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Preheating and Incubation of Cane Juice Prior to Liming: A Comparison of Intermediate and Cold Lime Clarification

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In the U.S., cold lime clarification remains the clarification process of choice in raw sugar manufacturing. A comparative study of cold vs intermediate lime clarification was undertaken at a factory that operated intermediate liming (~30% mixed juice (MJ) of pH 5.2 ± 0.3 was preheated to 87−93 °C to help maintain clean limed juice heaters, incubated at ~54 °C, and then limed) but still had the pipes to revert to cold liming (MJ incubated and limed at ~40 °C) for this study. Hourly samples were collected over a 6 h sampling period across cold and intermediate clarification processes on two consecutive days, respectively, and this was repeated three times across the 1999 grinding season. A total of 1.57% less sucrose was lost to inversion reactions across intermediate rather than cold liming. In intermediate liming, which required ~4.6% less lime, preheating of only 30% of the MJ markedly removed color (−29%), dextran (−10%), and starch (−24%) and caused large flocs to form that settled faster in the clarifiers. Faster settling led to an impressive 4.6% (season average) more turbidity removal across the clarifiers in intermediate rather than cold liming. Intermediate clarified juice (CJ) turbidity (season average 2028 ICU ± 675) was approximately half of cold CJ turbidity (average 3952 ICU ± 1450) with over 2-fold more CJ turbidity control. Subsequent turbidity values and control were significantly improved in the final evaporator syrup samples too. For both processes, juice incubation caused ~10% color removal, but this was offset by color formation on liming, because of the alkaline degradation of invert; however, overall, more color was removed than formed in intermediate liming. Starch was reduced in the incubator tank, for both processes, because added filtrate reduced the acidity enabling natural diastase from the cane to degrade starch. Some dextran occasionally formed in the incubator tank, in both processes. Summed across measured parameters, intermediate liming appears to offer several advantages over cold liming.

KEYWORDS: Cane juice incubation; cold lime clarification; dextran; intermediate lime clarification; heated cane juice; raw sugar manufacture; starch

INTRODUCTION

The major aim of clarification in raw sugar manufacturing is to remove from the mixed juice (MJ) the maximum quantity of impurities at the earliest stage. The degree of clarification has a great impact on boiling house operations, sugar yield, and refining quality of raw sugar. Several lime clarification systems have been developed over the years including cold, hot, intermediate, fractional, and saccharate liming. Moreover, variations also occur within a particular clarification system, from factory to factory. Although many other parts of the world have changed from cold liming mostly to hot liming, including South Africa (*1*) in the 1960s, cold liming is still usually operated in the U.S. Generally, in cold liming, sufficient lime is added at ambient temperature or slightly above ambient temperatures to neutralize organic acids present in the MJ and form a heavy precipitate, primarily of calcium phosphate. The separation or settling of the precipitate, aided by flocculating polymers, occurs in the clarifier. Up until now, the two major advantages of cold liming over other liming processes were considered to be its simplicity of operation and less sucrose inversion (*2*). However, recent factory studies (*3*−*5*) have unequivocally shown that excessive inversion occurs in cold liming clarifiers, ~10% color is formed on liming, and pH and turbidity control are erratic. Furthermore, with the 1990s introduction of mechanical harvesting of green and burnt billeted sugar cane in the U.S., to increase sugar yields per acre, there has been an unfortunate large increase of impurities that require factory processing. These impurities occur because of associated trash, i.e., leaves, top and field soil, and sugar destruction between harvesting and crushing. Therefore, there is currently

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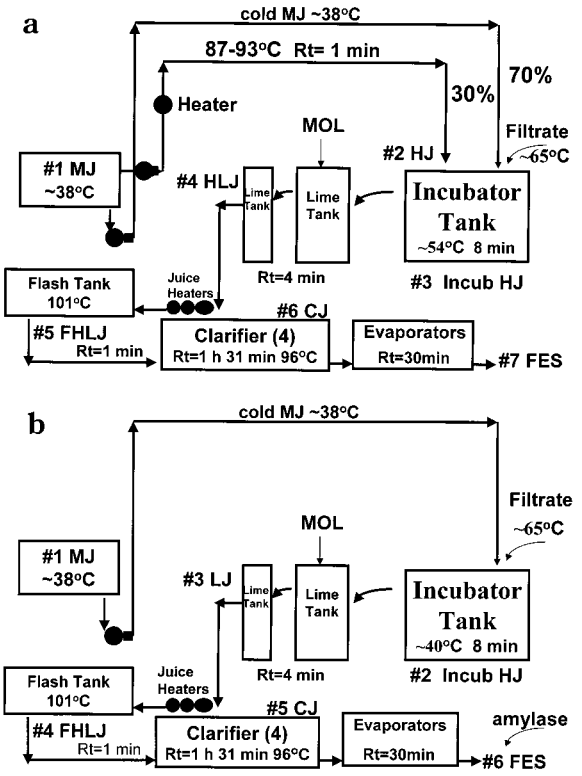

Figure 1. (a) Flowchart of the intermediate lime clarification process. (b) Flowchart of the cold lime clarification process.

a greater need to remove these extra impurities during clarification by using more advanced clarification systems than cold liming.

Although researchers and sugar technologists (see ref *4*) have investigated the merits of various clarificiation systems, particularly hot liming, most of this work was conducted on laboratory study samples. However, laboratory studies did not always reflect the complexity of factory processing streams, which can change in seconds, and gave no or little information on process control, which is essential for engineers. To the best of the authors' knowledge, no systematic factory comparison of intermediate liming as described in this study and cold liming has been undertaken. This is particularly true of factories that process mostly billeted cane. This investigation was, therefore, undertaken to assess the relative effectiveness of cold and intermediate liming processes in the factory.

MATERIALS AND METHODS

Factory Processing Conditions. This study was performed at a Louisiana raw sugar factory, during the 1999 grinding season. The season average factory cane grinding rate and flow rate were 510 short tons/h and ~2380 gallons/min, respectively, and ~95% of the cane processed was billeted (mostly burnt). The factory operated intermediate lime clarification for the last four years but still had the pipes to revert to cold liming for this study. Flow diagrams of the factory intermediate and cold lime clarification processes are shown in **Figures 1a**,**b**, respectively. In intermediate liming, a pump that was separate from the two MJ pumps pumped 30% of the MJ to heaters, heating the juice to 87−93 °C before entering a juice incubation tank. The factory had an incubation tank to keep the juice at optimum temperature and pH conditions for (i) starch degradation by natural cane diastase enzymes and (ii) dextran degradation by added commercial dextranase when deteriorated cane was being processed. The remaining 70% of unheated MJ was pumped directly into the incubator tank without preheating (note: all MJ was first prescreened). Filtrate from the clarifiers was added in the incubation tank, and after it was incubated, the juice was pumped into two lime tanks operated at ambient temperature, where lime was added automatically as a water slurry (average baume ~12) to give a limed juice target pH of 7.2. The limed juice was then flash-heated to ~101 °C to maintain constant temperature and remove bubbles, and polyelectrolyte flocculants were added (4 ppm on clarified juice (CJ)) before entering the clarifiers. The factory had four clarifiers. In this study, CJ was taken from the #4 Dorr Oliver 444 clarifier; the target pH of the CJ was 6.6. The evaporation station consisted of two pre-evaporators and three triple-effect Robert's type calandria evaporators. Commercial α-amylase (2.5 lbs/500 tons of cane) was added in the last evaporator body (maximum temperature, 70 °C). For the conversion to cold liming, the recirculation pump after the MJ tank was stopped so that all of the MJ entered the incubation tank without first being preheated (see **Figure 1b**).

Collection of Factory Samples−General. Because stored cane at the factory deteriorates more rapidly overnight, samples across intermediate and cold liming were taken between 8 am and 5 pm, on two consecutive days, respectively. The factory converted to cold liming at least 1 h prior to sampling to flush out the intermediate lime juice streams. Juices and syrups were carefully collected to prevent further chemical degradation reactions and/or microbial growth. Each sample was first collected in a large container (250 mL), and then, ~25 mL was poured into a small container (50 mL). Sodium azide (biocide 0.02−0.04%) was added to the small container before putting the container in dry ice. Glucose, fructose, and sucrose concentrations were measured in juice from the small containers, usually on the next day. Juice in the large containers was immediately cooled on ice. Brix and pH were measured at the factory. Sodium azide was then added, and the juice was stored in dry ice until transportation to, and storage in, a −80 °C laboratory freezer, before laboratory analyses. Flow rates in any factory fluctuate constantly; therefore, samples across each clarification system were taken at hourly intervals over a 6 h sampling period to obtain precise averages. Each sampling period was repeated three times across the grinding season, to cover cane variety, environmental, and process parameter variations. The three cold liming sampling period dates were as follows: 1 (27 Oct), 2 (30 Nov), and 3 (30 Dec). For intermediate liming, the dates were as follows: 1 (26 Oct), 2 (1 Dec), and 3 (29 Dec).

Sampling−Intermediate Liming. MJ, heated juice (HJ), incubated HJ (incHJ), heated limed juice (HLJ), flocculated HLJ (FHLJ), CJ, and final evaporator syrup (FES) were collected hourly over a 6 h period (see **Figure 1a**). Retention times in the pipes and tanks were taken into account. Consequently, there was a 1 min delay between sampling MJ and sampling HJ, an 8 min delay between HJ and incHJ, a 4 min delay between incHJ and HLJ, a 1 min delay between HLJ and FHLJ, and a 91 min delay between sampling HLJF and sampling CJ. The residence time in the clarifier was calculated using tank dimensions and average flow rate. There was a further 30 min delay between sampling CJ and sampling FES (only an approximation).

Sampling−Cold Liming. Sample collection was the same as for intermediate liming, except that there was no HJ sample as MJ was pumped directly into the incubator tank (**Figure 1b**).

Quantitation of Sucrose, Glucose, and Fructose by Gas Chromatography (GC). Samples were derivatized following the oximation−silylation procedure in ICUMSA GS7/4-22 (1998), and heating blocks were used to heat the derivatized samples at 80 °C. Gas chromatograph conditions varied considerably from ICUMSA GS7/4-22 (1998) because further separation of sugars was required for increased accuracy. The separation of sugars occurred on a DB-5 capillary (5%-phenyl)methyl polysiloxane column (30 m × 0.25 mm id, column film thickness was 0.1 mm) on a Hewlett-Packard 5890A gas chromatograph, equipped with a flame ionization detector. Operation conditions were as follows: injection port, 300 °C; detector, 310 °C; column started at 100 °C for 3 min then was programmed at 5°/min until 150 °C; then 10°/min until 300 °C; and remaining at 300 °C for 10 min. The head pressure was 21 psi with a 25:1 split ratio; the sample volume was 1 μL. Trehalose dihydrate (Alltech) was the internal standard for sucrose, and methyl-α-D-glucopyranoside (Sigma) was the internal standard for glucose and fructose.

Calculation of Sucrose Losses. The percentage of sucrose losses was calculated using the following formula of Schaffler et al. (*6*):

$$\% \text{ sucrose lost} = \frac{[(\% \text{ Glu/Brix})_{out} - (\% \text{ Glu/Brix})_{in}] \times MW_{Suc} \times 100}{(\% \text{ Suc/Brix})_{in} \times MW_{Glu}}$$

where MW = molecular weight, Suc = sucrose, Glu = glucose, and Brix = percent dissolved solids.

Settling Rates and Mud Volumes of FHLJs. Settling and mud volume (MV) measurements and calculations were based on the methods of Schmidt (*7*) and Lionnet and Ravno (*8*), with modifications. The FHLJ samples were heated to 96 °C, with constant stirring, before they were poured into a settling tube (5 cm × 34 cm). The settling tube was mounted in a glass waterbath maintained at the clarifier temperature. Each sample was poured into a settling tube to a volume of 300 mL and stoppered immediately, and mud level readings were taken between 0.0 and 18.0 min. A preliminary test had established experimental reproducibility of this settling method. **Calculations.** Mud height was plotted against time. The initial settling rate (ISR) in milliliters per minute was determined graphically from the initial linear slope. MV of FHLJ samples after 18 min (MV_{18}) were read directly. The final equilibrium FHLJ MV (MV_{∞}) or final height of the mud after infinite time was obtained from the intercept on a plot of MV percent vs $1/t$ where t is time in minutes.

Brix (Percent Dissolved Solids). The mean Brix of triplicate samples was measured using a Leica Abbe Mark II refractometer with a crosshair reticule.

pH was measured at room temperature (~25 °C), using an Ingold combination pH electrode calibrated at room temperature using two different pH buffers (pH 7 and pH 10). The electrode was connected to a Metrohm 716 DMS pH meter.

Color and turbidity were measured as the absorbance at 420 nm and calculated according to the official ICUMSA method GS2/3-9 (1994). Samples (5 g) were diluted in triethanolamine/hydrochloric acid buffer (pH 7) and filtered through a 0.45 μm filter.

Dextran concentrations for duplicate composite samples (10 g of each hourly sample were combined) were measured using the Robert's (*9*) copper method.

Starch concentrations for duplicate composite samples were measured using a colorimetric method (*10*), based on the starch−iodine complex.

Calcium of composite samples as CaO was by ethylenediaminetetraacetic acid titration following the ICUMSA method GS8/2/3/4-9 (1994).

Statistical Analysis. Data were analyzed using PC-SAS 8.1 (SAS Institute, NC) software. The process (intermediate vs cold liming) and sample type were considered as fixed effects, whereas the sampling period was regarded as a random effect. Hourly samples within a given sampling period were considered as replications. Initially, a four factor model (process, sample type, sampling period, and sampling hour) was tested using PROC GLM. Because sampling hour was not significant by the *F*-test, it was dropped as a model factor to increase error degrees of freedom. The remaining three factors were tested as main effects using PROC GLM. Means comparisons were undertaken using Duncan's New Multiple Range Test.

RESULTS AND DISCUSSION

pH and Inversion Control Across Both Liming Processes. Although there were no marked differences in sample pH values and standard deviations (process control) between the two processes (**Table 1**), the pH of the CJs, and particularly ($P < 0.05$) FES, was slightly higher in intermediate than cold liming. Although the decrease in pH from CJ to FES can be attributed to the precipitation of lime salts, concentration of H^+ ions, and release of small quantities of ammonia, it also led to reduced sucrose inversion occurring across the clarifiers and evaporators in intermediate liming (**Table 2**). Acid inversion of sucrose in sugarcane factories is more easily indicated by an increase in glucose and fructose, which are the first degradation products of inversion (*10*). This is because, in comparison to the high concentrations of sucrose, they occur at lower and, therefore, more measurable concentrations (*6*), and changes in their concentrations are more easily detected. However, although sucrose inverts into equal amounts of glucose and fructose, fructose is more labile than glucose (*6*) under acid conditions, making it an inaccurate measurement of inversion. As Schaffler and co-workers (*6*) demonstrated that glucose and sucrose referenced to Brix (percent dissolved solids) can be used to accurately calculate the percent sucrose loss, their calculation formula (see Materials and Methods section) was used in this study, using the data presented in **Table 2**. However, it must be noted that some glucose may also be destroyed, and so, the calculation formula is a conservative estimate of sucrose loss.

In cold liming, a season average of 0.44% sucrose was lost across the flash heater, 0.66% across the clarifiers, and 1.54% across the evaporators. With intermediate liming, only 0.26 and 0.81% were lost across the clarifiers and evaporators, respectively, with no previous inversion on flash heating. This is a substantial 1.57% difference in sucrose losses, and the savings to the factory using intermediate liming were approximately

Table 1. Season Average pH Values ($N = 18$)

sample	cold	intermediate
MJ	5.49 ± 0.12a[a]	5.48 ± 0.14a
HJ	n/a	5.40 ± 0.16
incub J	5.61 ± 0.14a	5.54 ± 0.16a
LJ/HLJ	7.47 ± 0.34a	7.52 ± 0.43a
FHLJ	7.13 ± 0.57a	7.07 ± 0.37a
CJ	6.22 ± 0.21a	6.39 ± 0.35a
FES	5.98 ± 0.15a	6.09 ± 0.10b

[a] Lower case letters represent statistical differences ($P < 0.05$) between the two clarification processes for season averages.

Table 2. Sucrose, Glucose, and Fructose Concentrations (Season Averages)[a]

		season avg. concns ± SD ($N = 10$[b])		
sample	avg. Brix ± SD	sucrose	glucose	fructose
		Cold		
MJ	15.13 ± 0.70a[c]	85.50 ± 0.92a	1.61 ± 0.53a	1.55 ± 0.70a
incub J	14.71 ± 0.67a	85.97 ± 1.52a	1.44 ± 0.20a	1.39 ± 0.18a
LJ	14.95 ± 0.49a	86.06 ± 1.59a	1.39 ± 0.16a	1.31 ± 0.16a
FHLJ	14.89 ± 0.49a	86.40 ± 2.05a	1.59 ± 0.40a	1.53 ± 0.33a
CJ	14.71 ± 0.48a	86.45 ± 1.37a	1.89 ± 0.61a	1.77 ± 1.54a
FES	68.04 ± 2.57a	86.80 ± 2.86a	2.59 ± 1.82a	2.35 ± 1.54a
% change MJ to CJ			+0.28	+0.22
% change CJ to FES			+0.70	+0.58
		Intermediate		
MJ	14.51 ± 0.64a	86.01 ± 1.58a	1.81 ± 0.87a	1.71 ± 0.74a
HJ	14.97 ± 0.40	86.24 ± 1.61	1.58 ± 0.55	1.52 ± 0.38
incub J	14.20 ± 0.67a	86.02 ± 1.45a	1.70 ± 0.68a	1.59 ± 0.48a
HLJ	14.39 ± 0.72a	86.13 ± 1.55a	1.61 ± 0.39a	1.51 ± 0.34a
FHLJ	14.38 ± 0.57b	86.48 ± 1.75a	1.55 ± 0.33a	1.51 ± 0.26a
CJ	14.42 ± 0.53a	86.95 ± 1.30a	1.67 ± 0.35a	1.58 ± 0.30a
FES	67.49 ± 2.13a	87.47 ± 2.12a	2.04 ± 1.16a	1.89 ± 0.99a
% change MJ to CJ			−0.14	−0.13
% change CJ to FES			+0.37	+0.31

[a] Sugar concentration is percent sugar (measured by GC)/Brix. [b] At least three randomnly chosen hours from each sampling period were analyzed. [c] Lower case letters represent statistical differences ($P < 0.05$) between the two clarification processes for season averages.

Table 3. Color Across Cold and Intermediate Clarificition Processes[a]

Cold Liming

sample type	avg. color (420 nm) ± SD: sampling period 1	2	3	season avg. ± SD
MJ	13 446 ± 2634A	11 634 ± 2320AB	10 370 ± 938B	11 817 ± 2360a
incub J	12 257 ± 2266A	9852 ± 1474B	9719 ± 803B	10 545 ± 1950a
LJ	13 914 ± 3075A	11 724 ± 2433AB	9719 ± 803B	11 786 ± 2796a
FHLJ	10 249 ± 2918A	8022 ± 831AB	7134 ± 485B	8495 ± 2197a
CJ	8776 ± 1848A	7180 ± 931B	7031 ± 479B	7662 ± 1410a
FES	14 218 ± 1774A	11 011 ± 559B	10 023 ± 642B	11 751 ± 2129a
% MJ to incub J	−8.8	−15.3	−8.1	−10.8
% incub J to LJ	+13.5	+19.0	+2.0	+11.8

Intermediate Liming

sample type	avg. color (420 nm) ± SD: sampling period 1	2	3	season avg. ± SD
MJ	11 782 ±1910A	11 818 ± 1016A	10 449 ± 1599A	11 324 ± 1574a
HJ	8536 ±1689A	8011 ± 514A	7402 ± 976B	7951 ± 1147
incub J	10 374 ± 118A	9045 ± 1138B	7837 ± 496B	9009 ± 1376b
HLJ	11 911 ± 1435A	9253 ± 561B	8783 ± 1535B	9869 ± 1799b
FHLJ	9645 ± 449A	7737 ± 537B	7274 ± 328B	8188 ± 1116a
CJ	7974 ± 508A	7062 ± 263B	6841 ± 443B	7252 ± 623a
FES	11 712 ± 1177A	11 674 ± 389A	10 503 ± 615B	11 272 ± 924a
% MJ to incub J	−12.0	−23.5	−25.0	−20.4
% incub J to HLJ	+14.8	+2.3	+12.1	+9.6

[a] Capital letters represent statistical differences ($P < 0.05$) across the sampling periods, and lower case letters represent statistical differences between the two clarification processes for season averages.

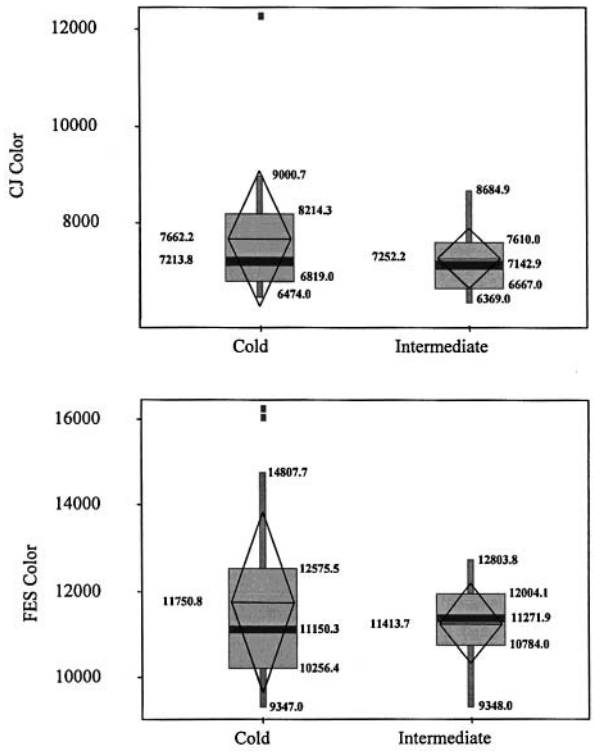

Figure 2. Box plot comparison of process control of CJ and FES color by cold and intermediate liming. (The horizontal line in the middle of the box marks the median. The top and bottom edges of the box mark the 25th and 75th quartiles. Whiskers extend to the farthest observation not farther than 1.5 times the distance between the quartiles. The horizontal line in the diamond marks the mean. The height of the diamond is two standard deviations (one on either side of the mean)).

$748,033 for the season, taking into account the pounds of raw sugar produced by the factory in 1999, cane sucrose recovery rate, average sucrose content of the raw sugar, and current average price of raw sugar (20 cents/lb).

A possible problem associated with the preheating of acidic MJ in intermediate liming is sucrose inversion. However, as seen in **Table 2**, there was an overall season decrease in glucose and fructose concentrations in the HJ from the MJ samples. The 1 min retention time of heating (see **Figure 1a**) most likely minimized inversion. Some glucose and fructose may have been removed with the heat-induced precipitation of macromolecules and color (*12*); the slight increase in HJ sucrose concentrations over those of MJ (**Table 2**) is further evidence of this.

For both processes, glucose and fructose were lower in the incubated juice than the MJ (**Table 2**). Again, this would indicate that excessive inversion did not occur under the acidic incubation conditions, and the slight decrease in glucose and fructose is because of a dilution factor from additional recycled filtrate. This is also indicated by the slight lowering of Brix.

Color Removal and Formation. The color of the incoming MJ varied slightly across the season for both processes, with the lowest MJ color at the end of the season (**Table 3**). In intermediate liming, preheating 30% of the MJ (HJ) before incubation caused marked color (season average ∼29%) removal. In previous studies on hot liming, where MJ is heated to an even higher temperature (∼107 °C) before liming, color was in a similar manner consistently removed from the MJ (*4*, *13*−*14*). This heat-induced color removal is considered to be associated with the precipitation of colloids and macromolecules, including starch and dextran polysaccharides and proteins (*12*). The high color removal in the HJ caused the incubated juice color to be significantly ($P < 0.05$) lower than that formed in cold liming (**Table 3**). Furthermore, for both processes across the season, ∼10% color was also removed on incubation (**Table 3**). The calcium salts contained in the recycled filtrate from the clarifiers may have precipitated some color.

The removal of color before liming was, however, offset by color formation (season average ∼10%; see **Table 3**) on liming, for both processes. This color formation was caused by the alkaline degradation of invert (see **Table 2**) and is a function of temperature and retention time. Eggleston (*3*, *4*) similarly observed that approximately 10% color formed on cold liming in various Louisiana factories across the 1996−1998 seasons.

After liming in both processes, color was removed by the settling process in the FHLJ and CJ samples. Color formed, as usual, across the evaporators because of further reactions of degradation products from the inversion of sucrose. The season average CJ color (7252 ± 623) in intermediate liming was still less than in cold liming (7662 ± 1410 ICU), as was FES color (see **Table 3**). However, while analysis of variance showed no sign of significant differences between cold and intermediate lime CJ and FES samples (**Table 3**), the box plots presented in **Figure 2** strongly suggest better processing control in intermediate liming. Good process control is vital for smooth factory throughput.

Overall, as compared to cold liming, markedly more color was removed than formed in intermediate liming, and color control was better. However, unlike in hot liming (*4*) where lime tanks are not required because lime is added to the juice

Table 4. Differences in Dextran Concentrations (Composites)

Cold Liming			
	avg. dextran (ppm on solids)		
	sampling period		
sample type	1	2	3
MJ	374	458	505
incJ	379	556	419
LJ	631	457	726
FHLJ	721	924	787
CJ	531	670	646
FES	746	630	883

Intermediate Liming			
	avg. dextran (ppm on solids)		
	sampling period		
sample type	1	2	3
MJ	402	587	506
HJ	355	475	486
incHJ	374	397	448
HLJ	510	521	794
FHLJ	526	560	726
CJ	723	490	624
FES	729	546	914

Table 5. Differences in Starch Removal (Composites)

Cold Liming			
	avg. starch (ppm on solids)		
	sampling period		
sample type	1	2	3
MJ	1275	913	204
incJ	1057	771	183
LJ	1036	665	136
FHLJ	852	706	73
CJ	1303	900	167
FES	1220	993	148

Intermediate Liming			
	avg. starch (ppm on solids)		
	sampling period		
sample type	1	2	3
MJ	1406	1197	304
HJ	824	717	329
incHJ	759	596	97
HLJ	575	202	75
FHLJ	556	472	200
CJ	689	688	213
FES	296	295	165

directly in pipes, color forms in the lime tanks in intermediate liming, because of the higher retention time of actual liming.

Dextran Removal and Degradation. Dextran is a polysaccharide formed by *Leuconostoc* bacteria, which is viscous in solution. Its rate of formation is dependent on many factors including cane harvest method, time and method of storage at the factory, temperature, aeration, and humidity. Maintaining low levels of dextran is a major priority not only to reduce sucrose losses but also to ensure adequate factory flow throughput, crystallization rates, and to avoid raw sugar penalities from the refinery.

The cold and dry weather across the 1999 grinding season kept dextran levels relatively low, which is reflected in the MJ values in **Table 4**. In intermediate liming, dextran was consistently removed on the preheating of the MJ (see HJ samples in **Table 4**). This agrees with recent hot liming results at another Louisiana factory (*4*).

It appears that occasionally, dextran was slightly formed in the cold and intermediate lime tanks (**Table 4**). Lillehoj et al. (*15*) observed that there is growth of *Leuconostoc* dextran forming bacteria in cold limed juice. The pH rise in liming can slow *Leuconostoc* growth, but in cold liming, growth still proceeds because the optimum conditions for organism growth, although not necessarily for dextran production, are about pH 7 (*15*). The slightly higher temperature of intermediate liming probably also caused bacteria to grow. In comparison, the much higher temperatures of liming in the hot liming process disallow any bacterial growth.

For both processes, dextran generally decreased in the CJ across the season, and this reflects the efficiency of the settling process. There was a buildup of dextran across the evaporators. Clarke et al. (*16*) observed a similar buildup of dextran across evaporators in phosphatation bone-char and carbonation bone-char sugarcane refineries, and Eggleston (*4*) observed it in cold liming and hot liming mills in Louisiana. This buildup is attributable to increased concentrations of impurities across the factory, which occlude more dextran in the sucrose molecules. Results here indicate that generally, the preheating of MJ (see HJ values) before incubation and liming, as in intermediate liming, helps to offset dextran problems.

Starch Removal and Degradation. Sugarcane, unlike most other plants, stores sucrose rather than starch as its major carbohydrate energy source. Nevertheless, a small amount of starch, usually <1% total solids, is always present throughout tissues of the cane plant, especially in the immature growing points, the leaves and nodes (*10*), and varies with cane variety. Although starch concentration is low in sugarcane, starch, for the same concentration as dextran, imparts a much greater viscosity and can cause major processing problems, particularly the slowing of factory flow throughput and reducing the filterability of raw sugar. Granular, insoluble starch is expressed into the juice during milling and becomes progressively solubilized and gelatinized across the factory. Godshall et al. (*10*) observed that a significant proportion of total starch was already solublilized and gelatinized (up to 80%) coming off the mill into the MJ.

As seen in **Table 5**, starch concentrations in MJ decreased dramatically across the grinding season because of increasing cane maturity. For both clarification processes, starch was degraded in the incubator tank, because the addition of recycled filtrate from the clarifier reduced the MJ acidity, enabling the natural juice diastase to degrade starch, especially solubilized and gelatinized starch (*17*). Furthermore, in intermediate liming, prior heating of MJ before incubation (HJ) had already markedly reduced starch concentrations (the slight increase in sampling period 3 is most likely because experimental error is magnified at such low starch concentrations). It is known (*1*, *18*) that heat can induce floc and subsequently scum and scaling formation without the application of lime. Furthermore, other researchers (*4*) have previously observed that heating juice removes impurities including high molecular weight denatured proteins such as casein (*19*), colloids, and other compounds such as color (see **Table 3**) and oligosaccharides (*20*).

In both liming processes, additional starch was removed on liming (HLJ samples). The calcium phosphate complex formed on liming would have precipitated some starch. However, the increase in temperature in the FHLJ and CJ caused an increase in measured starch. At such high temperatures, any insoluble starch granules would have been solubilized and gelatinized, and the colormetric method employed is obviously sensitive to

Table 6. Settling Performances

sampling period	ISR (mL/min)	MV after 18 min (%)	MV at infinity (%)	sampling period	ISR (mL/min)	MV after 18 min (%)	MV at infinity (%)
	Cold				**Intermediate**		
1	163[a] ± 86	18.6 ± 3.0	17.2 ± 2.5	1	235 ± 81	15.5 ± 6.3	14.6 ± 5.4
2	265 ± 111	11.1 ± 2.9	10.2 ± 2.4	2	234 ± 96	13.8 ± 1.6	13.0 ± 1.2
3	171 ± 53	12.0 ± 1.2	11.9 ± 1.1	3	261 ± 117	12.3 ± 1.2	10.9 ± 0.5
season avg.	205 ± 100a[b]	14.0 ± 3.5a	13.4 ± 3.3a	season avg.	242 ± 91a	13.7 ± 3.7a	13.0 ± 3.4a

[a] $N \geq 4$. [b] Lower case letters represent statistical differences ($P < 0.05$) between the two clarification processes for season averages.

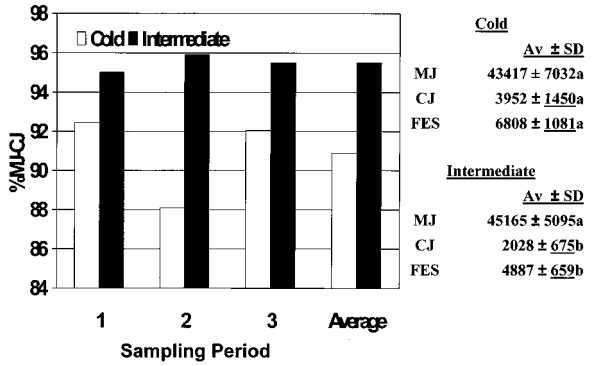

Figure 3. Percentage turbidity (at 420 nm) removal and values for each clarification process.

the solublized and gelatinized states of the starch. Godshall et al. (*10*), using this same starch method, also observed occasional increased starch in factory CJs and evaporator syrups as compared to MJs, although the type of clarification system utilized was not stated.

A final reduction in starch concentrations generally occurred, across the season, in the FES. It is the practice of this and many other raw sugar factories to add commercial amylase enzyme in the FES tanks to degrade mostly solubilized starch. The enzymic degradation of starch in FES was substantially greater and more consistent in the intermediate than cold liming samples. This large difference reflects the fact that a much higher removal of starch had occurred prior to this final stage in intermediate clarification.

Turbidity Removal. The major aim for any clarification process is to remove turbid "impurity" particles. For every sampling period, turbidity removal (MJ to CJ) was significantly ($P < 0.05$) higher in intermediate than in cold liming (see **Figure 3**). Intermediate CJ turbidity (season average 2028 ICU ± 675) was approximately half of the cold CJ turbidity (average 3952 ICU ± 1450) with over 2-fold more CJ turbidity control ($P < 0.0001$). This led to lower FES turbidity values in intermediate (average 4887 ICU ± 659) rather than cold (average 6808 ICU ± 1081) liming, again with improved control (**Figure 3**), which will be reflected in the raw sugar quality. In hot liming (*12*, *21*, *22*), higher turbidity removal, as compared with cold liming, is because of the faster settling of larger flocs, which are created in the 100% preheating of MJ at ~104−107 °C (*12*, *21*, *22*). In the case of intermediate liming, only 30% of the MJ was preheated and at a lower temperature of 87−93 °C, but there was still an impressive 4.6% increase in turbidity removal over cold liming across the season, and the intermediate flocs were usually larger. These results, therefore, indicate that all of the MJ does not have to be preheated to significantly gain in turbidity removal, nor does the preheating temperature have to be as high as 104−107 °C. The lower temperature would also have the additional advantage of reducing the rate of acid inversion of sucrose.

Table 7. Differences in Lime Addition (Composites)

process	sampling period	calcium (ppm on Brix basis) MJ	incJ	LJ/HLJ	CJ	change incJ to LJ/HLJ
cold lime	1	31.6	33.2	48.5	35.2	15.3
	2	33.1	31.9	47.2	34.6	15.3
	3	33.6	32.7	44.8	34	12.1
mean ± SD		32.8 ± 1.0	32.6 ± 0.7	46.8 ± 1.9	34.6 ± 0.6	14.23
intermediate lime	1	32.7	32.7	47.1	36.8	14.4
	2	30.4	31.7	45.7	36.6	14.0
	3	28.3	30.7	43.1	35.1	12.4
mean ± SD		30.5 ± 2.2	31.7 ± 1	45.3 ± 2.0	36.2 ± 0.9	13.6

Settling Performance. Differences in settling performance, across the grinding season, were also assessed in terms of ISR and MVs (**Table 6**) of the FHLJs. For both processes, where a very slow ISR (i.e., <70 mL/min) was measured, the FHLJ mud samples were usually markedly higher, i.e., >25%. Schmidt (*7*) previously observed that settling rates "fall off quite markedly as the mud concentration increases" in cane juices. MVs were larger at the beginning of the season for both processes, particularly cold liming, which caused the ISRs to be lower (**Table 6**). This was most likely because more trash and mud are often associated with immature, Polado-ripened cane. Generally, there were no significant differences in the settling rates between the two processes, although season average ISRs were slightly higher in intermediate liming with less variability. Moreover, there were no general differences in MVs, whereas in hot liming higher but less dense MVs have often been observed in comparison to cold liming (*13*, *22*, *23*). The small differences in this study are most likely because real factory samples were analyzed rather than model laboratory samples often reported (*13*, *22*, *23*), and the variations could also be due to cane varieties and age, district grown, time of year, and flocculant added (*8*).

Lime Addition. Across the season, ~4.6% more lime was added in cold liming, which is most likely because liming temperatures were not very different (**Table 7**). This led to no significant differences in the calcium contents of CJs from both processes. In comparison, various authors have observed around a third less lime is required in hot liming (*13*, *21*).

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