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Research paper

Bentonite, temperature and pH effects on purification indexes of raw sugar beet juice to production of inverted liquid sugar

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ABSTRACT

The present study deals with modeling of the effects of processing temperature (30–95 °C), concentration of Bentonite (Bent) (1–5 g/L) and pH (3.5–6) on color, turbidity, purity, ash and invert sugar content of purified raw sugar-beet juice. The used methodology was the face central composite design of response surface to optimize the key parameters (temperature of processing, concentration of Bent and pH) of the process. The main goal was to focus on the possibility to produce invert sugar directly from raw sugar-beet juice in conjunction with an alternative purification method and also comparison with the common conventional lime processing in the industry. In this way, second-order polynomial models were developed for dependent responses using least-square fit of regression analysis. The results of analysis of variance indicated that all three investigated independent parameters have significant influence on purity indexes of raw beet-juice. The optimum condition was a constant reaction time (30 min) at temperature of 75 °C, pH 4.47 and Bent concentration of 1.7 g/L which were determined on minimization of color, turbidity and ash content and maximization of invert sugar and purity. At this optimum point: color, turbidity, purity, ash and invert sugar content were found to be 1664 ICU_{420}, 6.3 NTU, 93.9%, 0.55% and 1.6%, respectively. Also the clarified juice quality obtained by Bent method was improved greatly when compared with conventional limed–carbonated as the color and turbidity in clarified juice were removed by the rate of 35.55% and 76.09%, respectively.

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1. Introduction

Composition of sugar beet is variable based on several factors including: soil agronomy and fertilization factors, weather conditions during the growing season, degree of maturity and storage conditions at the time between harvesting and consumption. Sugar beet generally contains 75% water, 17.5% sucrose, 5% mark, 1.1% nitrogen based organic material, 0.5% mineral and 0.9% non-nitrogen based organic materials (Van der Pol et al., 1998). Non-sugar compounds of beet raw juice include: coloring compounds, polysaccharides, proteins, and minerals that exist either in the form of true or colloidal solution. Raw juice should be administered under purification processes, including chemical and mechanical methods. One of the available chemical purification methods is application of clay. There are different opinions about the required properties of clays for their industrial applications in purification of sugar beet (Erdogan et al., 1996). Rather ion-exchange resins have also been introduced instead of clay (Bazhal et al., 1980). But in fact, high cost of constructions for ion-exchanger units, problems encountered in their use and environmental problems forced researchers to find alternative methods for removing of color and turbidity in sugar industry (Novotny, 1985). Refining of raw sugar beet juice by using Bent can be considered as an alternative chemical method.

Bent is composed of primarily montmorillonite (Mt) from the smectite group (Karakaya et al., 2011) which is generally classified into sodium (Na) or calcium (Ca) types depending on dominant exchangeable ion (Zhansheng et al., 2006). Ca-Bent, which contains Ca^{2+} as the dominant exchangeable cation, is characterized by its low water absorption and swelling capabilities and its inability to stay dispersed in water. Na-Bent is characterized by its ability to adsorb large amounts of water and form viscous, thixotropic dispersions (Eisenhour and Brawn, 2009). Bent is recommended as a desiccant or as dehydrator of gases, due to its excellent ability to adsorb water vapor. In particular, the presence of mycotoxins in various food producing chains has made the application of desiccants rather popular. In recent years, some new applications in the food industry were introduced (Bulut et al., 2009); such as: controlled or modified atmospheres for storage of fresh or processed foods, new high barrier polymers (plastics) for packaging, adsorbent or desorbent substances for different vapors, flavoring compounds and antimicrobial agents as parts of active packaging. Other applications include those used extensively as a binding agent in foundry sand and iron

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ore pelletization, as a barrier for water and nuclear waste, and in pharmaceuticals, cat litter, paint, rubber, perfumes, ceramics, drilling mud, plastics (Harvey and Lagaly, 2006; Murray, 2007), enzyme immobilization (Sedaghat et al., 2009), purification, decolorization and stabilization of vegetable oils in terms of market consumption (Rossi et al., 2001; Didi et al., 2009) and clarification of fruit juice by removing the proteins, heavy metal ions, pesticides and dark compounds (Koyuncu et al., 2007).

The inflation rate and the bleaching properties of Bent are not desirable in normal and primary form. Therefore, it is necessary to make some modifications to be exacerbated to obtain the desired properties. For example, calcium Bent can be treated with sodium carbonate, which results to ion exchange and substitution sodium instead of calcium and production of modified Bent with expanding ability. This new compound is called sodium–calcium Bent (or Na–CaBent). Na–CaBent is the granulated Na–Ca Bent used in beverage technology.

Effective factors on the clarification performance of Bent include: temperature, pH, methods of Bent preparation and stirring (Farmani et al., 2006a). The negative charge of Bent depends on the environmental pH and also on the type of Bent. In pH = 3–4 Zeta potential of sodium Bent is variable between −15 and −20 mV and Na–CaBent between −20 and −25 mV. The pH-value of the beverage affects the efficiency of Bent. The protein adsorption of Bent is higher with lower pH-values. Most probably this is caused by changes in the charge of dissolved proteins in the beverage, which are already influenced by the slightest pH changes; also the quality of Bent has a huge impact on the efficiency. Bent with low swelling capacity (pure calcium Bent) is less protein adsorbing. This issue becomes highlighted when pH-values are greater than 3.5. Special Bent such as Na–CaBent, with higher swelling capacity, shows much better protein adsorption above all, when pH-values are 3.4 and higher. Therefore the pH-value must be taken into consideration by using Bent (Blade and Boulton, 1988).

To make use of the full efficiency, Bent must be wetted in water prior to application. The insertion of water by pre-swelling leads to a bigger distance between the silicate layers, thus both of the volume for adsorption and also the capacity of adsorption will be increased. Swelling can be accelerated by higher temperatures of water (but the maximum is 60 °C). Supernatant can be considerably influenced by water quality. A thorough and intense mixing during Bent–CaBent dispersion preparing is necessary, especially during pre-swelling stage. Allow it to swell and settle for at least 4–6 h, recommended for 12 h. Pre-swelling with soft water is much better than using hard water, since soft water contains less calcium ions which this condition is favorable for the swelling process (Hymore, 1996).

The effect of five Turkish Bent on removing color and turbidity in sugar beet juice with and without quaternary ammonium salt (Quartamin D86) was investigated by Erdogan et al. (1996). The results showed that without quartamin the percentage of change in color and turbidity reached 29%, while with Bent and quartamin, these values reached approximately 33% (Erdogan et al., 1996).

In this study the effects of Bent concentration, pH and temperature were investigated based on minimization of color, turbidity, purity, and ash and maximization of invert sugar content of raw sugar beet juice. It should be noted that due to acidic conditions of experiments and also production process of invert sugar, this study was organized to produce inverted liquid sugar. Therefore, the purpose of this study was to evaluate the performance of Bent in elimination of impurities in raw sugar beet juice and its treatment to produce inverted liquid sugar for use in various industries, including beverages and cannery without the need to solve again and hydrolyze sugar crystals.

2. Materials and methods

This research was carried out in a laboratory of the sugar factory of Shirin Khorasan, Iran. Raw juices were sampled from diffusion units of the same sugar factory. The Bent used was sodium–calcium Bent (NaCalit ®PORE-TEC Premium Na-Ca-Bentonite ERBSLÖH, Geisenheim, Germany) that was prepared from the nectar plants of Mashhad, Iran. Used Chemical's materials had laboratory purity and were prepared by companies Merck, Germany and Fluka, Swiss.

2.1. Analyses of the Bent

The chemical compositions (in mass%) of the Bent that were determined by X-ray fluorescence (XRF) spectrometry using a Philips PV 1480 spectrometer with lithium tetraborate fused discs are L.O.I., SO_3, P_2O_5, MnO, Ti_2O, MgO K_2O, Na_2O CaO, Fe_2O_3, and AL_2O_3,SiO_2 in concentrations of 11.14, 0.01, 0.20, 0.027, 0.26, 2.30, 1.23, 0.94, 1.15, 3.44, 14.3 and 64.49(%), respectively. XRF analysis showed that SiO_2 was the important composition of the Bent, and on the other hand with the increasing amount of SiO_2 in Bent, the strength of clarification will increase (Erdogan et al., 1996).

2.2. Experimental

2.2.1. Methods

For refining the raw sugar beet juice, 200 mL of raw juice is poured in an Erlenmeyer 500 mL. The Bent which acts as ion exchange in this state is activated when it hydrated. Before using Bent it must be hydrated in water before and therefore the solution 20% of Bent was prepared 8–12 h before the experiment at 50 °C (Xifan et al., 2007). Then sufficient amount of 20 percent solution Bent was added to an Erlenmeyer. Due to the acidic properties of Ben, the pH of the mixture reduced slightly. Hence first, the Bent was added and then the pH of the mixture was adjusted by the citric acid at the sufficient pH. Blend of raw juice and Bent was mixed about 15 min by the magnetic mixer at room temperature (25 °C). The Erlenmeyer containing samples was transferred into the water bath and it was cooled by cold water after half an hour at the desired temperature. Cold solution was filtered and parameters were measured immediately.

2.2.2. Measurement

The properties of the raw and thick juice were determined according to the method of ICUMSA (2000)[1]. The color was measured by a spectrophotometer (Cambridge, England) at a wavelength of 420 nm according to the mentioned method and was calculated by using the following equation:

$$F_{IE} = 10^5 \frac{A}{L \times C \times \rho} \tag{1}$$

where F_{IE} is the color according unit ICUMSA (kg/cm^2), A is the adsorbance, C is the solid (or dry substance) content, L is the path length of light or cell thickness in cm, and ρ is the density (g/cm^3) of juice (Erdogan et al, 1996).

Turbidity was measured by a turbidimeter (Model 6035, turbidimeter, Jenway, UK) and ash content was measured by a conductivity meter (Model 380 BA, UK) and by Eq (2). Invert sugar was measured according to the method of Berlin institute (Asadi, 2007).

$$W_A = f_A \times f_5 t \times (A_5 - A_W) \times \left(\frac{4.5}{m} + \frac{Bx^\circ}{1000}\right) \times 0/0018 \tag{2}$$

where W_A is the ash content by conductometry method (%), F_A is the correction factor, F_t is the heating correction factor, A_5 is the electrical conduction for adjustment solution (μs), A_w is the electrical conduction for water in use (μs), m is the mass of syrup sample (g) and Bx° is the Brix (%).

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[1] International Commission for Uniform Methods of Sugar Analysis.

Purity (Quotient) was calculated using the following equation:

$$Q = \frac{POL + Invertsugar}{Brix} \times 100 \qquad (3)$$

where POL is the percent of sucrose that was measured by a polarimeter (saccharomat, schmidt + haensch) and Brix is dry substance and was measured by a Digital Refractometer for Sugar Analysis (Model HI 96801, Hanna Instruments, USA).

2.2.3. Experimental design and statistical analysis

Response surface methodology (RSM) was used to estimate the effect of independent variables (Bent concentration, x_1; pH, x_2; temperature of the process, x_3) on the color (ICU), turbidity (NTU), ash content (%), purity (%) and invert sugar (%). Face central composite design was employed for designing the experimental data.

Experimental data was modeled using the Design Expert software version 6.01 (Statease Inc., Minneapolis, USA) and three-dimensional representations of the response surface generated by the model. The design included 20 experiments, and it is adopted by adding 6 central points and 6 axial points to 2^3 full factorial design. The center runs provide a means for estimating the experimental error and a measure of lack of fit (Myers and Montgomery, 2002). The axial points were added to the factorial design to provide estimation of curvature of the model. The design was carried out in random order (Bradley, 2007). The response functions (Y) were color, turbidity, ash content, purity and invert sugar. These values were related to the coded variables (x_i, i = 1, 2, and 3) by a second-order polynomial using the equation below. Stepwise analysis was used to reduce the number of non-significant terms of model (reduced model).

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{11}{x_1}^2 + b_{22}{x_2}^2 + b_{33}{x_3}^2 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + \varepsilon \qquad (4)$$

The coefficients of polynomial model were represented by b_0 (constant term), b_1, b_2 and b_3 (linear effects), b_{11}, b_{22} and b_{33} (quadratic effects), and b_{12}, b_{13} and b_{23} (interaction effects).

The significant terms in the model were found by analysis of variance (ANOVA) for each response. The adequacy of the model was checked accounting to R^2 and adjusted R^2. Numerical and graphical optimization technique of the Design-Expert software was used for simultaneous optimization of the multiple responses. The desired amounts for each variable, and response were chosen. All the independent variables were kept within available range while the responses were either maximized or minimized.

3. Results and discussion

3.1. Model fitting

The coefficients of determination (R^2), adjusted R^2 and lack of fit are used to verify the model. The model is statistically appropriate when lack of fit is not significant and has the highest value of R^2 and adjusted R^2 (Koocheki et al., 2009). Based on the results obtained from the statistical analysis (Table 1), the second-order polynomial response surface model Eq. (4) was fitted to each of the response variables (Y).

Regression analysis and ANOVA were used for fitting the model and to examine the statistical significance of the terms. The estimated regression coefficients of the quadratic polynomial reduced models, R^2 and adjusted R^2 for the response variables are given in Table 2.

Coefficients of determination (R^2) can be expressed as proportion of variation described by the model to total changes that it is a criterion for desirable degree of fit. Therefore when R^2 approaches to the unity, performance of fitted model is described as a better representative of the response changes in terms of independent variable changes. Joglekar and May (1987) suggested that for a good fit of a model, R^2 should be at least 0.8. The R^2 values for color, turbidity, ash content, purity and invert sugar were found to be 0.95, 0.87, 0.88, 0.89 and 0.96, respectively (Table 2). The results showed that the R^2 values for these response variables were higher than 0.80, indicating that the regression models were suitable to explain the behavior. The lack of fit is an indication of the failure for a model representing the experimental data at which points were not included in the regression or variations in the models cannot be accounted for random error (Koocheki et al., 2009). If there is a significant lack of fit which could be indicated by a low probability value, the response predictor is discarded. The statistical analysis indicated that the proposed model was adequate, showing no significant lack-of-fit ($p > 0.05$) for all responses (Table 2).

Table 1
Statistical analysis results of fitted model on the response data.

Source	Standard deviation	R^2	Adjusted-R^2	p-Value for lack of fit
Color				
Linear	1261.24	0.58	0.50	0.0013
Quadratic	503.1	0.96	0.92	0.066^{ns}
Turbidity				
Linear	2.49	0.80	0.77	0.058^{ns}
Quadratic	2.29	0.89	0.81	0.062^{ns}
Quotient				
Linear	4.63	0.77	0.73	0.23^{ns}
Quadratic	0.85	0.91	0.84	$0.\ 5^{ns}$
Ash				
Linear	0.025	0.82	0.79	0.27^{ns}
Quadratic	0.023	0.90	0.82	0.32^{ns}
Invert sugar				
Linear	1.11	0.62	0.55	0.0007
Quadratic	0.4	0.97	0.94	0.055^{ns}

ns – not significant.

It should be noted that a large value of R^2 does not always imply that the regression model is a good one. Adding a variable to the model will always increase R^2, regardless of whether the additional variable is statistically significant or not. Adjusted R^2 is a modification of R^2 that adjusts for the number of explanatory terms in a model. Unlike R^2, the adjusted R^2 increases only if the new term improves the model more than would be expected by chance. Thus, it is recommended to use an adjusted R^2 to evaluate the model adequacy (Montgomery, 2005).

High adjusted R^2 in this project showed that non-significant condition has not been observed in the model.

3.2. Color

The effect of independent variables on reduced color of refined juice with Bent was presented in Fig. 1.

Regarding the effectiveness of color from Bent concentration, according to the significance ($p < 0.05$) coefficients of regression model (Table 2) and also response surfaces, it is clear that the Bent concentration affected the color values and at low contents reduced the color, but with increasing amount of Bent, the color of refined juice has increased (Fig. 1). This shows that excess of Bent in the environment can act as a cause of color. The study of Farmani et al. (2006a) shows that reducing the color related to adsorbing the dye material by Bent particles. Colored materials in sugar beet juice have complex structures and different sizes and mostly non-polar properties, and some may even are somewhat polar. Many minerals, especially montmorillonite (Mt) readily adsorb organic molecules. Action to adsorb depends upon many factors, including size and shape of molecular adsorption material, its concentration in the environment, the effect of van der Waals forces, long chains of particles adsorbed and entropy effects (Erdogan et al., 1996).

The effect of pH on reducing the juice colors is shown in Fig. 1. As it showed that the pH had a major impact on reducing of color, so by reducing pH removal of color increased. Significant effects of linear

Table 2
ANOVA and regression coefficients of the second-order polynomial model for the response variables (actual values).

Source	Color (ICU)		Turbidity (NTU)		Quotient (%)		Ash (%)		Invert sugar (%)	
	Coefficient	p-Value	Coefficient	p-Value	Coefficient	p-Value	Coefficient	p-Value	Coefficient	p-Value
Model linear	16,808.3	<0.0001	16.04	<0.0001	87.6	<0.0001	0.68	<0.0001	13.41	<0.0001
b_1	−1261.2	0.048	1.29	0.0014	−0.44	0.003	0.032	0.103	–	–
b_2	−7485.8	<0.0001	2.7	0.0001	0.59	<0.0001	−0.03	<0.0001	−6.94	<0.0001
b_3	58.05	0.0022	−0.53	<0.0001	0.2	<0.0001	4.3×10^{-4}	0.079	0.17	<0.0001
Quadratic										
b_{11}	128.03	0.09	–	–	–	–	–	–	–	–
b_{22}	1003.9	0.0001	–	–	–	–	–	–	0.80	<0.0001
b_{33}	–	–	0.003	0.015	–	–	–	–	–	–
Interaction										
b_{12}	140.15	0.069	–	–	–	–	8×10^{-3}	0.02	–	–
b_{13}	–	–	–	–	–	–	–	–	–	–
b_{23}	−16.51	0.0042	–	–	−0.033	0.0009	–	–	−0.03	<0.0001
Residual	–	–	–	–	–	–	–	–	–	–
Lack of fit	–	0.081	–	0.12	–	0.67	–	0.45	–	0.14
Pure error	–	–	–	–		–	–	–	–	–
Total	–	–	–	–	–	–	–	–	–	–
R^2	0.95	–	0.87	–	0.89	–	0.88	–	0.96	–
R^2 adjusted	0.92	–	0.83	–	0.86	–	0.84	–	0.95	–

($p < 0.001$) and quadratic terms ($p < 0.05$) confirm this subject (shown in Table 2). In removing impurities from beet juice two main factors, including adsorption and interaction charges, are involved. As mentioned previously, concentration of negatively charged Bent is variable depending on pH of environment and type of Bent. In the acidic environment, pH of zeta potential of Na–CaBente is more when it is compared with other one of negatively charged. Therefore, this type of Bent has a better function for adsorption in the acidic environment (Farmani et al., 2006a).

According to Shen et al. (2009) and Li et al. (2010) with research on the absorption of anionic dyes by various Bent; this result revolved that the efficiency of color removal by Bent increases with decreased pH. They reported that the surface charge of Bent was positive in the examined pH range (pH = 3–11). At low pH most of the color molecules can be adsorbed by Bent. SO_3^- groups are existent in the structure of anionic color. The higher removal efficiency of the dye by Bent adsorption at low pH values may be due to neutralization of the negative charge at its surface as $–SO_3^-$ anion, which increased the protonation and the electrostatic attraction between the negatively charged $–SO_3^-$ anion and the positively charged adsorption site (Ozcan et al., 2007). In alkaline mediums, the Zeta potential of the Bent reduced with increasing pH. On the other hand, there was a competitive adsorption between the OH^- ions and the dye anions. Hence, the removal efficiency of the dyes decreased with increasing pH (Shen et al., 2009). These results were similar to those results obtained by Kang et al. (2009).

Temperature also had a great effect on color removal by Bent (Fig. 1a). Considering the coefficients of the fitted model on the color data, it can be seen that color changes with temperature without relation to other variables and will follow a linear trend ($p < 0.05$) (Fig. 1a), so with increasing temperature, the removal of colored material increases.

Similar results have been reported by Gokmen and Serpen (2002) and Koyuncu et al. (2007), using adsorbent resin and Bent, respectively; they stated that with increasing temperature, adsorption efficiency of dark-colored compounds from apple juice increased. Farmani et al. (2006b) also concluded that with increasing temperature, the removal of colored compounds and turbidity of raw sugarcane juice by Bent increased.

Based on the sum of squares, the importance of the independent variables on color could be ranked in the following order:

$$\text{pH} > \text{processing temperature} > \text{Bent concentration}.$$

3.3. *Turbidity*

The results presented in Table 2 showed that the linear effect of process temperature and pH was highly significant on turbidity ($p < 0.001$) while Bent concentration was only significant at 1%.

Among the quadratic terms, only process temperature was significant ($p < 0.05$) for the turbidity. Based on the sum of squares, the importance of the independent variables on turbidity could be ranked in the order of processing temperature, pH and Bent concentration.

Turbidity changes with the varying amounts of Bent and it showed similar trend of color changes with the Bent content. There was a general trend showing an increase in turbidity of juice as the Bent concentration increased (Fig. 2a).

Bent can separate proteins, heavy metal ions and other impurities from the juice through adsorption and thereupon prevent the turbidity or secondary sedimentation (Blade and Boulton, 1988; Xifan et al., 2007). However, as mentioned excess of Bent in the environment can increase turbidity and color in the juices (Farmani et al., 2006a, 2006b).

In practice, it is observed that the impurities within the juice that were dispersed as colloidal particles causing turbidity and color in the juice, flock formed and with pass juice from the filter, remain behind the filter and eliminated from the juice. The effect of temperature and pH was linear on the turbidity change (Table 2). As it can be seen in Fig. 2 with increasing temperature and decreasing pH turbidity removal efficiency was increased by Bent.

Heat treatment clot colloidal material in juice and in presence of Bent in the juice, flocculate materials, accumulation and this action it makes easier smooth operation of juice (Anonymous, 1982).

As mentioned for the color changes, the reason of the better activity of Bent at low pH, in the separation of turbidity and color components is more negatively charged in the acidic conditions and with increase concentration of negative charge, strength of Bent clarification will increase (Bergaya and Vayer, 1997). Similar results have been reported by Xifan et al. (2007), they reported that the adsorption capacity of protein (as causing turbidity) in wine increased slightly from pH 2.69 to 4.20 and decreased sharply to pH 11.00. This suggested that the pH is the most important factor affecting the adsorption process, which affects the end surface charge of the Bent and the degree of ionization of the protein. Owing to the strong electrostatic interaction between the negatively charged adsorptive sites and the positively charged protein, the high adsorption capacity was obtained at lower pH. As the pH of the solution increased, the positive charges on the protein surface

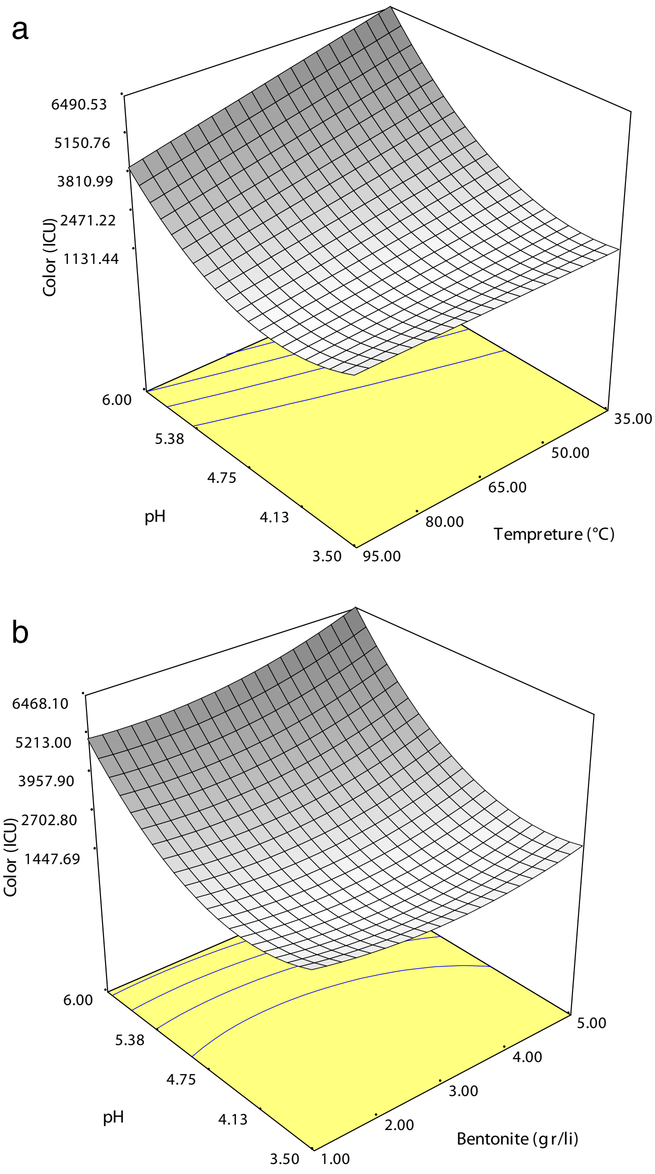

Fig. 1. Response surface for the effect of (a) Bentonite concentrations and pH (Temperature = 65 °C), and (b) Temperature and pH (Bentonite concentrations = 3 g/L) on the color of raw beet juice.

decreased and the negative charges increased. The Bent surface was negatively charged and had lower adsorption at higher pH. The active site of a clay mineral (either as an adsorbent or as a catalyst) is an acidic site, so Bent activity increases in acidic conditions (Bergaya and Vayer, 1997).

3.4. Ash

The analysis of variance of independent variables on the regression model of juice ash indicated that the linear effects of pH were significant in the model ($p < 0.001$) while the effects of Bent concentration and temperature variables were not significant (Table 2). Fig. 3 illustrates the effect of Bent concentration, temperature and pH on changes in the ash content of juice.

Due to the lack of significant effect of Bent concentration and temperature on the ash content ($p > 0.05$), it can be concluded that the concentration of Bent and the reaction temperature are not too sensitive to change in the ash percentage of juice. This independence is evident in the response surfaces as well (Fig. 3). So with the change in the levels of these variables, the extent of changes in the juice ash was very small.

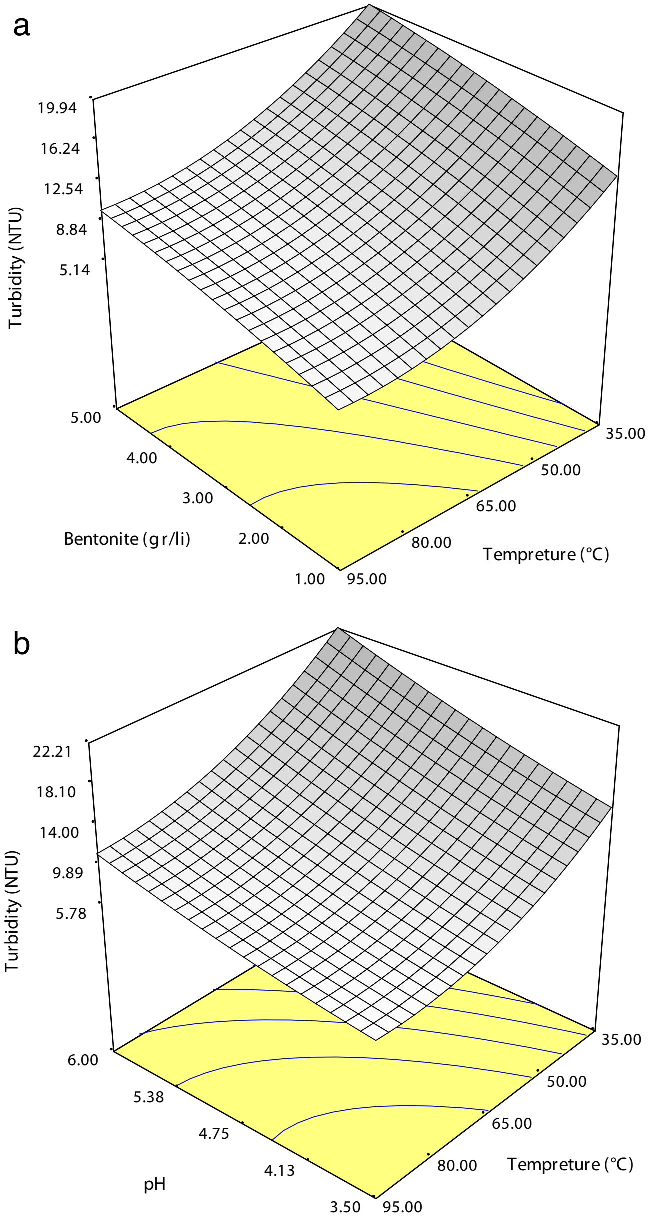

Fig. 2. Response surface for the effect of (a) Bentonite concentrations and Temperature (pH = 4.75), and (b) Temperature and pH (Bentonite concentrations = 3 g/L) on the turbidity of raw beet juice.

Mohammed (2010) reported that the efficiency of removal of Fe^{+3} ions in aqueous solution using natural Bent increases with increasing temperature.

Two main reasons are available for the increasing efficiency of ash removal by Bent. First, the higher temperature activated the metal ions for enhancing adsorption at the coordinating sites of the adsorbent, and the metal cation moved faster (Babel and Kurniawan, 2003), and second, acceleration of some originally slow step(s) and creation of some new activation sites on the adsorbent surface (Khalid et al., 1999).

The coefficients of the fitted model on the ash data of juice showed that the only significant effect for pH was the linear effect (Table 2). So with increasing pH, there was a reduced amount of ash in juice (Fig. 3).

The similar results about absorption of some heavy metals such as Zn, Cu, Ni and Cr by Bent at pH = 3 were obtained by other researchers (Alvarez-Ayuso and Garcia-Sanchez, 2003). Altin et al. (1999) reported

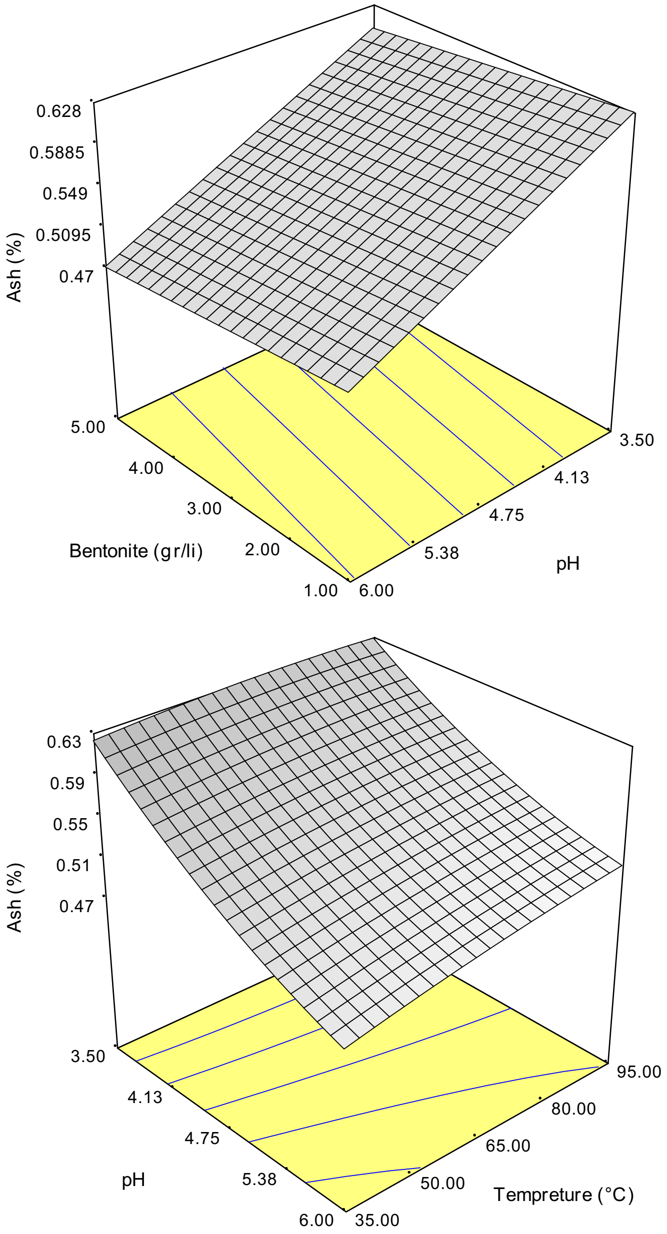

Fig. 3. Response surface for the effect of (a) Bentonite concentrations and pH (Temperature = 65 °C), and (b) pH and Temperature (Bentonite concentrations = 3 g/L) on the ash content of raw beet juice.

that the efficiency of removal of Pb^{2+} ions by Mt decreases at low pH. The mechanisms that influence the adsorption characteristics of Bent can be given by dissolution, ion exchange/adsorption, and precipitation (Karakaya et al., 2011). The same behavior for adsorption of Zn^{2+} ions was also observed by Kaya and Oren (2005), they observed that adsorption capacity of Na-Bent increased when the initial pH of the solution was increased from 3 to 8 and the lowest Zn^{2+} sorption rates were obtained at pH 3. This could be due to the increase in competition for adsorption of the sites by H^+ (Alvarez-Ayuso and Garcia-Sanchez, 2003) and dissolution of Al^{3+} ions from the aluminosilicate layers (Altin et al., 1999).

3.5. Invert sugar

The data given in Table 2 indicate that the invert sugar content in juice was directly related to the linear and quadratic effect of pH and linear effect of temperature ($p < 0.001$). Among the mutual interaction terms, pH and temperature of the process had a significant effect ($p < 0.001$) on invert sugar content of the raw beet juice. The importance of the independent variables to the invert sugar content would be ranked in the following order: pH > processing temperature > Bent concentration.

Fig. 4 shows the effect of Bent concentration, pH and reaction temperature on the invert sugar content of raw sugar beet juice. As can be seen, invert sugar content increased with increasing the temperature of the process (Fig. 4a).

Owing to, with increasing temperature the hydrolysis of sucrose to reducing sugars in the raw sugar beet juice increased, so amount of invert sugar was higher. The effect of pH on the invert sugar content was shown in Fig. 4b.

The results revealed that when the pH is reduced, it causes an increase in the amount of invert sugar in the juice. The sucrose chemically is somewhat unstable, especially in acidic solution and combined easily with water and hydrolysis to glucose and fructose. At pH = 8–9, the

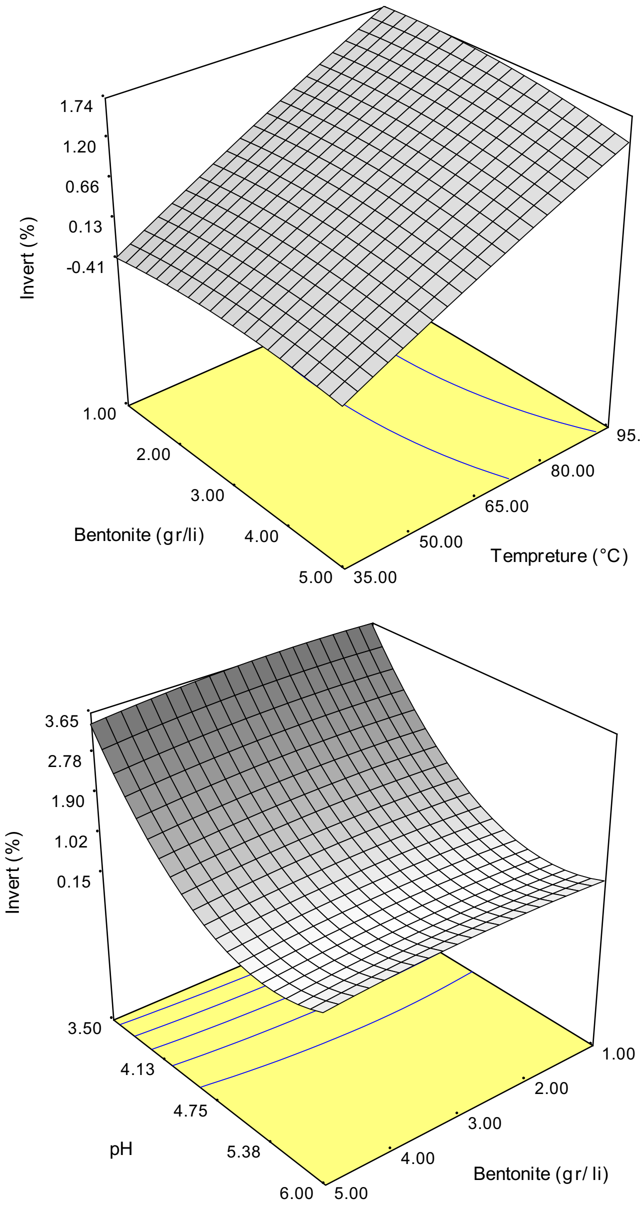

Fig. 4. Response surface for the effect of (a) Bentonite concentrations and Temperature (pH = 4.75), and (b) Bentonite concentrations and pH (Temperature = 65 °C) on the invert sugar content of raw beet juice.

hydrolysis rate of sucrose is at its lowest amount, and it is estimated that with a decrease per unit in pH or an increase of 10 °C in temperature, sucrose hydrolysis can be increase three times (Van der Pol et al., 1998).

3.6. Purity

The results in Table 2 indicated that pH and temperature parameters were significant ($p < 0.001$) for purity of refined beet juice in the model while the concentration of Bent was significant at 1%. Quadratic effects were not significant in any of the independent variables, so there were no observed curvature in the model. The mutual interaction between temperature and pH was only found to be significant ($p < 0.001$).

The variables with the largest effect were the linear terms of pH and reaction temperature followed by the linear term of Bent concentration (Table 2).

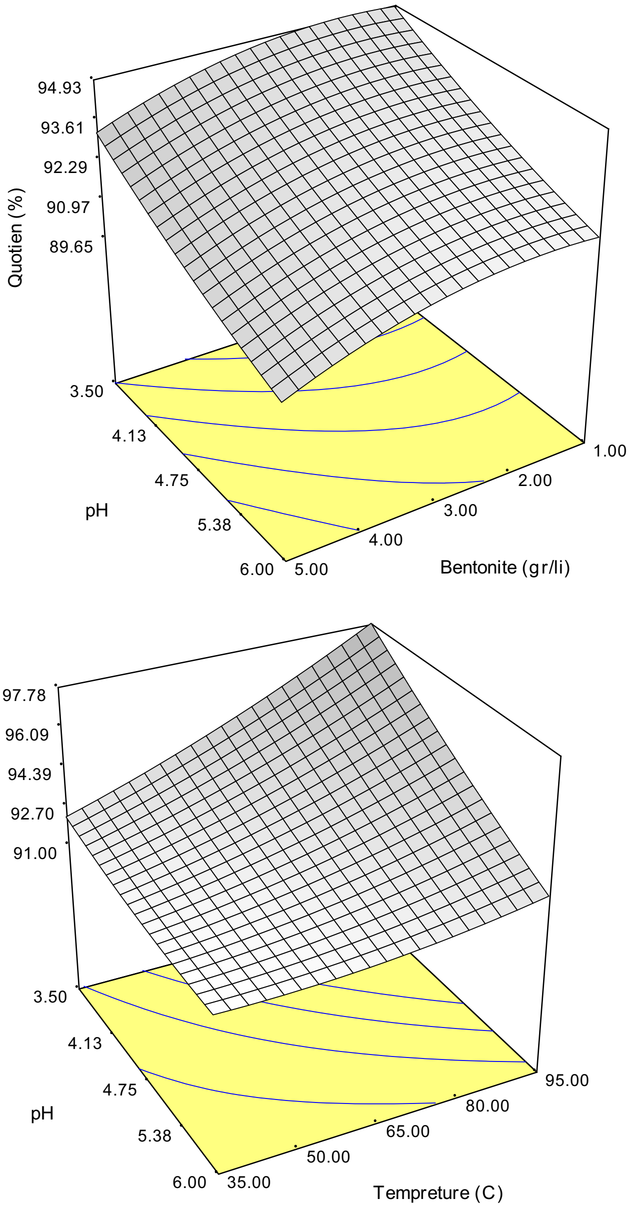

Fig. 5. Response surface for the effect of (a) Bentonite concentrations and pH (Temperature = 65 °C), and (b) Temperature and pH (Bentonite concentrations = 3 g/L) on the purity of raw beet juice.

The variation of purity with pH and concentration of Bent at a constant temperature (65 °C) and with reaction temperature and pH at constant Bent concentration (3 g/L) are presented in Fig. 5.

Generally, the results of this study showed that with increasing temperature of the process and decreasing pH value, adjusted purity of refined juice increased. With increasing temperature and decreasing pH value due to better activity of Bent and further removal of impurities of beet juice, Brix has been reduced. On the other hand, due to the sensitivity of sucrose to this condition and inverted to the reducing sugars, the amount of invert sugar also increases.

As a result, according to Eq. (3), both these factors (reduce of Brix and increase of sucrose inversion) can increase the adjusted purity. It could be mentioned that from hydrolysis per molecule of sucrose obtained two molecules of glucose and fructose. Therefore, the increasing in the amount of invert sugar is more than the decrease in sucrose, so the purity increases.

The effect of Bent was linear on purity variations. Bent at the lower values increased the purity of juice, but at higher content of it, purity decreased (Fig. 5). Bent at the lower content by removal of coloring, turbidity and ash agents caused to decrease the brix and to increase the purity of juice. However, at higher levels of Bent, the purity diminished. Probably, the increased amount of Bent is a stimulus to increase the impurity of juice.

3.7. Optimization

Optimum condition for purification process of raw sugar beet juice is determined to obtain maximum invert sugar and purity content with minimum color, turbidity and ash content using numerical optimization of Design Expert software. This optimum condition is tabulated in Table 3 that provides the highest value of invert sugar = 1.6 (%), purity = 93.9 (%) with lowest color = 1664 (ICU420), turbidity = 6.3 (NTU) and ash content = 0.55 (%). This means that the optimum conditions for raw sugar beet juice purification process were: process temperature of 75 °C, pH = 4.47 and Bent concentration of 1.7 g/L (Table 3).

3.8. Comparing the characteristics of optimal point obtained by Bent with the raw juice and conventional clarified juice

Finally, to evaluate the efficiency of this method in the refining of raw juice, characteristics of clarified juice obtained by Bent were compared with the raw juice and conventional clarified juice.

Classical juice refining steps include preliming, main liming, first carbonation and second carbonation presented in Fig. 6.

The thin juice characteristics improved greatly by Bent and classical processes rather than raw sugar beet juice, as for Bent process, the purity increased 6.9%, also, invert sugar content increased 4.28 times, whereas the color, turbidity and ash decreased 82.02%, 98.99% and 15.38%, respectively, as compared to the raw sugar beet juice values (Table 4).

In addition, the clarified juice quality obtained by Bent method was improved greatly when compared with conventional limed–carbonated as the color and turbidity in clarified juice were removed by the rate of 35.55% and 76.09%, respectively. Although the classical method reduced ash content of raw juice more than Bent method, this difference was not statistically significant at $p < 0.05$ level.

Table 3
Predicted optimum condition for raw sugar beet juice purification process.

Factor	Low	High	Optimum
Temperature (°C)	35	95	75
Bentonite concentration (g/L)	1	5	1.7
pH	3.5	6	4.47

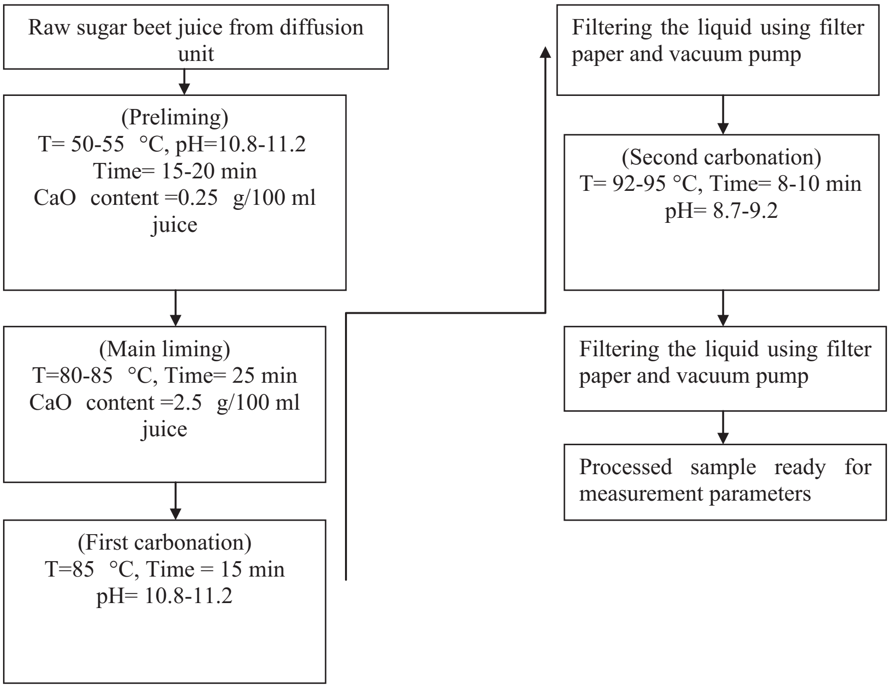

Fig. 6. Flow chart of the classical juice refining process.

4. Conclusion

Numerous processes have been developed over the past years to achieve efficient and cost-effective removal of impurities in order to produce low color juice. Appreciation of the types of color and turbidity compounds present in raw beet juice is important when choosing and operating refinery processes, as different processes may remove different types of color bodies. Therefore, combinations of processes are usually required to produce the best quality refined juice.

In this work, sodium–calcium Bent has been used successfully as an adsorbing agent for the removal of impurities especially colored compounds from raw beet juice. Adsorption was influenced by various parameters such as pH, temperature and Bent concentration. The maximum uptake of substances causing color and turbidity by Bent occurred at pH of 4.47 and adsorption increased with increasing temperature. The result provides clear evidence that the pH of beet juice and process temperature than Bent concentration influence greatly the refining capacity of Bent. While the process temperature increased the purity and invert sugar content, it decreased the coloring and turbidity agents of the clarified juice. Comparison of the bleaching capacity shows that the clarified juice quality obtained from Bent method was improved as compared to conventional limed–carbonated clarified juice in color, turbidity, inverted sugar and quotient. Application of this method can be advisable for production of inverted liquid sugar in various industries. However, to achieve best results further research to evaluate the use of this method and combine it with other processes has been recommended.

Table 4
The characteristics of clarified juice obtained by bentonite as compared with the raw juice and conventional clarified juice.

Purification index	Raw sugar beet juice	Conventional clarified juice	Clarified juice by bentonite (optimal point)
Color (ICU)	9256	2582	1664
Turbidity (NTU)	625.6	26.35	6.3
Purity (%)	87.9	90.35	93.9
Ash (%)	0.65	0.486	0.55
Invert sugar (%)	0.374	0.042	1.6

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