



Sugar beet molasses purification by bentonite addition: Analysis of quality enhancement and treatment conditions



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ABSTRACT

Reduction of non-sugar compounds in sugar beet molasses through various treatments is a promising method for molasses quality enhancement, improved sugar recovery and prevention of possible changes during storage. The presented study was conducted in order to examine the effect of sodium and calcium bentonite treatment on sugar beet molasses colour, turbidity and unavoidable sucrose content reduction during the purification process. Box-Behnken experimental design was employed with 3 independent parameters: pH (3–7), molasses dry substance (30–50° Brix) and bentonite suspension concentration (9–21 g/L) while treatment temperature was constant (60 °C) in all conducted experiments. Lower pH values and increasing bentonite concentrations expressed positive influence on molasses colour and turbidity reduction. Corresponding parameters also had slightly negative impact on molasses sucrose content. Compared to the calcium bentonite, sodium bentonite exhibited better results in terms of molasses colour and turbidity reduction. Optimal treatment parameters for achieving a 29–39% reduction in colour and 91–98% in turbidity with minimal sucrose content reduction (5%) were: pH 5.11–5.17, molasses dry substance 38.5–40.3° Brix and bentonite concentration 16–21 g/L.

1. Introduction

In the sugar production process molasses formation is inevitable. As the final residue obtained from the sugar crystallization unit, molasses is usually produced at about 4–5% (w/w) on sugar beet and has high viscosity and dark brown colour (Asadi, 2007). Molasses characteristics originate from the high content of fermentable sugars (sucrose, glucose, fructose, raffinose) and variety of organic non-sugar compounds like betaine, lactic acid, amino acids, minerals, vitamins, phenolic compounds and dark colour compounds. Colourants present are mainly (at least 80%) melanins, melanoidins and alkaline degradation products of hexoses (ADHs) (Bernal, Ruiz, Geanta, Benito, & Escudero, 2016; Valli et al., 2012). The majority of the corresponding colourants are not naturally present in sugar beet juice. They are formed during sugar beet processing due to the high temperature and specific pH values as well as interactions with organic non-sugar compounds (Asadi, 2007; Schlumbach, Pautov, & Floter, 2017). Melanoidins, as Maillard reactions products, have the most significant impact on juice colour increase. In contrast to sugar cane, sugar beet processing, even at low

processing temperatures (25 °C), induce significant melanoidins formation due to the higher nitrogen content in sugar beet compared to sugar cane (Coca, Garcia, Gonzalez, Pena, & Garcia, 2004). In the sugar juice purification stage, particularly liming and carbonation, most of the non-sugars, including colourants, are removed but high temperature operating conditions (70–90 °C) during evaporation and crystallization enable further melanoidins formation (Asadi, 2007; Schlumbach et al., 2017). Residual water and all non-sugar compounds including melanoidins and other colourants remain concentrated in molasses after sucrose crystal formation during final crystallization stage. Moreover, the list of non-sugar compounds in molasses is enlarged with the application of various insecticides and herbicides in the sugar beet cultivation process as well as upon various bactericides, antifoam agents and antiscalants application in the sugar production process. Considering molasses widespread application in food, feed and fermentation industries (Castañeda-Ayarza & Cortez, 2017; Mišljenović; Čolović, Vukmirović, Brlek, & Salas-Bringas, 2016; Urbaniec & Grabarczyk, 2014; Valli et al., 2012) reduction of non-sucrose compounds (particularly colourants) is preferable in order to facilitate the

Abbreviations: ADHs, alkaline degradation products of hexoses; SC, sucrose content; DS, dry substance; RSM, response surface methodology; ICUMSA, International Commission for Uniform Methods of Sugar Analysis

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efficient recovery of sucrose (molasses desugarization) or betaine by chromatographic process as well as minimize changes that may occur during molasses storage. Currently used methods related to sugar beet juice decolourisation are: juice sulfitation which particularly inhibits Maillard reactions, activated carbon filtration and ion-exchange resin application (Asadi, 2007). Some alternative filtration aids, particularly bentonite were also investigated for potential utilization in sugar beet juices decolourisation with promising results. Erdogan, Demirci, and Akay (1996) described how the application of five commercial bentonites used independently or in combination with sepiolite, diatomite and quaternary ammonium chloride reflected on sugar beet thick juice colour. The activated bentonite treatment reduced sugar juice colour by 29% (Erdogan et al., 1996). In the study of Laksameethanasana, Somla, Janprem, and Phochuen (2012) raw sugarcane juice was treated by using bentonite independently and in combination with activated carbon in order to produce less coloured syrup. The obtained syrup had significantly reduced colour compared to the conventional lime treated syrup (Laksameethanasana et al., 2012). By using a response surface design Jahed, Khodaparast, and Mousavi Khaneghah (2014) were able to optimize the raw sugar beet juice purification treatment with bentonite. The authors reported great improvements in colour (35.55%) and turbidity (76.09%) reduction of purified sugar beet juice compared to the conventional limed-carbonated juice (Jahed et al., 2014). Besides sugar industry, bentonite application received considerable attention over the years and was also investigated in wine (Lambri, Dordoni, Giribaldi, Violetta, & Giuffrida, 2012; Lira et al., 2014) and juice (Koyuncu, Kul, Çalimli, Yıldız, & Ceylan, 2007) clarification processes. To the best of authors' knowledge, there have been no studies regarding bentonite application in the sugar beet molasses decolourisation treatment. Therefore, presented investigation would be significant contribution to the existing knowledge of bentonite application in the food industry.

This study was conducted in order to investigate the influence of bentonite treatment on sugar beet molasses quality parameters: colour, turbidity and sucrose content (SC). The goal was to examine bentonite efficiency as molasses pre-treatment which would enable easier sucrose and betaine separation in the subsequent chromatographic process. Furthermore, the aim was to highlight the effectiveness of two different bentonite types towards colour reduction.

2. Materials and methods

2.1. Materials

Molasses (85° Brix) used in this research was sampled from the sugar beet factory “Šajkaška” (Žabalj, Serbia) during the sugar beet campaign in 2016. For the purpose of corresponding experiments and experimental design, molasses samples dry substance was adjusted to 30, 40 and 50° Brix using distilled water. Colour, turbidity and sucrose content measurements in samples were conducted in duplicate prior to every set of experimental runs.

2.1.1. Bentonite suspensions preparation

Molasses samples were treated by using two different types of fine powder formulated bentonite, sodium bentonite (BP Bentonite-Na) and calcium bentonite (BP Bentonite-Ca) (montmorillonite content 88–92%, moisture content 9–10%, Bentoproduct, Šipovo, Bosnia and Herzegovina). Bentonite suspensions were prepared according to the producer's recommendations: sodium bentonite: 100 g in 1.5 L of water, calcium bentonite: 100 g in 0.3 L of water. Hydration was achieved through bentonite steeping into distilled water heated at 40–50 °C followed by intense mixing in the initial hydration stage until uniform suspension was obtained (about 15 min). Afterwards, the distance between the bentonite silicate layers is enlarged during 12 h swelling period at room temperature (25 °C). As a result, bentonite adsorption capacity is increased (Jahed et al., 2014).

Table 1

The Box-Behnken design applied in the sugar beet molasses treatment with calcium and sodium bentonites.

Independent variables	Code units	Levels		
		Low (−1)	Medium (0)	High (+1)
pH	x ₁	3	5	7
Molasses dry substance (DS) [° Brix]	x ₂	30	40	50
Bentonite concentration (BC) [g/L]	x ₃	9	15	21

2.2. Methods

Prepared bentonite suspensions were added to 200 ml of diluted molasses (30, 40 and 50° Brix) in sufficient amount to achieve the resulting concentration (9, 15 and 21 g/L) given by the experimental design (Table 1). Citric acid (50 g/100 mL) was used to adjust selected pH (Lach-Ner s.r.o., Neratovice, Czech Republic) upon bentonite addition. Erlenmeyer flasks containing prepared blends were closed and placed into the water bath (GDE Enzymatic Digester equipped with Multistirrer 6, VelpScientifica®, Ustmate, Italy) heated at 60 °C and mixed for 30 min. Cooled blends (25 °C) were filtered through filter paper (nr. 604 ½, Selecta faltenfilter, Carl Schleicher and Schüll, Dassel, Germany) and the obtained filtrate was used for measurements.

2.2.1. Analytical measurements

Quality parameters of molasses samples were measured according to the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) methods. Colour was determined using spectrophotometer (MA 9522-Spekol 220, Iskra, Horjul, Slovenia) measuring the samples absorbance at the 420 nm wavelength. Dry substance content was determined using refractometer (modell G, Carl Zeiss, Jena, Germany). Colour was calculated using following equation:

$$\text{Colour [IU]} = \frac{A \times 1000}{c \times b} \quad (1)$$

where the colour is in ICUMSA unit (IU), A is the solution absorbance at 420 nm, b is the cuvette length in cm (1 cm cuvette for molasses) and c is dry substance content in g/cm³ calculated by using brix-density tables for sugar solutions. Molasses colour reduction was calculated using the following equation:

$$\text{Colour reduction [\%]} = [(C_i - C_f)/C_i] \times 100 \quad (2)$$

where C_i is the initial colour of the diluted molasses (IU) and C_f is the final colour in molasses samples after treatment (IU).

Molasses samples turbidity was measured using turbidimeter (Turb 550IR, WTW, Weilheim, Germany) and given in nephelometric turbidity units (NTU). Reduction in molasses turbidity was calculated as:

$$\text{Turbidity reduction [\%]} = [(T_i - T_f)/T_i] \times 100 \quad (3)$$

where T_i is the initial turbidity of the diluted molasses (NTU) and T_f is the final turbidity in molasses samples after treatment (NTU).

Sucrose content (w/w) in the corresponding samples was determined using polarimeter (Model 410, Metra, Prague, Czechoslovakia). Changes in molasses sucrose content were calculated as:

$$\text{Sucrose content reduction [\%]} = [(SC_i - SC_f)/SC_i] \times 100 \quad (4)$$

where SC_i is the initial sucrose content of the diluted molasses in % and SC_f is the final sucrose content in molasses sample after treatment (%).

All analytical measurements were performed in duplicate and expressed as mean values in Table 2.

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