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The Use of Diatomite to Remove Color and Turbidity in Sugar Industry

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ABSTRACT

Diatomite is a pale colored, light weight rock composed mostly of silica microfossil of algae, being known as diatoms. The experiments of using natural diatomite from Lampang, central Thailand to remove color and turbidity in sugar cane juice processed in a sugar factory gave satisfactory results. The color in ICUMSA (International Commission for Uniform Methods of Sugar Analysis) and turbidity of the treated juice were better, i.e., lower than that of the clarified juice from the industrial process lines, while sugar content tended to be higher. The two most appropriate conditions were obtained; when diatomite was used not more than 4% by weight of the juice or 2% diatomite followed by 1 ppm anionic polymer. Analyses of properties of the treated juice and physical and chemical properties of diatomite revealed physical and chemical adsorption properties of diatomite.

Key words: diatomite, ICUMSA, turbidity, brix, sugar cane juice, color

INTRODUCTION

Sugar cane juice is used as the raw material in sugar industry in Thailand. Color and turbidity in sugar cane juice are important because color is one of the factors that measure the quality of white sugar. The color of sugar products depends upon the color and turbidity of juice from which sugar is produced. Color and turbidity in sugar cane juice come from undissolved or suspended constituents in the juice. A variety of chemicals, including ion-exchange resin have been used to remove color and turbidity of juice in sugar technology. Sugar industry is forced to investigate other alternatives due to the expensive working conditions of chemicals and ion-exchange resins (Novontony, 1985). The use of natural resources to remove color and turbidity of juice in sugar technology is a way to reduce chemicals used in sugar industries and also will make the most usefulness of natural resources.

Diatomite or a diatomaceous earth is a pale colored, soft, light weight rock composed principally of the silica microfossils of aquatic unicellular algae known as diatoms.

Diatomite has a wide variety shapes of diatoms, typical size is 10-200 mm. Major components in diatomite are silica (SiO_2) and alumina (Al_2O_3), which are about 70 and 11-18% respectively. Minor components are Fe, K, Mg, Na, K and Ca. Diatomite has highly porous structure, low density and high surface area which results in a number of applications such as filter-aid in processing of liquid foodstuffs and chemical fluids (Inglethorpe,1992; Martinovic *et al*., 2006), sorption of inorganic and organic chemicals

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(Akyüz *et al*., 2001; Huttenloch *et al*., 2001; Martirosyan *et al*., 2003; Shawabkeh and Tutunji, 2003; Khraisheh, *et al*., 2004; Lin *et al*., 2006) as well as an adsorbent for oil spills (Lemonas, 1977). Erdogan *et al*. (1996) reported the use of diatomite and other minerals to remove color and turbidity in sugar beet juice and they recommended diatomite to use in the sugar processing.

In this work, diatomite from Lampang province, central Thailand was used to remove color and turbidity in sugar cane juice from a sugar industrial process. The results were compared with a clarified juice of the sugar industry. Clarified juice was the juice in the industrial process lines that had been removed color and turbidity by using lime and 5 ppm anionic polymer.

METERIALS AND METHODS

Materials

Diatomite samples were obtained from Lampang, central Thailand. The samples were ground manually , washed with distilled water, dried at 110°C and ground again to get smaller sizes before use (an average particle size was 6.88 μm, determined by using mastersizer model S.ver. 2.19 , Malvern Instruments). Sugar cane juice samples were obtained from a sugar industrial process line of a sugar factory in central Thailand and used right after sampling. The experiments were performed at the factory laboratory. Two types of sugar cane juice were used, mixed juice and limed juice. Mixed juice was the first crushed juice and limed juice was the mixed juice with pH adjusted to neutral by lime.

Removal of color and turbidity of sugar cane juice

To remove color and turbidity of sugar cane juice, two experimental conditions were used; the first condition was to use only natural diatomite and the second condition was to use diatomite followed by a polyanionic polymer (polyacrylamide, commercial name is Mirfloc 125 UH). When only diatomite was used, diatomite was added directly to the mixed juice or limed juice (2, 3, 4 and 5% by weight in 500 ml of the juices). The mixture was then heated and stirred at various temperatures (70-80, 80-90 and 90-103°C) for 10 min and set aside for 10 min before the precipitate was filtered off, using filter paper and Bushner funnel. When polyacrylamide was also used, it was added to the juice solution after separating precipitate from the solution then stirred gently for a few minutes and filtered off again. The concentration of polyacrylamide was varied from 1 to 7 ppm by 1 ppm intervals.

Characterization of the juice

After the experiments, the juice was characterized by pH, Brix, ICUMSA color, turbidity and purity to compare with that of the clarified juice of the industrial process lines. The ICUMSA color and turbidity were determined by measuring absorbance of the juice solution at 420 and 900 nm, respectively, and calculated by the equations below;

$$\text{ICUMSA} = \frac{A_{420\text{ nm}} \times 100{,}000}{b \times C} \quad (1)$$

$$\text{Turbidity} = \frac{A_{900\text{ nm}} \times 1{,}000}{\text{Brix}} \quad (2)$$

(Donovan and Lee,1995)

Where A = absorbance, b = path length of light (cell dimension) = 1 cm, C = concentration of sucrose in solution (g/100 ml); obtained by measuring brix of solution by using refractometer and looked for the value of C from “Table of brix and grammes of sucrose per 100 ml of sugar solutions” (Kerr,1970), brix = total dissolved solid in juice solution. Ultraviolet-visible spectrometer, spectronic 301, Milton Roy was used to measure absorbance of the solution.

Purity is the percentage of sucrose in dissolved solid. Purity of sucrose was determined by adding lead (II) acetate basic anhydrous, $(Pb(CH_3COO)_2).Pb(OH)_2$ to get clear solution then measured polarity (pol) of the solution by using polarimeter; Rudolph Research model Autopol IIS. Then pol was converted to % pol by using the table "Pol or Sucrose Factors According to Refractometer Solids" (26 g pure sucrose dissolved in 100 g of water is considered to be 100% pol).The purity of sucrose was then calculated by the following equation;

$$\% \text{ purity} = \frac{\% \text{ pol} \times 100}{\text{Brix}} \quad (3)$$

All the measurements were repeated three times and the average values were used.

Characterization of diatomite

The elemental compositions of diatomite were determined by x-ray fluorescence spectrometry (XRF) (Philips PW 2510).The specific surface area, pore volume and pore size were measured by BET N_2 adsorption method; surface area and porosity analyzer (Micromeritics model ASAP 2020) . The texture and morphology of diatomite were studied by scanning electron microscopy (JOEL model JSM 5410). Functional group interactions at the surface of diatomite were confirmed by fourier transform infrared spectroscopy (FT-IR) (Perkin Elmer System 2000).

RESULTS AND DISCUSSION

The pH of the juice was almost unchanged after treating with diatomite or diatomite followed by anionic polymer, the pH of mixed juice and limed juice were about 5.3 and 7.5, respectively. Two optimum conditions to remove color and turbidity were obtained, i.e, to use not more than 4% natural diatomite or 2% diatomite followed by 1 ppm anionic polymer.

When 2% diatomite was used without anionic polymer, the ICUMSA color of the juice was better (lower) than that of the clarified juice, in both mixed juice (Figure 1) and limed juice

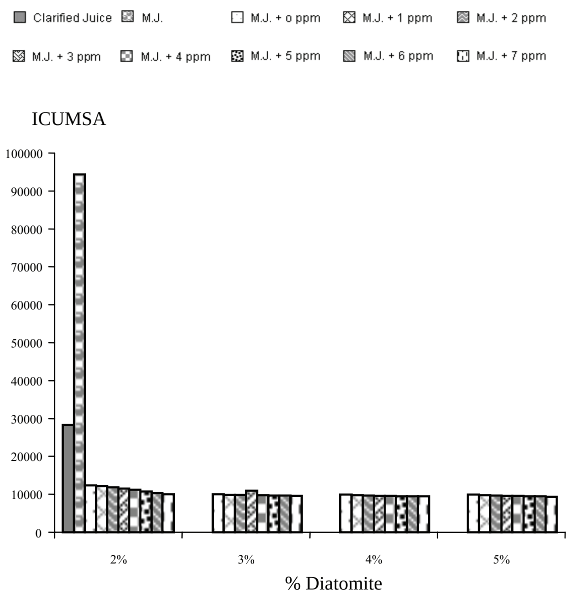

Figure 1 ICUMSA color of clarified juice and mixed juice when different amounts of diatomite and anionic polymer were added.

Abbreviation in Figure 1:

M.J. = mixed juice.

M.J. + 0 ppm, M.J.+1ppm, M.J.+ ...ppm = mixed juice followed by 0 to 7 ppm of polymer.

(Figure 2), but the turbidity was still higher (Figures 3 and 4). This might be because diatomite reacted better with the colorants in the juice than with haze-active protein, which is responsible for the turbidity of the juice (Siebert and Lynn, 1997; Siebert, 2006). However, both the ICUMSA and turbidity were lower than that of clarified juice when the amount of diatomite was increased to 4%. When 2% diatomite was added followed by 1 ppm anionic polymer, both color and turbidity of the treated juice were lower than that of the clarified juice (Figures 1 - 4). In each case the best result was obtained when the juice temperature was 90-103°C .

At the optimum conditions, i.e., 4% diatomite or 2% diatomite followed by 1 ppm polymer, in most cases, purity of the treated juices was higher than that of clarified juice except when 2% diatomite followed by 1 ppm polymer was used in limed juice (Figures 5 and 6). This implied that the use of diatomite or diatomite followed by anionic polymer not only reduced the color and turbidity of sugar cane juices but also increased the purity of sucrose in the juices. However, at neutral pH, co-precipitation of some sucrose in the juice might occur which resulted in the slightly lower purity of the juice, but this juice still could be processed further to produce sugar since the purity was higher than 80%.

The chemical compositions of Thai diatomite determined by x-ray fluorescence spectrometer (XRF) are shown in Table 1. The results indicated that SiO_2 which is the main constituent in diatomite was only 63.59%. Although the silica content in Thai diatomite was rather lower than diatomite from other resources, and the Fe content was rather high, we found that its adsorption properties was good enough to use

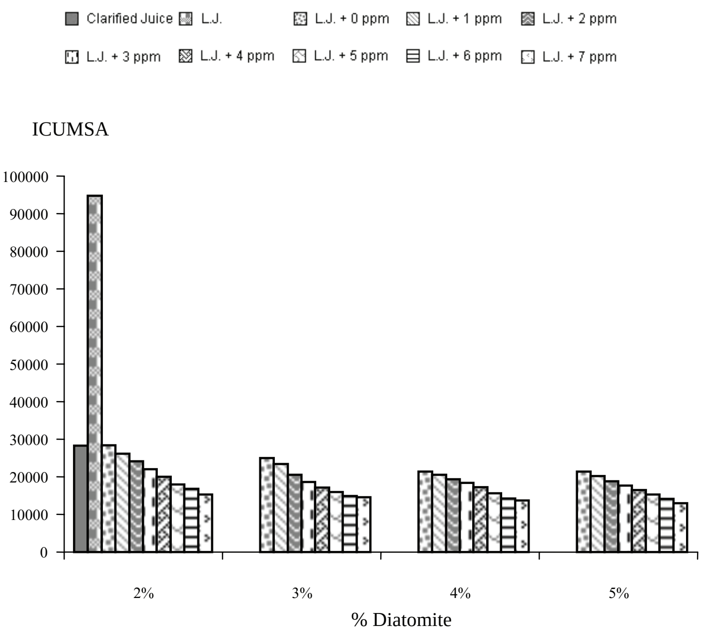

Figure 2 ICUMSA color of clarified juice and limed juice when different amounts of diatomite and anionic polymer were added.

Abbreviation in Figure 2 :

L.J. = limed juice.

L.J.+ 0ppm, L.J.+ 1ppm, L.J.+…ppm = limed juice followed by 0 to 7 ppm of polymer.

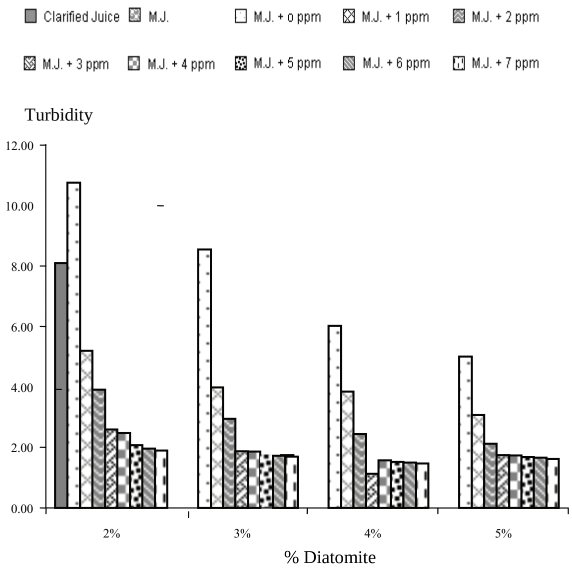

Figure 3 Turbidity of clarified juice and mixed juice when different amounts of diatomite and anionic polymer were added.

Abbreviation in Figure 3:

M.J. = mixed juice.

M.J. + 0 ppm, M.J.+1ppm, M.J.+ ...ppm = mixed juice followed by 0 to 7 ppm of polymer.

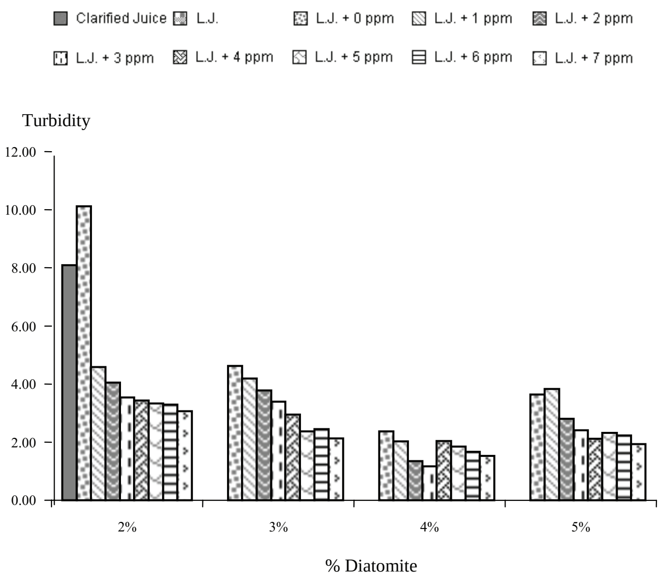

Figure 4 Turbidity of clarified juice and limed juice when different amounts of diatomite and anionic polymer were added.

Abbreviation in Figure 4 :

L.J. = limed juice.

L.J.+ 0ppm, L.J.+ 1ppm, L.J.+…ppm = limed juice followed by 0 to 7 ppm of polymer.

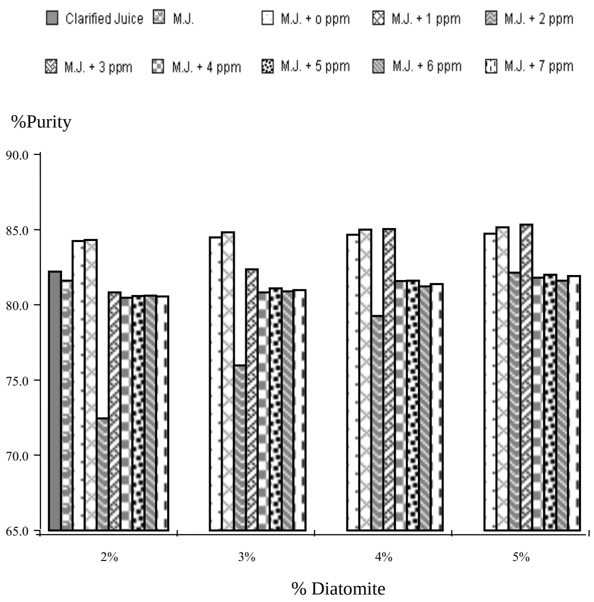

Figure 5 Purity of clarified juice and mixed juice when different amounts of diatomite and anionic polymer were added.

Abbreviation in Figure 5 :

M.J. = mixed juice.

M.J. + 0 ppm, M.J.+1ppm, M.J.+ ...ppm = mixed juice followed by 0 to 7 ppm of polymer.

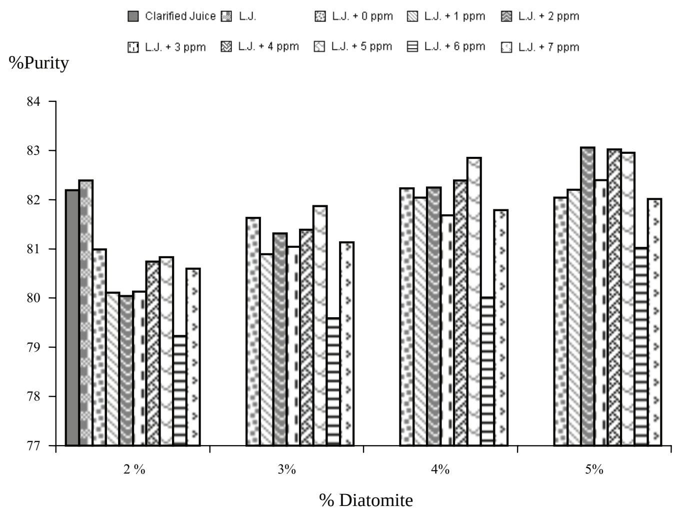

Figure 6 Purity of clarified juice and limed juice when different amounts of diatomite and anionic polymer were added.

Abbreviation in Figure 6 :

L.J. = limed juice.

L.J.+ 0ppm, L.J.+ 1ppm, L.J.+…ppm = limed juice followed by 0 to 7 ppm of polymer.

as an adsorbent in sugar cane juice.

Mineralogical analysis of natural diatomite used in this work is shown in Figure 7. Since natural diatomite composes mostly of amorphous SiO_2, a high purity diatomite such as those from Burney, California, usually gives a broad band in XRD pattern (Inglethorpe, 1992). The natural diatomite used in this work was about 70% pure, as confirmed by XRF analysis (Table 1) and this is consistent with the data that has been reported before (Inglethorpe, 1992) which stated that mineral impurities in Thai diatomite was about 30%, including clay minerals such as kaolinite and montmorillonite, quartz, goethite and hematite, the last two minerals were oxide and hydroxide of iron. All the intense peaks in XRD spectrum are due to the mineral impurities in diatomite. It is understood that the XRD band of an amorphous material is always obscured by the intense peaks of crystalline materials when the amorphous material contains such a high percentage of impurity. The 30% clay impurities in Thai diatomite was also confirmed by the particle size measurement which was found that the particle size smaller than 2 μm was about 30% (Benjamas, 2004). It was found that the XRD pattern and chemical compositions of diatomite in this work did not change after diatomite was used to remove color and turbidity of sugar cane juice. This means that the removal of color and turbidity in sugar cane juice did not affect the mineral or chemical compositions of diatomite.

Table 1 Typical chemical compositions of Thai diatomite (Benjamas, 2004).

Oxide	Compositions (%)
SiO_2	63.59
Fe_2O_3	17.76
Al_2O_3	14.49
K_2O	1.56
MgO	0.90
TiO_2	0.46
Na_2O	0.38
P_2O_5	0.29
CaO	0.13

The texture and morphology of diatomite before and after used were studied by Scanning Electron Microscope (SEM) as shown in Figure 8, as can be seen in Figure 8a, the natural diatomite before used had cylindrical structure with a number of pores on the surface. After it was used to remove color and turbidity in mixed juice and limed juice, the pores were diminished as seen in Figure 8 (b and c), respectively.

The surface area of diatomite was 32.96 m^2/g, which is in agreement with other research (Al-degs *et al*., 2001) and it was reduced to 4.69 m^2/g after used. The pore volume had the same trend with the surface area, adsorption of colorants

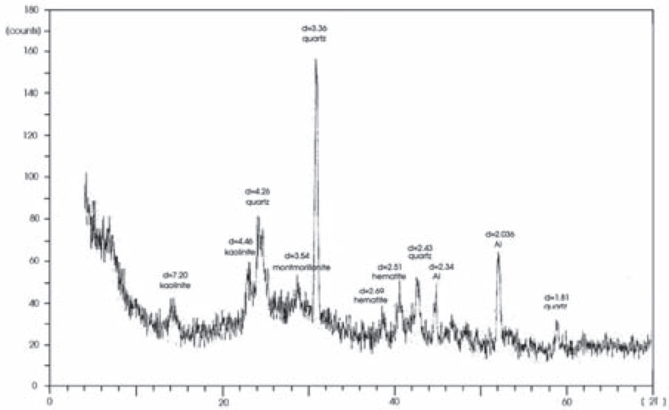

Figure 7 XRD pattern of natural diatomite used.

and turbidity-produced compounds in sugar cane juice caused the pore volume of diatomite to be decreased from 0.14 to 0.05 cm^3/g.

FT-IR spectra of diatomite before and after used are shown in Figure 9 (a and b). In the FT-IR spectra, the OH stretching frequencies of silanol and silanediol groups at the surface of diatomite were shifted from 3,432 to 3,394 cm^{-1}. The Si-O stretching was shifted from 1,099 and 1,030 cm^{-1} to 1,108 and 1,036 cm^{-1}, respectively. When diatomite reacted with the pigments in sugar cane juice such as anthocyanins, tannins and Fe^{3+}-phenol compounds which also consist of OH groups, hydrogen bonds were formed and led to the lowering of OH stretching at the surface of diatomite. This resulted in strengthening the Si-O bonds at the surface, so that Si-O stretching frequencies were raised. The lowering of OH stretching was consistent with the results reported by Yuan *et al.* (2004) .

The use of diatomite to remove color and turbidity in sugar cane juice involved both physical and chemical adsorptions. Physical adsorption was revealed by the reduction of surface area and pore volume of diatomite due to adsorption of colorants and turbidity-produced compounds into pores of diatomite. Chemical adsorption was revealed by the formation of hydrogen bonding between OH of silanol groups at the surface of diatomite with OH groups of color and turbidity-produced compounds in the juice.

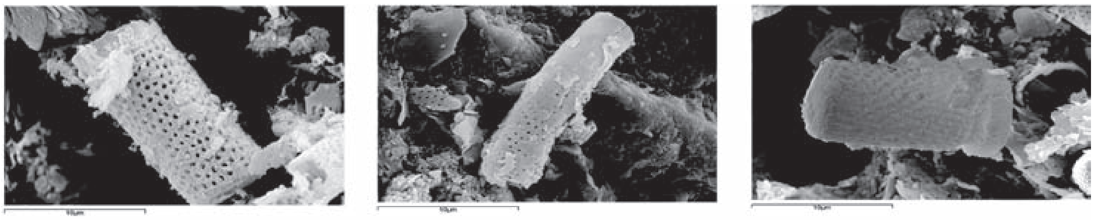

Figure 8 **(a)** SEM image of natural diatomite before used.
(b) SEM image of natural diatomite after use in mixed juice.
(c) SEM image of natural diatomite after use in limed juice.
All magnifications at 5,000.

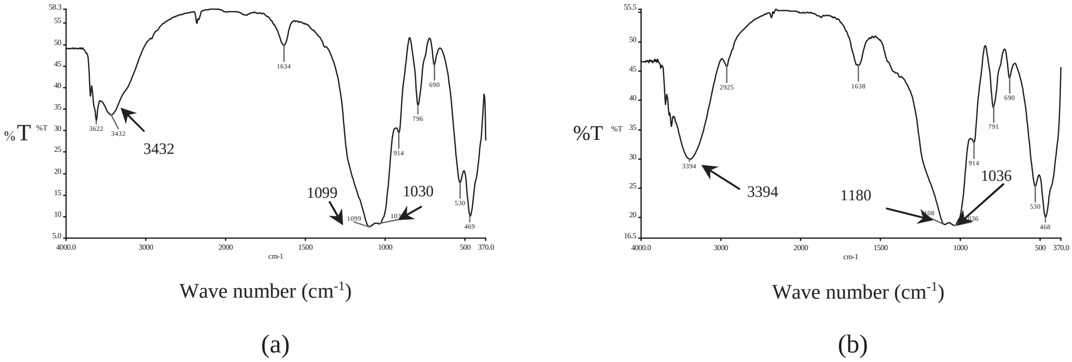

Figure 9 **(a)** FT-IR spectrum of natural diatomite.
(b) FT-IR spectrum of diatomite after used to remove color and turbidity in sugar cane juice.

CONCLUSION

Natural diatomite from Lampang province of central Thailand can be used to remove color and turbidity in sugar cane juice in industrial process lines. The ICUMSA color and turbidity of the treated juice were better (lower) than those of the clarified juice of the sugar factory, while the purity was higher. Two optimum conditions were obtained, to use not more than 4% diatomite or 2% diatomite followed by 1ppm polyanionic polymer, polacrylamide. Sugar produced from diatomite-only treated sugar cane juice is considered an "organic sugar" because no chemical was involved in the bleaching process. Analyses of the treated juice and diatomite revealed physical and chemical adsorption properties of diatomite.

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