +80 g/hL. Bentonites with added sodium in the activator achieved much better juice clarification compared to pure Ca bentonite. Better clarification of apple juice was obtained by bentonites with the higher sodium content in the activator and also in treatments with higher dosages (g/hL). All bentonites with added sodium in activator released significantly more sodium into the juice (6.7–8.4 mg/L) compared to the control and pure Ca bentonite (3.2–3.4 mg/L). At higher doses of bentonite, the increase in sodium release into the juice was greatest with bentonite B-70. Bentonite with a small addition of sodium in the activator (B-30) was the best compromise in terms of juice clarification and sodium release.

Key words: apple juice, clarification, bentonite, sodium

INTRODUCTION

In the production of clear apple juice, it is important that the juice does not contain any sediment after bottling. In addition to pasteurization, which prevents subsequent haze formation due to microbiological processes, it is important to remove certain compounds before heating, which would become insoluble in the heating process and thus cause turbidity. These compounds are primarily thermo-instable proteins. These need to be removed with the addition of bentonite. It also removes heavy metals, polyphenols, as well as turbid particles from fruit processing. The final stable clarity of the juice is usually a combination of the use of fining agents (bentonite, gelatin) and subsequent filtration. For good filtration, it is important that the stabilization process itself, e.g. protein with bentonite, produces a satisfactory clear juice (Günther and Junker, 1995).

Protein instability and use of bentonite

Juice and wine protein instability depends on several factors, but wine grape proteins are the main haze factors, being mainly caused by pathogenesis related proteins (thaumatin like proteins and chitinases) with a molecular weight between 1040 kDa. Bentonite is a type of clay and its main part is the mineral montmorillonite. It consists of lamellae among which, depending on the type of bentonite, there is a different amount of crystalline water and cations (Ca^{2+}, Na^{+}, Mg^{2+}). In the aqueous solution, the mineral emits cations and the surface of the lamellae becomes negatively charged (Ribéreau-Gayon et al., 1997).

The negative charge of bentonite attracts positively charged proteins. The immense internal surface area between the individual alumina-silica plates provides an abundant number of attachment sites. The particles of bentonite swell readily in water and separate into sheets of alumina-silicate. The sheets are about 1 nm thick and 500 nm wide. The separation of the sheets provides an immense surface area over which cations exchange and adsorption and hydrogen bonding can occur. When fully expanded in water, sodium bentonite has a surface area of about 700-800 m2/g. Swelling the bentonite before addition significantly improves its efficacy (Jackson, 2008).

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Many products and technological solutions have been studied in recent years, however, sodium bentonite is still the most efficient and most commonly used treatment to remove unstable proteins (Cosme et al., 2020). Beside removing heat-unstable proteins, bentonite stabilises unstable colour in red wine and has an impact in lowering the copper casse (Jackson, 2008).

Use of bentonite with other fining agents and the impact on juice clarification

Often the bentonite is used with gelatin. Adding gelatin to apple juices or clarified ciders can induce hazes in response to content of haze-active polyphenols. At an appropriate gelatin concentration, turbidity is nearly linear with the polyphenol concentration. Treatment with bentonite prior to gelatin addition, appeared to remove the endogenous protein (Siebert and Lynn, 1997a).

Increasing the amounts of gelatin and bentonite used for the pretreatment of apple juice clearly improves the flux by means of delaying the membrane fouling. The molecules in apple juice, which are responsible for membrane fouling could be successfully retained by the gelatin and bentonite formed aggregate (Gokmen and Cetinkaya, 2007). The gelatin and bentonite combination has an impact on the better elimination of tannins, total polyphenol, condensed tannins and anthocyanins in comparison with fining only with bentonite in freshly squeezed grape juice (Mazrou et al., 2020).

Ultrafiltration with a bentonite pretreatment is an optimal process for the effective clarification and high quality of apple juice and causes the permeate fluxes during membrane filtration to effectively increase (Youn et al., 2004; Bahceci, 2012). Rai et al. (2007) are reporting, that using bentonite after an enzymatic treatment can improve the permeate flux by ultrafiltration in case of mosambi (sweet citrus fruit) juice. Filtration characteristics of apple juices pretreated with various filter-aids were studied (Youn et al., 2004) to select an appropriate process for clarification with higher permeate flux and a lower membrane fouling, and for the production of high-quality juice. Bentonite was a superior filter-aid to polyvinylpolypyrrolidone (PVPP), activated carbon of 10-14 mesh, and the enzyme mixture of pectinase and amylase in reducing juice turbidity, although PVPP was best in tannin rejection. Permeated fluxes during membrane filtration were effectively increased by filter-aid pretreatment, especially bentonite (Youn et al., 2004).

Bentonite side effects in fining

Using 150 mg/L of gelatin and 750 mg/L of bentonite for fining apple juice can also significantly reduce the total phenolic content (Bahceci, 2012). Koyuncu et al. (2007) are reporting that the adsorption efficiency of dark-coloured compounds from apple juice is better with acid-activated bentonite, than with heat-activated and native bentonite.

The methanol in apple wine also restricts its quality. Bentonite is an excellent methanol interrupter. It is suggested that bentonite treated and fermented at 10 °C was the most effective way to decrease methanol content, retain pectin content and preserve the wine quality (Han et al., 2022).

Fining with bentonite in apple juice also reduces Patulin – the mycotoxin produced by a number of moulds involved in fruit spoilage. Total toxin reduction using filtration, enzyme treatment, and fining with bentonite were 70%, 73%, and 77%, respectively (Bissessur et al., 2001).

Activated bentonites and sodium release in juice or wine

Bentonites are rarely used in their natural state, they are usually activated with sulfuric acid or alkaline salts. It is possible to load (activate) bentonites with H^+, Na^+ or Ca^{2+} ions to form acid, calcium or sodium bentonites to impact their ion exchange capacity. Ca bentonites can be dispersive, causing little sediment, but the treated material remains fairly turbid. Sodium bentonites are more difficult to disperse, they clear juices or wines well, but cause more sediment. At the same dosages, Na bentonites stabilize more proteins than Ca bentonites (Ribéreau-Gayon et al., 2006).

Ca bentonites have a low water adsorption capacity (<10 mL/g), while sodium bentonites have a high adsorption capacity (>20 mL/g). The efficiency of Ca bentonites decreases sharply at higher pH values (> 3.5). They thus have a greater ability to bind proteins even at higher pH values with activated bentonites (Eschnauer and Görtges, 1999; Günther and Junker, 1995). Calcium bentonite tends to clump on swelling and provides less surface area for fining. Nevertheless, it has the advantage of producing a smaller and heavier sediment that is easier to remove (Marchal et al., 1995). Sodium bentonites swell more and have a higher protein adsorption capacity. When added to juice or wine, they make a higher deposit leaving a clear liquid (Ribéreau-Gayon et al., 2006).

The wines stabilized with sodium-activated bentonite have a higher content of volatile compounds and a lower volume of lees than those treated with pure sodium bentonite, an effect that was greater when the fining treatment was done before the start of the fermentation (Salazar et al., 2017). High levels of sodium in foods and drinks can be a health risk for consumers. According to the Dietary Guidelines for Americans (FDA, 2020), diets higher in sodium are associated with an increased risk of developing high blood pressure, which can raise the risk of heart attacks, heart failure, stroke, kidney disease and blindness. Most Americans exceed the recommended limits for sodium in their diet. On average, Americans eat about 3,400 milligrams (mg) of sodium per day. The Dietary Guidelines for Americans recommend limiting sodium intake to less than 2,300 mg per day (FDA, 2020). Sources of sodium in wine or juice can be from using various additives such as: Na chloride, Na sorbate and Na bentonite. Natural contents in wine are around 35 mg/L (Ough and Amerine, 1988). The maximum permitted Na content in wine is 60 mg/L (OIV, 2022a).

High concentrations of calcium (Ca), magnesium (Mg), potassium (K), and sodium (Na) are often responsible for imparting a salty character on wines, which is often described as a negative sensory attribute (Cabello-Pasini et al., 2013).

MATERIALS AND METHODS

In the experiment, four Ca-bentonites (which were activated with Na-carbonate during their factory production) were used for the fining of apple juice before pasteurization. The shares of sodium carbonate in the activator of bentonites were 0% (B-0), 30% (B-30), 50% (B-50) and 70% (B-70). The control in the experiment was apple juice without the addition of bentonite. For the impact determination of different bentonites on the turbidity of apple juice, the same dosage of 60 g/hL and an additional control with pasteurisation were used. For the determination of correlation between bentonite dosage and juice turbidity after fining, the apple juice was treated with the following dosages: +40 g/hL, +50 g/hL, +60 g/hL, +70 g/hL and +80 g/hL. After the sedimentation, turbidity (NTU) was measured using a turbidimeter. After pasteurization, sodium contents were measured via atomic spectrometry (OIV, 2022b). All treatments were performed in triplicates.

Data analysis

Data obtained in the experiment were statistically analysed using the statistical programme IBM SPSS Statistics Data Editor (Version 25). An analysis of variance (ANOVA) was performed. The significance of differences between means at $P \leq 0.05$ were tested using the Tukey test. Correlations were also calculated between dosages and juice turbidity and between dosages and sodium content after bentonite fining.

RESULTS AND DISCUSSION

Juice clarity after bentonite fining

Figure 1 presents the turbidity values (NTU) in apple juice 24 hours after the addition of bentonite, where only one dosage of bentonite was used (+60 g/hL). The turbidity results were also compared with the pasteurized sample – CONT2 (without bentonite + pasteurization). In the sample (CONT2) a characteristic phenomenon of apple juice turbidity after heating, if bentonite is not used, was observed (Jackson, 2008). Compared to the unheated sample (CONT 1), the turbidity increased from 4.5 to 15.8 NTU. This was also confirmed by (Tajchakavit et al., 2001) where omission of the fining procedure resulted in an increase in turbidity and visual observation of larger quantities of haze. Fining with a gelatin/bentonite ratio of 1:1 in their experiment resulted in the least haze. Appropriate sedimentation of coloid particles before pasteurization is very important for good permeated flux during subsequent filtrations (Rai et al., 2007). Maximum permeate flux was observed with an enzymatic treatment followed by adsorption using bentonite.

At the same time, the results of our experiment showed that the samples with the addition of activated bentonite (B-30, B-50, B-70) were much clearer than the ones with pure Ca-bentonite (B-0). That was also confirmed by Ribéreau-Gayon et al. (2006), where sodium bentonites had higher protein adsorption capacity. Pure Ca-bentonite (B-0) showed very poor sedimentation of turbid particles, resulting in problems in the subsequent filtration of juices. There is no statistical difference among activated bentonites in turbidity with respect to different (%) activation with sodium, which means that 30% of the sodium content in the activator is already sufficient for appropriate bentonite performance. These results were confirmed for activated bentonites by Siebert and Lynn (1997b), where unstabilized apple juice was treated with various amounts of bentonite, silica gel, or polyvinylpolypyrrolidone (PVPP). In their experiment, bentonite was very effective in removing haze protein, taking out about to 92% at the highest treatment level, while silica gel removed only to 25% of the haze protein.

Figures 2-5 show the effect of increasing bentonite dosages (g/hL) on the clarity of apple juice before pasteurization. The first finding is that the NTU values for pure Ca bentonite (B-0) (Figure 2) were directly proportional to the increasing dosage of bentonite. At an increasing dosage of 10 g/hL, the turbidity of the juice increased by 4.2 NTU.

On the other hand, figures 3–5 show that in bentonites with sodium in their activator (B-30, B-50 and B-70), the increased effect of juice clarification was observed everywhere at higher dosages of bentonite (lower NTU values). These results were also confirmed by Youn et al. (2004), who stated that ultrafiltration with bentonite pretreatment was the optimal process for apple juice production.

The reduction in turbidity for every 10 g of bentonite addition was 0.64 to 1.9 NTU. This effect was strongest in bentonites with the highest Na-content in their activator (B-70). Brugirard (1997) also stated that calcium bentonites, after being activated with sodium carbonate can have similar or higher protein removal capacities in comparison with bentonite without sodium in its activator.

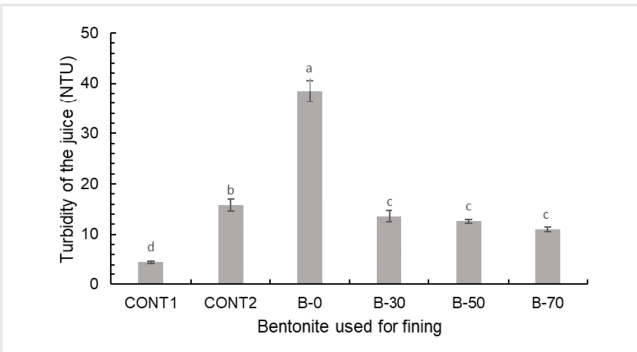
Our results indicate, that in cases where higher doses of bentonite are needed due to the composition of apple juice, it makes sense to use bentonite with a higher Na content in the activator. All correlations between bentonite dosage and turbidity (NTU) are pronounced (r = 0.86 to 0.98).

Sodium content in apple juice after fining with bentonite

Figure 6 shows the sodium contents in apple juice after clarification with different bentonites at a dosage of +60 g/hL, compared to the control (without the addition of bentonite). The results clearly point towards a significant increase in sodium content in apple juice after fining with bentonites with sodium in activator, in comparison with pure Ca-bentonite (B-0) and the control (without bentonite).

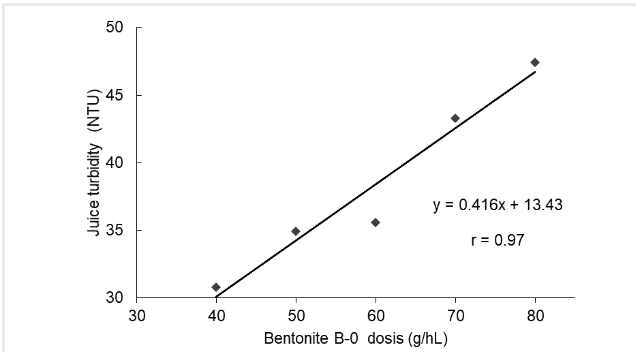
The sodium content in juice was highest in bentonite B-70 (with 70% of sodium in the activator). The pure Ca-bentonite showed no increase in sodium content. That can be confirmed by (Marchal et al., 1995) where calcium bentonite does not liberate sodium ions into wine. Regardless of the increase in content of sodium, we do not expect extreme values that could be controversial from a health point of view for the consumer.

Figures 7, 8, 9 and 10 show the effect of increasing the bentonite dosage on the sodium content in apple juice. The results showed that the effect of increasing the bentonite dosage is as big as the proportion of sodium in the bentonite activator. In pure Ca bentonite (Figure 7), there was no effect on the increase in Na (r = 0.50). For bentonite B-30 (Figure 8), an increase in sodium content of 0.59 mg/L can be expected. Bentonite B-50 increased the sodium content by 0.62 mg/L (Figure 9) and bentonite B-70 by 1.3 mg/L, by



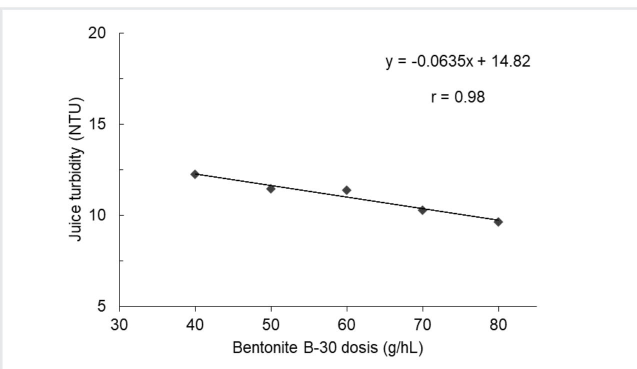
NTU – nephelometric turbidity unit; CONT1 – no bentonite and no pasteurisation; CONT2 – no bentonite + pasteurisation; B-0 – 0% of Na in activator; B-30 – 30% Na in activator; B-50 – 50% Na in activator; B-70 – 70% Na in activator, a,b,c – different characters indicate statistically significant differences (Tukey test, P ≤ 0.05)

Figure 1: Turbidity (NTU) of apple juice after bentonite fining (60 g/hL)



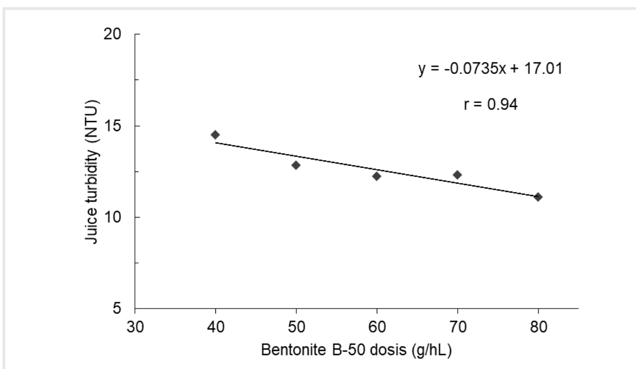
NTU – nephelometric turbidity unit; CONT1 – no bentonite and no pasteurisation; CONT2 – no bentonite + pasteurisation; B-0 – 0% of Na in activator; B-30 – 30% Na in activator; B-50 – 50% Na in activator; B-70 – 70% Na in activator, a,b,c – different characters indicate statistically significant differences (Tukey test, P ≤ 0.05)

Figure 2: Correlation between dosage (g/hl) and juice turbidity after bentonite (B-0) fining



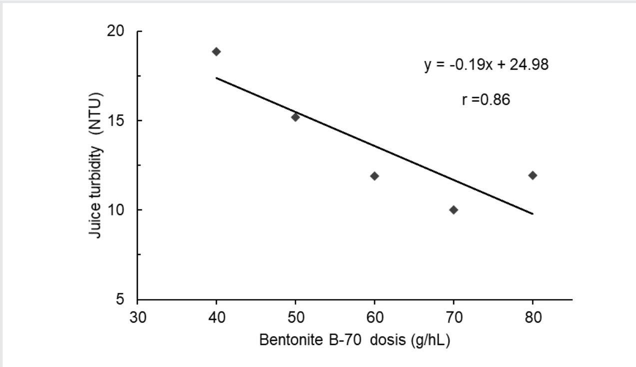
NTU – nephelometric turbidity unit; y – trend line slope; r – coefficient of correlation; B-30 – 30% of Na in activator

Figure 3: Correlation between dosage (g/hl) and juice turbidity after bentonite (B-30) fining



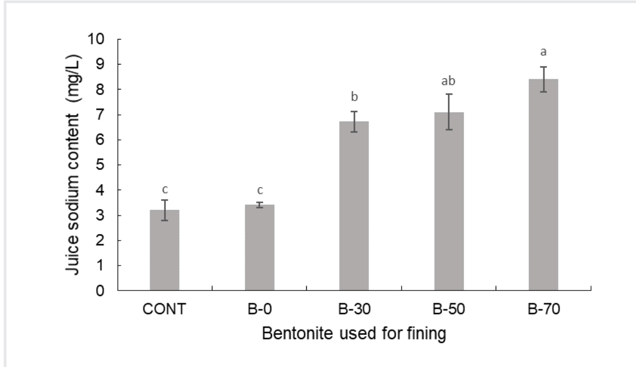
NTU – nephelometric turbidity unit; y – trend line slope; r – coefficient of correlation; B-50 – 50% of Na in activator

Figure 4: Correlation between dosage (g/hl) and juice turbidity after bentonite (B-50) fining



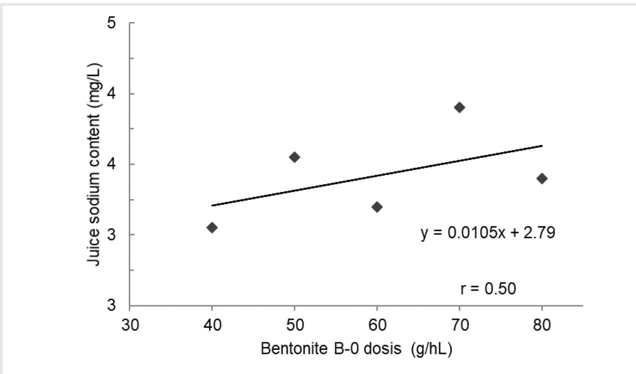
NTU – nephelometric turbidity unit; y – trend line slope; r – coefficient of correlation; B-70 – 70% of Na in activator

Figure 5: Correlation between dosage (g/hl) and juice turbidity after bentonite (B-70) fining



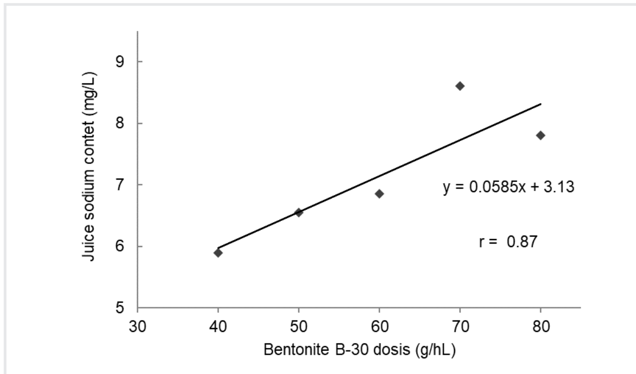
CONT – no bentonite; B-0 – 0% of Na in activator; B-30 – 30% Na in activator; B-50 – 50% Na in activator; B-70 – 70% Na in activator. a, b, c – different characters represent statistically significant differences (Tukey, P ≤ = 0.05)

Figure 6: Sodium (Na) content in apple juice after clarification with different bentonites at a dosage (+60 g/hL)



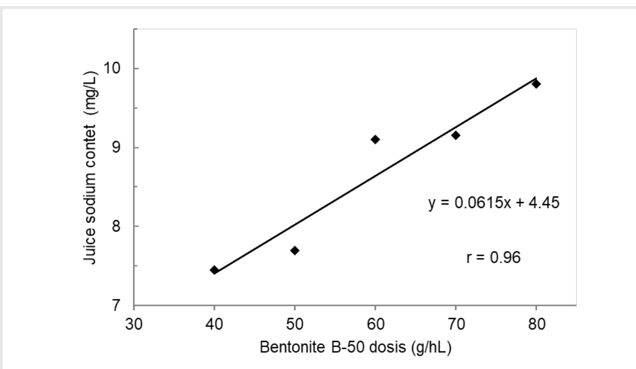
y – trend line slope; r – coefficient of correlation; B-0 – 0% of Na in activator

Figure 7: Correlation between dosages (g/hl) and sodium content (mg/L) after bentonite (B-0) fining



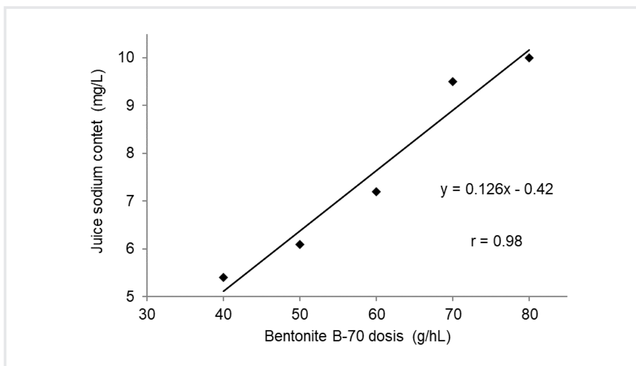
y – trend line slope; r – coefficient of correlation; B-30 – 30% of Na in activator

Figure 8: Correlation between dosage (g/hl) and sodium content (mg/L) after bentonite (B-30) fining



y – trend line slope; r – coefficient of correlation; B-50 – 50% of Na in activator

Figure 9: Correlation between dosage (g/hl) and sodium content (mg/L) after bentonite (B-50) fining



y – trend line slope; r – coefficient of correlation; B-70 – 70% of Na in activator

Figure 10: Correlation between dosage (g/hl) and sodium content (mg/L) after bentonite (B-70) fining

increasing the dose by 10 g/hL (Figure 10). For bentonites B-30, B-50 and B-70 the correlations between bentonite dosage and sodium content are pronounced (r = 0.87 to 0.98).

CONCLUSION

Four Ca-bentonites with different sodium proportions in the activator were used for the fining pretreatment of apple juice. Bentonites with a higher sodium content clarify the juice slightly better, but also release more sodium at higher dosages. In the experiment, bentonite with a 30% sodium content in the activator showed the best results in terms of juice clarification, as well as in the release of sodium into the juice and should therefore be used when considering the risks in consumer health.

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Vpliv čiščenja z bentonitom na bistrost in vsebnost natrija v jabolčnem soku

POVZETEK

V poskusu smo za bistrenje jabolčnega soka pred pasterizacijo uporabili štiri kalcijeve bentonite (Ca bentonit) z različno vsebnostjo natrija v aktivatorju. Deleži natrija v aktivatorju so bili naslednji: 0% (B-0), 30% (B-30), 50% (B-50) and 70% (B-70). Za določitev korelacije med odmerkom bentonita in motnostjo soka po bistrenju smo uporabili različne odmerke: +40 g/hL, +50 g/hL, +60 g/hL, +70 g/hL in +80 g/hL. Bentoniti z dodanim natrijem v aktivatorju so dosegli veliko boljše bistrenje soka v primerjavi s čistim Ca bentonitom. Jabolčni sok se je bolje bistril pri bentonitih z višjim deležem natrija v aktivatorju ter hkrati ob višjih odmerkih (g/hL). Vsi bentoniti z dodanim natrijem v aktivatorju so sprostili bistveno več natrija v sok (6,7–8,4 mg/L) v primerjavi s čistim Ca bentonitom (3,2–3,4 mg/L). Pri večjih odmerkih bentonita je povečanje sproščanja natrija v sok največje pri bentonitu B-70. Bentonit z majhnim dodatkom natrija v aktivatorju (B-30) je najboljši kompromis v smislu bistrenja soka in sproščanja natrija.

Ključne besede: jabolčni sok, bistrenje, bentonit, natrij