APPLICATION OF HABER COLOUR PRECIPITANT - IN SUGAR MILL SYRUP CLARIFICATION PROCESS FOR COLOUR REDUCTION

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ABSTRACT

Sugar is an important commodity in world of agricultural trade. It is mainly sucrose; a disaccharide made up of glucose and fructose, and is sourced from either sugar cane (Saccharum officinarum L.) or sugar beet (Beta vulgaris L.). Sugar obtained from sugar cane contributes to 70% of the world's sugar production. One of the most important parameters in raw sugar quality is colour. Every sugar technologist knows that, without good clarification of sugar cane, colour removal in the juice and syrup the production of good quality raw sugar is impossible. The purpose of clarification is the precipitation and removal of all possible non-sugars, organic and inorganic, and the preservation of the maximum sucrose and reducing sugars possible in the clarified juice. Poor clarification of cane juice and syrup complicates the entire process of sugar manufacture. The costs of sugar refining increase with the amount of colouring matter in the raw sugar feedstock. A reduction of colour in raw sugar or a cheap and effective method of removal in processing would lead to lower refining costs. In thisresearch paper, Haber 8031 - colour precipitant was used based on the laboratory evaluation to confirm its functional chemistry and efficiency which is a high charge medium molecular weight cationic polyamine polymer. HABER 8031 is completely water soluble and acts as a highly effective colour precipitants.

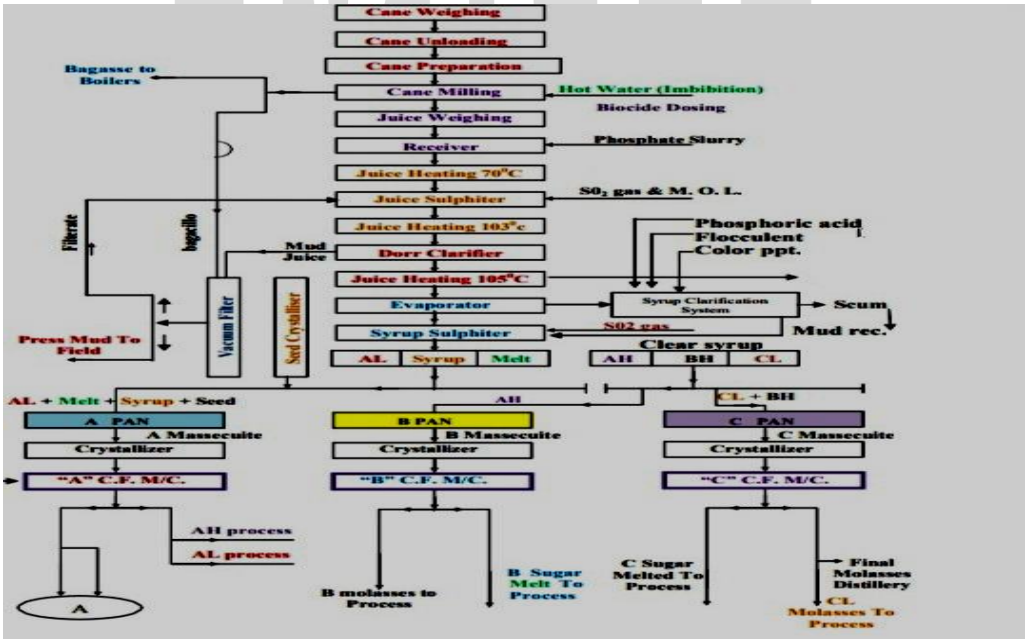
Key word:polyamine, poly DADMAC, syrup colour precipitant, ICUMSA, Colour Reduction

1.0 INTRODUCTION:

Sugar cane is harvested and cut on a seasonal basis.Harvested sugar cane is transported in large containers or bins to the sugar mill. The cane is then shredded and crushed (i.e., milled) to extract the juice. The juice is incubated and limed to remove impurities (e.g., starch) that affect subsequent processes and minimize sucrose inversion. The limed juice is then boiled (≥ 100 °C) and flashed before a flocculant is added to enhance the bridging of impurity aggregates.

The treated juice is then clarified to separate and remove flocculated impurities, fiber and soil. Clarified juice then passes to the evaporation stage, where water is removed to form syrup. In the crystallization process, the syrup is seeded and the crystals grow in vacuum pans, followed by separation of crystals by centrifugation. The separated crystals are washed and then dried to produce raw sugar ready for export or transferred to a sugar refinery for the production of white sugar. The flow sheet of the process given below.

SUGAR MANUFACTURING PROCESS FLOW SHEET NO: 1



1.1 MATERIALS AND METHODS

Color: The ICUMSA color method 4 using a wavelength of 420nm was applied for testing the color of light colored syrup, as usually made on clarified juice to measure effectiveness of clarification. Apparatus used for the purpose were; spectrophotometer equipped with 10, 5 and 1cm cells, membrane filters of 47mm diameter, pore size 0.45 µm, precision refractometer and reagents used was 0.1M TEA solution and 0.05M HCl, 0.05M NaOH.

Procedure: A diluted solution of sample to be tested, (50 brix solution) was prepared. For the brix of syrup was 65, then 100g of 50 brix solution was weighed 100/65 x 5 = 7.69g of syrup, made up to 100g and mixed. The pH of the solution was adjusted to 7.0 ± 0.2 with TEA buffer solution and mixed. (The use of TEA buffer solution is better than a 1M HCl or 1M NaOH solutions). The solution was filtered through a membrane filter with pore size 0.45µm. The solution was placed in an optical cell and measure absorbance, at 420nm in a spectrophotometer using distilled water as reference standard of zero color. (The cell length was chosen so that the instrument reading was between 20-80% transmittance and 0.1, 0.7 absorbance.) .The result was expressed as the following. ICUMSA 420 color unit = Where: b. Cell length (cm) c. Concentration of total solids (gm/ml).

Turbidity: The turbidity of clarified juice or syrup is a measure of the effectiveness of the clarification process. The ICUMSA Colors of unfiltered and filtered samples was measured at 420nm and the difference between the readings was the turbidity of the sample. Apparatus used were: Precision refractometer, pH meter, Buchner funnel, spectrophotometer, 1cm cell, Whatman No. 5 filter paper and the reagents were NaOH 1M solution, HCl 1M solution, kieselguhr filter aid. Syrup clarification isusing flotation system with optimized dosages of phosphate and anionic flocculant.

The original brix of the clarified syrup was determined and prepared a 65 brix (2) solution. The original brix = 60, then for 100g of 65 brix solution, the weight of the original clarified syrup taken for dilution = 6.5/60 x 100 = 10.833g. The amount of clarified syrup was weighed out necessary to make 65 brix solutions (in this case 10.833g of clarified syrup) in a beaker and made up to 100g with distilled water. The solution was divided into equal portions in two 100ml beakers. The first portion of the sample was taken and filtered through Buchner funnel with Whatman No 5 filter paper using kieselguhr filter aid. The first 10ml running was discarded. The pH of the filtrate was adjusted to 7.0 ± 0.2 using either 1M NaOH. Absorbance (A1) in 1cm- cell at 420nm was measured against distilled water blank. ICUMSA 420 Color C1 = Concentration of total solids (g ml-1) = Brix x true density/100. The 2nd portion of the sample (do not filter) was taken and adjusted to the pH to 7.0 ± 0.2 using NaOH. Absorbance (A2) was measured in 1cm cell at 420nm against distilled water blank. ICUMSA420 Color C2 = the result was expressed as the following: Turbidity of Clarified syrup= C2-C1 = 3.4.3

The pH : PH of sugar solution (50g/100g concentration) was determined using glass electrode attached to pH meter after calibration at pH 4.00, 7.00 and 9.00 at 20°C following ICUMSA method GS1/2/3/4/7/8-23 (1994)

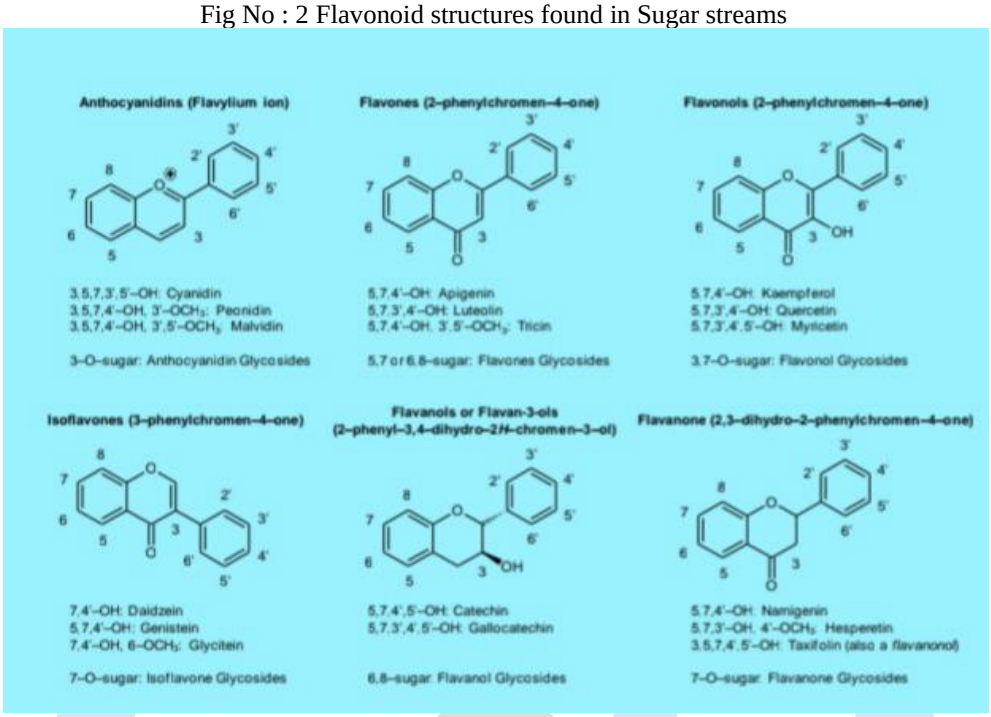
1.2: COLOUR CHEMISYRY IN SUGAR JUICE AND SYRUB

Colour in sugar process streams consist of a complex mixture of compounds. They are introduced naturally from the cane plant or produced during processing in the factory. The compounds formed have different molecular weights, chemical structures and properties as a result of degradation and polymerization reactions caused by changes in process parameters such as pH and temperature. The colorants that are difficult to remove are mainly hydrophobic in nature and persist throughout the sugar manufacturing process occluding within the sugar crystals.

Naturally Occurring Colourants: Chlorophylls and Carotenoids Sugar cane pigments are predominantly made up of chlorophylls, carotenoids (carotenes and xanthophylls) and flavonoids. These colourants are present in expressed juices after the milling of cane. Extraneous matter such as the tops and leaves of the sugar cane plant contribute significantly to colour in juice (3) colloidal in nature, chlorophylls and carotenoids are insoluble in water. Therefore, they do not contribute to the colouring of the final product as they are easily removed during clarification.

Flavonoids: Flavonoids are soluble and weakly acidic in nature and persist throughout the milling and refining processes. These compounds are essential for the growth of the sugar cane plant. However, their presence in processing significantly impacts on the colouring of raw sugar. Flavonoids contribute up to a third of the colouring in raw sugar according to Smith and Paton (4). This amount can considerably rise with juices expressed from whole green cane crop that contain tops and leaves. The colouring of raw sugar from flavonoids is attributable to the occlusion of flavonoid glycosides in the sugar crystals during crystallization. These naturally occurring compounds are divided into various sub-groups such as flavones, flavanols and anthocyanins and only differ in the numbering and positioning of hydroxyl groups on the C_6–C_3 flavonoid backbone structure as shown below.

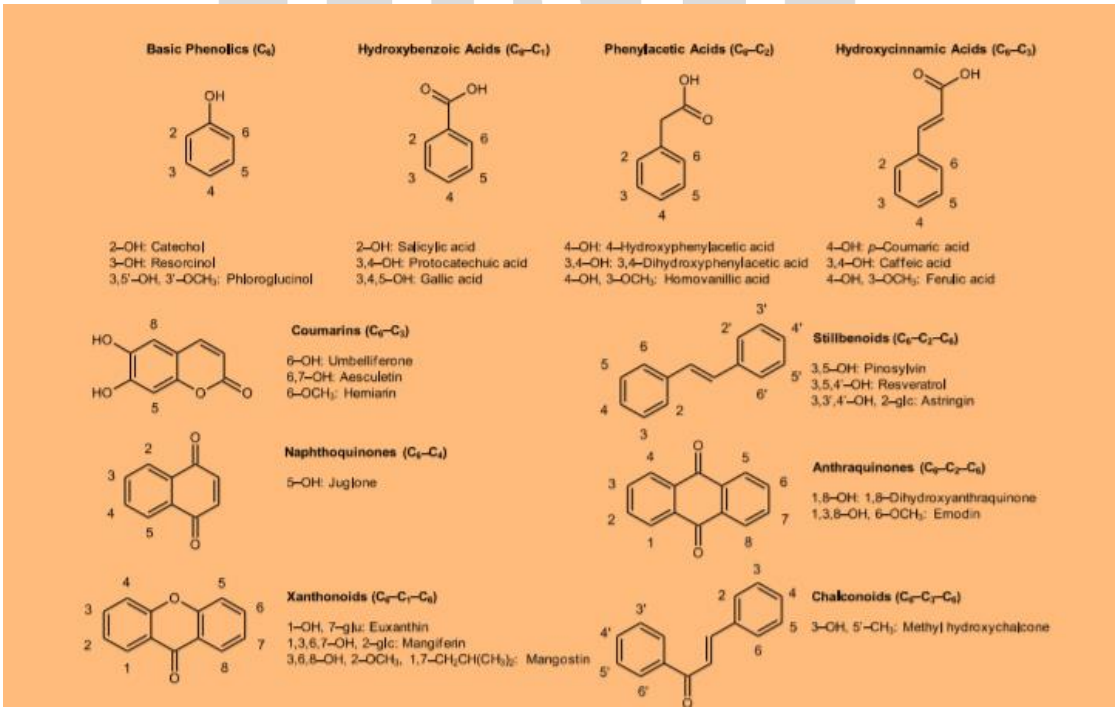
Fig No : 2 Flavonoid structures found in Sugar streams



Polyphenolic Compounds:Polymers consisting of multiple phenolic units are termed polyphenols. The number of repeating phenolic units varies; hence each polymer has a different molecular weight and structure. The simplest polyphenols are dimers of the monomeric phenolic units such as ellagic acid (i.e., gallic acid dimer). Intermediate polyphenols consist of two or more dimers of monomeric phenolic units (e.g., ellagitannin). The molecular weights and structures of simple and intermediate polyphenolscan be determined. However, this is not possible for complex polyphenols (e.g., lignin) which consist of repetitive monomeric phenolic units resulting in a macromolecule with an extremely HMW (5, 6).

Sugar cane polyphenols include lignins and tannins. Lignin is a complex macromolecule present in the cell wall of plants. The rigidity of plant stems is attributable to the presence of lignin with cellulose. Lignin comprises of three different phenolic units (viz., p–hydroxyphenyl, guaiacyl and syringyl); the proportions vary according to the type of cane plant and the extraction conditions (7). Tannins are polymeric products of phenolic compounds. They have the ability to form strongly coloured complexes with proteins to form stable, hydrophobic co-polymers.

Fig No :3 Poly phenols structures found in Sugar streams



Nitrogenous Compounds:The main group of nitrogen-containing compounds present in cane juice is amino acids and proteins. Proteins are complex HMW compounds made up of amino acids. The amount of proteins in juice is dependent on the cane variety,

soil type and harvesting conditions. Moreover, the levels of proteins in juice are relatively lower in sugar cane than in sugar beet. Proteins are of different isoelectric points some of which are removed during clarification while the remainder persists in the later stages of processing. Proteins denature and degrade to individual amino acid units as a result of heat and changes in pH. Amino acids produced from protein denaturation coupled with those endogenous to the cane plant are not removed during clarification and can react with reducing sugars via the Maillard reaction to form HMW dark coloured compounds.

Decolourisation using Chemical Additives:Chemical additives in the form of oxidants, precipitants, coagulants and inhibitors have been used to assist in the colour removal of sugar process streams. Arguably, SO_2 is one of the best performing decolorizing agents. The use of SO_2 as a bleaching gas, during sulfitation for plantation white sugar production, is known to produce very low colored sugar with a lustre appearance (8). It is widely used in under developed countries but discouraged in developed countries because of the residual sulfur contamination that is hazardous to human health. In addition, the low colour in these treated sugars are only temporary, with residual iron compounds, not removed during the sulfitation process, oxidize and colorize sugar crystals within a few months of storage (9). Organic polymers, such as polyacrylamides and polyamines, are commonly used for coagulation, flocculation and sedimentation processes during the clarification of juice andsyrup(10). However, the amounts of polymeric material added are limited due to cost and the possible presence of toxic residual monomers at higher dosages (11); hence these are usually dosed at lower concentrations with an additional process for optimum colour removal (12)

TABLE NO: 1

PHYSICAL AND CHEMICAL PROPERTIES OFHABER 8031

Physical state and appearance	Liquid
Odour	Amine-like
Chemical Nature	High charge medium MW cationic polyamine polymer.
Molecular Weight	Not applicable
Colour	Colourless to amber
pH	3.0 - 7.0
Specific Gravity	1.05 - 1.25
Viscosity	400 - 1000 cps
Freeze Point	7.0 °C
Flash Point	Not applicable
Solubility	Completely soluble in water
Halal & Kosher Certificate No	**2770300722**
FDA certificate No	**2773170822**

Reactivity of Colourants during Sugar Manufacturing

Enzymatic Browning Enzymatic browning is a colour forming reaction involving a phenolic and a nitrogenous compound, occurring prior to the heating of sugar cane juice to form melanins. The reaction is likely to take place after crushing and milling of sugar cane when the juice makes contact with atmospheric air. In general, the reaction involves an enzyme that acts as a catalyst to oxidize o–diphenolic substrates to o–benzoquinones (13). The o–benzoquinone can further react with a phenolic compound or an amino acid to produce a highly dark colored condensation product (i.e., melanins) (14, 15).The presence of PPO catalyses two reactions: the production of a diphenol (16) and the oxidation of the diphenol to an o–benzoquinone .

Factory Produced Colourants:

Melanins:Polyphenolic products formed by the enzymatic oxidation of phenolic compounds during processing are called melanins. The enzymatic browning is catalyzed by the polyphenols oxidase (PPO) enzyme responsible for the conversion of phenols into quinones (17).Quinones can then bind to proteins to form coloured polymers or undergo condensation to form dark colourants.

Melanoidins:

Melanoidins by definition are the coloured end products of the Maillard reaction between an amine (e.g., amino acid) and a carbonyl compound (e.g., reducing sugar). Also known as the non-enzymatic browning reaction, the reaction mechanisms are complex, consisting of repetitive condensation, dehydration and polymerization reactions resulting in dark brown coloured substances (18). The coloured Maillard reaction products (MRPs) formed are of varying molecular 17 weights, which are dependent on temperature and reaction time.

Aroma compounds:

Aroma compounds are reaction intermediates formed as a result of sucrose degradation, sucrose fragmentation and amino acid degradation. Some of these products are similar to those obtained from Maillard and caramelisation reactions. Intermediate products are capable of further reacting amongst each other to yield volatile products such as pyrazines, imidazoles and thiophenes. These products act as precursors of melanoidins since they either possess amino nitrogen or carbonyl groups, initiating the Maillard reaction.

Caramels:

Caramels are produced by the polymerization of thermally degraded products of sucrose at high temperatures (19). The products contain mixtures of oligosaccharides, polysaccharides and coloured matter (20). These colloidal compounds formed have a tendency to remain on the outer surface of the sugar crystals, which affect the quality of the final raw sugar product.

Hexose Alkaline Degradation Products (HADPs):Alkaline degradation products of hexose sugars are coloured products formed as a result of the thermal decomposition of reducing sugars. The end products mainly consist of carboxylic acids, carbonyl compounds and lower molecular weight (LMW) polymers, which can lead to inversion of sucrose and further colour formation. The degradation

rate and composition are heavily dependent on temperature, juice pH and the presence of divalent cations (21). The alkaline degradation rate of hexose sugars is much faster than under acidic conditions.

Colour in Sugar Process Streams:

As discussed in the formation of colourants produced during factory processing is mainly due to sugar degradation reactions. Reducing sugars, such as glucose and fructose, formed by the inversion of sucrose, play an important role in the formation of colour. These sugars degrade due to changes in operating conditions such as pH and temperature to form highly reactive intermediates, which undergo condensation and polymerization reactions to form highly coloured polymers. Colour precursors are of interest as they are not removed during juice clarification and polymerise to HMW coloured polymers and subsequently contribute to the colour in raw sugar (22). A wide range of cane pigments and natural colourants are introduced intomanufacturing process as a result of milling and crushing of harvested cane. The cane plant primarily consists of LMW compounds that contribute approximately 30% of the colouring in raw sugar (23). The remaining 70% is attributable to colourants produced in the factory, mostly polymeric and of HMW with different chemical structures and properties. Lindeman and O'Shea (24) reported that 50–60% of colourants by weight were of HMW and its contribution of these to the total colour in the final product, based on a standard spectrophotometric procedure at 420 nm, was approximately twice that of LMW colourants. Generally in Australia and most other parts of the world, colour is measured at pH 7.0; however the colour at pH 7.0 is the least stable.

For example, HMW colourants (e.g., caramels, melanoidins) are pH insensitive; therefore their colour does not change across pH 4.0–9.0. On the other hand, flavonoids and phenolic compounds (i.e., colour precursors) are highly pH sensitive. The colours of these compounds are lightly coloured at pH 4.0 but darken greatly at pH 9.0 (25). This is because at pH 9.0, the ionization of these compounds is almost complete. Hence, these compounds are more highly coloured in their anionic form than in their neutral form. As the pH significantly affects the molecular structure and association-dissociation equilibria of 32 colourant types in sugar process streams, it is possible to determine the different mechanisms of colour formation taken place during processing by measuring the indicator value (IV) (26). The IV is the ratio of colour at pH 9.0 to that at pH 4.0 and reflects on the pH sensitivity of the colourants present in sugar process streams (27). For example, a decrease in IV value shows a higher presence of HMW colourants and may be attributable to the Maillard and/or caramelisation reactions taking place. It is also important to note that lower pH sensitive compounds (i.e., HMW compounds) will appear to be visually darker than higher pH sensitive compounds (i.e., LMW compounds). This is due to the higher absorption of the lower pH sensitive compounds over most of the visible region and can be avoided if colour is only measured spectrophotometrically at 420 nm (28).

It was examined the colorations rate as functions of temperature, time, juice (sucrose concentration) and pH, which may impact on the coloring of raw sugar during the clarification and evaporation stages. An Arrhenius expression for the rate of colour formation in sugar manufacture was proposed as described in Equation given below

$$y = (0.8930 \times 10^{9}) e^{-\left(\frac{8502}{T}\right)}$$

where y is the colour formation rate (% of the initial colour per min)

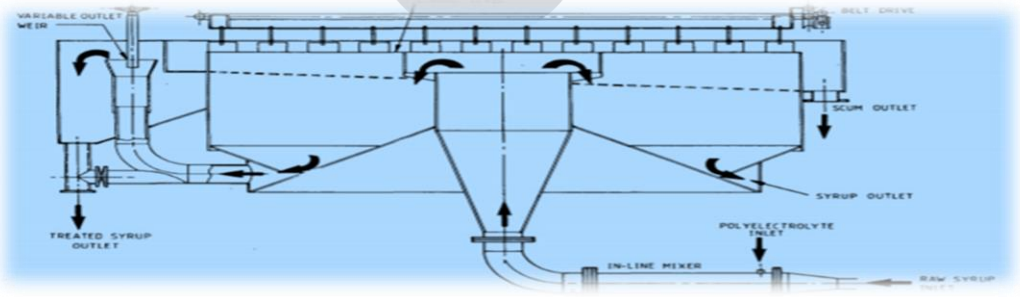
T is the absolute temperature (K).

right and Jegaraj (29) concluded that the extent of colour in raw sugar can be minimized by reducing the residence time of process streams at high temperatures (i.e., lower residence times during clarification, evaporation and crystallization processes), lowering the ESJ pH and reducing the content of nitrogenous compounds (i.e., proteins and amino acids) present in sugar cane plants.

1.3: RESULTS AND DISCUSSSIONS

Before conducting the plant trial, the basic principles involved in the coloring matters present in sugar mill juice and syrup was understand very clearly. And then the present clarifier conditions and chemical dosing area and patterns, quantity was noted down.

Fig No: 4Syrup clarifier Diagram



Present conditions: phosphoric acid dosage: 300 ppm on syrup solid %
Anionic flocculant dosage: 15 ppm on syrup solid %

Colour precipitant dosage: 120 ppm on syrup solids %
Avg Inlet brix % : 60
Avg Outlet brix% : 61
Avg Flow : 48 m3/hr
Avg Syrup Inlet pH : 6.0
Avg Syrup Outlet pH : 5.8
Colour Value as per ICUMSA method and Turbidity as NTU.
With this above basic details, the plant trial was conducted .Its results were tabulated as below.

Table No:2 Haber 8031 Colour precipitant plant trial Report

HABER COLOUR PRECIPITANT PLANT TRIAL REPORT @ SRYUB CLARIFIER ON SEP-2022						
Inlet Colour	Outlet colour	% Colour Reduction	Inlet Turbidity	Outlet Turbidity	% Turbidity Reduction	Remarks
15610	13416	14.06	18.2	5.5	69.78	Blank Colour Reduction
16438	14599	11.19	39.8	4.7	88.19	6.52%
14369	12552	12.65	25.2	1.2	52.38	Haber Trial Avg Colour reduction % 13.22
17403	14543	16.43	21	7.6	63.64	
16876	14616	13.63	32.4	6.6	79.63	
16967	14427	14.97	29.5	5.5	81.36	
16523	15007	9.65	24.2	8.9	63.22	

Fig No: 5 Haber 8031 Dosing Point

Fig No:6 Haber 8031 Dosing Point

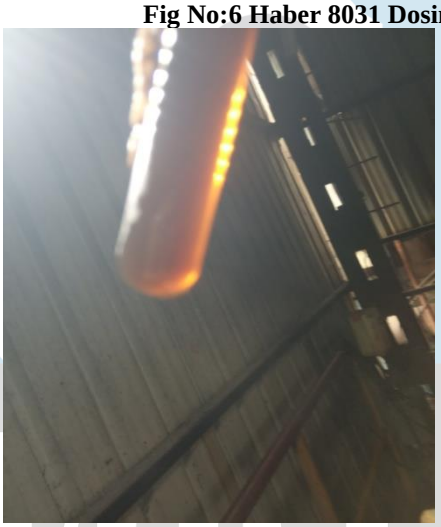
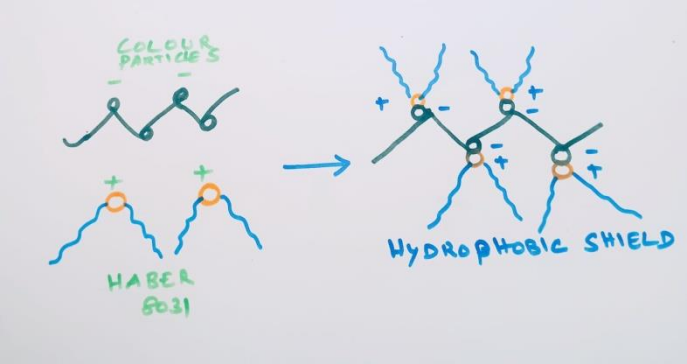


Fig No:7 Syrup Clarifier

Detailed descriptions of various chemicals tested and their structures are presented by Bennett et al (30) and Elvin (31), and these papers should be consulted for exact formulae.However, colour precipitants must have three properties for effective colour removal:
1. A strongly basic (cationic) centre which can attach to weakly acidic (anionic) centers on colour molecules. This basic centre is normally an amino nitrogen group. The ease of precipitation increases with the anionic charge on the colour molecule.
2. A long chain or cyclic hydrocarbon, often with a fatty acid component, which is hydrophobic.
3. A balance between the above two components such that the precipitant is readily dispersable in sugar solutions.
On addition to the sugar liquor, the basic centre is rapidly attracted to and binds with the acidic centers of the soluble colour molecules, leaving the hydrocarbon portion projecting away from the colour body. This creates a hydrophobic layer around the

complex (see Figure 1), rendering the colour body insoluble and it precipitates out of solution forming small black particles about 0.5 µm in size.

Fig No:8 Hydrophobic Shield by Colour precipitant



These particles are too small to be easily removed by conventional filtration, but are effectively scavenged by carbonatation and phosphatation precipitates.
Mochtar (32) mentions three stages in decolourisation by the Talo process.
1. The cationic surfactant reacts with the anionic colour to form an insoluble precipitate.
2. This precipitate is scavenged by the calcium phosphate to form a primary floc.
3. The addition of an anionic polyacrylamide polymer then increases the effective size of these flocs by bridging (secondary flocculation).
Bennett mentions further that the fatty acid chains reduce the interfacial tension between air and liquor at the surface of the flocculated particles, and hence improve adherence of bubbles to the precipitate during aeration. Hence, Haber 8031 has a greater benefit with phosphatation than with carbonatation. It should be noted that over-addition of the colour precipitant may cause the hydrophobic groups of the excess precipitant to attach to those already complexed with the colour bodies. This results in a further layer with ionic centers in contact with the solution, rendering the complex soluble once again. Hence, there is an optimum dosage of the precipitant that should not be exceeded for maximum effectiveness. Bennett et al give extensive results in this regard for a number of chemicals, with the optimum addition rate being in the range 100-150ppm. The range of the optimum dosage is relatively narrow, and it is recommended that periodic testing be carried out in factories to ensure best performance and efficient utilisation of the colour precipitant.

Conclusion:
A summary of their study is as follows: The formation of colour in the sugar mill juice and syrup was clearly observed .Rapid increase in colour formation was observed over time. The contribution of caramel colourants was small with or without an amino acid. Consistency in the behavior of amino acid (i.e., did not affect colour greatly) in both synthetic and factory .Lower levels of reducing sugars showed slower colour formation. Lower pH retarded colour formation, however higher pH levels rapidly produced more colour. Colour formation was prominent at 100 °C and negligible at 65 °C.• Higher brix content in juicesamples resulted in an increase of melanoidins and a decrease in HADP.Haber 8031 yields a better % Colour reduction as compared to existing one. The pan / Evaporator wash water affects the performance of colour precipitant.

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