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Behavior of Cationic Color Precipitants as Alternatives of Hydrogen Peroxide in Raw Sugar Melt Clarification Process

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Abstract

There is regenerate interest in the sugar industry to get to high performance in color removal during clarification process of raw sugar melt. Many efforts have conducted to approach the most proper alternative decolorants. The present study is focusing on investigation of cationic color precipitants "CCP" as alternatives of "H_2O_2" in clarification of raw sugar melt in Egypt. The experiments have performed using a pilot plant at Quos sugar factory, Qena governorate, Egypt. In these experiments, a comparison between two types of "CCP" (S TYPE 20 and TACELENE 4015) have been evaluated from the standpoint of decolorization efficiency. The behavior of the most effective matter has studied via the effect of dosage, pH, retention time and feeding point on the decolorization efficiency. A comparison between S TYPE 20 and hydrogen peroxide H_2O_2 in decolorization of raw sugar melt has conducted. The results show that, (S TYPE 20) is more effective than (TACELENE 4015) with percentage of color removal of 33.11% and 20.70% respectively. In addition, the results revealed that the proper experimental conditions for application S TYPE-20 are 7.4-pH, 30-minutes retention time and 100-ppm dosage. Addition of S TYPE 20 after clarification is the most proper feeding point. The results of comparison between S TYPE 20 and H_2O_2 show that decolorization percent are 40.40% and 27.70% respectively. Depending on these results, we concluded that the cationic color precipitants can be used successfully as decolorizing agents to replace hydrogen peroxide.

Keywords: ***Color, Decolorization, Cationic Precipitants, Hydrogen Peroxide.***

1- Introduction

Producing low color white sugar is the most important objective in sugar factories. The quantity of coloring matter in cane juice is very small on a percentage basis, amounting only to about 17% of the 1% organic non-sugars in cane juice. It has a very pronounced effect on the appearance of the juice or sugar. Its character is somewhat dependent upon the type of cane, soil and growing conditions, geographical area, and the milling and refining processes employed [(1)]. There are many methods has been done to develop a simple and economical decolonization process to remove colored impurities. These methods include modified clarification techniques; dissolved air floatation, membrane filtration, chemical precipitation, ion exchange, activated carbon adsorption and chemical oxidation via ozonolysis and hydrogen peroxide [(2)]. As part of efforts to find alternative materials to hydrogen peroxide as decoloring agent in sugar industry processes in Egypt, alternative decolorizing agents attracted the attention of researchers during the last period. Since most of the high molecular weight impurity and most of colorants being anionic in alkaline conditions [(3)]. So cationic color precipitants can be used to precipitate colorants and other anionic impurity from sugar liquors and this provides the basis for a new process of simultaneous clarification and decolorization[(4)]. Cationic color precipitants must have three properties for effective color removal as follows:

They should have a strongly basic (cationic) center, possess a long chain or cyclic hydrocarbon moieties and a balance between the above two components such that the precipitant is readily dispersible in sugar solutions. Cationic centers can attach to weakly acidic (anionic) centers on color molecules. This basic center is normally an amino nitrogen group. The ease of precipitation increases with the anionic charge on the color molecule. The long chain of n-alkyl hydrocarbon of not less than 16 carbon atoms, often with a fatty acid component which is conferred water insolubility (hydrophobic) upon the anionic impurity, then the impurity would be precipitated (5). In this regard, extensive results are approached for several chemicals, where the most active cationic precipitants were found to be the dialkyl quaternary ammonium compounds containing (two) C_{16} or two C_{18} hydrocarbon chains (4). One product that has developed for the sugar industry is Talofloc (dioctadecyl dimethyl ammonium chloride), where R= C_{18} (Figure 1).

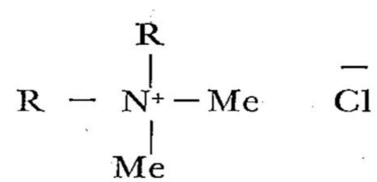

Fig (1): (TALOFLOC) model

The model of interaction of Talofloc with colorant molecules (6) is shown in Fig (2).

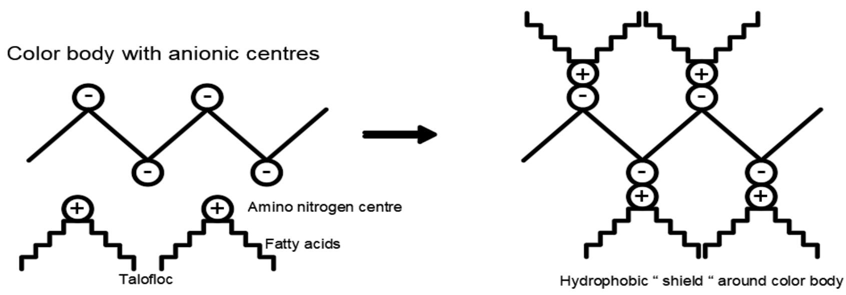

Fig (2): Reaction of Talofloc with colorant

There are several advantages for using cationic color precipitants "CCP" such as; they can been successfully used to reduce color of mixed juice and syrup at raw sugar and refining clarification process. They are an engineered processing aids added to mixed juice or syrup to inhibit color formation reactions, remove colorants and color precursors. CCP are high active chemicals traps colorants for further removal during juice clarification with the mud or floated scum. They are food grade liquid products with high advantages in safety handling and lower environmental impact [7].

Many efforts have been done for application of cationic color precipitants in decolorization of cane juice and syrup according to the conditions of every country [8-11]. In addition, cationic color precipitants can be used to precipitate colorant and other anionic impurity from washed raw sugar melt. In this line, in palmar factory in Venezuela, it was found that cationic color precipitant can been used with hydrogen peroxide in clarification of raw sugar melt [12].

In this study, we will evaluate the behavior to make use of cationic color precipitants in clarification of raw sugar melt as alternative decolorants to hydrogen peroxide. Quality parameters (pH, retention time, dosages and feeding point) with using CCP during clarification process of raw sugar melt will be taken into consideration.

2- Materials and Methods

Materials, analyses, and methods of experiments were performed on-site at the pilot plant for sugar research at Quos sugar factory. The experiments were designed according to the equipment's available at the pilot plant where the study was performed. Assessment of the clarified melt was made by analyzing for brix, purity, color, ash % brix, CaO % brix, pH and turbidity. All the methods used for analyses were in accord with the manual adopted for analysis in Egyptian Sugar Integrated Industrial Company (ESIIC) laboratories.

2- 1 Materials

The raw sugar used was obtained from the sugar mill, the color value was varying between 1500 and 2500 (m.a.u.) % brix. In a 150 liter, receiving tank equipped with mixer and steam coil, raw sugar was slowly added to hot water (± 82 °C) under stirring forming sugar solution of 60 to 65 % brix. The liquor was divided into equal aliquots to which various clarifying agents were dosed. Lime as milk of lime of Baume "± 5" obtained from the sugar mill, phosphoric acid concentration "5% w/v" where 1ml contain 39 ppm P_2O_5 and anionic flocculent solutions of 0.1 % (w/v) concentration were prepared on the day of experimentation and were stable for one week. Hydrogen peroxide (H_2O_2) was food grade, 50 % w/w, Al-N asr co. EGYPT. Two cationic color precipitants S TYPE-20 (20-±0.5 %, Carbo Solutions, USA) and TACELENE 4015 (50 %, Kemperlite, INDIA) were selected. Cationic color precipitants were diluted (1:10 w/v) before application.

2.2 Experimental method for clarification of Raw Sugar Melt

The experiments are performed to study the comparison between cationic color precipitants S-TYPE 20 and TACELENE 4015 with adding the same dosage of 300 ppm before clarification of raw sugar melt by phosphoflotation to evaluate decolorization efficiency for the clarified melt at semi-industrial scale. The trials were carried out to study the behavior of the most proper matter via the effect of dosage at (50, 100 and 200) ppm of CCP. The effect of pH of treated liquor at pH values of 6.8, 7.1 and 7.4, point of feeding (before and after) and retention time at 30 minutes and 60 minutes for resulted clarified melt and evaluating the clarification efficiency and its effect upon Brix, color, turbidity, pH, purity, ash % brix and calcium oxide content in each case were investigated.

On a pilot scale, the processes were conducted according to the following steps: a sugar solution of 62 ±1 °Brix was clarified by the phosphatation process. A batch of 80 liter of melted raw sugar was transferred and divided equally to reaction tanks and heating the melt to 84 ±1 °C. Phosphoric acid “5% w/v" was added until 300 ppm P_2O_5 content on solid. Lime “$Ca(OH)_2$ of Baume “± 5” obtained from the sugar mill was added to pH ± 7.2 with allowing a retention time of 8 min for complete reaction between phosphate and calcium hydroxide. Cationic color precipitants and hydrogen peroxide were added before or/and after adding phosphoric acid and milk of lime. A sample of reactant was taken to check the pH value. Aeration was done by recycling the melt to the same tank by the pump for 30 seconds followed by adding 10 ppm of anionic flocculant. The melt was let to float for about 40 min and then the trials were performed for choosing the most proper cationic color precipitant. A comparison was made with hydrogen peroxide at the same dosage according to the previous steps.

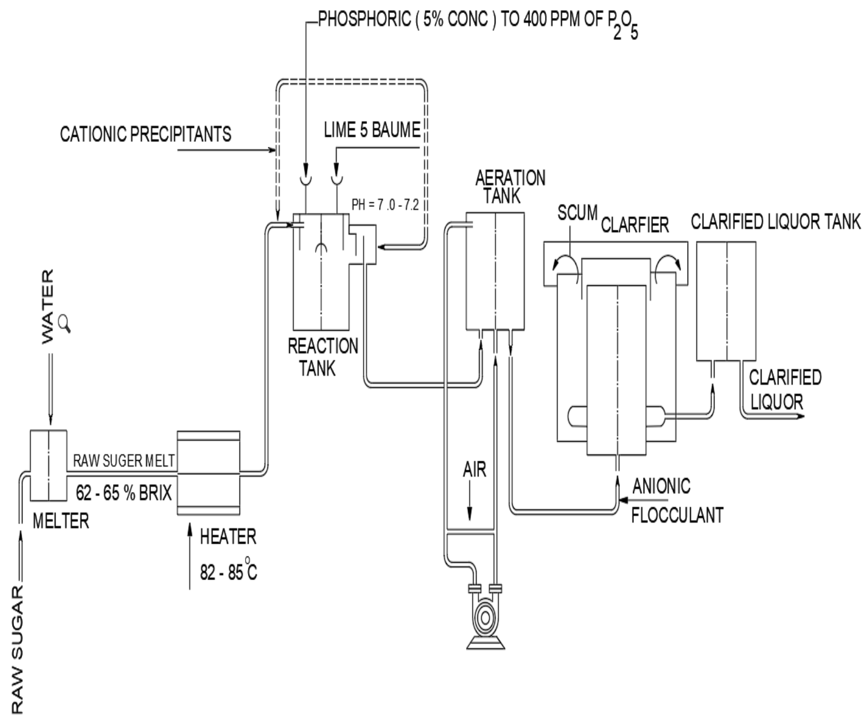

Fig (3): Decolorization of Raw Sugar Melt using phosphoflotation by Cationic Color Precipitants.

2.3 Analysis

The color of sugar solution was determined using the ICUMSA method at 420 nm by 7300 spectrophotometer - Bibby scientific (Jenway) U.E. Turbidity was determined by measuring the absorbance (A) at 975 nm in 1 cm cuvette cells against Mille-Q water (Louisiana). Brix was measured at ambient temperature using a digital refractometer (PTR 46, Index Instruments Ltd and UK). pH was measured using a pH/m v/temp pH meter (Jenway 3510, England 027500). Sucrose content or Pol % cm^3 was typically determined by measuring the positive rotation of polarized light via the double polarization method, using a PolAAr 3001 (Optical Activity Ltd., UK). Calcium oxide was determined by titration with EDT A according to ICUMSA method GS8/2/3/4-9 (2000). The conductivity ash was measured using conductivity meter type Jenway 4510 [13].

3- Results and Discussion

3.1 Comparison between different cationic color precipitants

A comparison between two types of cationic color precipitants, namely (S TYPE 20 and TACELENE 4015) is investigated from the standpoint of decolorization efficiency for clarification of raw sugar melt. The procedure was to add 200 ppm of S TYPE-20 and 200 ppm of TACELENE 4015 for the first tank and second tank respectively before clarification of melt. The experiment is conducted according to the previous steps and evaluated the effectiveness of these types on color, purity, ash % Brix, calcium oxide content, and turbidity for the clarified melt. The results of the trials are listed in Table (1).

Table (1): Effect of color precipitants on clarification of raw sugar melt at 200-ppm dosage of S TYPE-20 and TACELENE CHEM 4015.

Analysis	Raw Sugar Melt	Clarified Melt + S TYPE 20	Clarified melt + TACELENE 4015
BRIX (%)	62.61	61.65	61.87
PURITY (%)	96.55	96.63	96.60
COLOR (m.a.u) % BRIX	2507	1677	1988
COLOR REMOVAL (%)	----	33.11	20.70
TURBIDITY(IU)	186	6	1
TURBID. REMOV. (%)	----	96.77	99.46
pH	6.3	6.7	6.7
CaO % BRIX	0.120	0.087	0.094
ASH % BRIX	0.43	0.42	0.42

It seems clear from the Table (1) that the color removal percent achieved using S TYPE 20 (33.11 %) is better than that of color removal in case of TACELENE 4015 (20.70 %). Furthermore, the turbidity removal associated with TACELENE 4015 (99.46 %) is better than that of S TYPE 20 (96.77%). Nevertheless, the turbidity using STYPE-20 was still in the acceptable limit (±9) according to the manual adopted for analysis in ESIIC laboratories in Egypt. On the other hand, the results of analysis of purity, pH, ash % brix and calcium oxide % brix for the clarified melt are non-significant in both cases. As color removal was the primary objective of the evaluations, the cationic color precipitant S TYPE 20 was chosen to be under study for the following pilot plant tests.

From the previous results, the effect of some variables such as dosage, pH, retention time and feeding point on the decolorization efficiency of raw sugar melt by using the cationic precipitant S TYPE 20 will been studied.

3.2 Effect of dosage

The pilot plant trials are conducted to investigate the optimum dosage rates of STYPE-20. In this regard 50,100 and 200 ppm of (STYPE-20) were added on raw sugar melt before clarification. The results were evaluated from the standpoint of the quality parameters (brix, color, purity, pH and turbidity) of the resulted clarified melt. The average results of the trials are recorded in Table (2).

Table (2): Effect of color precipitant (STYPE_20) dosages on the quality of clarification of raw sugar melt.

Analysis	Raw Sugar Melt	DOSE (ppm)		
		50	100	200
BRIX (%)	61.17	59.77	60.29	59.12
PURITY (%)	97.69	97.59	97.90	98.15
COLOR (m.a.u) % BRIX	2313	1487	1370	1322
COLOR REMOVAL (%)	----	35.80	40.77	42.84
TURBIDITY (IU)	60	8	6	12
TURBID. REMOV. (%)	----	86.67	90.00	80.00
pH	6.5	6.7	6.8	6.7

It is evident from Table (2) that with increasing the dosage of (STYPE-20) from 50 to 100 ppm the color removal percent of the clarified liquor increases from 35.80 % to 40.77 %, after that the increase in color removal percent (42.84 %) at 200 ppm was non-significant. While the best turbidity removal percent (90.00 %) was achieved at 100-ppm dosage.

Even though the best color removal has been achieved at 200-ppm dosage, but the best turbidity is at 100-ppm dosage and due to cost considerations so the most proper dosage (100 ppm) must be considered.

3.3 Effect of Feeding Point.

The pilot plant trials are conducted to investigate the most suitable feeding point of cationic color precipitant STYPE-20 before or after clarification process. Using 100 ppm of STYPE-20 that have been added before and after clarification of raw sugar melt. The results are evaluated from the standpoint of the quality parameters (brix, color, purity, pH and turbidity) of the resulted clarified melt. The average results of the trials are cited in Table (3).

Table (3): Effect of adding 100 ppm of S TYPE-20 (CCP) on feeding point of clarification of raw sugar melt.

Analysis	Raw Sugar Melt	clarified melt+(CCP) before clarification	clarified melt+(CCP) after clarification
BRIX (%)	61.80	61.02	60.45
PURITY (%)	97.86	97.78	97.84
COLOR (m.a.u) % BRIX	2824	1819	1787
COLOR REMOVAL (%)	----	35.66	36.80
TURBIDITY(IU)	23	7	4.0
TURBID. REMOVAL (%)	----	70.00	82.61
pH	6.4	6.9	6.9

The results represented in Table (3) revealed that the color removal percent of resulted clarified melt is 36.80 % using S TYPE-20 after clarification of raw sugar melt, whereas the color removal percent of resulted clarified melt is 35.66 % using S TYPE-20 before clarification of raw sugar melt. Nevertheless, the difference was in fact not significant. The turbidity of resulted clarified melt using S TYPE-20 after clarification of raw sugar melt is 4 IU, whereas the turbidity of resulted clarified melt adopting also S TYPE-20 before clarification of refinery liquor is 7 IU.

Depending on the above results, the best results have been achieved when application of S TYPE_20 after clarification. These results confirmed those published by Bennet [(4)] who proved that this is the nature of cationic color precipitants where different cationic color precipitants show a variety of relationships.

3.4 Effect of pH Values

The pilot plant trials are performed to investigate also the effect of pH values on the efficiency of clarification of raw sugar melt, where 100 ppm of STYPE-20 were added to the raw sugar melt after clarification. The parameters are monitored to determine the efficiency of clarification according to quality parameters (Brix, purity, pH, color, turbidity, CaO % Brix and ash % brix) of the resulted clarified melt. The results of pilot plant trials are listed in Table (4).

Table (4): Effect of pH values on the clarification of raw sugar melt in presence of 100 ppm STYPE-20.

Analysis	Raw Sugar Melt	pH of treated raw sugar melt		
		6.8	7.1	7.4
BRIX (%)	60.96	62.09	62.14	62.15
PURITY (%)	98.31	98.41	98.25	98.40
COLOR (m.a.u) % BRIX	2934	2103	2131	1786
COLOR REMOVAL (%)	----	28.32	27.37	39.13
CaO % BRIX	0.138	0.114	0.116	0.117
ASH % BRIX	0.59	0.51	0.53	0.51
TURBIDITY (IU)	213	4.0	5.0	3.0
pH	6.4	6.4	6.8	7.1

It can be observed from Table (4) that the best color removal percent (39.13 %) after clarification of raw sugar melt is achieved at pH 7.4, while the color removal percent at pH's 6.8 and 7.1 was 28.32 %, 27.37 % respectively. This may be explained via the ionization strength of coloring matter that increases in alkaline medium, thus the negative charge of coloring matter was more concentrated on the surface and ready to be conjugated with the active center of cationic color precipitants as mentioned before[4]. The lowest turbidity of the resulted clarified melt was achieved at pH 7.4 (3 IU). The use of STYPE 20 with variation of pH values has non-significant effect on purity, ash %

brix, calcium oxide % brix as well as pH of the resulted clarified melt.

3.5 Effect of Retention Time

The pilot plant clarification tests are applied at different retention times (30 minutes and 60 minutes) using 100 ppm of STYPE-20. The parameters are monitored to investigate the efficiency of the resulted clarified melt. These are brix, purity, pH, color, and turbidity. The average results of pilot plant trials are listed in Table (5).

Table (5): Results of clarification of raw sugar melt in presence of 100 ppm of S TYPE-20 at different retention times.

Analysis	Raw Sugar Melt	30 min.	60 min.
BRIX (%)	60.33	59.19	59.19
PURITY (%)	97.39	97.92	97.81
COLOR (m.a.u) % BRIX	2641	1702	1713
COLOR REMOVAL (%)	----	35.60	35.14
TURBIDITY(IU)	43	7.0	10.0
TURBID. REMOVAL (%)	----	83.72	76.74
pH	6.5	6.8	6.8

It is of interest to mention here from Table (5) that the color removal percent of the resulted clarified melt at retention time 30 minutes (35.60 %) is better than the color removal percent of resulted clarified melt at 60 minutes (35.14 %). Nevertheless, the difference was non-significant. In addition, the turbidity achieved at retention time 30 minutes is 7 IU whilst the turbidity at retention time 60 minute is 10 IU.

From the above results, 30 minutes as retention time for clarification of raw sugar melt using S TYPE-20 must be considered.

3.6 Comparison between cationic S TYPE-20 and hydrogen peroxide.

Cationic color precipitant (S TYPE-20) was tested and compared with hydrogen peroxide (H_2O_2) in clarification of raw sugar melt. This was done by adding 200 ppm from STYPE-20 and 200 ppm H_2O_2 individually before clarification of melt. The experiment evaluated the effectiveness of these matters on clarified melt color, purity, ash % Brix, calcium oxide content and turbidity. The results of the trials are listed in Table (6).

Table (6): Comparison between the efficiency of S TYPE 20 and H_2O_2 on clarification of raw sugar melt at 200 ppm.

Analysis	Raw Sugar Melt	STYPE-20	H_2O_2
BRIX (%)	62.13	60.48	61.50
PURITY (%)	98.60	98.73	98.78
COLOR (m.a.u) % BRIX	2119	1263	1532
COLOR REMOVAL (%)	----	40.40	27.70
TURBIDITY(IU)	129	5.0	3.0
TURBID. REMOVAL (%)	----	96.12	97.67

pH	6.3	6.8	6.8
CaO % BRIX	0.100	0.081	0.095
ASH % BRIX	0.44	0.43	0.42

From Table (6) it can be concluded that the color removal percent of raw sugar melt using S TYPE-20 is 40.40 % whereas the color removal with using hydrogen peroxide is 27.70 %. The increase in percent of color removal adopting cationic color precipitant (STYPE-20) may be explained via the mechanism of the decolorization in each case. Where, in case of cationic color precipitants cationic centers can be attached to weakly acidic (anionic) centers on color molecules. This basic center is normally an amino nitrogen group. The ease of precipitation increases with the anionic charge on the color molecule. The long chain of n-alkyl hydrocarbon confers water insolubility (hydrophobic) upon the anionic impurity, then the impurity will precipitate this confirming results published by Davis [5]. However, in case of hydrogen peroxide H_2O_2 the molecule acts as a good oxidizing agent when it ionizes giving the hydroperoxide anion HOO^- that can oxidize some color matter such as polyphenols [14]. In our conditions of clarification, the negatively charged coloring matters are precipitated at alkaline condition with positively charged cationic color precipitant STYPE-20.

During the clarification process of raw sugar melt using H_2O_2, the quantity of lime suspension must be increased to a fixed P_2O_5 dose (300 ppm) to compensate the decrease of pH value inherent in H_2O_2 addition and to maintain a suitable clarified liquor pH value [14]. The turbidity removal percent of the resulted clarified melt by using S TYPE-20 is 96.12%, whereas the turbidity of resulted clarified melt in case of H_2O_2 is 97.67%. However, there is no appreciable difference between both of turbidity removal. In addition, the differences in results of analyses regarding purity, ash % brix and calcium oxide % brix for the clarified melt are not significant as well.

Fig (4): Comparison between the efficiency of S TYPE 20 and (H_2O_2) on clarification of raw sugar melt at 200 ppm.

Conclusion

In this study, experiments at a pilot plant scale were conducted for investigating the behavior of cationic color precipitants during clarification of raw sugar melt and choosing the most effective one.

The results indicate that S TYPE 20 is more effective than TACELENE 4015, where color removal percent is 33.11% and 20.70% respectively. The most proper conditions upon using S TYPE-20 are at 100-ppm dosage, 7.4 pH value and 30-minute retention time. This study showed that cationic color precipitants can be used before or after clarification process. The comparison between S TYPE-20 and hydrogen peroxide indicated that S TYPE 20 is more effective than hydrogen peroxide, where color removal percent is 40.40% and 27.70% respectively at the same dosage. In addition, the turbidity removal is convergent in the range from 96.12 % to 97.67 %. The experiments revealed that there is no change in ash % brix and calcium oxide % brix for clarified melt. Thus, we concluded that application of cationic color precipitants does not lead to any drop in pH of clarified melt besides not causing effect upon both CaO and ash content of raw sugar melt.

Depending on the above results, we can conclude that S TYPE 20 can be used successfully instead of hydrogen peroxide "H_2O_2" in decolorization of raw sugar melt.

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الملخص العربي

دراسة سلوك مرسبات اللون الكاتيونية كبديل لفوق أكسيد الهيدروجين
في عملية معالجة السكر الخام المذاب

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هدفت هذه الدراسة الي استخدام مرسبات اللون الكاتيونية كبديل لفوق أكسيد الهيدروجين في عملية معالجة السكر الخام المذاب. تم اجراء التجارب في وحدة البحوث النصف صناعية بمصانع سكر قوص. في هذه التجارب تم اجراء مقارنة بين مادتين من مرسبات اللون الكاتيونيه وهما "S TYPE 20 & TACELENE CHEM 4015 " لاختيار أفضلهما من حيث كفاءة الازالة اللونية ومن ثم دراسة سلوك هذه المادة خلال عملية معالجة السكر الخام المذاب للوصول الي أفضل ظروف يمكن عندها تطبيق هذه المادة، حيث شملت دراسة أثر الجرعة وموضع الإضافة وزمن الاحتجاز. كما تمت مقارنة بين مادة " " S TYPE 20وفوق أكسيد الهيدروجين وتأثير كل منهما على كفاءة الازالة اللونية للسكر الخام المذاب. وقد أوضحت النتائج ان S TYPE 20 أكثر فاعلية من TACELENE 4015، حيث كانت الازالة اللونية 33.11%، 20.70% على الترتيب. وعند دراسة سلوك S TYPE 20 اظهرت النتائج ان جرعة ppm 100، وزمن الاحتجاز 30 دقيقة، pH 7.4 تمثل أفضل الظروف التي يمكن عندها الوصول الي أعلي إزالة لونية اثناء معالجة السكر الخام المذاب. كما يمكن إضافة هذه المواد سواء قبل او بعد عملية المعالجة. عند مقارنة

استخدام S TYPE 20 وفوق أكسيد الهيدروجين أوضحت النتائج ان S TYPE 20 هي أكثر فاعلية من فوق أكسيد الهيدروجين حيث كانت الازالة اللونية 40.40 % و27,7% على الترتيب.

من هذه النتائج نستخلص انه يمكن استخدام مرسبات اللون الكاتيونية كبديل مناسب لفوق أكسيد الهيدروجين في عملية معالجة السكر الخام المذاب.