MIXED JUICE CLARIFICATION REVISITED

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Abstract

The clarification of mixed juice has been the subject of research for decades, due mostly to its large impact on sugar quality. Much, if not all, of the work has however been done on mixed juice originating from milling rather than from diffusion. This paper examines a back-to-basics, laboratory approach, involving cane juices produced from conditions simulating milling and diffusion. Furthermore, different types of cane, for example fresh versus deteriorated, clean stalks versus stalks plus tops and trash, have been used. Saccharate and milk of lime have also been compared. Analytical techniques were carefully considered and methods such as X-ray fluorescence were used, and material balances across clarification were done. The laboratory work shows that, for the same cane quality, clear juice quality in terms of phosphate, calcium, magnesium, silica and sulphated ash is the same whether milling or diffusion has been used. Saccharate liming always yields better clear juice in terms of turbidity, colour, phosphate and calcium contents. Material balances closed well across clarification, except for phosphate and for silica; this highlights the need for sampling/analytical improvements for these two species. Finally, the effect of clarifier mud carryover on sugar quality is discussed.

Keywords: clarification, saccharate, turbidity, phosphate.

Introduction

The main objective of this project has been to improve mixed juice (MJ) clarification. Although clarification has been the subject of many investigations, there has been no recent major breakthrough in the industrial process used for cane juice. Processing conditions have changed, for example there has been a major shift to diffusion in Southern Africa, short residence time clarifiers have been introduced, and more recently, filter stations have been eliminated at some diffusion factories.

The main objectives of the present work were:

- To re-examine cane juices produced from different cane types.
- To re-examine cane juices produced from conditions simulating milling and diffusion.
- To compare milk of lime (m-o-l) and saccharate (sacch) liming in the laboratory, with emphasis on investigating sacch liming in areas such as turbidity removal, impurity of the resultant clear juice (CJ), colour, and the effect of evaporation on turbidity.
- To construct material balances for some of the main impurities, across clarification.
- To demonstrate on a pilot scale the effect of clarifier mud carryover on sugar quality.

Analytical methods

All the analyses for juices and sugars were analysed according to the methods given in the SASTA Laboratory Manual (Anon, 1985). Mud was analysed by X- ray fluorescence.

Procedures for laboratory trials

The size and complexity of this investigation caused limitations in the experimental procedures. For example sacch had to be prepared using pure sucrose rather than the normal clear juice or syrup as used industrially. The deterioration trials used unburnt cane which is not representative of industrial conditions. Finally the simulated diffuser conditions cannot be fully representative of all industrial practices.

Cane type
Four types of cane were used viz. clean fresh cane stalks, clean fresh cane stalks plus tops, clean fresh cane stalks plus trash, and deteriorated clean cane stalks which were obtained by leaving harvested cane stalks in the field for two weeks prior to processing. All the cane was unburnt and was of the same variety. The samples originated from a demarcated area in one of the fields at the South African Sugar Association Experiment Station (SASEX). The cane stalks were shredded at the Sugar Milling Research Institute (SMRI) in a Jeffco cutter grinder, and the pulps sub-sampled, mixed as required and the juice extracted in a Pinette Emidecau press and clarified the same day.

M-o-l and sacch were used for the laboratory clarification. Sacch was prepared by using a pure sucrose solution (ratio of 7 parts sucrose to 1 part calcium oxide). The juice was heated, limed, degassed by gentle boiling and settled at 100°C in the SMRI settling kit for 30 minutes, using 3 mg/l of a conventional polyelectrolyte flocculant.

Cold versus hot extraction (simulation of milling and diffuser juices)
The juice for the cold extractions was obtained by pressing the cane pulp under standard conditions at ambient temperature.

For the hot extractions, the required masses of cane pulp and of water were mixed, using water at 80°C, placed in a plastic sachet, and kept submerged in a water bath at 80°C for 30 minutes. The press was warmed by pouring hot water through it and the hot cane/water mixture pressed under standard conditions. Clarification was then done in the normal way.

Milk of lime versus saccharate - effect of evaporation on turbidity and on selected impurities
Clear juices were obtained from cane pressed at the SMRI as described earlier. Syrup was then produced by laboratory evaporation and tested for turbidity. Four batches from each of the juices (m-o-l and sacch) were evaporated to brixes simulating the four effects of an evaporator. The apparatus used for the evaporation was the boiling down apparatus (Bruijn, 1977) which simulates a single tube evaporator with steam side heating and absolute pressure control.

The juice and syrup samples were analysed for brix, turbidity and some selected species.

Material balance
For these balances, masses of the MJ, m-o-l or sacch, flocculant and mud were recorded. The mass of CJ was obtained from the following equation:

Mixed juice + flocculant + m-o-l/sacch = CJ + mud (dry)

MJ, CJ and mud were sampled for analysis.

Effect of clarifier mud carryover on sugar quality
Cane of the same variety was demarcated at SASEX so that all the tests could be done on the same type of cane. MJ was produced by crushing cane at the SMRI firstly on a once through basis in a three roller pilot mill, adding imbibition water to the crushed stalks and then passing through the mill again. The

resultant MJ was clarified using the hot liming process and the mud retained for the carryover tests. The CJ was evaporated to 65° Brix at the SMRI in a pilot forced circulation plate evaporator at atmospheric pressure. The syrup was crystallised in the SMRI pilot pan (Lionnet, 1995) to make A-massecuite which was then separated in a centrifuge to give A-sugar. Two control tests (no carryover) and two with carryover (5% m/m mud added to the CJ prior to evaporation) were done.

Procedures for factory juices

Identical experimental procedures to those used for the SMRI trials viz. laboratory clarification, sampling and analysis were done but on MJ from factories. M-o-l versus sacch were investigated and material balances were determined using MJ from Maidstone (MS), Sezela (SZ) and Gledhow (GH).

Milling tandem and diffuser juices from GH were used since the cane to these two extraction plants is allocated randomly; the MJ obtained should thus reflect the different effects of milling and diffusion.

The tests to determine the effect of evaporation on syrup turbidity were done on CJ from factory MJ, but the CJ was evaporated at the SMRI.

Laboratory trials results

Cane type

Averaged analytical results (Table 1) for selected species from eight tests each for:

- clean cane versus others (cane+tops, cane+trash and clean deteriorated cane)
- clean cane versus clean deteriorated cane
- clean cane versus clean cane+tops+ trash.

Table 1: Results averaged for cane type

MJ	P_2O_5	Ca^{2+}	Mg^{2+}	SiO_2	S.ash
	mg/kg brix				%
Clean	1274	1469	1165	591	0.36
Others	1976	2271	2009	702	0.45
Clean Deteriorated	1391	1414	1735	546	0.29
Cane+Tops+Trash	2565	3128	2283	868	0.62
CJ					
Clean	370	1576	1026	325	0.30
Others	382	3054	1791	360	0.50
Clean Deteriorated	208	2309	1587	315	0.34
Cane+Tops+Trash	556	3789	1995	404	0.65
Mud	Clean cane		Others		% difference
g/kg juice	0.54		1.04		93

The results in Table 1 show that:

- Deterioration does not affect MJ in terms of P_2O_5, Ca^{2+}, Mg^{2+}, SiO_2 and S.ash. CJ quality however is negatively affected as follows; calcium (Ca^{2+}), magnesium (Mg^{2+}) and sulphated ash (S.ash) are higher.

- Tops and trash have very negative effects on both MJ and CJ qualities. S.ash in CJ has increased by 115%.
- The combined effect of deterioration and of extraneous matter, which would represent industrial cane more closely, is also shown. The concentration of all the species in CJ has increased. Similarly mud mass increases by 93%.

Cold versus hot extraction (simulation of milling and of diffusion)

The averaged results for MJ, CJ and for mud are shown in Table 2.

Table 2: Results averaged for cold and hot extractions

Sample	P_2O_5		Ca^{2+}		Mg^{2+}		SiO_2		Total ash	
	mg/kg brix								% mass/mass	
	cold	hot	cold	hot	cold	hot	cold	hot	cold	hot
MJ	1557	1927	1978	2029	1706	1749	693	644	0.39	0.40
CJ	376	380	2604	2512	1531	1541	361	336	0.43	0.43

Mud	Moisture	Mud	LOI	Total ash
	%	g/kg juice	% mass/mass	
Cold	4.2	0.963	75	25
Hot	4.2	0.766	58	42
% difference	-	-20	-23	+68

Except for phosphate (P_2O_5), where there is about 20% more from the hot extraction, the cold versus hot process does not impact markedly on MJ. For CJ, there appears to be no marked difference in juice quality as a result of hot or cold extractions. It is interesting to note that Mg^{2+} is found in both MJ and CJ at high concentrations which are however lower than those of Ca^{2+}.

The quantity of mud from the hot extraction is about 20% lower than that from the cold extraction, however the loss on ignition (LOI) is much higher in the mud from the cold extraction (75% vs. 58%). Since the organic and total ash (100-LOI) contents add to 100%, the total ash content in the mud from the cold extraction is lower.

Milk of lime versus saccharate

The averaged results for MJ, CJ and mud derived from m-o-l and sacch are shown in Table 3.

Except for a lower phosphate level in CJ for sacch, the CJ qualities with respect to the species measured are generally similar.

The quantity of mud produced with sacch is about 16% higher than that resulting from the use of m-o-l, and the organic content of the mud is also higher; hence the increase in mass of the mud from sacch liming is due to a higher content of organic matter. Sacch liming yields higher mud volumes than m-o-l, however the amount of dry mud is also higher with sacch indicating that more impurities are removed by the latter.

Table 3: Results averaged for m-o-l and sacch liming

MJ	pH	Brix	K^+	P_2O_5	Ca^{2+}	Mg^{2+}	SiO_2	S.ash
			mg/kg brix					%
	5.2	10.10	9365	1830	2102	1875	703	0.43
CJ m-o-l	7.1	10.65	9685	465	2690	1560	365	0.43
CJ sacch.	6.9	10.99	8970	330	2700	1660	365	0.43

Mud	Moisture	Mud	LOI	Total ash
	%	g/kg juice	% mass/mass	
m-o-l	4.0	0.802	64	36
sacch	4.2	0.949	70	30
%difference	-	+16	+9	-16

Material balance

The average results from 6 runs each for m-o-l and sacch are shown in Table 4. Pure calcium hydroxide ($Ca(OH)_2$)was used, i.e. no Mg^{2+} was added, and the Ca^{2+} added as lime was measured and subtracted.

Table 4: Material balance in grams (g) over clarification

M-o-l	K^+	P_2O_5	Ca^{2+}	Mg^{2+}	SiO_2	S.ash
In MJ	3.79	0.74	0.81	0.70	0.28	15.60
In m-o-l	-	-	0.93	-	-	3.15
Total (g)	3.79	0.74	1.74	0.70	0.28	18.75
In CJ	4.12	0.20	1.14	0.66	0.16	17.10
In Mud	0.001	0.48	0.41	0.055	0.20	1.20
Total (g)	4.12	0.68	1.55	0.71	0.36	18.30
% difference	+9	-8	-11	+2	+28	-2
Sacch	K^+	P_2O_5	Ca^{2+}	Mg^{2+}	SiO_2	S.ash
In MJ	3.74	0.73	0.88	0.76	0.28	16.00
In sacch	-	-	0.853	-	-	2.90
Total (g)	3.74	0.73	1.73	0.76	0.28	18.90
In CJ	3.94	0.15	1.19	0.74	0.16	17.60
In Mud	0.002	0.47	0.39	0.049	0.04	1.20
Total (g)	3.94	0.62	1.58	0.79	0.20	18.80
% difference	+5	-15	-9	+4	-28	-1
% difference = (mass in CJ + Mass in Mud - mass in MJ) * 100 / mass in MJ						

The mass balances show that sampling and/or analytical problems are experienced with phosphate and silica determinations. The balances for the other species close to within 10%, which is acceptable considering the complexity of these tests.

Most of the S.ash, potassium (K^+)and Mg^{2+} from MJ or added through the liming agent exit in the CJ. P_2O_5 however exits in the mud, while Ca^{2+} and silica (SiO_2) are distributed between CJ and mud.

Effect of evaporation on turbidity and selected impurities
A full scale test at one mill (Scott, 1988) showed that the lower CJ turbidity obtained with sacch did not persist in syrup and sugar.

The average turbidities and Ca^{2+} concentrations in juices extracted at the SMRI are shown in Figures 1A and 1B respectively**.**

The initial turbidity of the CJ produced from sacch is lower than that produced from m-o-l. The turbidity of both juices increases on evaporation and stabilises after the simulated 3 rd effect but the use of sacch still results in lower turbidities.

The Ca^{2+} levels do not change markedly on evaporation, however the levels in syrup (4th effect) using m-o-l or sacch appear to be lower, indicating scaling.

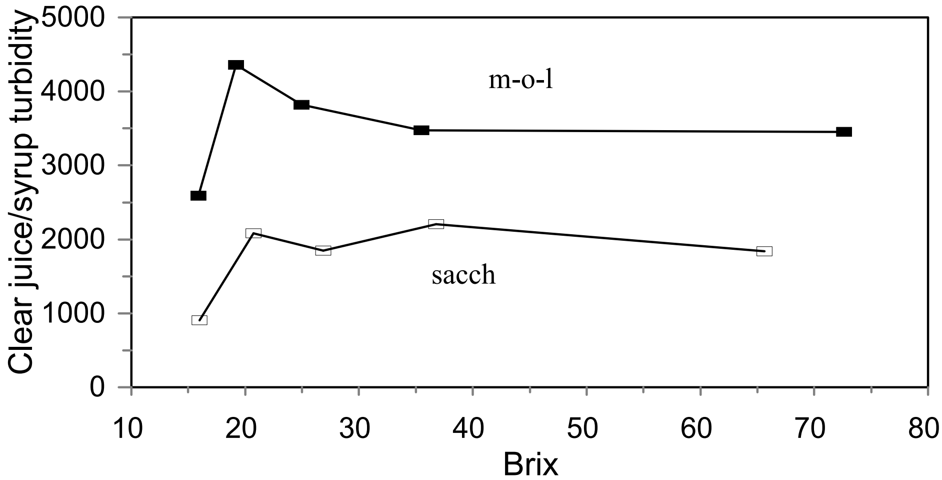

Figure 1A: CJ/ SYR turbidity versus brix, simulated evaporator effect.

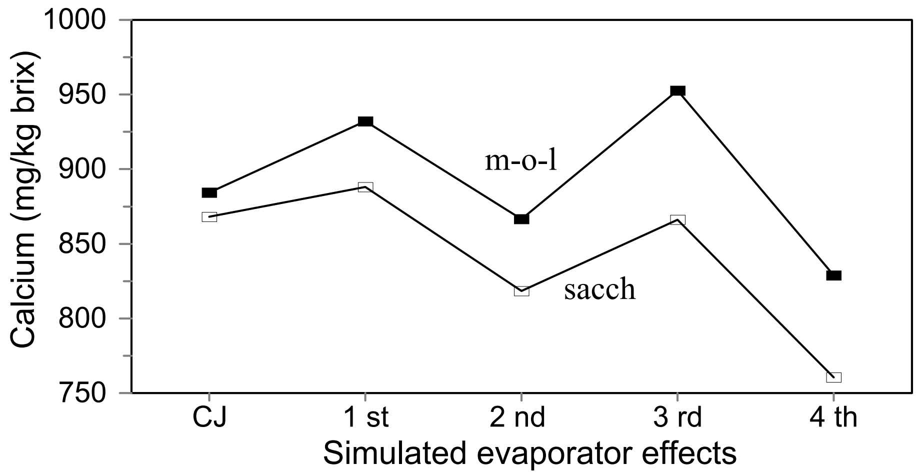

Figure 1B: Ca^{2+} content of syrup after each simulated evaporator effect

Effect of mud carryover on sugar quality
MJ, CJ, syrup (SYR), affinated sugar (AFFS) and A-molasses (A-mol) as produced in the SMRI pilot plant were analysed for the following:

Colour, Brix, P_2O_5, Ca^{2+}, K^+, S.ash, fructose(F), glucose (G), sucrose (S) and pH @ 25°C.

Colour and S.ash profiles from MJ to A-mol, for the control and the carryover tests, are shown in Figures 2A and 2B where it is clearly evident that carryover of muds results in an increase for these two species in all the products.

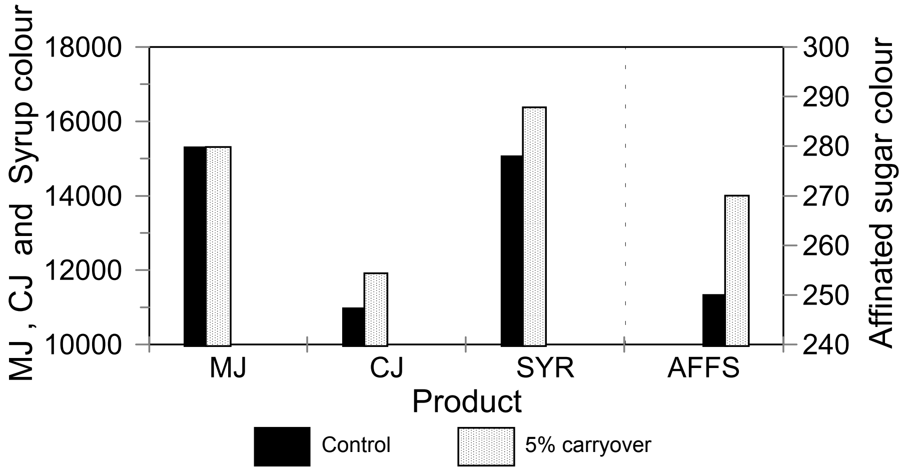

Figure 2A: Effect of mud carryover on colour.

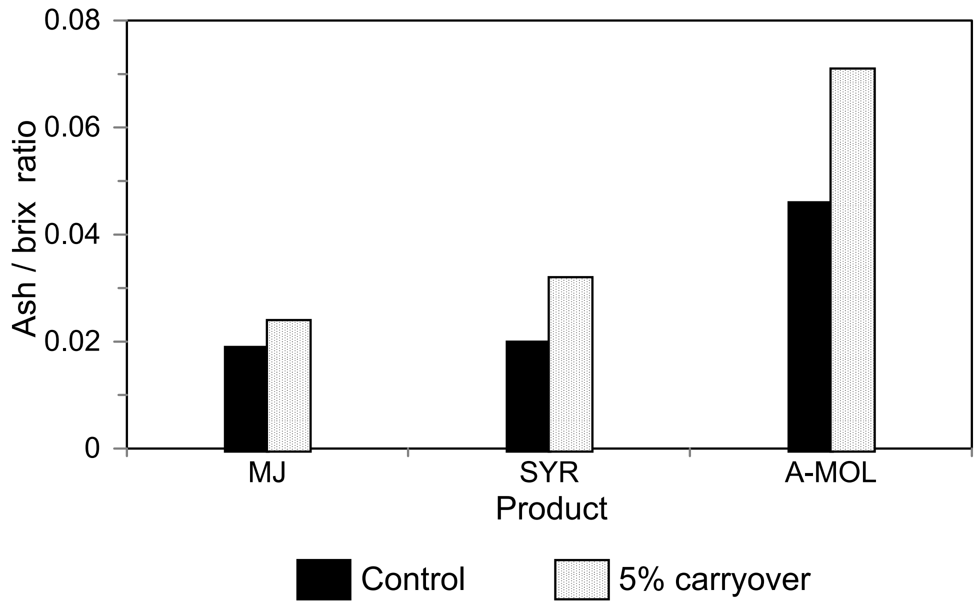

Figure 2B: Effect of mud carryover on S.ash.

Further effects of carryover on affinated sugar quality are shown in Table 5.

Table 5: Effect of mud carryover on affinated sugar quality

	Colour	P_2O_5	Ca^{2+}	K^+
		mg/kg brix		
Control	250	4	5	6
+ 5% carryover	270	47	35	8
% increase	8	1075	600	33

There are very marked increases in the Ca^{2+} and P_2O_5, levels, and a smaller change in the K^+ level. The AFFS colour is also higher.

Factory trial results

Milling and diffuser juices
Comparisons are shown in Tables 6 and 7 for MJ and CJ respectively.

Table 6: Comparison of milling and diffusion juices from GH

	MJ Mill	MJ Diffuser	%Difference (Diffuser-Mill)x100/ Mill
S.ash%	0.39	0.37	-5
K^+ (mg/kg brix)	9148	8757	-4
P_2O_5 (mg/kg brix)	1396	1677	+20
Ca^{2+} (mg/kg brix)	719	919	+28
Mg^{2+} (mg/kg brix)	1418	1012	-29
SiO_2 (mg/kg brix)	435	523	+20
Colour	19010	23016	+21
Turbidity	167351	41110	-75

Table 7: Comparison of clear juices when m-o-l and sacch are used on MJ from GH

	m-o-l		sacch	
	CJ(mill)	CJ(diffuser)	CJ(mill)	CJ(diffuser)
S.ash %	0.45	0.46	0.45	0.45
K^+ (mg/kg brix)	9369	8775	8929	8974
P_2O_5 (mg/kg brix)	434	484	335	327
Ca^{2+} (mg/kg brix)	1862	1892	1610	1669
Mg^{2+} (mg/kg brix)	1266	911	1122	901
SiO_2 (mg/kg brix)	359	413	288	357
Colour	19936	24606	20103	21332
Turbidity	35250	20474	22195	8568

The following observations may be made from the results.

- There are differences in the MJ quality between milling and diffusion. The most noticeable is the lower turbidity in diffusion juice.
- For m-o-l and sacch, diffuser CJ shows lower concentrations of Mg^{2+}, but higher colours and higher concentrations of SiO_2.
- Again, the use of sacch results in very much lower CJ turbidities.

The muds are compared in Table 8.

Table 8: Comparison of muds produced from milling and diffusion juices from GH

	m-o-l		sacch	
	Mill	Diffuser	Mill	Diffuser
Dry mass (g/kg juice)	3.5	0.8	3.7	1.0
P_2O_5 (mg/kg mud)	27383	75835	22118	54371
Ca^{2+} (mg/kg mud)	23687	109007	24766	80595

Diffusion juice clarified with m-o-l yields significantly lower mud quantities, about one quarter of that for milling juice.

Higher P_2O_5 and Ca^{2+} concentrations are obtained in mud from diffusion; but the total mass of P_2O_5 in the mud is only slightly lower with diffusion while the mass of Ca^{2+} is roughly the same as for milling.

Starch and gum levels in juice
MJ from the diffuser and the milling tandem at GH and the corresponding CJ were analysed for starch and gums.

The averaged results from five tests are shown in Table 9.

Table 9: Starch and gums in MJ and CJ from milling and diffusion juices from GH

	MJ		
	Mill	Diffuser	% difference
Starch (mg/kg brix)	1617	443	-73
Gums (mg/kg brix)	10385	5204	-50
	CJ		
Starch (mg/kg brix)	1321	411	
Gums (mg/kg brix)	3717	4523	
Starch (% change from MJ)	-18	-7	
Gums (% change from MJ)	-64	-13	

The amounts of starch and of gums are clearly higher in the mill juice compared to diffuser juice.

The change in the starch levels from MJ to CJ, i.e. across clarification, is small, but the level of starch in the diffuser juice is lower.

There is a reduction in gums across clarification for mill juice which results in the concentration in CJ being similar to that in the CJ from the diffuser. Indications are that diffusion removes only the type of polysaccharides that would otherwise be removed during clarification and that amount which is not removed by conventional clarification goes through with CJ.

Milk of lime versus saccharate
Laboratory clarification tests were done on factory juices to compare the effect of m-o-l and of sacch on the concentrations of monosaccharides and of ash in CJ. The average results from tests on MJ from SZ and MS show that there are no changes in the reducing sugar/ash ratio with sacch or with m-o-l. The findings from these tests are in good agreement with those from the laboratory trials on juice extracted at the SMRI. The most significant findings here concern colour and turbidities, as shown in Table 10. Clearly there are small changes in colour but with an indication that sacch gives lower values. The major improvement however is in CJ turbidity, with reductions of about 40% with sacch.

Results of the effect of m-o-l and sacch on selected species in MJ and CJ are also shown in Table 10.

Table 10: Effect of m-o-l and sacch on selected impurities in industrial juices.

Sample	P_2O_5	Ca^{2+}	K^+	Mg^{2+}	SiO_2	Colour	Turbidity
	mg/kg brix						
SZ							
MJ	1 572	572	10 838	1 147	671	17 105	26 052
CJ (m-o-l)	355	1 601	10 170	905	293	18 767	11 560
CJ (sacch)	149	1 390	9 750	893	246	17 330	4 246
MS							
MJ	1 651	1 468	12 986	1 416	836	20 840	34 707
CJ (m-o-l)	272	2 212	12 603	1 336	428	21 083	14 133
CJ(sacch)	192	1 967	12 160	1 311	421	19 758	5 858

The expected drop in the CJ phosphate levels and an increase in the calcium levels occurred for both liming methods. However, for sacch, the CJ had lower phosphate and calcium levels. K^+, Mg^{2+} and SiO_2 levels in CJ are similar for both liming methods.

Material balances

The results obtained with the factory juices were very similar to those from the SMRI juices. Thus the comments made for mass balance in the laboratory trials section apply here. The complete data are given in a technical report (Sahadeo, 2001). For the same final CJ pH, the actual amounts of Ca^{2+} from m-o-l or from sacch used as liming agent are similar. This is illustrated in Figure 3.

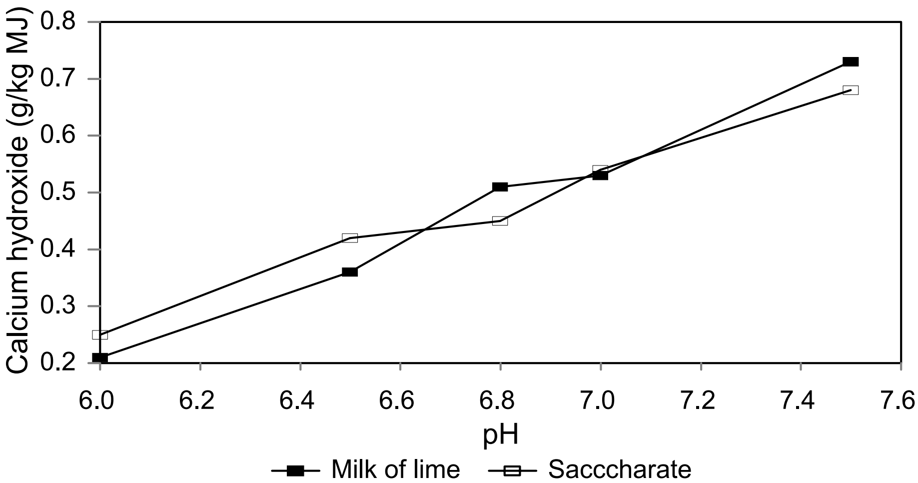

Figure 3: Mass of calcium hydroxide used for liming (g/kg MJ) as a function of CJ pH

Effect of evaporation on turbidity and selected impurities

The results for factory juices are similar to the those found for juice extracted at the SMRI with respect to CJ turbidity, ie. sacch yields lower turbidities. In addition, the effect of evaporation on P_2O_5 is shown in Figure 4. The drop in phosphate levels with evaporation indicates scaling, a result which is similar to that found with calcium in the juices extracted at the SMRI.

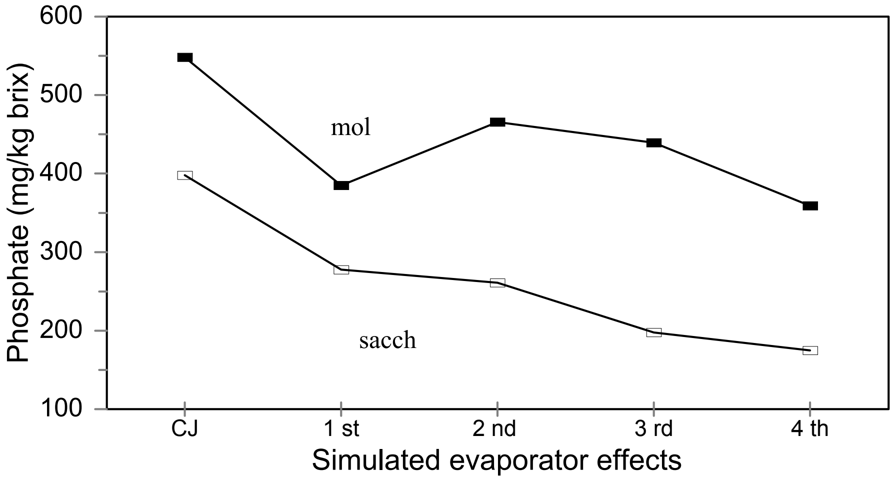

Figure 4:Phosphate contents, simulated evaporator effects

Conclusions

- MJ and CJ derived from clean cane and clean deteriorated cane had similar ash levels whereas the presence of trash and tops raised ash levels by about 50%.
- Comparing dry mud masses, clean cane produced about 90% less mud than cane with trash and tops.
- Hot or cold extractions to simulate milling and diffusion did not impact on the quality of CJ in terms of phosphate, calcium, magnesium, silica and sulphated ash.
- For all the tests done on juices extracted at the SMRI and for all factory juices, sacch liming gave a better quality CJ in terms of turbidity, colour, phosphate and calcium. The amounts of calcium used for both methods of liming, to the same CJ pH, were similar. The mud volumes from sacch liming were higher, however the dry matter content of muds from sacch liming was also higher, indicating the precipitation of more impurities. In view of these results, consideration should be given to the use of sacch liming at the factories.
- Material balances have shown large amounts of inorganic phosphate and of silica to be unaccounted for. It is of concern that phosphate, a fundamental species in clarification, cannot be analysed with the precision and accuracy required. Silica, which can cause serious scaling problems, suffers from the same limitations. It is recommended that the analysis of these two components be reviewed in detail to establish acceptable methods.
- Laboratory simulations of evaporation (four effects) of CJ to syrup show no evidence of turbidity returning in syrup when either sacch or m-o-l is used at the clarification stage. The syrup with sacch liming tests showed lower calcium and phosphate contents, possibly indicating more scale deposits in the evaporator. This would be advantageous in terms of sugar quality but at the expense of evaporator scaling.
- It is evident from pilot scale tests that carryover of muds results in an increase in colour in all the products, viz. MJ, CJ, syrup, affinated sugar and A-molasses. There is a significant increase in the calcium and phosphate levels in affinated sugar, but small changes in the potassium level. There is an increase in the ash levels of syrup and molasses as a result of carryover. Clearly mud carryover should be avoided in factories.

- The fundamental purpose of clarification is to produce sugar of the required quality, particularly colour and filterability. This work has not carried the investigation into sugar quality. However it is logical to assume that good clear juice quality in terms of phosphate, calcium, magnesium, silica and sulphated ash should impact positively on sugar quality. A next stage in this work would be to link sugar quality to clear juice quality.

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