Effect of incorporating alum in cane juice clarification efficiency and sucrose losses

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Abstract

The effect of incorporating alum in the clarification stage of raw juice in sugarcane processing on the juice quality and sucrose loss was investigated. Alum was incorporated in both intermediate and hot liming clarification processes of cane juicing. One portion of the cane juice was used for With Pre-treatment Treatment (WPT) while the other portion constituted No Pre-treatment (NPT) juice. Alum at levels of 0 mg L^{-1}, 50 mg L^{-1}, 100 mg L^{-1} and 150 mg L^{-1} was incorporated in both intermediate and hot liming clarification processes in each of the two cane juice portions. Sugar concentration (sucrose, glucose and fructose), °Brix, pH, colour, settling performance (initial settling rates (ISR), final mud volume (MV_∞), and turbidity) and residual aluminium ion concentration were evaluated. Any significant variations (p < 0.05) in these parameters were assessed. The study found significantly lower (p < 0.05) sucrose losses in clarified juice from intermediate liming of WPT after alum treatment than in the rest of the clarified juices. Colour and turbidity in the pre-treated cane juice of intermediate liming was reduced by 36.9% and 98.1%, respectively at 150 mg L^{-1} alum level. An initial settling rate of 260 ml min^{-1} in WPT cane juice of intermediate liming at 150 mg L^{-1} alum level resulted in the most compact final mud volume of 10.3%. The residual aluminium concentration (0.025 to 0.048 mg L^{-1}) in alum treated clarified juices was lower than the natural aluminium concentration (0.088 mg L^{-1}) in untreated cane juice. This study showed the potential for the use of alum in cane juice clarification to improve on clarification efficiency and lower sucrose loss.

Keywords: Alum; Clarification; Cane juice; Turbidity; Sucrose loss

1 Introduction

Conventional technology used in the processing of sugarcane involves extraction of juice from the sugarcane, either by milling or by diffusion, clarification of the raw juice extracted, concentration to syrup and crystallization of the sugar in the syrup (Rein & Bento, 2006). Sugar losses associated with sugarcane processing can be chemical (degradation under acid or alkaline thermal conditions), microbial or enzymatic, with the latter being mostly limited to sugarcane harvesting, storage and extraction (Eggleston & Amorim, 2006; Eggleston, Vercellotti, Edye, & Clarke, 1996). Once extracted, the juice is heated to high temperatures, during which time sugarcane enzymes are denatured and most of the microbes killed, resulting in chemical losses becoming dominant (Saska, Zossi, & Liu, 2010b; Eggleston & Amorim, 2006). The major consequence of chemical losses of sucrose at the sugarcane factory is the decrease in sugar yields, and therefore reduction in profits (Eggleston & Amorim, 2006). The worldwide change from the harvesting of burnt to green (unburnt)

sugarcane, has resulted in more salts,invert sugars and impurities that are associated with the trash (leaves, tops and soil), being delivered to the factory. These extra impurities, apart from making clarification difficult, catalyse the hydrolysis of sucrose so adversely affecting its recovery and the refining quality of the raw sugar (Eggleston, Monge, & Pepperman, 2002; Eggleston, Grisham, & Antoine, 2010, 2014).

One area of potential improvement is in clarification, where the factory should achieve additional removal of colour, turbidity and polysaccharides, and at the same time keep sucrose inversion at a minimum. Work on the potential of cationic aluminium coagulants in cane juice clarification has been ongoing since 2004 (Sugar Research Processing Institute (SPRI), 2006). Preliminary experiments, treating raw cane juice with polyaluminium coagulants on a bench scale, have shown improvement in removal of turbidity, colour, and ash and an increase in pH, when compared to traditional clarification (Godshall, Mckee, Triche, & Moore, 2006). The analysis of the treated samples showed there was no aluminium carryover in the clarified juice. These results showed the potential for the use of aluminium coagulants in juice clarification. However, the effect of incorporating alum in cane juice clarification on cane juice quality and sucrose inversion losses has not been evaluated. Alum (also soda alum, filter alum, pearl alum or pickle alum) is the name used for aluminium sulphate ($Al_2(SO_4)_3.18H_2O$) (Ravina & Moramarco, 1993). When added to water hydrous oxides of aluminium are formed, the simplest of these being aluminium hydroxide ($Al(OH)_3$), which is an insoluble precipitate (Pernitsky & Edzwald, 2006). Several more complex positively charged soluble ions are also formed including: $Al(OH)^{2+}$, $Al(OH)_2^+$, $Al_3(OH)_5^{5+}$, $Al_6(OH)_{15}^{3+}$, $Al_7(OH)_{17}^{4+}$ and $Al_8(OH)_{20}^{4+}$ and $Al_{13}O_4(OH)_{24}^{7+}$ (Jiang & Graham, 1998; Zouboulis & Traskas, 2005). The mechanism of coagulation by alum includes both charge neutralization and sweep floc mode. These positively charged complexes can interact strongly with the negative surface charge around the particles that cause turbidity, resulting in destabilization and thus leading to formation of flocs that are denser and able to settle (Muisa, Hoko, & Chifamba, 2011; Zouboulis & Traskas, 2005; Degremont, 1991). Simultaneously, aluminium hydrox-

ide precipitates will form and grow independently of the colloid population, enmeshing the colloids in the sweep floc mode (Ravina & Moramarco, 1993). Alkalinity is required for the alum reaction to successively proceed otherwise the pH would be lowered to the point where soluble aluminium ion (Al^{3+}) is formed instead of aluminium hydroxide. Use of polyaluminium in sugar processing is not widespread. However, it has been reported that polyaluminium chloride has been used in Brazil at the level of 400-800 mg L^{-1} to produce direct consumption white sugar to avoid sulfitation and also in India for the same purpose at the level of 1000 mg L^{-1} (SPRI, 2006). The current study was aimed at investigating the effect of incorporating alum in cane juice clarification on juice quality and sucrose inversion losses.

2 Materials and Methods

2.1 Factory processing conditions

Mixed cane juice samples were collected from Mumias Sugar Company (MSC), Kenya. Lime, as a water slurry (average Baume $\sim 15°$) is automatically dosed into the extracted cane juice to a target pH of 7.2. The limed juice is passed through primary and secondary heaters where it is superheated under pressure (113 kPa abs) to 103 °C. The heated limed juice is then released into an open vessel (Flush tank) where it boils instantaneously releasing air bubbles that would otherwise be trapped in the floc and cause settling difficulties. The flushed juice is dosed with anionic polyelectrolyte flocculant (INDOFLOC®) at 3mg L^{-1} before entering a continuous clarifier maintained at 96 °C. The clarifier residence time is 45 minutes. The clarified juice is pumped to the evaporator station while the mud is recycled to the diffuser and eventually leaves with the bagasse.

2.2 Cane juice collection and treatment

Mixed cane juice samples each of sixteen litres were taken from the diffuser at 2-hour intervals over a 6 hour sampling period. The sampling period was repeated three times over a period of three months to cover cane variety, environmental and

process parameter variations. Each sample was split into two halves of eight litres. The first half was used for With Pre-treatment (WPT) which involved preheating and incubation, while the other half was used for the No Pre-treatment (NPT) analysis. The protocol followed is presented in Figure 1.

Cane juice with pre-treatment

The eight litres of cane juice were divided into two equal parts. One part was preheated to 90 °C and mixed with the other half before incubating at ambient temperature for 8 minutes. After incubation the juice was again split into two portions; one half was used for the intermediate liming process while the other half was used for the hot liming process. In the intermediate liming process, lime was added as a water slurry (average Baume $\sim 15°$) with constant stirring to a target juice pH of 7.2. The limed juice was heated to boiling before subdividing it into four equal portions. Four alum levels (0 mg L^{-1}, 50 mg L^{-1}, 100 mg L^{-1}, and 150 mg L^{-1}) were added to each portion and mixed thoroughly. Anionic polyelectrolyte flocculant (INDOFLOC®) at 3 mg L^{-1} was added to each portion and stirred thoroughly. A sample of 300 ml was drawn from each portion for determination of settling rates and mud volumes. The remaining juice in each portion was allowed to clarify in an incubator at 96 °C for 45 minutes. For settling rates and mud volume determinations, the 300 ml was poured into a settling tube (5cm x 34cm) in a water bath and stoppered. The water bath was maintained at 96 °C. Samples for analysis were drawn from the mixed juice(MJ), juice after incubation (AFTINCB), heated limed juice (LJ) and clarified juice (CJ) from the four alum treated portions (CJ$_0$, CJ$_{50}$, CJ$_{100}$ and CJ$_{150}$).For hot liming process, the preheated and incubated juice was first heated to boiling before liming to a target pH of 7.2. The limed juice was subdivided into four equal portions which were treated like the intermediate liming process before drawing samples for settling rate and mud volume measurements from each portion. Samples for analysis were drawn from heated limed juice (LJ) and clarified juices from the four alum treated portions (CJ$_0$, CJ$_{50}$, CJ$_{100}$ and CJ$_{150}$). Each sample in both liming processes was collected in a 250 ml

flask. Exactly 25 ml of this sample was transferred into a 50 ml flask and sodium azide (0.02 %) added to reduce/prevent chemical and microbial degradation of sucrose. The sodium azide-treated samples were stored in dry ice at $\sim$ -78 °C. Glucose, fructose and sucrose concentrations were measured in juice from the stored samples. After removal of 25 ml sugar cane juice from each sample, the remaining juice was used for colour, turbidity, °Brix and pH determinations.

Cane juice without pre-treatment

The other eight litres of cane juice were split into two portions. Half was used for the intermediate liming process while the other half was used for the hot liming process. The intermediate and hot liming processes were carried out as described for cane juice with pre-treatment. Samples for analysis from the intermediate liming process were drawn from heated limed juice (LJ) and clarified juices from the four alum treated portions (CJ$_0$, CJ$_{50}$, CJ$_{100}$ and CJ$_{150}$). A similar protocol was followed for the samples from the hot liming process. Each sample from the two liming processes was collected in a 250 ml flask. The samples were then treated exactly as those from the cane juice with pre-treatment.

2.3 Determination of sucrose, glucose and fructose by HPLC

The determination of sucrose, fructose and glucose in cane juices was done by isocratic HPLC analysis based on the Official International Commission of Uniform Methods of Sugar Analysis (ICUMSA) method GS7/4/8-23 (ICUMSA, 2002). Ten grams of the cane juice were diluted using a small quantity of distilled water in a 100 ml volumetric flask. The volumetric flask was made up to the mark with distilled water and the solution mixed well by shaking. At the same time three working mixed standards each containing sucrose (Fisher Scientific, Leicestershire-UK), fructose (Duchefa Biochemie, The Netherlands) and glucose (Prolabo, Paris-France) were prepared by dissolving in distilled water the following quantities of the sugars: standard 1 (sucrose 0.2521 g, fructose 0.2509 g, glucose 0.2528 g), standard 2 (sucrose 0.5040
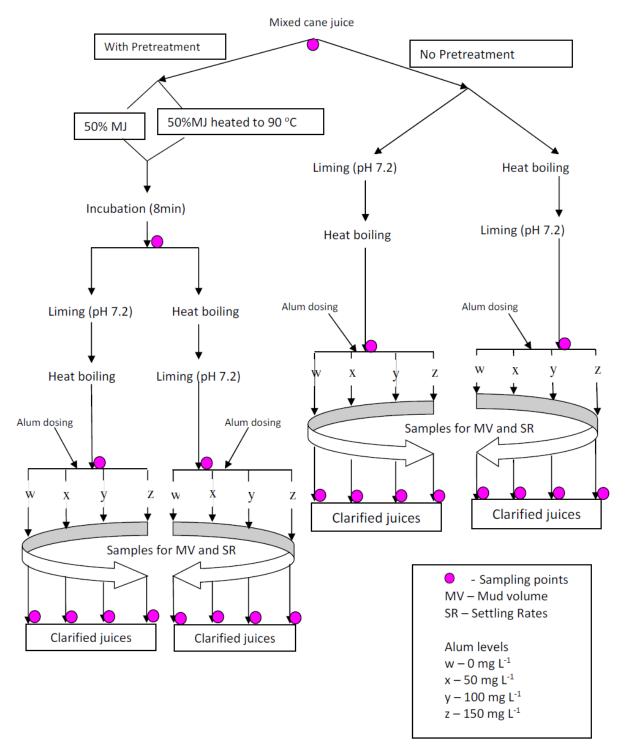
Figure 1: Mixed cane juice treatment protocol

g, fructose 0.5041 g, glucose 0.5098 g) and standard 3 (sucrose 0.7563 g, fructose 0.7554 g, glucose 0.7518 g) in a 100 ml volumetric flask and made up to the mark. The samples and the standards were then filtered into 2.5 ml vials using a syringe filter and a membrane filter of 0.45 μm pore size. Isocratic analyses for the sugars was done using a Shimadzu liquid chromatograph system SCL-10A (Tokyo, Japan) equipped with a pump, LC-10AS, thermostatically controlled Shimadzu column oven (CTO-10A) and a Shimadzu Refractive Index Detector (RID-6A). The analyses were done on a reversed phase amino column (Nucleodur Amino-RP, 4.6x250 mm, 5 μm, Machery

Nigel, Germany). The mobile phase used was acetonitrile:water (75:25 v/v). Chromatographic conditions used were; flow rate 0.9 ml min^{-1}., column temperature of 35 °C and a head pressure of 23 – 31 kgf cm^{-2}. A volume of 20 μL of the working standards or the diluted samples was injected and 1 μL loaded into the HPLC system by a sampling loop. The peak areas obtained were plotted on a Shimadzu C-R7A PLUS CHROMATOPAC data integrator. Using the working standards, peak areas, average response factors (g unit area^{-1}) for each sugar was calculated.

Each sugar concentration in the samples was calculated as follows:

$$Sugar\ conc.(\%) = \frac{[response\ factor\ \times\ sugar\ peak\ area\ ]}{sample\ weight} \times 100 \tag{1}$$

Percent sucrose losses were calculated using the following formula by (Schaffler, Muzzell, & Schorn, 1985):

$$Sucrose\ lost = \frac{[(\frac{Glu}{o_{Brix}})_{out} - (\frac{Glu}{o_{Brix}})_{in}]}{(\frac{Suc}{o_{Brix}})_{in} \times MW_{Glu}} \times MW_{suc} \times 100 \tag{2}$$

Where: MW = molecular weight
Suc = sucrose
Glu = glucose
$°Brix$ = percent dissolved solids

2.4 pH measurement

The pH of the sample juices were measured and corrected to 25 °C using a Control Dynamics pH Meter (Model: APX 175E/C C.D. Instrumentation PVT Ltd, Bangalore, India) calibrated using pH buffers pH 4 and pH 7.

2.5 Determination of °Brix (% Refractometric Dissolved Solids)

The mean °Brix (corrected to 20 °C) of triplicate samples was measured using a temperature-controlled refractometer accurate to ±0.01 °Brix (Model GPR 12-70X. Index Instruments Ltd, England).

2.6 Determination of cane juice colour and turbidity

Colour and turbidity of the juice were measured as the absorbance at 420 nm and calculated according to the official ICUMSA method GS2/3-

10 (ICUMSA, 2005). Samples (5 g) were diluted in triethanolamine / hydrochloric (TEA/HCl) acid buffer solution (pH 7) and filtered under vacuum through a 0.45 μm filter in to a clean dry conical flask to remove turbidity. The filtrate was de-aerated by immersing the conical flask containing the filtrate in an ultrasonic bath for 3 minutes. Absorbancy (A_s) of the de-aerated filtrate was measured at 420 nm using filtered and de-aerated TEA/HCl buffer solution as the standard for zero colour/turbidity. The refractometric dry substance (RDS) of the sample was also measured as described in ICUMSA method GS4-13. ICUMSA colour units were calculated as follows:

$$ICUMSA_{colour} = \frac{10_8 \times A_s}{b \times (correctedRDS) \times \rho} IU_{7.0}$$

Where: A_s = Absorbancy at 420 nm
b = length (cm) of the absorbing path
ρ = density of test solution (kgm^{-3}).

2.7 Determination of settling rates and mud volumes

Settling rates and mud volume measurements and calculations were based on the method of Lionnet and Ravn (1976), with modifications by Eggleston, Monge, & Pepperman (2002). The samples were heated to the clarifier temperature (96 °C) with constant stirring before pouring into a settling tube. The settling tube was mounted in a water bath maintained at the clarifier temperature. Each sample was poured into a settling tube to a volume of 300 ml and stoppered immediately and mud levels taken between 0.0 – 18.0 minutes. Mud height (ml) was plotted against time. The initial settling rate (ISR) in ml per minute was graphically determined from the initial linear slope. Mud volume after 18 minutes (MV_{18}) was read directly. The final equilibrium mud volume (MV_{∞}) or final height of the mud after infinite time was obtained from the intercept on a plot of MV percent against $1/t$, where t is time in minutes.

2.8 Determination of residual aluminium ions

Aluminium ions were determined in the clarified juice using the atomic absorption spectroscopy technique (AOAC, 2000). Clarified juice samples were filtered using a Millipore 0.45 μm cellulose acetate filters. An Atomic Absorption Spectrophotometer, (Shimadzu, Model AA-6300, Tokyo-Japan) with nitrous oxide-acetylene flame was used. Standards of known concentrations and blanks were used for calibration. Aluminium ions were measured by atomic absorption as the ions absorbed radiation from element specific hollow cathode lamp set at a wave length of 309.3 nm. The absorbencies obtained were used to estimate the concentrations of the metal ions in the samples from the standard curve.

2.9 Statistical analysis

Data analysis was done using Statistical Analysis Software, PC-SAS 9.1 (SAS, 2002-2003). Univariate analysis of variance and General Linear Model procedure were carried out to determine the effect of pre-treatment, liming process and alum on clarified juice quality and sucrose loss. Treatment means separation was done by Fisher's Least Significant Difference at α=0.05.

3 Results and Discussion

3.1 °Brix and sugar concentrations

The mean °Brix and mean sugar concentrations in NPT alum treated cane juice for both intermediate and hot liming processes are presented in Table 1. In the NPT sample, °Brix deceased significantly (p<0.05) from 15.35±0.32 in theMJ to 14.88±0.51 in the LJ for the hot liming process. This coincides with a significant decrease in the mean concentration of the invert sugars from MJ to LJ. In the intermediate liming, the decrease in °Brix from 15.35±0.32 in MJ to 15.04±0.26 in the LJ was not significant (p>0.05). The mean concentration of the invert sugar for both the intermediate and hot liming processes decreased significantly (p<0.05) upon liming the MJ. In the intermediate liming process, glucose decreased from

1.82±0.07 in the MJ to 1.62±0.12 in the LJ while fructose decreased from 1.74±0.07 in the MJ to 1.57±0.07 in the LJ (Table 2). In the hot liming process, the decrease in glucose and fructose from the MJ to the LJ was 1.82±0.07 to 1.59±0.12 and 1.74±0.07 to 1.48±0.16, respectively. The decrease in the invert sugar concentrations was considered to be due to alkaline degradation of these sugars, a reaction that increases with temperature and retention time (Eggleston, Clarke, & Pepperman, 1999; Eggleston, 2000; Eggleston, Monge, & Ogier, 2002) leading to colour formation. A similar trend in invert sugar concentrations was reported by Eggleston, Monge, and Pepperman (2002) while comparing cold and intermediate liming processes and Eggleston, Monge, and Ogier (2002) while assessing factory performance of cold, intermediate and hot liming clarification processes. The decrease in °Brix from MJ to LJ could be further evidence to this. It was observed that in all the samples, lower fructose concentrations than glucose concentrations were recorded. The increase in invert sugar concentrations from LJ to CJ, the first degradation products of inversion, is an indicator of acid inversion of sucrose in sugarcane factories (Godshall, Clarke, & Dooley, 1991). This is because in comparison to the high concentrations of sucrose, they occur at lower and therefore measurable concentrations and changes in their concentrations are more easily detected (Schaffler et al., 1985). Although sucrose inverts to equal amounts of glucose and fructose, fructose is more labile than glucose in acid conditions (Schaffler et al., 1985) making it an inaccurate measurement of inversion. It is on this basis that Schaffler and co-workers (1985) demonstrated that glucose and sucrose referenced to °Brix can be used to accurately calculate the percent sucrose loss and their calculation formula (Equation 2) was used in this study. In the WPT sample where the cane juice was preheated and incubated before liming, there was an insignificant decrease (p>0.05) in °Brix from the MJ to the LJ in both liming processes (Table 2). The preheating and incubation of the MJ led to a significant decrease (p<0.05) in invert sugar concentration from 1.82±0.07 in the MJ to 1.61±0.12 in the juice after incubation (AFTINCB) for glucose and from 1.74±0.07 in the MJ to 1.49±0.11 in the AFTINCB for fructose. After liming the incubated juice, the invert sugars again

decreased significantly (p<0.05) in the intermediate liming. In hot liming, there was a significant decrease (p<0.05) in the invert sugar concentrations upon liming the AFTINCB. Acid inversion of sucrose in sugarcane factories is easily indicated by an increase in glucose and fructose, the first products of inversion (Godshall et al., 1991). According to Eggleston, Monge, and Pepperman (2002), a possible problem associated with preheating of acidic mixed juice as is the case with the WPT, is sucrose inversion. However, as observed in Table 2 there was a mean decrease in invert sugar concentrations from the MJ to the AFTINCB samples. The one and a half minute heating time probably minimised inversion and could be that some invert sugars may have been removed with heat-induced precipitation of colloids, macromolecules (starch, dextran polysaccharides and proteins) and colour (de Armas, Martinez, Vicente, & Legaz, 1999; Saska, Zossi, & Liu, 2010a). The decrease in °Brix from the MJ to the AFTINCB could be further evidence of this. In both liming processes, there was a decrease in invert sugar concentrations from the AFTINCB to the LJ (Table 2). This was associated with the alkaline degradation of these sugars, a reaction that is a function of temperature and time (Eggleston, Monge, & Ogier, 2002; Eggleston, 2000; Eggleston et al., 1999). In the clarified juices, higher increases in glucose concentrations were recorded in the hot liming process than in intermediate liming process, a pointer to higher sucrose losses in the hot liming process.

The sucrose loss during the clarification in the NPT and the WPT for both liming processes is summarised in Table 3. There was no significant (p>0.05) sucrose loss differences in the juices clarified without alum addition. However, the sucrose loss after clarification in the WPT intermediate liming was significantly lower for each alum level than in the rest of the clarified juices. Alum addition in each of the liming processes did not significantly (p>0.05) affect the sucrose loss except in the WPT intermediate liming where the loss with the addition of alum at 50 mg, 100 mg and 150 mg was significantly lower than the control. A study by Eggleston et al. (2014) indicated that the higher pH of clarified juices after saccharate liming than after adding milk of lime led to lower sucrose loss due to acid inversion of sucrose across the clarifier. Results from this study strongly suggest that

intermediate lime clarification of WPT cane juice with addition of alum could be an important tool for sugar processors to reduce expensive sucrose losses.

3.2 Colour and pH

Colour in sugar process streams consists of a complex mixture of compounds that originate naturally from the cane plant or produced during processing in the factory and its appearance in raw sugar plays a key role in the marketing strategy of sugar industries worldwide (Nguyen & Doherty, 2012a, 2012b; Davis, 2001). The colour and pH in the NPT cane juice for both intermediate and hot liming processes are presented in Table 4. In the NPT cane juice, the pH of the clarified juices in intermediate liming were not significantly different ($p < 0.05$) and ranged from 6.43 ± 0.19 in CJ_0 to 6.54 ± 0.17 in CJ_{150}. The pH of the clarified juices in hot liming were also not significantly different ($p < 0.05$) and ranged from 6.40 ± 0.11 in CJ_0 to 6.51 ± 0.15 in CJ_{150}. In both liming processes, the pH reduced significantly ($p < 0.05$) from the limed juice to the clarified juices. This drop in pH upon clarification of the limed juice is as a result of the precipitation of lime salts during settling of the mud (Eggleston, Monge, & Pepperman, 2002; Eggleston, Monge, & Ogier, 2002) and the inversion of sucrose with associated production of acids (Eggleston et al., 2010) could also have contributed to this. Similarly, in the WPT cane juice, the pH of the clarified juices in intermediate liming and hot liming (Table 4) were not significantly different ($p < 0.05$). However, there was a significant ($p < 0.05$) drop in pH from limed juice to clarified juices in both liming processes.

Colour formation is an indicator of sugar degradation and subsequently the quality of the raw sugar (Eggleston & Monge, 2005). In the NPT cane juice, colour significantly increased ($p < 0.05$) upon liming the MJ in both the intermediate and hot liming processes (Table 4). In the intermediate liming, there was a 12.4% colour increase from MJ to LJ while in the hot liming the colour increase from the MJ to the LJ was 13.9% (Table 4). The colour formation upon liming of the MJ was due to the alkaline degradation of the invert sugars, a reaction that is relatively fast and is a func-

tion of temperature and retention time (Eggleston, Monge, & Ogier, 2002; Eggleston, 2000; Eggleston et al., 1999). The higher colour formation in hot liming could have been due to the heating of the acidic MJ before liming. When cane juice is heated under moderate acid conditions (between pH 5.5 and 6.8), colour is formed from the acid degradation of sucrose and invert sugars (Eggleston & Amorim, 2006). On settling the limed juice, colour was significantly ($p < 0.05$) removed in the clarified juices of both liming processes. The highest colour reduction of 33.6% in intermediate liming was recorded in CJ_{150} which was significantly higher than the colour reduction of 24% in CJ_0 (Table 4). In hot liming, a colour reduction of 30.6% and 29.4% was observed in CJ_{150} and CJ_{100}, respectively and these were not significantly different (Table 4). The least colour removal (23.1%) was recorded in CJ_0. It can be concluded from these results that addition of alum at $150 mgL^{-1}$ of cane juice in intermediate liming gave clarified juice with the highest colour removal.

In the WPT cane juice, pre-heating and incubating the mixed juice led to increased colour removal in the clarified juices for both intermediate and hot liming processes. By pre-heating and incubating the MJ, juice colour was significantly removed ($p < 0.05$) from 13271 IU in MJ to 11041 IU in AFTINCB. The colour removal preceding the incubation is considered to be heat-induced and is associated with the precipitation of colloids and macromolecules, including starch, dextran polysaccharides and proteins (de Armas et al., 1999; Saska et al., 2010a). Eggleston, Monge, and Pepperman (2002) reported a 29% colour removal in intermediate liming from the mixed juice when 30% of the mixed juice was pre-heated before incubation. However, this colour removal was offset by colour formation on liming the juice. On liming the AFTINCB, significant colour formation was recorded in both liming processes (Table 5). In intermediate liming, colour increased by 8.4% from AFTINCB to LJ compared to an increase of 11.9% in hot liming from AFTINCB to LJ (Table 5). This colour formation was caused by alkaline degradation of invert sugars, an observation made earlier by Eggleston (2000) and Eggleston et al. (1999). There was significant ($p < 0.05$) colour removal upon clarifying the limed juice in both liming processes. In intermediate liming, significantly

Table 1: °Brix and [ω]Sugar concentrations (mean ± SD) for No Pre-treatment cane juices

Sample	°Brix	Sucrose/°Brix	Glucose/°Brix	Fructose/°Brix
Intermediate Liming				
Mixed juice	15.35 ± 0.32^a	87.82 ± 2.78^a	1.82 ± 0.07^b	1.74 ± 0.07^b
Limed juice	15.04 ± 0.26^{ab}	87.71 ± 2.36^a	1.62 ± 0.12^c	1.57 ± 0.07^c
CJ_0	14.98 ± 0.27^{abc}	87.22 ± 1.73^a	2.23 ± 0.10^a	1.98 ± 0.13^a
CJ_{50}	15.19 ± 0.45^{ab}	86.16 ± 2.68^a	2.22 ± 0.11^a	2.07 ± 0.12^a
CJ_{100}	14.88 ± 0.39^{bc}	88.21 ± 2.53^a	2.20 ± 0.12^a	2.02 ± 0.12^a
CJ_{150}	14.68 ± 0.69^c	88.29 ± 4.23^a	2.21 ± 0.10^a	2.00 ± 0.13^a
Hot Liming				
TMJ	15.35 ± 0.32^a	87.82 ± 2.78^{ab}	1.82 ± 0.07^b	1.74 ± 0.07^b
Limed juice	14.88 ± 0.51^{bc}	89.01 ± 4.22^a	1.59 ± 0.12^c	1.48 ± 0.16^c
CJ_0	14.97 ± 0.33^{abc}	87.70 ± 2.05^{ab}	2.23 ± 0.12^a	2.07 ± 0.11^a
CJ_{50}	15.03 ± 0.41^{ab}	87.28 ± 2.73^{ab}	2.22 ± 0.13^a	2.04 ± 0.13^a
CJ_{100}	14.61 ± 0.53^c	89.82 ± 2.98^a	2.21 ± 0.10^a	2.03 ± 0.10^a
CJ_{150}	15.13 ± 0.42^{ab}	86.12 ± 2.78^b	2.20 ± 0.08^a	2.10 ± 0.11^a

Means in the same column followed by the same superscript are not significantly different at $p<0.05$. n=9. [ω]Sugar concentration is percent sugar (measured by HPLC)/°Brix; CJ_0, CJ_{50},CJ_{100}, and CJ_{150} = clarified juice after treatment with 0, 50, 100 and 150mg of alum, respectively.

($p<0.05$) higher colour removal was observed in CJ_{50}, CJ_{100} and CJ_{150} than in CJ_0. Similarly in hot liming, CJ_0 had significantly lower colour removal than in CJ_{50}, CJ_{50}, CJ_{100} and CJ_{150}.

3.3 Settling performance

Initial settling rate

Mud settling during clarification is important because any carryover of slow settling particles causes difficulties later in the process and in subsequent processes such as refining (Schoonees-Muir & Gwegwe, 2008). Mud volume is important as too high a volume puts an extra burden on the mud filter station and there is a greater possibility of having unwanted carryover of mud into the evaporator station (Eggleston et al., 2014) Differences in settling performance in the various limed cane juices were assessed in terms of initial settling rate (ISR) and final mud volume (MV_∞). The initial settling rate (ISR) and final mud volume (MV_∞) results for NPT and WPT are presented in Table 6. In the NPT intermediate liming, ISRs ranged from 203 ml min-1 in CJ_0 to 256 ml min^{-1}

in CJ_{150}. The ISRs for CJ_{50}, CJ_{100} and CJ_{150} did not differ significantly ($p<0.05$) from each other but were significantly different from the ISR for CJ_0. In NPT hot liming, the ISRs ranged from 196 ml min^{-1} in CJ_0 to 240 ml min-$^{-1}$ in CJ_{150}. The ISR for CJ_0differed significantly ($p<0.05$) from the ISR for CJ_{50}, CJ_{100} and CJ_{150} which did not differ significantly from each other. Generally, lower ISRs were observed in hot liming process than in intermediate liming process for the NPT. In the WPT, ISRs in intermediate liming process ranged from 212 ml min^{-1} in CJ_0 to 260 ml min^{-1} in CJ_{150}. The ISRs for CJ_{50}, CJ_{100} and CJ_{150} did not differ significantly ($p<0.05$) from each other. The lowest ISR (208 ml min-1) in WPT for hot liming was recorded in CJ_0 and differed significantly ($p<0.05$) from CJ_{50}, CJ_{50} and CJ_{150}. Generally, lower ISRs were observed in hot liming process than in intermediate liming process for the WPT. The higher settling performance in the alum dosed cane juices could be explained by the fact that when the flocculant is combined with the aluminium coagulant, the suspended material is aggregated into larger and heavier particles that settle faster (Anon, 2005).

Table 2: °Brix and $^\omega$Sugar cconcentrations (mean ± SD) for With Pre-treatment cane juices

		Intermediate Liming		
Sample	°Brix	Sucrose/°Brix	Glucose/°Brix	Fructose/°Brix
TMJ	15.35 ± 0.32^{cd}	87.82 ± 2.78^a	1.82 ± 0.07^b	1.74 ± 0.07^b
AFTINCB	15.01 ± 0.14^e	86.78 ± 1.56^{ab}	1.61 ± 0.12^c	1.49 ± 0.11^c
Limed juice	15.17 ± 0.26^{de}	86.73 ± 1.84^{ab}	1.49 ± 0.08^d	1.36 ± 0.09^d
CJ_0	15.43 ± 0.20^{bc}	86.15 ± 1.48^b	2.09 ± 0.13^a	1.90 ± 0.09^a
CJ_{50}	15.65 ± 0.14^{ab}	86.14 ± 1.65^{ab}	2.02 ± 0.11^a	1.82 ± 0.08^{ab}
CJ_{100}	15.42 ± 0.31^{bc}	85.99 ± 1.56^b	2.03 ± 0.17^a	1.87 ± 0.10^a
CJ_{150}	15.70 ± 0.16^a	86.57 ± 1.02^{ab}	2.04 ± 0.11^a	1.84 ± 0.09^a
		Hot Liming		
TMJ	15.35 ± 0.32^{bc}	87.82 ± 2.78^a	1.82 ± 0.07^b	1.74 ± 0.07^b
AFTINCB	15.01 ± 0.14^d	86.78 ± 1.56^{ab}	1.61 ± 0.12^c	1.49 ± 0.11^c
Limed juice	15.31 ± 0.13^c	86.39 ± 0.46^{ab}	1.52 ± 0.13^c	1.42 ± 0.14^c
CJ_0	15.65 ± 0.26^a	84.04 ± 1.40^c	2.13 ± 0.06^a	1.86 ± 0.08^a
CJ_{50}	15.62 ± 0.23^a	84.26 ± 1.18^c	2.11 ± 0.12^a	1.83 ± 0.09^{ab}
CJ_{100}	15.35 ± 0.35^{bc}	85.27 ± 1.78^{bc}	2.11 ± 0.09^a	1.82 ± 0.11^{ab}
CJ_{150}	15.54 ± 0.31^{ab}	84.58 ± 1.54^c	2.10 ± 0.09^a	1.87 ± 0.12^a

Means in the same column followed by the same superscript are not significantly different at p<0.05. n=9. $^\omega$Sugar concentration is percent sugar (measured by HPLC)/°Brix; CJ_0, CJ_{50},CJ_{100}, and CJ_{150} = clarified juice after treatment with 0, 50, 100 and 150mg of alum, respectively.

Table 3: Sucrose loss (mean ± SD) (%) after clarification for the various alum levels

		Alum levels			
	Treatment	0mg	50mg	100mg	150mg
NPT	Intermediate liming	1.32 ± 0.03^{aA}	1.30 ± 0.02^{aA}	1.26 ± 0.02^{aA}	1.27 ± 0.03^{aA}
	Hot liming	1.37 ± 0.06^{aA}	1.34 ± 0.02^{aA}	1.32 ± 0.02^{aA}	1.30 ± 0.02^{aA}
WPT	Intermediate liming	1.31 ± 0.05^{aA}	1.16 ± 0.06^{bB}	1.18 ± 0.06^{bB}	1.20 ± 0.02^{bB}
	Hot liming	1.34 ± 0.07^{aA}	1.30 ± 0.09^{aA}	1.30 ± 0.04^{aA}	1.28 ± 0.05^{aA}

Means followed by the same lower case superscript in the same row are not significantly different at p<0.05. Means followed by the same upper case superscript in the same column are not significantly different p<0.05. n=3.

Table 4: Colour and pH (mean ± SD) for No Pre-treatment cane juice

Sample	Intermediate Liming		Hot Liming	
	Colour (IU)	pH	Colour (IU)	pH
TMJ	13271 ± 396^b	5.69 ± 0.13^c	13271 ± 396^b	5.69 ± 0.14^c
Limed juice	14917 ± 456^a	7.17 ± 0.08^a	15116 ± 422^a	7.12 ± 0.19^a
CJ_0	11337 ± 114^c	6.43 ± 0.19^b	11624 ± 139^c	6.40 ± 0.11^b
CJ_{50}	10636 ± 131^d	6.47 ± 0.10^b	10944 ± 163^d	6.43 ± 0.12^b
CJ_{100}	10397 ± 143^d	6.50 ± 0.15^b	10672 ± 213^e	6.45 ± 0.18^b
CJ_{150}	9905 ± 180^f	6.54 ± 0.17^b	10490 ± 84^e	6.51 ± 0.15^b

Means in the same column followed by the same superscript are not significantly different at p<0.05. n=9; CJ_0, CJ_{50}, CJ_{100}, and CJ_{150} = clarified juice after treatment with 0, 50, 100 and 150 mg of alum, respectively; IU= ICUMSA Units.

Table 5: Colour and pH (mean ± SD) in alum and With Pre-treatment cane juice

Sample	Intermediate Liming		Hot Liming	
	Colour (IU)	pH	Colour (IU)	pH
TMJ	13271 ± 396^a	5.69 ± 0.14^d	13271 ± 396^a	5.69 ± 0.14^c
AFTINCB	11041 ± 544^c	5.77 ± 0.17^d	11041 ± 544^c	5.77 ± 0.17^c
Limed juice	11965 ± 624^b	7.08 ± 0.22^a	12350 ± 468^b	7.12 ± 0.12^a
CJ_0	8925 ± 675^d	6.49 ± 0.12^c	9288 ± 248^d	6.41 ± 0.09^b
CJ_{50}	7799 ± 569^e	6.66 ± 0.21^b	8539 ± 244^e	6.44 ± 0.13^b
CJ_{100}	7672 ± 569^e	6.61 ± 0.14^{bc}	8186 ± 420^{ef}	6.47 ± 0.15^b
CJ_{150}	7548 ± 626^e	6.59 ± 0.16^{bc}	8006 ± 623^f	6.52 ± 0.13^b

Means in the same column followed by the same superscript are not significantly different at p<0.05. n=9; CJ_0, CJ_{50}, CJ_{100}, and CJ_{150} = clarified juice after treatment with 0, 50, 100 and 150 mg of alum, respectively; IU= ICUMSA Units.

Final mud volumes

The final mud volumes (MV_∞) among the clarified juices in NPT and WPT for both intermediate and hot liming processes are presented in Table 6. In the NPT intermediate liming, the MV_∞ of the clarified juices at all alum levels were significantly (p<0.05) different. There was a decrease in the MV_∞ of these juices as thealum level was increased. The highest MV_∞ of clarified juice (15.5%) was recorded in CJ_0 while the lowest MV_∞ of clarified juice (10.5%) was recorded in CJ_{150}. In the NPT hot liming, a similar trend of a decrease in theMV$_\infty$ of clarified juiceswith the increasing alum level was observed. The highest MV_∞ of clarified juice (18.2%) in CJ_0 was significantly (p<0.05) different from the rest of the MV_∞ of the clarified juices. Generally, higher MV_∞ ofclarified juiceswere observed in NPT hot liming than in NPT intermediate liming for each alum level. In the WPT intermediate liming, the MV_∞ of the clarified juice from all the alum treatments were significantly different (p<0.05). The lowest MV_∞ (10.8%) was recorded in CJ_{150} and was significantly different from CJ_0. In the WPT hot liming, CJ_0 MV_∞ was significantly high (p<0.05). The lowest MV_∞ of 11.6% in CJ_{150} was not significantly (p<0.05) different from 12.0% in CJ_{100}. The larger mud volumes in hot liming explain the slow initial settling rates compared to intermediate liming. A similar observation was made by Eggleston, Monge, and Pepperman (2002) when comparing settling rates in cold and intermediate liming processes.

Table 6: Settling performance (mean ± SD) in With Pre-treatment and No Pre-treatment cane juices

| Sample | No Pre-treatment | | | | With Pre-treatment | | | |
| | Intermediate Liming | | Hot Liming | | Intermediate Liming | | Hot Liming | |
	ISR(ml min^{-1})	MV∞(%)	ISR(ml min^{-1})	MV∞(%)	ISR(ml min^{-1})	MV∞(%)	ISR(ml min^{-1})	MV∞(%)
CJ$_0$	203±36^b	15.5±0.65^a	196±38^b	18.2±0.89^a	212±32^b	13.2±0.54^a	208±36^b	13.7±0.78^a
CJ$_{50}$	248±34^a	12.6±0.60^b	226±52^a	13.4±0.76^b	256±32^a	12.4±0.60^b	230±32^a	12.8±0.53^b
CJ$_{100}$	250±24^a	11.3±0.80^c	232±42^a	13.2±0.63^b	254±35^a	12.6±0.74^{ab}	241±37^a	12.0±0.75^c
CJ$_{150}$	256±27^a	10.5±0.49^d	240±55^a	12.8±0.49^c	260±32^a	10.3±0.61^c	250±27^a	11.6±0.49^c

Means in the same column followed by the same superscript are not significantly different at p<0.05. n=9. CJ$_0$, CJ$_{50}$,CJ$_{100}$, and CJ$_{150}$ = clarified juice after treatment with 0, 50, 100 and 150mg of alum, respectively.
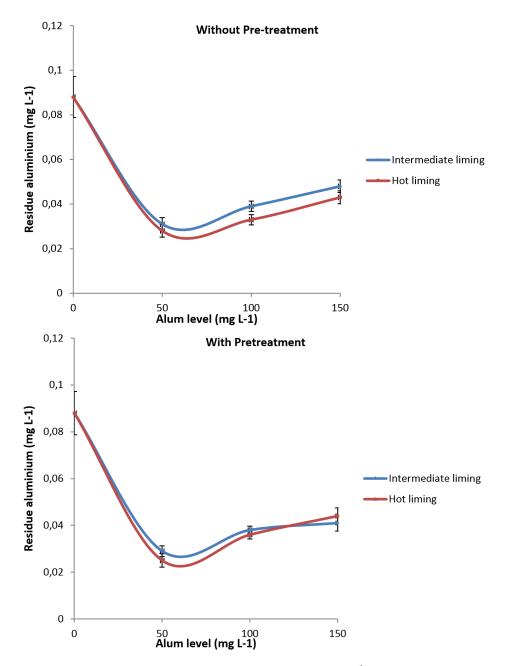
Figure 2: Concentration of residual aluminium (mg L^{-1}) in clarified juice

Table 7: Turbidity (IU) (mean $\pm$ SD) in No Pre-treatment and With Pre-treatment cane juices

Sample	No Pre-treatment		With Pre-treatment	
	Intermediate Liming	Hot Liming	Intermediate Liming	Hot Liming
MJ	54685 ± 4498^a	54685 ± 4498^a	54685 ± 4498^a	54685 ± 4498^a
CJ_0	4594 ± 359^b	4958 ± 234^b	3719 ± 461^b	3831 ± 305^b
CJ_{50}	2618 ± 377^c	2968 ± 429^c	1459 ± 234^c	1870 ± 301^c
CJ_{100}	2389 ± 342^c	2855 ± 285^c	1187 ± 209^c	1380 ± 227^c
CJ_{150}	2172 ± 339^c	2634 ± 404^c	1024 ± 222^c	1272 ± 288^c

Means in the same column followed by the same superscript are not significantly different at $p<0.05$. n=9. CJ_0, CJ_{50}, CJ_{100}, and CJ_{150} = clarified juice after treatment with 0, 50, 100 and 150 mg of alum, respectively.

Turbidity measurement

Turbidity is an optical property of a liquid that describes the clarity or haziness of the liquid. It is not colour related, but rather relates to the loss of transparency due to the effect of suspended particulates, colloidal material, or both. The ICUMSA turbidity measurement is a spectrophotometric technique that measures absorbance at 420nm and is an indication of suspended matter in cane juice (Maseko, Singh, & Motsa, 2011). The removal of turbidity is indicative of the removal of these components (Hamerski, da Silva, Corazza, Ndiaye, & de Aquino, 2012). To determine the extent of turbidity removal after clarification of the various treatment cane juices, turbidity values of the MJ and the clarified juices were measured and the results are summarised in Table 7. In the NPT intermediate liming, turbidity was significantly ($p<0.05$) removed on clarification of the MJ. The highest turbidity removal of 96% in CJ_{150} was not significantly different from a turbidity removal of 95.6% in CJ_{100} and 95.2% in CJ_{50}. The lowest turbidity removal of 91.6% was in CJ_0. In the NPT hot liming, turbidity was significantly ($p<0.05$) removed from MJ on clarification. Turbidity removed in CJ_0 (90.9%) was significantly ($p<0.05$) lower than 94.6%, 94.8% and 95.2% removed in CJ_{50} and CJ_{100} and WCJ_{150}, respectively. In the WPT intermediate liming, turbidity removed in CJ_{50}, CJ_{100} and CJ_{150} was not significantly ($p<0.05$) different. However, turbidity removed in CJ_0 was significantly ($p<0.05$) lower. In the WPT hot liming process, turbidity removed in CJ_0 (93%) was significantly lower than turbidity removed in CJ_{50}, CJ_{100} and CJ_{150}. It is evident

that the WPT clarified juices had better turbidity removal than the NPT clarified juices. This could be partly due to the faster settling of the larger flocs that are formed in preheating of the 50% of the MJ and partly due to the presence of the resulting complex positively charged ions from alum, which adsorb to the negative turbidity particles thus neutralizing their charge. This could also be seen in the initial settling rates where higher ISRs were observed in the WPT than in the NPT cane juices (Table 6). This was in agreement with studies by Eggleston, Monge, and Pepperman (2002) and de Armas et al. (1999) who reported formation of larger flocs and faster settling rates leading to higher turbidity removal upon preheating of the mixed cane juice before liming. These results therefore suggest that incorporating alum in WPT intermediate liming has a great potential for turbidity removal in cane juice clarification.

3.4 Residual aluminium ions

To determine the aluminium carryover in the clarified juice, residual aluminium concentration in the clarified juice was quantified and compared to the aluminium concentration in the untreated cane juice. The results are summarised in Figure 2. In all the clarified juices, for both WPT and NPT, the residual aluminium concentration was significantly lower ($p < 0.05$) than in the untreated juice. This showed that even with the cane juice treatment with alum, the clarification process was able to remove aluminium from the cane juice to levels lower than the natural aluminium concentration in the untreated cane juice. This was in agreement with studies by Anon (2005, 2006) who noted no

aluminium carryover in the clarified juice when using polyaluminium coagulants in conjunction with normal lime clarification.

4   Conclusions

Incorporation of alum in the clarification of cane juice showed improvements in colour and turbidity removal with the preheating and incubation of the MJ in WPT resulting in higher colour and turbidity removal than in NPT. Similarly, lower sucrose losses were observed in intermediate lime clarification of WPT cane juice, strongly suggesting that alum could be used by sugar processors to improve clarification and reduce sucrose losses. Higher ISRs and compact MV_∞ were found in the alum treated cane juices than in the control juice in both NPT and WPT. This observation could be an important step in reducing the possibility of mud carryover to the evaporator station and in lowering the burden of mud volume in the mud filter station. This study indicates the potential of the applicability of incorporating alum in intermediate lime clarification of WPT without the risk of aluminum carryover in the clarified juice.

Acknowledgements

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