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AN OVERVIEW ON THE CHEMISTRY OF CLARIFICATION OF CANE SUGAR JUICE

By

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Abstract

The main aim of the clarification process is to supply juice of optimum quality to the back end of the factory. All Australian raw sugar mills clarify mixed juice by simple defecation. This process is followed by air-flashing and gravity sedimentation. Despite advances made in recent times to improve the technological aspects of the clarification process, the variability of the cane supply still effects variability in clarification performance. This is evident from inconsistent control of juice quality and clarity, and mud levels. This paper reviews the present knowledge of the chemistry of clarification, identifies knowledge gaps and discusses potential areas of research that may lead to increased process capacity without expansion of the back end of the factory and improved raw sugar quality.

Introduction

In the simplest terms, clarification is defined as the separation of solids from liquid. The success of a clarification process depends on the correct application of the theories of precipitation, coagulation and flocculation. The aims of the clarification process in the raw sugar factory are to remove suspended and colloidal particles, remove non-sugar soluble impurities (e.g., proteins and polysaccharides) and raise the pH in order to minimise inversion of sucrose during subsequent processing. Clarification affects juice filterability, evaporator heat transfer coefficients, sucrose crystallisation and the quality and quantity of raw sugar produced. It therefore affects the colour, crystal morphology, crystal content, ash and polysaccharide content of raw sugar. In the raw sugar refinery, poor filtration and reduced refined sugar yields can often be traced back to poor clarification of mixed juice in raw sugar mill.

The scope of this review is to examine present knowledge of the chemistry of clarification of cane juice, identify knowledge gaps and discuss areas of research that may lead to improved raw sugar quality and increased process capacity, without expansion of the back end of the factory. Operational aspects, i.e., design of the clarifier and optimisation of the unit processes will not be discussed in the review, except where these operational aspects affect the chemistry of clarification. Outside the scope of the review is an account of the flurry of activity in the 1960s to early 1980s on the utilisation of flocculants and the performance of clarifiers in Australia. These activities are documented in many Sugar Research Institute (SRI) reports and papers published by this Society. However, it needs to be mentioned that SRI has made considerable improvements in the design of clarifiers. These design improvements have led to significant increases in capacity-to-size ratios of SRI clarifiers as compared to multi-tray clarifiers. The decrease in juice residence times has been remarkable—up to 3.5 h residence time for multi-tray clarifiers has been reduced below 20 minutes for the SRI clarifier. The impact that this decrease in residence time has had on clarification chemistry is not well understood.

Physico-chemical nature of cane sugar juice

Raw juice is a solution of sucrose mixed with a variety of insoluble and suspended matter such as bagacillo, soil, sand, clay, starch, waxes, fats and gums. It also contains soluble impurities such as proteins, glucose, fructose, oligosaccharides, polysaccharides, organic acids, amino acids etc. (Honig, 1953; Binkely and Wolfrom, 1953). The

amounts of impurities present in raw juice depend on the cane variety, maturity, soil type, cane condition and harvesting. The size of particles in raw juice range from about 2 mm to less than 0.5 μm. The number density in juice of particles up to 5 μm size varies between 2.5×10^8 and 1.2×10^9 particles per mL (Bennett, 1957). These particles give juice its turbidity. The surface properties of these particles are determined by the properties of adsorbed compounds. Although juice consists of heterogeneous materials, it was shown by Bennett (1957) that the materials in juice are electrophoretically homogeneous. The nature and surface properties of the particles are key determining parameters in the physico-chemical behaviour of juice to coagulation and flocculation.

There are basically four forces exerted on suspended particles in a liquid medium. These are gravity, van der Waals forces, Brownian motion and electrostatic repulsion. Gravity is the force which separates particles according to density, with the densest suspended solids moving towards the bottom of the vessel in which they are suspended. Van der Waals are short-range forces between suspended particles. These forces tend to draw suspended particles toward each other. Brownian forces impart motion to colloidal particles through collision of particles and molecules of the suspending medium. Electrostatic repulsion is the force between charged particles that tend to keep like charges apart.

The stability of cane juice is due to the following factors.

- The particles in cane juice carry a negative charge whose value depends on cane variety, cane condition and liming technique. The origin of the charge is due to the ionisation of functional groups of adsorbed materials on the particle surfaces. The charge on the particles in mixed juices varies with pH. The point of net zero charge is generally between pH 2.5 and 3.5. At the pH of net zero charge no coagulation will occur. At pHs even slightly above or below the net zero charge, pH coagulation begins to occur.

- The outermost adsorbed materials on the particles are mainly proteins, charged polysaccharides containing uronic acids or covalently linked phenolic acids or neutral polysaccharides that may carry an apparent charge through hydrophobic interactions with charged compounds such as phenolic acids. The extent of adsorption is primarily dependent on the solubility of the

materials, the pH and the ionic strength of the solution. For example, the presence of waxes and oil-soluble materials on particle surfaces influences the relative orientation of particles and inter-particle distances.

Two steps must be completed in order to remove suspended solids from cane juice. First, the surface charge of the particles must be neutralised sufficiently to overcome interparticle repulsion and for van der Waals forces to predominate.

Second, the coagulated particles must come together to form macrofloc particles. The century-old method for juice clarification of using lime (CaO) and heat meets these conditions. Neutralisation of the negative charge is achieved by the addition of lime and by dehydration or denaturation of adsorbed materials on the suspended particles. Calcium phosphate is the resultant material formed upon liming. The calcium phosphate particles and other materials adsorbed on the surfaces bind together to form floc particles.

Clarification processes

There are various methods used in clarification, all designed to remove impurities from juice. The clarification process used in the cane sugar industry is best described as simple defecation. The Australian process is based on the addition of lime as lime saccharate to heated juice at *ca.* 72–74 °C to a pH of *ca.* 7.8. This is followed by secondary heating of the limed juice to *ca.* 103 °C and flashing before entering the clarifier. Polyacrylamide flocculant is added at the feed launder in the clarifier. Insoluble calcium salts, mainly calcium phosphate from the reaction with inorganic phosphate, form flocs that settle in the clarifier. Where juice phosphate concentrations are low or clarification is poor, phosphate in the form of superphosphate or phosphoric acid is added to the juice tank prior to heating.

In other cane sugar milling industries, there are many variants of this simple defecation process. Milk of lime is often added instead of lime saccharate, lime addition may be in batches rather than continuous, and the point of addition may be to cold juice or to hot juice (heated in a single step)(Chen, 1993). These variations impact on the chemistry of clarification by affecting changes in calcium solubility, reaction time and temperature.

The carbonatation or carbonation clarification process is used extensively in the beet sugar industries of Europe and North America and in

some sugar refineries (Cleary, 1988). The process involves the addition of lime to juice to give an alkaline pH followed by addition of carbon dioxide to form a calcium carbonate precipitate. Other insoluble or sparingly soluble calcium salts co-precipitate, and colloids and other particles are adsorbed or enmeshed in the conglomerate precipitate (McGinnis, 1982).

Many sugar refineries use a phosphatation floatation process to clarify melt liquor. The process involves the addition of phosphoric acid or an acid phosphate to the process stream followed by lime (as a milk or as a saccharate) to give a slightly alkaline pH (7.0 to 7.4). The treated liquor is aerated and an insoluble calcium phosphate scum is separated by floatation. The resulting liquor is filtered on polish filters (Trott, 1988).

In many cane sugar producing countries, mills produce a 'white' sugar for direct consumption. The manufacture of blanco directo, turbinado or plantation white sugars requires more elaborate defecation methods and chemistries (Chen, 1993).
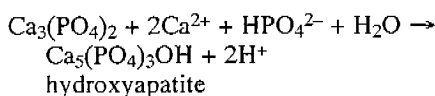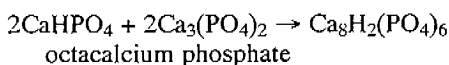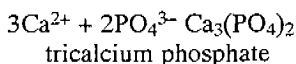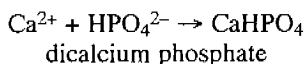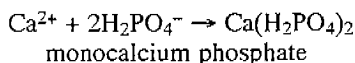
Some cane sugar mills use sulfitation to produce direct consumption and very low colour sugar. The process involves the addition of sulphur dioxide and lime simultaneously or in either order. Like simple defecation, variations on heating and point of addition exist in the process. The purpose of sulfitation is to prevent colour development in further processing and to reduce calcium salt carryover. Cane sugar mills in other countries may use a combination of carbonatation and sulfitation or phosphatation flotation and sulfitation to produce direct consumption sugar (Kulkarni, 1996).

The Talodura process developed by Tate and Lyle is used to clarify cane mill evaporator syrup by phosphatation floatation. The process is similar to that used in refineries for clarification of remelt sugar; phosphoric acid is added to the evaporator syrup followed by lime and flocculant addition and aeration (Chen, 1993). The process is used in addition to conventional juice clarification to produce a plantation white sugar or a raw sugar with very low colour and good refining characteristics. Since the resulting clarified syrup has a lower viscosity, crystallisation effects better molasses exhaustion.

Calcium phosphate chemistry

The literature contains publications on the chemistry of calcium phosphate precipitation in cane juice (Cameron, 1933; Kerr et al., 1952; Honig, 1953; Kerr et al., 1956; Bennett, 1967, 1971; Deben, 1976). The main reaction in clarification is that between lime and inorganic phosphate. The dominant calcium phosphate phases are as follows:

$$Ca^{2+} + 2H_2PO_4^- \rightarrow Ca(H_2PO_4)_2$$
monocalcium phosphate

$$Ca^{2+} + HPO_4^{2-} \rightarrow CaHPO_4$$
dicalcium phosphate

$$3Ca^{2+} + 2PO_4^{3-} \; Ca_3(PO_4)_2$$
tricalcium phosphate

$$2CaHPO_4 + 2Ca_3(PO_4)_2 \rightarrow Ca_8H_2(PO_4)_6$$
octacalcium phosphate

$$Ca_3(PO_4)_2 + 2Ca^{2+} + HPO_4^{2-} + H_2O \rightarrow Ca_5(PO_4)_3OH + 2H^+$$
hydroxyapatite

In addition to these compounds, tetracalcium phosphate $Ca_4(OH)_2(PO_4)_2$ is also formed. It was reported that the slowness of the calcium-phosphate reaction in reaching equilibrium is due to the slow dissolution of the intermediate $CaHPO_4$ and the slow rate at which $Ca_3(PO_4)_2$ precipitates. It therefore follows that the calcium phosphate obtained upon liming cane juice is made up of a number of compounds of varied composition and physical character. The fine particles of the calcium phosphate precipitated carry a positive charge which partially neutralises the negative charge of materials adsorbed on the particle surfaces.

As a rule of thumb, satisfactory clarification is achieved when cane juice contains about 300 mg of P_2O_5/L. However, there have been situations where good clarification was not achieved with double the normal requirement of P_2O_5 (Davies, 1936). It has been observed that too little lime will give poor settling and the juice will be cloudy. Too much lime causes the juice to be dark, ash content increases and the molasses output is high. Deben (1976) applied the theories of thermodynamics and kinetics of sparingly-soluble metal salts to account for some of the reported facts and is of the opinion that better information on juice behaviour to clarification could be obtained by looking at the calcium and phosphate molar ratio in juice. Deben (1976) in the same study reported that adjustment of the calcium-to-phosphate ratio to 1.75 in juice is necessary in order to achieve good clarification. As juices vary widely as to buffer capacity and calcium and phosphate contents, it would be

impossible to achieve optimum clarification with calcium saccharate alone.

A number of studies have been carried out on the interactions of some compounds on calcium phosphate precipitation (Honig, 1953; Kerr et al., 1956; Nancollas et al., 1979). Investigations have shown that citric acid, iso-citric acid and aconitic acid act as inhibitors to calcium phosphate precipitation due to the hydroxyl groups (Nancollas et al., 1979). Organic acids in cane juice play a primary role in increasing the buffering capacity of juice. The precipitation of calcium phosphate in juice is also adversely affected, either by the solubilising effect of the organic calcium salts on calcium phosphate, or by the failure to lime completely in the highly buffered juice. Large amounts of organic acids in evaporator supply juice cause severe scaling in the evaporators and pans.

Investigations have also shown that the presence of magnesium ions in juice stabilises amorphous tricalcium phosphate by decreasing its solubility (Feenstra et al., 1981). Magnesium ions have also been shown to inhibit the transformation of tricalcium phosphate to hydroxyapatite formation (Kibalczyc et al., 1988; Jourani and Bounahmidi, 1995). However, the exact nature of the magnesium ion influence is still not known.

Coagulation and flocculation

Coagulation (aggregation of small particles into big particles)/flocculation (linking up of big particles) would be spontaneous if not for factors which impart stability to suspended particles (viz. gravity, van der Waals forces, Brownian motion and electrostatic repulsion). These forces affect stable colloidal suspensions of microparticles.

However, flocculation of micro-particles in cane juice can be achieved by the addition of anionic polyelectrolytes based on copolymers of acrylamide and acrylate (Figure 1). Many factors affect flocculation. The main ones are polymer dosage, shear forces, particle sizes of suspended materials, molecular weight of polymer, solution density, pH of solution and temperature.

There are several hypotheses for the mechanism of formation of flocs, most of which remain unsupported by experimental evidence. One possibility, which must certainly play some part in floc formation, is the reduction of zeta potential (charge on the surface of a particle) by the adsorbed charged polymer molecules, allowing the particles to come together by Van der Waals

attraction. A second possibility is that the polymer attaches simultaneously to two particles by its two ends. Further adsorption then occurs, and as the polymer molecule is contracted, the particles are drawn closer together.

This theory seems unlikely, and since the polymer molecule is not linear, the ends will probably not be available for binding.

A third theory assumes the polymer molecules to be well tangled so that one polymer molecule will adsorb at several points on the surface of a particle, leaving loops, which may be of varying length, projecting out from the surface. Collision of these partially covered particles results in bridge formation (Ruehrwein and Ward, 1952).

Using this theory, it is possible to postulate that the polymer molecules may completely adsorb on individual particles before bridging formation can occur. This is most likely to happen at high polymer concentrations and explains why flocculation is seen to decrease above a critical dosage. In this case, flocculation will be further hindered by repulsion of the charges on the polymer molecules. This would seem to be the more feasible theory since it better explains the observed effects.

This bridging mechanistic pathway was found to explain cane mud flocculation by studies conducted by Whayman and Crees (1974, 1975) and Crees et al. (1991).

In one such study, a series of partially hydrolysed polyacrylamide flocculants was evaluated using the batch settling tests on limed juice.

It was found that the adsorption site for polymer-particle is through the acrylate ion. Figure 2 is a schematic representation of bridging of mud particles. In Figure 2(a) the polymer is used to link the negative charges of mud particles in juice via Ca^{++} ions. Figure 2 (b) is an illustration of the floc network formed.

Sugar refineries use cationic flocculants to remove colourants during the clarification process (Bennett, 1967, 1971; Bennett et al., 1971). There are some reports in which cationic flocculants are being used in conjunction with anionic flocculants in mixed juice to improve the quality of raw sugar (Li et al., 1996; Kampen, 1997). Investigations are being carried out by SRI on the effectiveness of cationic flocculants under Australian sugar mill conditions.

Although most water-soluble flocculant polymers possess linear configurations in solution, they are supplied as partially hydrated, coiled structures. For polymers to function at peak performance, they must be allowed to uncoil or
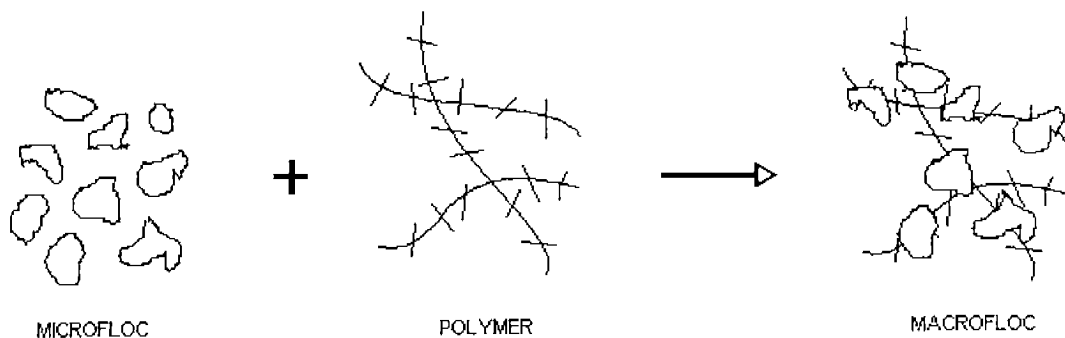
MICROFLOC + POLYMER → MACROFLOC

Fig. 1—Floc formation.
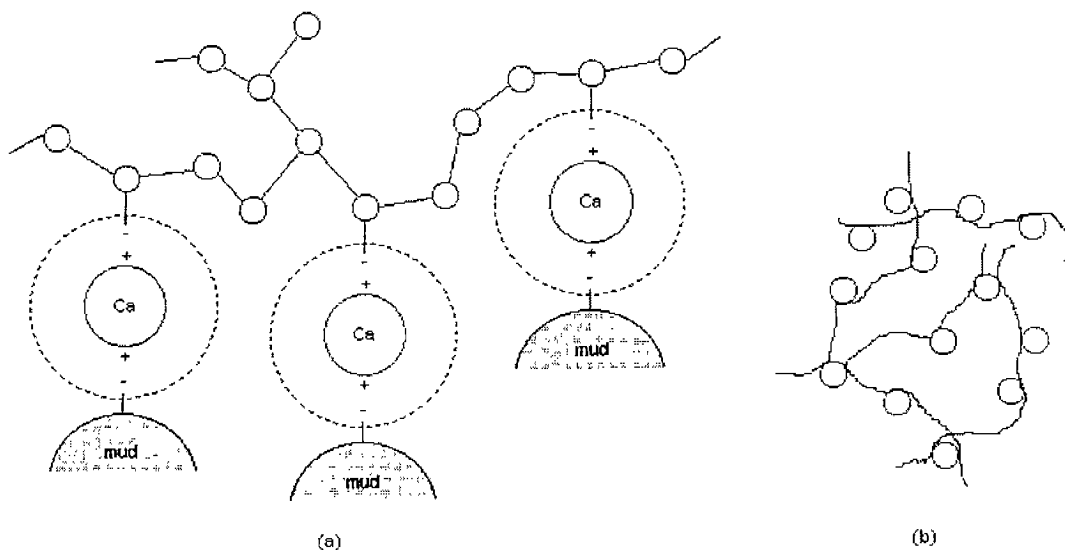

(a)   (b)

Fig. 2—Bridging of mud particles.

hydrate to their maximum capacity. As the charge density of the polymer increases, so does its ability to uncoil by virtue of internal charge repulsion. A polymer cannot stretch out to its greatest extent until it is completely dissolved.

Shephard (1978) investigated factors affecting the performance of polyacrylamide flocculant solution in juice clarification and came to the conclusion that, for optimum results, the flocculant solutions should be well mixed, the water used in dissolving the flocculant should be of low ionic strength and that the flocculant solution concentration should be low in order to facilitate efficient mixing with limed juice. The calcium and phosphorus concentrations in juice

were shown to influence differently the behaviour of the polyacrylamide flocculants with varying degrees of hydrolysis.

Opportunities to improve clarification

As clarification is the hub of sugar processing, further fundamental investigations into the subject are necessary since most studies in recent years have focussed on applied research. Despite a good understanding of the calcium-phosphate reactions there is still a dearth of knowledge on the interaction between juice constituents and the calcium phosphates and between the juice constituents themselves. With Australian sugar mills moving more and more to

green cane harvesting the impact this will have on the chemistry of clarification is not known. The following are outlines of areas that need to be investigated in order to understand better the chemical processes of clarification.

Pre-clarification

The main tasks during clarification are to clarify the juice and separate juice from mud. These processes are carried out in one step. This has made control of settling rate, mud level and turbidity extremely difficult. Variations in cane supply and quality from rake to rake affect clarification performance. A way to minimise variation in clarification performance is to carry out clarification in three steps:

1. Removal of sand, dirt and clayey materials;
2. Removal of bagacillo, pith, polysaccharides, gums, waxes, proteins etc;
3. Clarification by chemical means.

Clay particles are present in juice. They play key roles in the settling rate and turbidity due to their ion exchange capacity. Similarly, the other materials mentioned in steps (1) and (2) will affect clarification performance. It is proposed that a weir flotation centrifuge based on the counterflow centrifugal separation principle be developed for pre-clarification of juice prior to liming. The advantage of this technology is that steps (1) and (2) can virtually be carried out in one step.

Residual calcium

The contribution of residual calcium (i.e., calcium content) in raw juice to clarification is not well understood. There are many questions that remain unanswered. For example, how does residual calcium control the $CaO–P_2O_5$ relationship? What is the relationship between added calcium in cane juice and residual calcium in cane juice? What is the relationship between calcium in limed juice and calcium in clarified juice? What role do other juice constituents play in these relationships? What effect does the concentration of residual calcium have on scale formation, molasses output and the purity of raw sugar produced?

Clarification equation

It is apparent from the Deben (1976) studies, the Bogstra ratio (i.e. $P_2O_5/SiO_2 + Fe_2O_3 + Al_2O_3$) and from the investigations of Kampen and Tan (1997), that the use of the concentration of P_2O_5 in juice to predict clarification performance is inadequate. It is known that calcium ions tend to foster flocculation, magnesium ions and aconitate ions affect the volume of mud and sodium ions increase the zeta potential and hence the stability of colloidal particles in juice. Prediction of cane juice behaviour to clarification may be better achieved by expressions based on a comprehensive study on the composition of juice constituents. Such a study should include the identification of clay minerals which affect mud settling and the difference in composition as a result of harvesting green or burnt cane.

Relationship between juice composition and geochemistry

It is well known that the amounts of impurities present in raw juice depend on cane variety, maturity, soil type, cane condition and harvesting. Soils high in K_2O will increase SiO_2, Fe_2O_3 and Al_2O_3 in the clarified juice. Juice quality and scale formation are often specific to individual mill areas and may reflect environmental parameters. Research on environmental parameters such as base geology, soil chemistry, topography, soil mineralogy, fertiliser usage, groundwater and feedwater chemistry, and micro-mineral matter within the cane needs to be undertaken.

An integrated approach using juice composition and evaporator scale analysis, and the environmental parameters could give a better understanding of the many factors implicated in clarification performance. Empirical relationships could be developed which will assist in the prediction of clarification performance.

Polymers

The principle of colour removal is based on the necessity to employ a cation which interacts more strongly than calcium with organic impurity anions. This led to the synthesis of cationic polymers containing quaternary ammonium salts. These polymers have been reported to improve clarification in some refineries (Bennett 1967, 1971). Further research is needed into tailor-making cationic polymers of specific functionalities in order to achieve maximum complexation with colour compounds, colour precursors, negatively charged polysaccharides and organic acids present in cane juice. Also, research into preparing block polymers that have the ability to act both as a coagulant (a compound that effects instability in stable colloidal systems causing aggregation of microparticles) and a flocculant (a compound used to link aggregates and enhance sedimentation) should be pursued.

There is a report of a natural coagulant *Moringa oleifira* being used as an efficient coagulant for the treatment of turbid waters (Ndabigengesre *et al.* 1994). Although this coagulant has vaguely been described as a cationic peptide, research into its actual structure and mechanism of flocculation should be carried out. This could open up the way to a new group of cationic flocculants based on amino acids.

Flocculation mechanism

The chief chemist of a sugar mill will say that large fast settling flocs usually result in poor clarification, since the clarified juice contains increased quantities of small flocs and bagacillo, and has increased turbidity. As a result, a flocculant that will produce a medium sized floc with minimal carryover of bagacillo and small flocs, and low turbidity is used. Study into the mechanism of flocculation by anionic copolymers should be revisited since the bridging mechanism does not fully account for observed reactivity. Depending on the nature and type of anionic polymer, it seems that ion bridging, hydrogen bonding and charge neutralisation may all be important.

Despite the importance of the floc structure in flocculation, techniques available to characterise the structure of micron sized floc directly are still limited. Fractal theory, developed in the early eighties has been found to be able to characterise complex, random aggregate structures. Fractal geometry has been used for floc characterisation and in understanding floc growth. It is suggested that flocculation kinetics and dewaterability studies of calcium phosphate with cationic/anionic polymers be conducted using small angle laser light scattering (SALLS).

Hydrophobic modifiers

Hydrophobised polysaccharides have self-assembling properties and form complexes with hydrophobic and less hydrophilic compounds. Such polymers are expected to remove waxes, floc-forming components, colour compounds and colour precursors. The polymers have been prepared from water-soluble polysaccharides and polysaccharide derivatives by introducing hydrophobic moieties, such as long alkyl chains and cholesterol. A recent article describes the preparation of these compounds from wood components (Uraki *et al.*, 1997). It is proposed that a similar preparation should be tried with bagacillo in order to produce hydrophobicly modified bagacillo.

Conclusion

The basic chemistry of clarification of cane juice has been presented with emphasis on the simple defecation process. The processes involved in the coagulation and flocculation of cane juice were discussed and the important properties of flocculants presented. A number of areas were identified that may open up opportunities to improve juice quality and hence improved raw sugar quality.

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