

# ACETATE EXTRACTION IN A CANE DIFFUSER

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## Abstract

The effects of temperature, retention time, pH and different liming procedures on the extraction of acetate in a cane diffuser operating under normal production conditions have been studied. The lowest extraction of acetate was obtained when the diffuser was not limed. The quantity of acetate extracted was affected by the strength of lime used on the diffuser and was lower with more dilute lime. An appreciable increase in acetate in juice across clarification was found at a milling as well as at a diffusion factory.

## Introduction

In a comparative study of milling and diffusion, Ivin *et al*<sup>1</sup> found that under certain liming conditions on cane and bagasse diffusers, the level of acetate in the mixed juice was about ten times that when the diffuser was not limed. From the analysis of the mixed juice from some milling tandems and cane diffusers in South Africa, Schäffler *et al*<sup>3</sup> found that:

- The concentration of acetate in the mixed juices from different milling tandems was similar (about 300 ppm on brix).
- The concentrations of acetate in the mixed juice from the diffusers ranged from about 300 to 1 000 ppm on brix.

Relatively low acetate levels (about 300 ppm on brix) were found on the Maidstone and Amatikulu diffusers, while the levels on the Felixton diffusers were about 1 000 ppm on brix.

At Maidstone, single point liming into the stage 4 pump suction with 7 Bè lime, was used to control the pH over the diffuser at about 6,0 to 6,2 where the signal was obtained by measuring the pH at stage 1.

At Amatikulu, 4 Bè lime was added into the pump suction of stages 2, 8 and 14 to control the pH at about 6,5 to 7,0 at these stages while the pH at the unlimited stages was about 5,5.

At Felixton, 12 Bè lime was added into the pump suction of 8 stages. However, when no lime was added into the Felixton diffuser the concentration of acetate in the mixed juice fell to about 300 ppm, i.e. a level similar to that found in the mixed juice of the Maidstone and Amatikulu diffusers, and the Maidstone and Darnall milling trains.

The most damaging consequence of the extraction of acetic acid is the high level of corrosion of parts of the plant. Of greatest concern at Felixton has been the severe corrosion of parts of the pan calandrias and the cause has been traced to the relatively high concentrations of acetic acid in the V2 steam (Schäffler *et al*<sup>3</sup>).

The objective in the work described below was the establishment of the conditions which would permit liming of the Felixton diffusers in such a way that:

- Acetate extraction into the mixed juice would be minimised
- The benefits of liming, viz minimization of corrosion and of sucrose inversion, would be realised.

## Plant Layout

The extraction plant at Felixton consists of two similar cane diffusers. The knifed and shredded cane fed to each diffuser is distributed by a leveller and, although provision was made to vary the bed height, the usual practice is to operate at the minimum setting which gives a bed depth of about 1,5 metres.

The megasse from each diffuser is dewatered through two dewatering mills which operate in parallel. By running either two or one dewatering mill the diffuser is operated either at its nominal throughput rate of 300 tons cane per hour or at 150 tons cane per hour. The press water is returned directly to the diffuser in front of the second set of screws, i.e. stage 12.

The scalding juice temperature is maintained at above 90°C. Changes to the operating temperature of the diffuser are made by changing the set point of the controller which feeds steam (V2) directly under the cane bed into stages, 3, 5, 7, 11, 12 and 13.

The lime addition is via two controllers, each of which feeds lime to an equally partitioned four-way splitter box. The outlets of the one box are connected to the suction lines of the pumps for stages 1, 2, 3 and 4. The pH signal for that controller is the pH of the stage 2 juice measured after the pump. Similarly, the other controller feeds lime to stages 6, 8, 10 and 12 with the pH control signal being taken from stage 8. Figure 1 shows the diffuser layout.

The level of acetate in cane juice was obtained by analysing daily composite samples of DAC extract taken over 24-hour periods for one week in November 1988. Each composite was made up from 24 individual DAC extract samples. The data are presented in Table 1.

The analyses of the weekly composite samples of mixed juice taken over a period when the diffusers were operating under ostensibly identical conditions are given in Table 2.

The data allows a rough estimation to be made of the level of significant difference between mixed juice analyses. This is useful for the interpretation of the data below, since, in the tests, one diffuser was operated as the reference (usually no lime and operating temperature of 85°C) while the variable under consideration was changed on the other.

The variables investigated were:

- Lime strength
- Number of lime addition points
- Operating temperature
- Throughput rates (fibre retention times).

For the tests, which lasted about 8 hours, the diffusers were operated under the test conditions for a period of 12 hours prior to the test being performed.

To investigate the effect of lime strength, lime of 12 and 4 Bè was used. The results, given in Table 3, are for weekly runs.

In the tests, where the number of lime additions points was varied, lime of different strengths was inadvertently used. With one point liming (Table 4.1) into either stage 2 or 3 the lime strength was about 2–3 Bè.

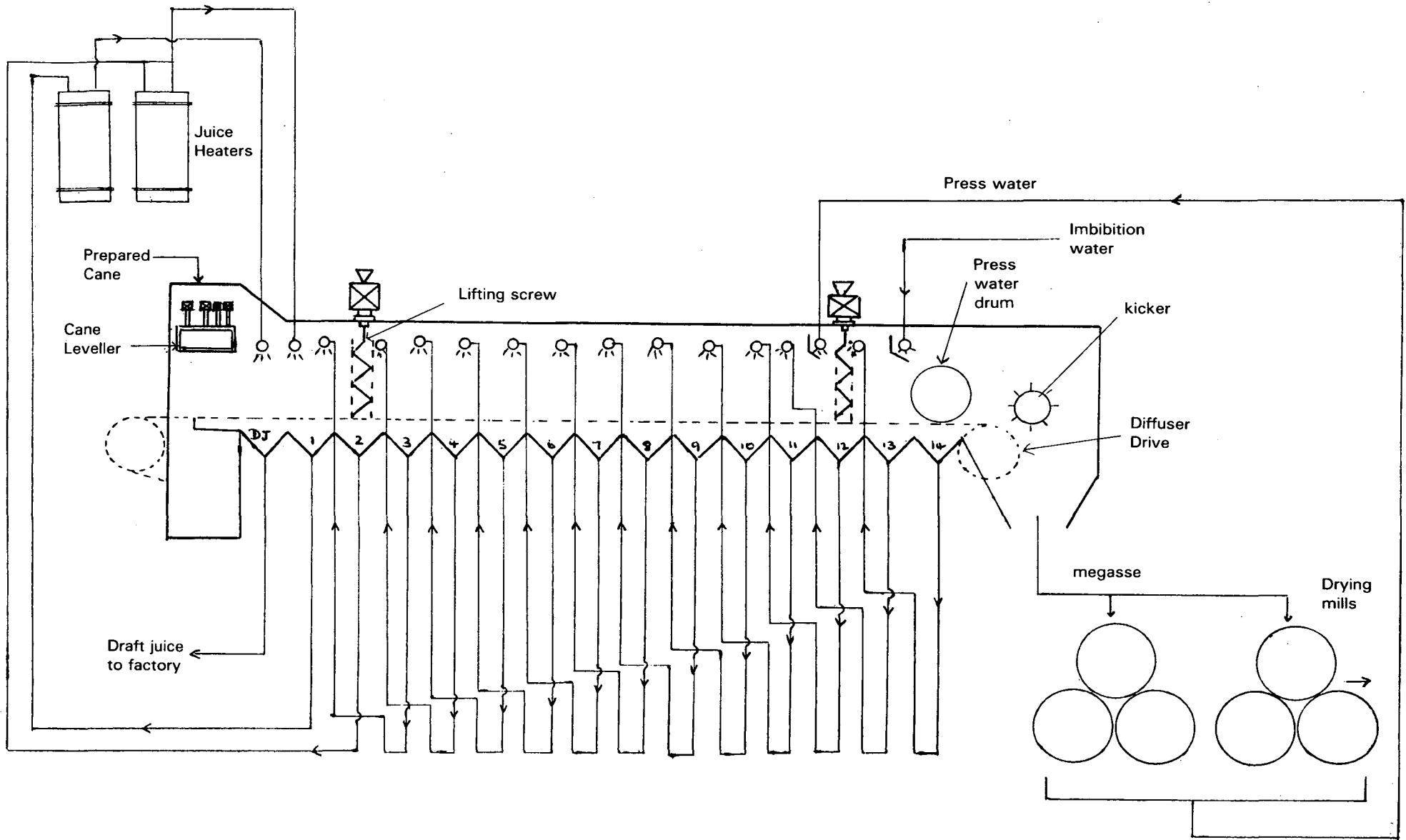


FIGURE 1

With the two point liming (Table 4.2), 4 Bè lime was added to stages 1 and 2. With eight point liming (Table 4.3), 1.0-1.5 Bè lime was added to stages 1, 2, 3, 4, 6, 8, 10 and 12.

The effect of temperature (Table 5) was measured under conditions of no liming.

The effect of fibre retention time (Table 6) was measured under both liming and no liming conditions.

Although not part of the main investigation it was found that the clarification process was responsible for a substantial increase in acetate in the juice being processed. The data in Table 7 give the analysis of daily composite samples of mixed and clarified juices from Darnall (a milling factory), Amatikulu (a diffusion factory) and Felixton.

Reliable information which could be used to quantify rates of corrosion in a cane diffuser is not available. An estimate of the rates of corrosion of mild steel parts of the diffuser, under liming to about 6,0 and no liming, was obtained under non-ideal test procedures using home-made mild steel corrosion coupons over a two week period. The juice corrosion rates were measured using coupons installed in the juice headers of stages 1 and 14, while vapour corrosion rates were obtained from coupons suspended from the inside of the diffuser roof above stages 5 and 12.

**Table 1**  
Acetic acid concentration in cane juice

DAC Extract for:	25-11	26-11	27-11	28-11	29-11
Acetic Acid: - ppm on Brix	230	210	260	310	280

**Table 2**  
Comparative mixed juice analyses under similar operating conditions

Week ending	Diffuser	Acetic acid ppm on		Brix of Mixed Juice	Acetic acid percentage difference
		Brix	Sample		
04.06.88	A	250	29	11,60	5
	B	237	28	11,79	
11.06.88	A	232	26	11,20	13
	B	206	23	11,16	
18.06.88	A	245	28	11,41	16
	B	211	25	11,83	
25.06.88	A	210	25	11,88	13
	B	237	28	11,80	
02.07.88	A	210	23	10,94	7
	B	197	23	11,69	
09.07.88	A	244	28	11,46	18
	B	207	24	11,62	
16.07.88	A	228	27	11,83	8
	B	247	28	11,34	
24.07.88	A	270	32	11,85	16
	B	232	29	12,45	
01.10.88	A	179	21	11,73	33
	B	238	29	12,20	
08.10.88	A	264	30	11,38	15
	B	230	28	12,18	
15.10.88	A	325	34	10,47	41
	B	457	52	11,37	
22.10.88	A	410	43	10,49	10
	B	374	41	10,97	
29.10.88	A	231	26	11,24	36
	B	314	37	11,70	
05.11.88	A	240	28	11,65	13
	B	271	30	11,06	
12.11.88	A	225	25	11,18	48
	B	332	38	11,44	
19.11.88	A	250	26	10,41	8
	B	270	31	11,49	

**Results and Discussion**

**Acetate in cane.** The concentrations of acetate given in all the tables have been expressed in terms of the free acid. The level in cane (Table 1) appears to be variable between about 200 and 300 ppm on brix, but values outside this range are likely to be found.

**Level of significant differences.** The percentage difference between the acetate levels in the mixed juices from the two diffusers when operating under similar conditions was usually less than 20%, but a few differences were substantially higher. The overall average difference was ca 19%. Differences under 25%, where the average of three or more tests have been compared, are unlikely to be significant.

**Lime strength.** It is clear that the effect of lime strength between 12 and 4 Bè is highly significant with the lower lime strength resulting in much lower levels of acetate extraction.

**Table 3**  
Effect of lime strength on acetate extraction

Sample for week ending	Diffuser	Type of sample	Temp °C	Lime strength Bè	Number of addition points	Acetic acid ppm on brix
05.12.87	A	M J	85	12	8	960
10.01.88	A	M J	85	4	8	300
	B	M J	85	Nil	Nil	270
30.04.88	A	M J	85	12	8	1 379
	B	M J	80	12	8	1 001
20.08.88	A	M J	85	Nil	Nil	281
	B	M J	85	4	8	427
27.08.88	A	M J	85	Nil	Nil	290
	B	M J	85	4	8	450

**Table 4.1**  
One point liming

Test No.	Date	Diff	Temp °C	Lime strength Bè	pH Average across diffuser	Number of addition points	Mixed jce acetic acid ppm on Brix	% Diff
1	28.07-29.06 1988	A	84	Nil	5,10	Nil	128	380
		B	85	2,0	6,20	1 (Stage 3 pH 8,2)	617	
2	21.07-22.07. 1988	A	85	Nil	5,15	Nil	178	60
		B	83	3,0	6,00	1 (Stage 2 pH 7,3)	280	
3	26.07-27.07. 1988	A	84	Nil	5,13	Nil	260	160
		B	84	2,8	6,13	1 (Stage 3 pH 8,2)	677 0	
4	26.07-27.07. 1988	A	83	Nil	5,18	Nil	338	110
		B	85	2,8	6,20	1 (Stage 3 pH 8,1)	707	
5	02.08.1988	A	85	Nil	6,03	1 (Stage 3 pH 7,8)	663	110
		B	85	3,2	5,00	Nil	320	

Some of the tests carried out subsequently, when the lime strength was as low as 1 Bè, did not reveal a further dramatic benefit with the even more dilute lime slurry, though there may have been a further marginal benefit.

**Number of lime addition points.** In these and subsequent tests the lime strength was always maintained at 4 Bè or less. When all the lime was added at one point and the average pH in the diffuser was held at 6,0–6,2, the concentration of acetate in the mixed juice was usually about double that in the reference mixed juice sample.

**Table 4.2**  
Two point liming

Test No.	Date	Diff	Temp °C	Lime strength Bè	pH Average across diffuser	Number of addition points	Mixed jce acetic acid ppm on Brix	% Diff
1	21.08.1988	A	85	4*	5,5	2 (Stage 1 and 2 pH 6,5)	340	60
		B	85	Nil	5,10		210	

**Table 4.3**  
Eight point liming

Test No.	Date	Diff	Temp °C	Lime strength Bè	pH Average across diffuser	Number of addition points	Mixed jce acetic acid ppm on Brix	% Diff
1	10.08–11.08.1988	A	85	Nil	5,12	6 (2 Stages blocked)	194	120
		B	84	1,5	6,13		427	
2	11.08–12.08.1988	A	85	Nil	5,17	Nil	159	60
		B	75	1,6	6,04	9	257	
3	16.08.1988	A	85	Nil	5,03	8	284	50
		B	75	0,9	6,03		423	
4	17.08.1988	A	84	Nil	5,09	8	168	125
		B	84	1,0	6,02		379	

**Table 5**  
Effect of temperature on acetate extraction

Test No.	Date	Diffuser	Operating temp °C	Acetic acid ppm on brix	% Diff
1	17.05.1988	A	82	271	74
		B	74	471	
2	18.05.1988	A	83	202	16
		B	75	235	
3	20.05.1988	A	83	291	1
4	24.05.1988	A	85	246	55
		B	74	381	
5	25.05.1988	A	90	290	10
		B	75	318	

With the two point lime addition the average pH in the diffuser was about 5,5. This was too low to permit meaningful comparison with the one or eight point lime addition tests. When 8 point liming was applied the average pH in the diffuser was just above 6,0, and the extraction of acetage showed some of the lowest levels obtained under liming conditions. However, the acetate concentration in the mixed juice was always higher than when no lime was added.

**Temperature.** All the tests were carried out without lime addition. In three of the test results the acetate concentrations were not significantly different, but in the other two the higher acetate concentration was associated with the lower temperature. Normal reaction kinetics would predict the opposite.

**Table 6**  
Effect of cane throughput rate on acetate extraction

Test No.	Date	Diff	Temp °C	Lime strength Bè	Number of addition points	Through-put T.C.H.	Mixed jce acetic acid ppm on Brix	% Diff
1	01.06.1988	A	84	Nil	-	256	225	2
		B	85	Nil	-	191	230	
2	02.06.1988	A	83	Nil	-	176	288	26
		B	85	Nil	-	271	228	
3	03.08.1988	A	85	2.8	Stage 3 (pH 7,6)	301	443	93
		B	85	2.8	Stage 3 (pH 7,9)	171	857	

**Table 7**  
Acetate in mixed and clarified juices

Date		Acetic acid ppm on	Brix of mixed juice	% increse M.J. to C.J.
		Brix		
19.07.88	Mixed juice	148	12,19	49
	Clear juice	220	11,82	
20.07.88	Mixed juice	157	12,10	75
	Clear juice	274	11,68	
21.07.88	Mixed juice	176	12,48	56
	Clear juice	274	12,03	
22.07.88	Mixed juice	152	12,53	
	Clear juice	250	12,02	
19.07.88	Mixed juice	268	12,30	63
	Clear juice	437	12,35	
20.07.88	Mixed juice	262	11,45	53
	Clear juice	400	10,98	
21.07.88	Mixed juice	211	12,78	45
	Clear juice	305	12,46	
18.05.88	Mixed juice	252	10,75	44
	Clear juice	362	10,51	
24.05.88	Mixed juice	336	11,60	32
	Clear juice	443	11,52	
25.05.88	Mixed juice	299	11,03	49
	Clear juice	444	11,03	
22.07.88	Mixed juice	271	12,16	50
	Clear juice	405	12,09	

**Fibre retention time.** Under the no liming conditions, the acetate concentrations in the juice from both diffusers were similar. The difference observed when both diffusers were being limed may be associated with a higher base rate of acetate formation, and is in broad agreement with model predictions from laboratory scale studies by Schäffler<sup>2</sup>.

**Mixed juice to clarified juice.** The data given in Table 7 showed an increase in acetate concentration from mixed juice to clarified juice of some 50% at three factories. This can be expected to contribute significantly to the vapour corrosion problem.

**Corrosion rates.** The benefit of liming is seen in the lower rates of corrosion of the coupons in the juice, but corrosion in the vapour phase appears to be very similar whether liming is practised or not.

**Table 8**  
Corrosion rates in diffusers

Diffuser operating conditions:		Location of coupons	Corrosion rate mpy
Temperature	Liming		
85	Yes	Juice header stage 1	4
85	Yes	Juice header stage 14	11
75	No	Juice header stage 1	17
75	No	Juice header stage 14	21
85	Yes	Vapour phase stage 1	34
85	Yes	Vapour phase stage 12	37
75	No	Vapour phase stage 5	42
75	No	Vapour phase stage 12	30

### General

An added benefit of using the reduced strength lime was a reduction in the total quantity of lime used.

Total lime consumption (12 Bè addition) = 1,20 kg/ton cane  
 Total lime consumption ( 4 Bè addition) = 0,70 kg/ton cane  
 Cost saving p.a. = R290 000

### Conclusions

The use of 4 Bè lime slurry instead of 12 Bè to lime a cane diffuser resulted in a substantial reduction in the quantity of acetate extracted. A further marginal reduction may be obtained if an even more dilute lime slurry is used. The quantity of lime used in the diffusion and clarification processes together was lower when more dilute lime was used.

Adding the lime to more than one juice stage appeared to give a marginal reduction in the amount of acetate extracted.

A doubling of the fibre retention did appear to have a significant effect on the quantity of acetate extracted when lime was added.

The effect of temperature varied between zero and an unexpectedly large increase in acetate extraction at the lower temperature.

A substantial increase in acetate across clarification was found and the effect was confirmed at one other diffusion factory as well as at a milling factory.

Liming a diffuser to about pH 6,0 reduced the corrosion rate of the mild steel in contact with the juice, but had no effect on the corrosion rate of mild steel in the vapour space.

### Future Work

Conduct tests to quantify the effect of the diffuser operating pH on corrosion rates. Using this information together with sucrose inversion rates calculated from the relationships derived by Schäffler (personal communication), would indicate the ideal minimum pH to be used.

Determine the acetate extraction levels at this ideal pH using the best liming conditions.

Study the extraction/formation of acetate in clarification so that steps can be taken to minimise it.

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