

IRON MEDIATED CLARIFICATION AND DECOLOURISATION OF SUGARCANE JUICE

By

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Abstract

IN ORDER TO operate most profitably, the sugar producers in Louisiana wish to engage in a cooperative arrangement with the sugar refineries. Because the sugar refinery is an industrial scale decolouriser that operates using natural gas as fuel, it makes sense that sugar with less colour, produced using bagasse-power, would likely have greater profit margins. The removal of phenolic colorants from raw juice using native cane protein as a vehicle and Fe^{3+} as an oxidative catalyst was studied. Colour was removed as phenol-protein conjugates which rapidly precipitated with the addition of a cationic flocculant. The decanted juice was clarified via cold-liming. The treatment yielded clarified juice with up to 70% lower colour than hot-liming juice. It appears that the phenolics were oxidised by Fe^{3+} which engaged a REDOX cycle yielding quinoid species. The free N- ϵ -amino groups of lysine in the albuminoid proteins appeared to add to the quinones. Stoichiometry indicated a degree of polymerisation of eight. Oligomer formation ceased at this length which appeared sufficient to facilitate irreversible cross-linking and/or capping of the protein. The aggregates of iron, lignol(s) and protein were insoluble and precipitated. The process was tested in a 150 L settling clarifier which was operated in both pulsed and continuous modes. The method scaled well and the product juice exhibited 50–60% less colour than a cold-limed control when Fe^{3+} was applied in quantities ranging from 100–200 mg/L.

Introduction

Commercial cane sugar in the U.S. is refined from a raw sugar (~800–2500 IU) to yield a product with a colour of 15–50 IU. This arrangement is currently in a state of flux as global competition is encouraging vertical integration of the industry (Brady, 2005). Refineries use natural gas to power their operations. The white sugar premium is approximately 6.6 cents (Todd, 1997) per kg but the cost of natural gas can offset as much as 39% of this premium. The cost of the fuel used to process raw sugar is integrated with the price of cane. This price differential is pushing refiners to demand lower colour raw sugar to reduce their cost of refining.

The prohibitive expense associated with the implementation of affination in the raw sugar mill makes alternative options attractive. A wide variety of techniques and additives have been tested to either improve clarification and/or reduce the colour of raw sugar. A comprehensive review has been presented elsewhere (Madsen, 2006). There are no industrial-scale decolourising processes operating at raw sugar mills in the United States. The base technology is in the form of the carbon and resins that are used in refinery facilities.

It has been noted that the phenolic materials present in sugar originate in the cane. Removal of these materials from the juice will prevent them from propagating through the process. The colour load of the clarified juice rapidly saturates decolourising carbon which necessitates frequent regeneration via kilning (thermal desorption). This can be surmounted by a chemical regeneration process (Bento, 2006; Rein and Bento, 2006).

Additives can be used to bleach or inhibit colour formation in processing streams or on sugar. For example, hydrogen peroxide has been considered as a pre-treatment method for resin/carbon decolourisation (Bento, 2004) or as a bleach for sugar when applied directly (Saska, 2006). Iron salts can be used to effect clarification and decolourisation when used in tandem with ultrafiltration (UF), peroxide, sulfitation and/or conventional liming procedures. A hybrid decolourisation/clarification method similar to the work presented here was reported by Zerban (1921) who applied 20 ppm of Fe^{3+} and observed little effect. A patent (Madsen *et al.*, 1984) describes the use of ferric chloride as a chelant/oxidant. The agent served to create floc which was removed via ultrafiltration and the resulting liquor was hot-limed.

Iron is present in cane, and some 30–40% (Seip, 1947) of it is extracted into the juice during milling. Additional iron enters the juice as soluble salts (Subbarao, 1935) via the action of acidity upon mild steel piping and equipment. The total amount is about 10–20 mg Fe per L (van der Poel *et al.*, 1998) of juice. This iron is implicated in the formation of colour in cane juice, presumably via formation of both coloured complexes and oxidation products (quinones and polymer) with intrinsic phenolic compounds (Riffer, 1986). This mechanism can be exploited to effect coagulation of the phenolic colorants which could facilitate removal via UF.

Described in this work is a process where ferric iron is used, in conjunction with a cationic flocculant and conventional cold-liming to reduce juice colour without the use of carbon, resin or ultrafiltration.

Materials and methods

Iron clarification: Settling rate of flocs from raw juice

Raw juice was treated with an aqueous solution of Fe^{3+} as $\text{FeCl}_{3(\text{aq})}$, (Mallinckrodt, hexahydrate, 99.8%). The material was standardised against a five-point curve (EM, IX0230-2, 1000 mg/kg Fe^{3+} in HCl, $R^2 = 0.9999$), as the Fe^{2+} tris(*o*-phenanthroline) complex via absorbance (Beckmann Coulter DU-800) at 510 nm. The determination was made both with and without hydroxylamine as a reductor. By difference, it was determined that the working solution, made with 18 M Ω de-ionised water, contained 49 232 mg/kg of iron, 1.71% of which was Fe^{2+} . This solution was used in all subsequent tests.

Directly following the addition of Fe^{3+} to raw juice, the mixture was stirred and cationic polyamine (Ecolab PCS-3106) was added at 10 mg solid/kg juice. The polyamine content of the liquid preparation was determined via gravimetry to be ~60 g/100 g.

Immediately following the addition of the cationic flocculant, the juice was stirred and poured into an Imhoff cone and the settling rates were determined using a stopwatch. The time was marked onto the cone and the supernatant juice was collected via syringe. The supernatant was weighed and expressed as the mass percent of juice recovered/time. Mud was defined as the difference.

The mud was filtered through a coarse paper and the filtrate was mixed with the decantate. The mixed juice was brought to a pH of 7.1 with slaked lime ($\text{Ca}(\text{OH})_2$ 5g/100g) and then rapidly brought to boil using a microwave oven. To the boiling mixture was added anionic polyacrylamide (Magnafloc LT-340), 5 mg/kg as polymer). This mixture was settled as before to yield a clarified juice. The juice was adjusted to pH = 7 ± 0.1 , filtered through 0.45 μm membrane filter and colour was measured by absorbance at 420 nm. This process was repeated for the samples containing 50–600 mg/kg Fe^{3+} , in 50 $\mu\text{g/g}$ increments.

Pilot Test

Briefly, the ‘dual clarification’ method involved:

1. Treatment of raw juice, at ambient temperature, with approximately 150 mg/L Fe^{3+} and 10–15 mg/L of cationic flocculant.
2. Settling of this mixture to yield ‘stage-1’ juice.

3. Stage-1 juice was limed 'cold' to pH 7.1 and rapidly boiled.
4. To this was added up to 5 mg/L anionic flocculant. The mixture was settled to provide a decolourised, 'stage-2' juice.

A pilot-scale settling clarifier was constructed to hold approximately 150 L of juice and was designed to operate continuously at approximately 7.7 L/min. The clarifier was used to test the efficacy of the iron mediated clarification and decolourisation (FeMCA) process. The equipment used for this work can be seen in Figure 1.

The goal was to evaluate the colour removal both in batch (single-tank of juice dosed at once) and continuous modes of operation (steady-state with in-line addition of chemicals).

In the batch tests, the iron was added in increments from 0 to 50, 100, 150 and 200 mg/L in 150 L of raw juice. In continuous mode, the clarifier was brought to steady-state. The 150 L vessel was fed raw juice at 7.7 L/min. Iron and cationic flocculant were dosed to supply 150 and 15 mg/L, respectively. The stage-1 juice was taken off at a rate of 6.7 L/min and mud was removed via a progressive cavity pump at a rate of 1.0 L/min.



Fig. 1—Pilot clarification module tested at Raceland (2007 campaign).

Irreversible coagulation and a covalent mechanism

Protein, fraction V bovine serum albumin (BSA, Cohn fraction V), was used to make a solution that delivered $9.04\text{E-}8$ mMol/ μL (5853 $\mu\text{g/g}$) in de-ionised water. Acetate buffer (1 M) was prepared from de-ionised water and sodium acetate and was adjusted to pH 5.00 ± 0.05 (Oakton 11 series with Ag/AgCl probe) using glacial acetic acid (Mallinkrodt, AR).

A model phenolic compound, 3,4-dihydroxycinnamic acid (caffeic acid, CFA), was used to make a solution of $4.95\text{E-}5$ mMol/ μL (8825 $\mu\text{g/g}$) in a degassed matrix consisting of absolute ethanol and water, 1:1.

A solution was prepared from analytical grade sucrose (Fisher, ACS, 99.8% [α]_D²⁵=+66.5°) and 18 M Ω de-ionised water.

This solution contained 40 g/100 g (brix) and was standardised by refractometry (Bellingham and Stanley RFM340). With dilution, this solution was used as the base for the model solutions.

A 0.2 M phosphate buffer solution (PBS) was prepared by dissolving 9.36 g NaH₂PO₄, and 32.73 g Na₂HPO₄ into 1 L of de-ionised water. Eluent for gel permeation chromatography (GPC) was prepared using 250 mL of this solution and 17.53 g NaCl diluted to 1 L.

The eluent contains 50 mMol PBS and 0.3 M NaCl, at pH= 7.0 \pm 0.1. This solution was degassed under vacuum (24' Hg) with sonication prior to use.

In order to establish the stoichiometry that exists between BSA, CFA and Fe³⁺, the aforementioned materials were used to prepare a series of samples according to the matrix given in Table 1. The amount of BSA applied to each sample equates to 9.04E-5 mMol of protein or 5.42E-3 mMol of N- ϵ -NH₂-lysine. Fe³⁺ was added at 5.65E-3 mMol, a slight excess over the BSA amino equivalent.

The samples CFA 3 and 7 (bold text), represent one and two equivalents of CFA, respectively. To 15 mL polyethylene centrifuge tubes was added, in this order: water, sucrose solution, BSA solution, CFA solution, AcONa buffer and, finally, FeCl₃ solution.

Table 1—Sample matrix.

Sample	BSA, μ L	FeCl ₃ , μ L	AcONa, μ L	Sucrose 40 Bx	CFA, μ L	Water, μ L	Total, μ L	g/mL	CFA mMol
CFA 0	1000	22	125	2500	0	1353	5000	0.00	0.00000
CFA 1	1000	22	125	2500	59	1294	5000	104	0.00289
CFA 2	1000	22	125	2500	85	1268	5000	150	0.00416
CFA 3	1000	22	125	2500	111	1242	5000	196	0.00544
CFA 4	1000	22	125	2500	137	1216	5000	242	0.00671
CFA 5	1000	22	125	2500	163	1190	5000	288	0.00798
CFA 6	1000	22	125	2500	189	1164	5000	334	0.00926
CFA 7	1000	22	125	2500	221	1132	5000	390	0.01083
CFA 8	1000	22	125	2500	241	1112	5000	425	0.01181
CFA 9	1000	22	125	2500	261	1092	5000	461	0.01278
CFA 10	1000	22	125	2500	281	1072	5000	496	0.01376

The samples were sealed and swirled (vortex mixer) to mix and allowed to stand at room temperature (24°C) for 10 minutes. The samples were centrifuged at 3600 r/min for 10 min. The tubes were sampled and analysed via GPC.

The operational parameters for the GPC are given in Table 2. The instrument was standardised for molecular weight using aprotinin (6.5 kDa), carbonic anhydrase (29 kDa) and BSA (66.4 kDa) via absorbance at 280 nm.

A quantitative calibration was made using a mixed standard containing BSA and CFA. Since only one wavelength is available at a time, 330 nm was chosen because it is an absorbance maxima for caffeic acid ($R^2=0.997$). BSA has a very small absorbance at this wavelength, so quantitation could be made using the DRI ($R^2=0.995$).

The samples were analysed via GPC. The plots acquired using the DRI were not used because the BSA rapidly fell below the limit of quantitation. Under the conditions specified in Table 2, caffeic acid had a retention time of 14.68 \pm 0.04 minutes.

A response factor (amount/area) of 6.022E-05 \pm 4.7% was used for quantification. The points at 200, 390, 568, 780, 992, and 1204 μ g/mL represent 1.0, 2.0, 3.0, 4.0, 5.1 and 6.2 BSA (60 * LYS) equivalents, respectively.

Table 2—GPC parameters.

Pump	Waters 510
Detector 1	Thermo Differential Refractive Index (DRI), 45°C
Detector 2	Applied Biosystems UV-VIS, 330 nm
Detector hold-up time	1 minute, DRI to UV-VIS
Column	Shodex Protein KW-903, 300 mm X 4.2 mm (ID), 5 µm, 40°C
Eluent	50 mMol PO ₄ buffer, pH 7.0±0.1 and NaCl, 0.3 M, 1 mL/min
Degasser, autosampler	Perkin Elmer, Vacuum; BioRad AS3500
Acquisition/run-time	Dionex ACI, Dionex Peaknet 5.2, 30 min
6.5, 29, 66.4 kDa, min	16.17, 12.53, 9.45
Quantity BSA, 7.29 min.	491, 978, 1949 µg/g, RF= 1.12E-3, R ² =0.995
Quantity CFA, 14.68 min	115, 203, 397 µg/g, RF= 1.26E-5, R ² =0.997

Results and discussion

Decolourisation, given in Figure 2, reconstructed using equation 1, was dependent upon the dose of Fe³⁺. The limitation imposed upon the decolourisation process is the mud pack volume.

This is illustrated in Figure 3, which describes the behaviour of the mud-pack relative to the Fe³⁺ dose. Decolourisation reached a maximum of 60% using decantation for mud removal. The mud-pack volume was 20% greater than that observed with the standard clarification process.

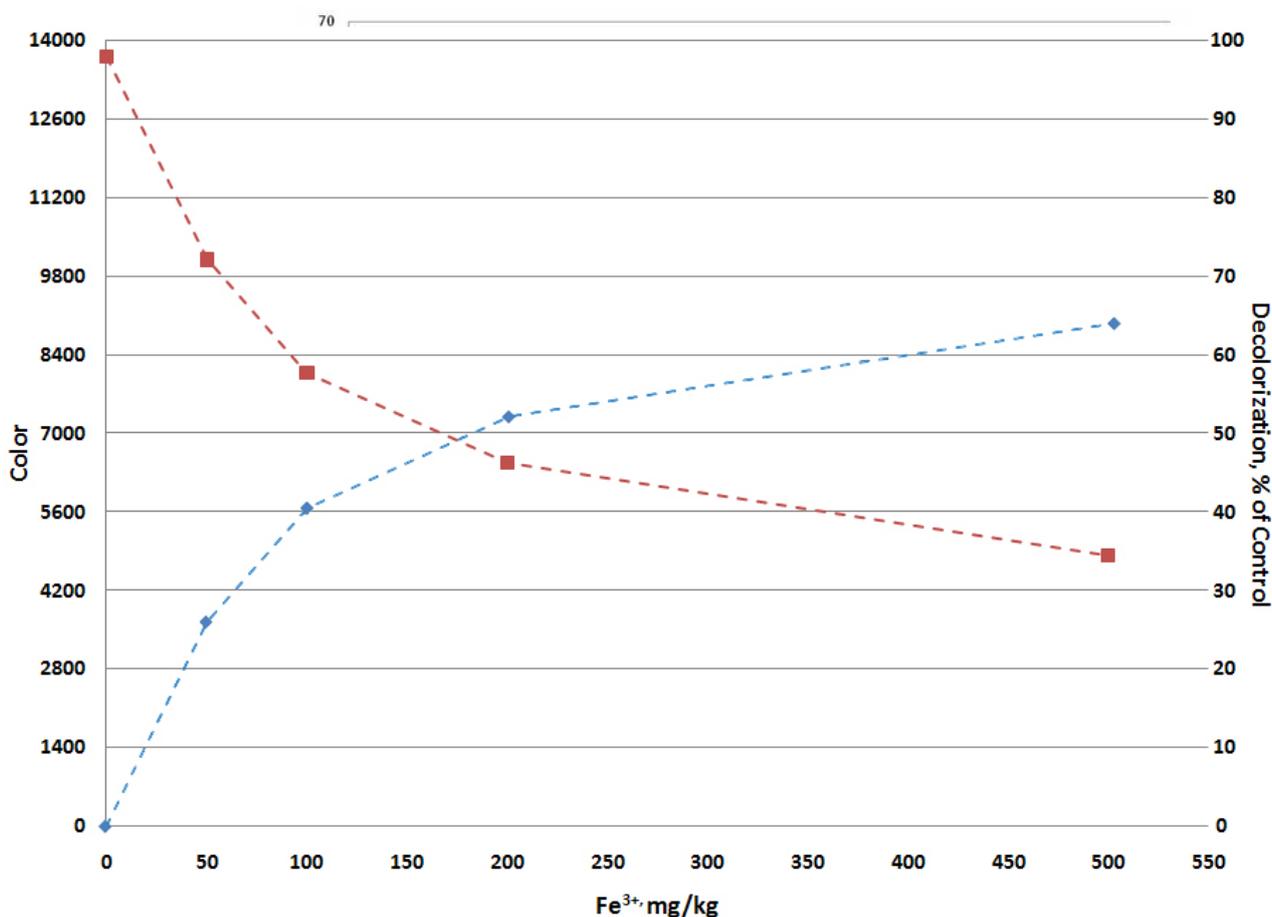


Fig. 2—The effect of iron dosage on colour removed with dual clarification (red), and extent of decolourisation as the percent of the control (blue).

The colour reduction, the colour removed relative to the control, was dependent on the amount of iron applied. This relationship appears to follow the following empirical equation, where Fe^{3+} is the amount of iron added in mg/kg.

$$Colour_removed, \% = \left(\frac{control_colour - 35289 * Fe^{3+^{-0.32}}}{control_colour} \right) * 100 \quad (1)$$

It was noted during the bench scale trials that, although the decolourisation continues to improve as iron levels are increased (up to a point), the settling rates decrease (and mud-pack volume increases). This can be seen in Figure 3 for juice settled for 20 minutes.

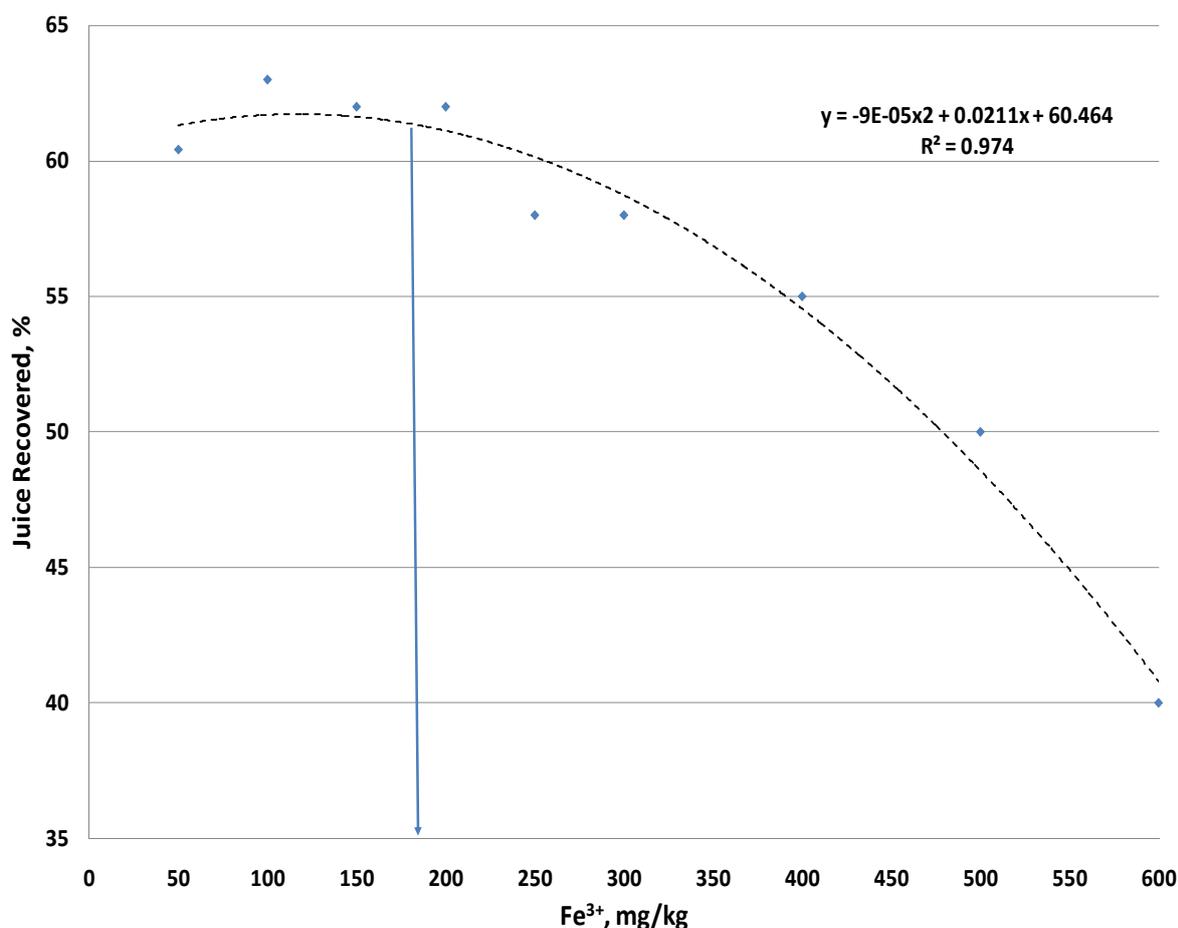


Fig. 3—Juice recovered in 20 minutes when increasing iron dosages beyond 200 mg/L.

In the laboratory, the juice can be filtered or centrifuged to provide a sample for analysis. Because this would be uneconomical to do in the mill, it was decided that dosing to 150–200 mg/L to provide a decolourisation of up to 50% was a practical limit.

While type/brand of anionic flocculant demonstrated little variation, the cationic materials were not equivalent. For example, products such as the Cytec Superflocs (regardless of charge density) performed marginally, while the PCS-3106 was exemplary.

A chart displaying the removal of juice colour, relative to a cold-limed control is given in Figure 4. The decolourisation % for each bar is calculated relative to a control taken at the same time from a tap preceding the dosing points on the clarifier module.

The behaviour of the pilot scale process is similar to that observed at bench scale and decolourisation behaves in a manner similar to that described by equation 1.

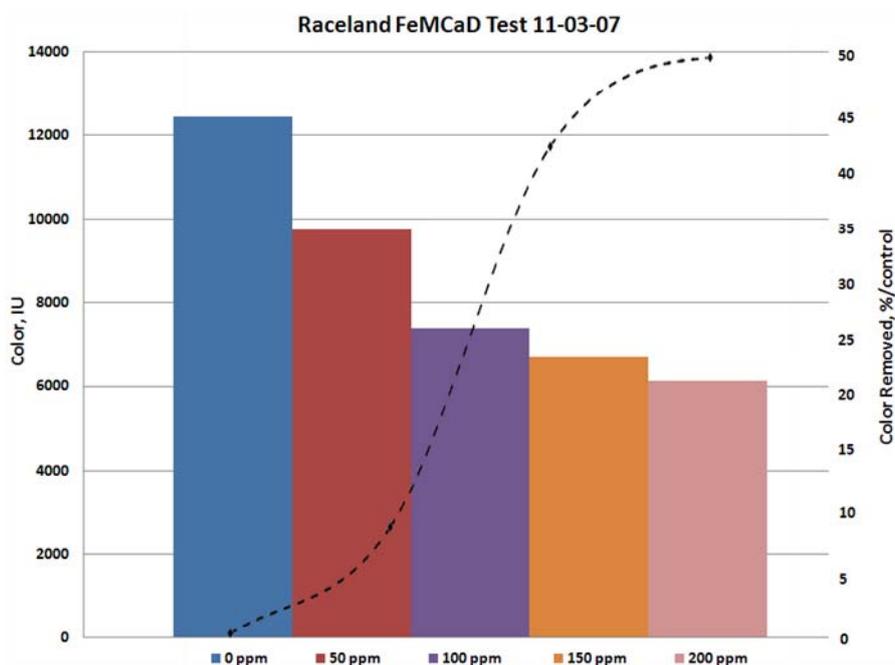


Fig. 4—Mill test; 150 L batches with increasing Fe³⁺.

The two step process yielded a low colour juice similar to what can be seen in Figure 2. After 45 minutes, the mud had a settled volume of 26 mL/100 mL. This volume of mud contained approximately 1.2 g of dry solids which was similar to that recovered during the bench-scale trials plotted in Figure 3.

The overall colour reduction, in pulsed tests, versus raw juice clarified by cold-liming (control) was approximately 50% when the iron was applied at 150–200 mg/L. When the iron dose exceeded this amount, the settling rate decreased. Thus, a dosage of 150 mg/L of iron was selected for tests where the clarifier would be operated continuously. The results were consistent with those achievable in the lab, in terms of dose-response and an equivalent nominal colour removal of ~50%.

The clarifier operated without incident for approximately six hours. During this time, the juice was collected, along with a concomitant control, limed, brought to boil using a microwave, flocculated (5 mg/L anionic flocculant) and settled to yield stage-2 juice which was assayed for colour.

This juice exhibited a level of decolourisation which was comparable to that observed in the batch test at equivalent dosage. It was concluded that the two-stage method scales well and can provide a clarified juice with 50% less colour than that achievable with normal hot or cold-liming procedures.

It was found that if cane juice is treated with iron, cationic flocculant and cold-limed, up to 50% of the colour in the juice can be removed at the clarifier. The liming step must take place before the juice is heated. If this order is reversed, the sucrose inverts and the juice will form colour on contact with air. The treated juice can then be limed and clarified normally.

The samples in Figure 5A appear to be identical. Upon the addition of the iron, it was immediately noted that samples containing two BSA equivalents (1.1E-2 mMol) of CFA precipitated immediately. The samples in Figure 5B were centrifuged. The precipitation of coloured material in Figure 5C is striking and increases with the amount of added CFA. Upon standing sealed for three days, all samples containing at least 1 BSA equivalent (5.4E-3 mMol) precipitated completely. The precipitate pellets in Figure 5D appear smaller because the mixtures were re-centrifuged to settle the flocs that had formed.

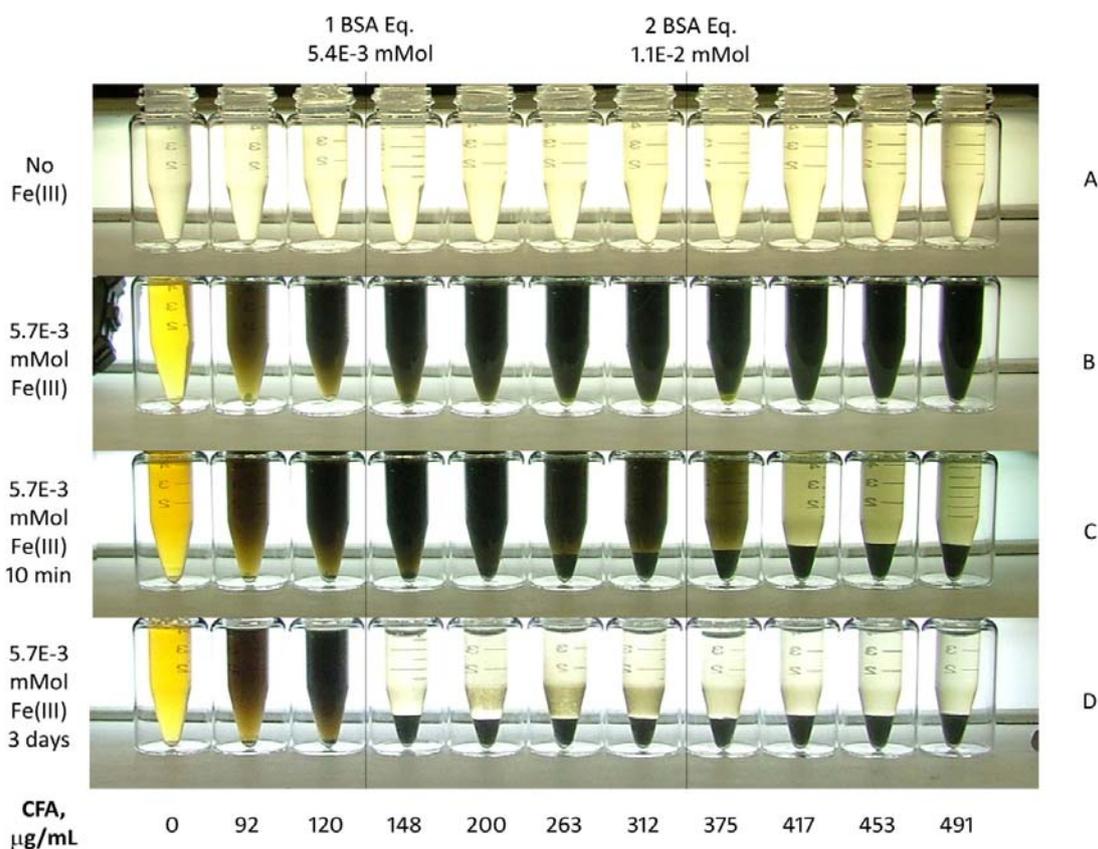


Fig. 5—Addition of iron and centrifugation of model samples. 148 and 375 µg/mL CFA correspond to CFA 3 and 7.

The amount of CFA consumed, expressed in Figure 6 in mg/L, inset, reached a maximum at 200 and was resolved at 500 µg/mL CFA. This is equal to 0.005 mMol and is very close to 1 N-ε-lysine BSA equivalent (0.0054 mMol).

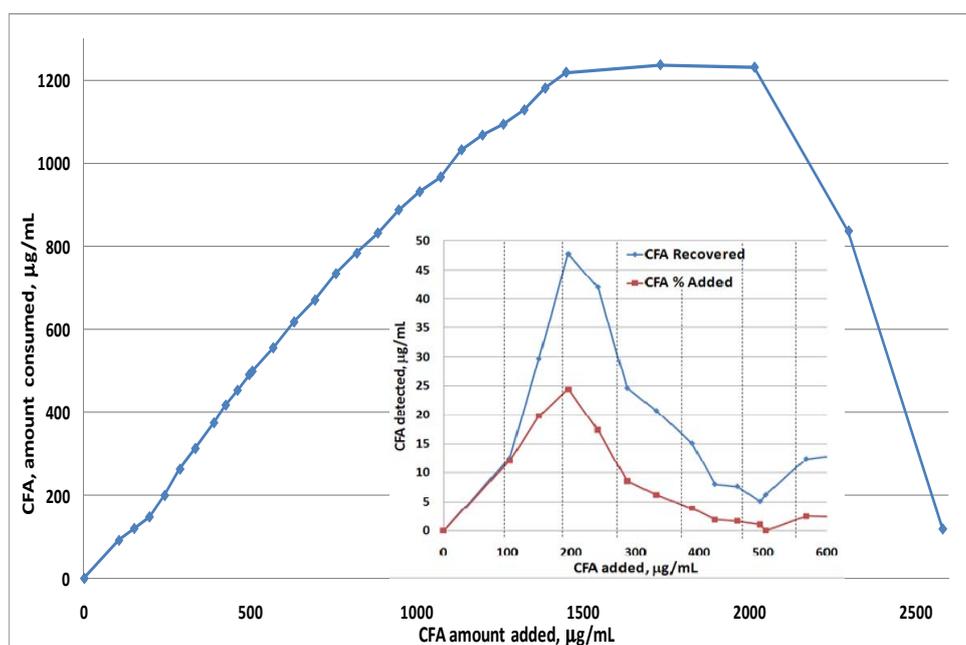


Fig. 6—Amount of CFA consumed. Note the plateau (steady state) and point of saturation (inset).

From this it appeared that at least two, simultaneous reactions were occurring. The first suggests a saturation effect and the second suggests polymerisation. Between 7.971 and 8.333 BSA equivalents of CFA are needed before the rate of consumption stabilises. Approximately 14.5 equivalents are needed before the amount of CFA detected equals the amount that was applied.

The plot in Figure 6 suggests that there is a fast initial consumption of CFA which ended when ~ 1 lys-BSA equivalent, or 0.05 mMol of CFA was added. This suggests that the free sites are reacting/binding until saturation. Following this, there is a short induction period, possibly the accumulation of an undetectable intermediate, followed by a linear increase in consumption. This implies that either the residues added in the prior step are reacting with further CFA and removing it from the bulk solution or the CFA is homopolymerising.

It appears that first, the free N- ϵ -amino-lysine groups are reacting with the phenolic species, probably in the quinone form. Then, the quinone end-groups are reacting further to extend phenolic chains outward. This continues until either the CFA reservoir is exhausted and/or the chains extending from two separate proteins meet and couple.

From the stoichiometry given in Figure 6, where the maximum CFA consumed amounts to 0.0343 mMol, it can be derived that the equivalence to BSA is approximately 380:1. On free amino-groups this amounts to a ratio of 6:1. There appears to be, depending upon the cross-linker used, of the 60 lysine residues in BSA, approximately 8–12 are able to crosslink with the N- ϵ -NH₂ lysine(s) of other protein molecules in the bulk solution (Huang *et al.*, 2004).

If the number of chains per protein is, on average, 10, the CFA to amino group ratio then becomes 38:1. Dividing this figure by two, for two interacting protein molecules gives a bridge length or degree of polymerisation of approximately 8.

Upon examination of the stage-1 (acidic Fe³⁺) process under a microscope, it was found that the application of 6 M urea failed to disrupt the floc, and the addition of EDTA caused only a marginal disassociation. Addition of *o*-phenanthroline caused the flocs to become larger and more robust. This is demonstrated in Figure 7. GC-MS analyses of mixtures of juice and phenanthroline with and without iron indicated that the ligand was either removed from solution or otherwise destroyed.

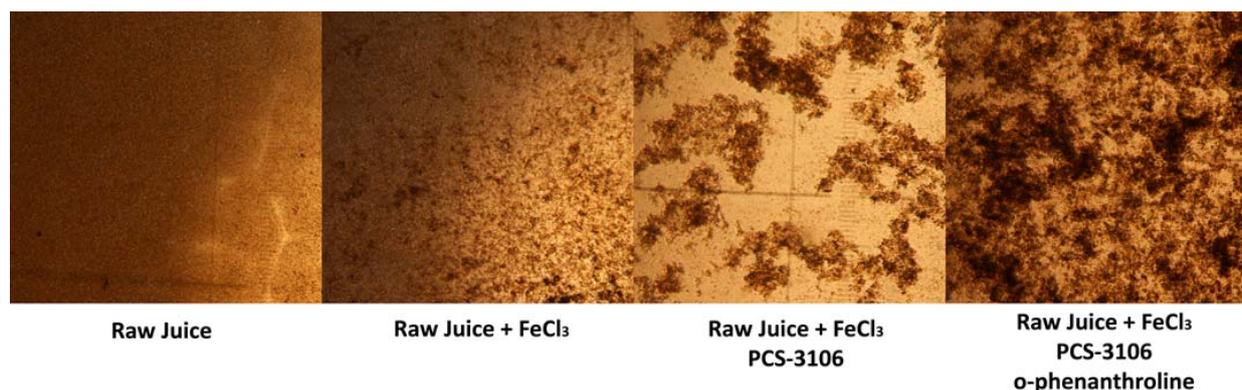


Fig. 7—FeMcaD trial in a juice droplet, 250X magnification.

The failure of 6 M urea to disrupt the floc argues that little in the way of hydrogen bonding is responsible for the stability of the BSA:CFA aggregates. The very slight disruption observed when the aggregate was treated with EDTA indicates that some, but not all of the structure is likely dependent upon chelation of iron. The effect of added *o*-phenanthroline was unexpected.

Rather than causing the material to disintegrate (at least partially) due to sequestration of any bound iron that might be structural, the addition of *o*-phenanthroline caused the flocs to become larger (Figure 7). Unsure of this result, the juice was treated with 'excess' *o*-phenanthroline with

and without added FeCl_3 . These samples were analysed via GC-MS and revealed a marked (>90%) removal of the ligand. In order to explain this, it was found that Cavalieri *et al.* (2002) had reported on the electrophilic addition of *o*-quinone species to purine bases in DNA. The reaction involves the radical semiquinone and is functional over a relatively wide range of pH, including physiological. An adapted scheme is given in Figure 8.

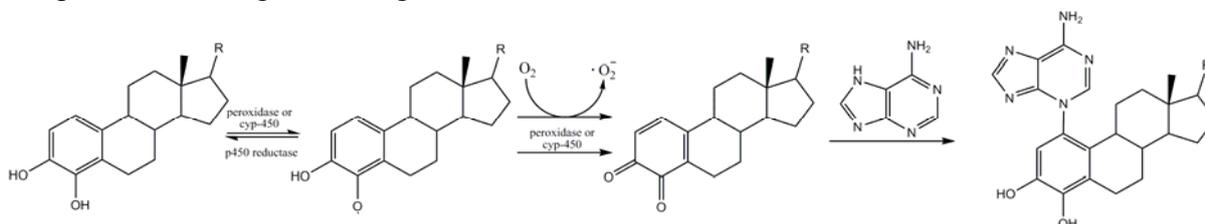


Fig. 8—Synthesis and reaction of a *o*-quinone with a purine base.

This scheme (Figure 8) is in line with observations. Instead of using cyp (cytochrome P) 450, Fe^{3+} serves as the initial oxidant to yield the semiquinone which is subsequently subject to autooxidation via O_2 . The resulting quinone then reacts with the purine base to yield the adduct.

We were unable to confirm that the *o*-phenanthroline was behaving in a similar way. It was suspected that the compound, being difunctional, might be serving a crosslinking species.

Because the aggregates appear to be covalent in nature, they are irreversibly formed. This irreversible nature indicates that the flocs once made, may be stable through liming, removing the need for the second stage of the process. With cold liming, it might be possible to do both steps, sequentially, in one reactor.

Conclusions

Ferric chloride can be used to remove phenolic compounds from cane juice via coagulation. As iron dosage is increased, the amount of floc increases as the settling rate decreases. Addition of cationic polyacrylamide improves the settling rates allowing for higher iron dosages to be used. Under the best conditions, it appears that a removal of approximately 50% ($55 \pm 4\%$) of the colour can be practically achieved while maintaining a satisfactory degree of clarification within 20 minutes of settling.

The method scaled well and performed as predicted by the bench-scale experiments. It was found that the 50% reduction of colour (relative to a cold-limed control) was achieved with the application of 150–200 $\mu\text{g}/\text{mL}$ Fe^{3+} and then liming to pH 7.1.

The mechanism appears to be a mixture of classical charge-neutralisation, complexation and covalent linkages between the phenolic compounds and native albuminoid protein found in the juice. The aggregates made via charge-neutralisation can be disrupted via addition of further coagulant while the complexes are labile to changes of pH, treatment with *o*-phenanthroline and/or application of urea. The flocs made here were especially resistant to the action of urea, were insensitive to pH and were demonstrated to aggregate with and remove *o*-phenanthroline.

This action was tested on BSA using varied amounts of the phenolic surrogate compound caffeic acid. It was found that the rate of consumption increases until flocs are no longer observed to form. At this point, steady consumption dropped to zero and the process was considered complete.

The observed consumption revealed an initial saturation of the protein which occurred at 200 $\mu\text{g}/\text{mL}$ CFA. This corresponded to the number of free N- ϵ -amino groups from BSA. Following this, the rate of consumption increased until 1500 $\mu\text{g}/\text{mL}$ of CFA had been consumed. At 2250 $\mu\text{g}/\text{mL}$, the rate of consumption drops to zero and the reaction is complete. Based on the number of N- ϵ -amino groups present, it was elucidated that crosslinks formed between the proteins which were between 6 and 10 phenolic residues in length.

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**CLARIFICATION INTERMEDIARE POUR LE FER ET LA
DECOLORATION DU JUS DE CANNE**

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**MOTS CLEFS: Couleur, Réduction de Couleur,
Clarification, Fer.**

Résumé

POUR AMELIORER la rentabilité, les producteurs de sucre en Louisiane souhaitent coopérer avec les raffineries de sucre. Étant donné que l'opération principale de la raffinerie est de réduire la couleur et que la raffinerie fonctionne à l'aide de gaz naturel comme combustible, il est logique que le sucre avec moins de couleur, produit à partir de bagasse, donnerait de meilleurs gains financiers. L'élimination des matières colorantes phénoliques du jus brut à l'aide de protéines présentes dans la canne et du Fe^{3+} comme un catalyseur oxydatif, a été étudiée. La couleur a été supprimée grâce à la précipitation de composés phénol/protéine causée par l'addition d'un flocculant cationique. Le jus décanté a été clarifié par le chaulage à froid. Le traitement a donné un jus clarifié avec une couleur réduite par 70% comparée au jus provenant d'un chaulage à ébullition. Il semble que les phénoliques ont été oxydés par le Fe^{3+} à travers une réaction RÉDOX produisant des quinoides. Les groupes N-ε-amino libres de lysine dans les protéines albuminoïdes s'ajoutent aux quinones. La stœchiométrie a indiqué un degré de polymérisation de huit. La formation d'oligomères cesse à ce point ce qui facilite la réification irréversible de la protéine. Les agrégats du fer, lignol(s) et protéines étaient insolubles et sont précipités. Le processus a été testé dans un décanteur de 150 litres qui a été exploité en mode pulsé et continu. La méthode a bien fonctionné et le jus produit présentait 50–60% moins de couleur comparé à un contrôle chaulé à froid, avec 100-200 mg/l Fe^{3+} ajouté au jus.

CLARIFICACIÓN CON HIERRO Y DECOLORACIÓN DE JUGO DE CAÑA

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Abstract

PARA OPERAR en un modo más rentable, los productores de azúcar en Louisiana desean emprender un arreglo cooperativo con las refinerías de azúcar. Debido a que una refinería es una estación reductora de color a escala industrial que opera usando gas natural como combustible, tiene sentido que un azúcar de menos color, producido usando energía del bagazo, pueda dar mejores márgenes de ganancia. Se estudió la remoción de colorantes fenólicos del jugo crudo usando proteína de la caña como un vehículo y Fe^{3+} como un catalizador oxidante. El color fue removido como conjugados de fenol-proteína que precipitaron rápidamente con la adición de un floculante catiónico. El jugo decantado fue clarificado vía encalado en frío. El tratamiento dio jugo claro con hasta 70% menos color que el jugo encalado en caliente. Parece que los fenólicos fueron oxidados por Fe^{3+} el cual entró en un ciclo REDOX produciendo especies quinoides. Los grupos libres N- ϵ -amino de lisina en las proteínas albuminoides parecieron sumarse a los quinoides. La estequiometría indicó un grado de polimerización de ocho. La formación de oligómeros cesó en esta longitud que parece suficiente para facilitar ligado cruzado irreversible y/o capping de la proteína. Los agregados de hierro, lignoles y proteína resultaron insolubles y precipitaron. El proceso se probó en un clarificador sedimentador de 150 L que operó en modo pulsante o en modo continuo. La escalabilidad del método fue buena y el jugo mostró 50–60% menos color que uno encalado en frío cuando se aplicó Fe^{3+} en cantidades entre 100–200 mg/L.