CLARIFICATION TRIALS AT DARNALL

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Abstract

Results of comparison trials indicated the superiority of enzymatic hydrolysis in the syrup stage, following simple defecation, over (1) vacuum clarification, (2) the 50-50 split stream process, (3) the 90-10 split stream process, as a means of producing low starch raw sugars cheaply. The defecation plus enzyme process is easier to control and appears to produce sugars of lower colour. The ash content of split stream sugars was disappointing.

Introduction

The cheap and efficient reduction in the starch content of juices has become one of the prime objectives of the South African Sugar Milling Industry. The introduction of the Rabe vacuum flotation process in 1967 provided not only an economic method for starch reduction, but also an incentive for further research and a basis for evaluating other processes.

Promising results of laboratory and pilot plant trials prompted a plant comparison, both as regards cost and efficiency, between vacuum flotation and starch reducing processes involving thermostable enzymes[1].

A child of these innovations, the Tongaat split stream process, was first discussed at the 1968 SASTA congress[1]. It was hoped that this process would provide a practical means of simultaneously reducing both the starch and the ash contents of raw sugars.

Darnall mill was chosen as a suitable venue for comparisons between the Rabe and enzyme processes. At the request of the Tongaat Sugar Company, it was agreed that the Split Stream Process would also be tested. Because of the rather extensive plant changes required, the trial run of the Tongaat Process was delayed for some weeks. During this time a variation, incorporating vacuum flotation of about 90% of the juice followed by back titration with the remaining 10% was developed in the laboratory and run for two weeks on a full plant scale. The variation, termed the 90% Split Stream process for purposes of this paper, was improved by retaining the smaller stream in a hold up tank to effect starch hydrolysis by natural enzymes.

In the comparison trials the processes were operated at weekly intervals in the following order:

Week	*Week Ending*	*Process*
1	3 November, 1968	Rabe Vacuum Flotation
2	10 November, 1968	Defecation + enzyme
3	24 November, 1968	90% Split Stream
4	1 December, 1968	Defecation + enzyme
5	8 December, 1968	Tongaat Split Stream

Throughout the comparisons, process details were recorded by means of the usual factory data sheets, while personal observations were noted on the ease of operation, and on side effects. Sugar samples, collected every shift, were analysed by Hulett's Central Laboratory.

Process Details

(a) Rabe Vacuum Flotation (Fig. 1)

Mixed juice, heated to 60°C, was limed to pH 8.8. Mono Calcium Phosphate was added to a pH of 8.2 and the floc stabilised by the addition of a coagulant. The mud and juice were separated by flotation under vacuum. The clear juice was boiled and passed through the subsiders where a small quantity of secondary precipitate was removed. The flotation mud was mixed with the sediment from the subsiders before filtration and the filtrate returned to the mixed juice stream.

(b) Defecation plus Enzyme (Fig. 2)

Mixed juice was clarified by simple defecation and a thermostable bacterial α -amylase added to the syrup in the feed line a short distance before the entry to the penultimate evaporator vessel. At this point the temperature of the syrup (75 to 80°C) was most suitable for enzymatic starch hydrolysis[1].

(c) 90% Split Stream (Fig. 3)

After heating to 60°C the mixed juice was divided into two streams:

1. The major stream, representing 85-90% of the mixed juice, was limed to pH 9.0. At this pH the superior activity of the coagulant, then added, allowed separation of mud and juice by vacuum flotation without the use of added phosphates. The clear juice from the vacuum clarifier was boiled and run to a flash tank.
2. The minor stream, 10 to 15% of the mixed juice, was heated to about 75°C and held in a retention tank for some ten minutes to allow the natural amylase in the juice to hydrolyse starch. The juice was then run into

the top of the flash tank where it was heated to near boiling by the flash from the major stream. The two streams were then mixed. The flash tank heating system was resorted to in the absence of a suitable juice heater.

Phosphoric acid was added at a constant rate to the combined streams and the pH of the phosphated juice controlled by automatic alteration of the proportioning of juice between major and minor streams.

The phosphated juice was passed through the subsiders and the mud from the subsiders filtered separately from the mud from the vacuum clarifiers. This was done to prevent re-solution of starch in vacuum clarifier muds by the large volume of higher temperature subsider muds. Filtrates were mixed and added to the mixed juice.

(d) Tongaat Split Stream (Fig. 4)

After heating to 60°C the mixed juice was split into two equal streams:

1. The juice of one stream was limed to pH 9.8 and passed through the vacuum clarifier following the addition of a coagulant.
2. The juice from the second stream was heated directly with steam to 75°C and held in a retention tank for ten minutes to allow for starch hydrolysis by natural enzymes.

The clear juice from the flotation stream and the whole natural enzyme stream were mixed, heated to boiling and fed to the subsiders.

The muds from the subsiders and from vacuum flotation were filtered separately and the mixed filtrates returned to the mixed juice.

It had been calculated and experienced during development at Tongaat that the combined juices fed to the subsiders would be about pH 8.2. In practice a pH of about 8.6 was obtained which resulted in a clear juice pH of 7.0 to 7.2.

Operating costs

Table I summarises the operating chemical costs of the various processes compared. All costs are given in cents per ton of sugar made.

Table II summarises the usage of the various chemicals, based on mixed juice.

TABLE I
Operating Chemical Costs, cents per ton sugar

Week Clarification	1 Rabe	2 Defecation +enzyme	3 90% S/S	4 Defecation +enzyme	5 Tongaat S/S
Lime	6	5	5	5	6
Phosphate	17	—	4	—	—
Coagulant	13	—	14	—	13
Enzyme	—	21	—	11	—
Total Cost	36	26	23	16	19

TABLE II
Usage of Operating Chemicals Based on Mixed Juice

Week	1	2	3	4	5
Lime lbs. per ton	1.14	1.06	1.03	1.06	1.11
MCP ppm	188	—	—	—	—
Phosphoric acid ppm	—	—	30	—	—
Coagulant ppm	6.8	—	7.4	—	6.9
Enzyme ppm	—	6.5	—	3.4	—

Analysis of sugar samples

A total of 90 raw sugar samples was sent to the Central Laboratory. After pruning the first sugars from each week to ensure that the samples were representative of the clarification method used, the sugars were analysed for starch, gums, available phosphate, wax, sulphated ash, calcium, magnesium, silica and chloride. The analytical results were averaged, and the arithmetic mean and standard deviations for each constituent are summarised in Table III.

TABLE III
Analysis of Individual Raw Sugar Samples—x=mean value s=standard deviation

Week Clarification		1 Rabe	2 Defecation +enzyme	3 90% S/S	4 Defecation +enzyme	5 Tongaat S/S
No. of samples		17	16	17	16	14
Starch	x	209	86	213	141	238
ppm	s	27	13	32	17	52
Gums	x	1769	1309	1648	1528	1741
ppm	s	332	135	160	165	424
Phosphate	x	70	101	69	97	83
ppm	s	7	11	10	9	15
Wax	x	238	385	171	96	293
ppm	s	39	83	60	31	111
Ash	x	0.568	0.551	0.566	0.644	0.469
%	s	0.054	0.095	0.072	0.072	0.112
Ca^{++}	x	242	233	182	197	170
ppm	s	19	41	16	20	51
Mg^{++}	x	211	156	163	178	124
ppm	s	22	25	21	12	50
Silica	x	159	212	246	218	220
ppm	s	59	120	32	83	94
Cl—	x	1084	853	1012	1239	664
ppm	s	238	130	106	227	241
Starch free gums ppm	x	1560	1223	1435	1387	1503

Weekly composites of the raw sugars were prepared and analysed for pol, moisture, reducing sugars, colour, sulphated ash and insolubles. These data are reported in Table IV, which also includes figures for reducing sugars expressed as a percentage of non-pol solids, and for "reduced colour", a contrived figure based on non-pol solids which is quoted for comparison purposes only.

Because of the differences in the pol and moisture contents of the sugars from different weeks, data pertaining to raw sugar samples have been related to non-pol solids. In Table V, the arithmetic means of the individual raw sugar analyses have been expressed as a percentage of the non-pol solids in the composites for the corresponding week.

The weekly composites were affinated and the air-dried affinated sugars analysed for the same constituents as the individual raw sugars, with additional analyses for colour and filterability. These data appear in Table VI.

Discussion of analytical results

If differences in the sugar analyses are to be used for comparing the clarification processes it is essential to be critical about the results obtained. Variations due to causes other than the weekly process changes must be uncovered and discounted.

In Table VII standard deviations of the analytical results for the individual raw sugars have been expressed as a percentage of the arithmetic mean.

TABLE IV
Analysis of Composite Raw Sugars

Week Clarification	1 Rabe	2 Defecation +enzyme	3 90% S/S	4 Defecation +enzyme	5 Tongaat S/S
Pol °S	97.80	97.75	97.65	98.01	98.35
Moisture %	0.46	0.48	0.81	0.36	0.32
Sulphated Ash %	0.56	0.54	0.57	0.64	0.49
Reducing Sugars %	0.44	0.51	0.47	0.46	0.32
Insolubles %	0.05	0.03	0.06	0.06	0.06
Colour a*c 560 mμ	1.17	0.91	1.25	0.99	0.92
Non Pol Solids %	1.74	1.77	1.54	1.63	1.33
R.S. % Non Pol Solids	25.29	28.82	30.52	28.22	24.06
Reduced Colour	67	51	81	61	69

TABLE V
Analysis of Individual Raw Sugar Samples. Results expressed as percentage of non-pol solids

Week Clarification	1 Rabe	2 Defecation +Enzyme	3 90% S/S	4 Defecation +enzyme	5 Tongaat S/S
Starch	1.20	0.48	1.38	0.92	1.79
Gums	10.17	7.40	10.70	9.92	13.09
Starch free gums	8.97	6.92	9.32	9.00	11.30
Phosphate	0.40	0.57	0.45	0.63	0.62
Wax	1.37	2.17	1.11	0.62	2.20
Ash	32.64	31.13	36.75	41.82	35.26
Ca^{++}	1.39	1.32	1.18	1.28	1.28
Mg^{++}	1.21	0.88	1.06	1.16	0.93
Silica	0.91	1.20	1.60	1.42	1.65
Cl—	6.23	4.82	6.57	8.04	4.99

TABLE VI
Analysis of Affinated Composite Samples

Week Clarification	1 Rabe	2 Defecation +enzyme	3 90% S/S	4 Defecation +enzyme	5 Tongaat S/S
Starch ppm	120	50	120	60	150
Gums ppm	750	610	840	670	1090
Phosphate ppm	10	12	35	49	61
Wax ppm	109	109	120	104	194
Ash %	0.08	0.11	0.08	0.08	0.14
Ca^{++} ppm	80	70	55	55	85
Mg^{++} ppm	15	15	10	10	20
Silica ppm	80	62	38	90	160
Cl— ppm	62	51	38	40	60
Starch free gums ppm	630	560	720	610	940
Filterability %	42	47	43	48	24
Colour a*c 560 mμ	0.26	0.18	0.21	0.18	0.31

TABLE VII
Statistical Data for Individual Raw Sugars
Standard deviation as percentage of arithmetic mean

Week Clarification	1 Rabe	2 Defecation +enzyme	3 90% S/S	4 Defecation +enzyme	5 Tongaat S/S
No. of Samples	17	16	17	16	14
Starch	13	15	15	12	22
Gums	19	10	10	11	24
Phosphate	10	11	15	9	18
Wax	16	22	35	32	38
Ash	10	17	13	11	24
Ca^{++}	8	18	9	10	30
Mg^{++}	10	16	13	7	40
Silica	37	57	13	38	43
Cl—	22	15	10	18	36

In the case of silica and wax, the standard deviation of the results is equivalent to more than 20% of the mean for four out of five weeks. It would appear that the wax and silica contents of Darnall sugars vary considerably from shift to shift, and as a result these constituents have not been considered in making comparisons between the clarification processes.

With all other constituents the spread of results was within acceptable limits for the first four weeks. During week five, the Tongaat split stream, the significant spread of results for all constituents may be attributed to the considerable difficulties experienced in controlling the process. As will be seen later these difficulties led to the early abandonment of the Tongaat process trial.

Methods of analysis

Calcium and Magnesium were determined by atomic absorption spectrophotometry.

Colour was measured by ICUMSA method 3[3].

A Potentiometric method with silver nitrate was used for the determination of chloride[2].

Reference to all other analytical methods can be found through Jennings and Van Keppel (1966) Proc. S. Afr. Sug. Technol. Ass. 40 (1966) 196-198[4].

Discussion

Although the accent has been on the production of a low starch sugar, other factors, including cost, ease of operation and overall sugar quality must be considered when comparing the clarification methods.

Comparisons of unit operations in sugar factories suffer because of variations in raw material quality. Averaging on a weekly basis goes some way towards overcoming this bogey, but it can be seen from Table VIII that the starch content of mixed juice varied considerably over the five weeks of the exercise, although other aspects of juice quality remained fairly constant.

Unfortunately no data are available on the quantity of dissolved starch in the mixed juice. In vacuum flotation, by which only starch not in solution may be removed to any extent, the amount of starch available for inclusion in sugar is dependent on the quantity and nature of starch in mixed juice. It has been shown[5] that 25% of the starch in mixed juice may be in solution, in which case the efficiency of removal in vacuum clarification may drop to below 70%. In the split stream processes, which rely on natural enzyme hydrolysis to reduce the starch content of part of the juice, overall removal may be even lower, for it is doubtful whether the efficiency of the natural enzyme process exceeds 70% even in ideal circumstances.

Because vacuum flotation is a physical separation of undissolved starch from mixed juice, the process cannot be controlled to yield a desired level of starch in sugar. If a factory using vacuum flotation was to produce sugar with 40 ppm starch at a cost of 45 cents per ton, there would be no means of reducing the cost at the expense of an acceptable higher starch content. Split stream processes also suffer from this lack of adaptability, though some control may be possible through variation of the juice split.

TABLE VIII
Juice Quality

Week Clarification	1 Rabe	2 Defecation +enzyme	3 90% S/S	4 Defecation +enzyme	5 Tongaat S/S
Brix % mixed juice	11.72	12.04	11.99	11.99	11.53
Mixed juice purity	83.98	83.32	83.45	83.55	83.17
P_2O_5 in mixed juice (ppm)	196	191	190	191	185
Starch (ppm on brix)					
in mixed juice	1845	1559	1779	2042	2227
in clear juice	385	1115	585	1452	844

In the trials at Darnall, raw sugars produced using vacuum flotation and split stream processes averaged more than 200 ppm starch. This was reduced to below 150 ppm using enzymes, despite an increase in the starch content of mixed juice during week four.

With experience, a factory manager using the enzyme process may be able to control the starch level of raw sugar by adjusting enzyme dosage according to the starch content of mixed juice. It may even prove possible to devise a system of automatic control for the operation. The economic advantages of such a system would be considerable.

An unexpected benefit of the enzyme process as applied at Darnall was the substantial removal of starch by the simple defecation process. This removal, amounting to 29% of the starch in mixed juice, is in conflict with earlier observations on simple defecation, but has been measured at other Hulett mills, notably Amatikulu. The advantages of applying enzymes to clear juice with 1450 ppm starch rather than to mixed juice with 2040 ppm starch are obvious.

Costs and operation

It is clear from Table I that the enzyme plus defecation process is not only the most efficient means tested for the removal of starch, but also the cheapest.

It will also be apparent, from Table II that the usage of coagulant for the Tongaat Split Stream process was no less than for Rabe flotation, despite the fact that only half the juice was passed through the vacuum clarifier. The excessive usage of coagulant is unexplained, and is contrary to observations on a laboratory and pilot plant scale. It is possible that some external influence caused the excessive usage, in which case the costs of the Tongaat process could be reduced considerably.

A pH of 9.8 to 10 for the flotation stream had been recommended if the maximum benefits were to be derived from the Tongaat process. There were two major problems with this system. The first was caused by the buffering action of the juice in this range which made control of the quantity of lime added very difficult. The second problem concerned the pH of the combined streams fed to the subsiders. Instead of the 8.2 expected, the pH was in the region of 8.6, yielding syrup of pH 7.1. The addition of a small quantity of phosphoric acid to the combined streams to adjust the final pH would overcome the second problem but would increase the costs.

The major advantage of the 90% Split Stream process over the Rabe Vacuum Flotation is the reduction in the usage of phosphate. Phosphoric acid was used as a means of controlling final pH and improving clarification in the subsiders. During the operation of the 90% Split Stream it was found that starch removal by the vacuum flotation was very poor, falling below 70% on occasions.

The Rabe flotation and both Split Stream processes are extremely sensitive to pH changes and require considerably more attention than simple defecation. To the drawbacks mentioned must be added the fact that, for unexplained reasons, some refractory juices perform very badly in the vacuum clarifier, necessitating the dosage of high concentrations of coagulant to maintain a satisfactory raw sugar quality. During the running of the Tongaat Split Stream process, conditions deteriorated to such an extent that the trial had to be abandoned a day ahead of schedule.

Although simple defecation is also affected by juice quality, the staff of defecation factories have experience in handling refractory juices and take action quickly and without great cost. In addition it can be said that the enzyme process as applied in the trials at Darnall is not influenced by mixed juice quality and is not dependent on the physical nature of the starch in mixed juice.

Overall sugar quality

Among the advantages claimed for the Tongaat process were a decrease in ash content of 5 to 15%, and a decrease in silica, calcium salts and magnesia[1].

There is, however, little evidence of improvement in the ash content of Split Stream sugars, indeed the crystal ash content of Tongaat process sugar is the highest of the five compared. The Rabe process appears to offer the best removal of ash, though the evidence from the two weeks of simple defecation is conflicting.

Little can be said of the Calcium and Magnesium content of the sugars. These constituents are likely to follow the trends of the total ash.

There is a definite indication that defecation sugars contain more phosphates than sugars produced by processes including vacuum flotation.

It would appear that the application of enzymes to hydrolyse starch provides a side benefit in the hydrolysis of other polysaccharides. Starch free gums in both raw and affinated enzyme sugars tend to be lower than for sugars produced using flotation. The lowering of the polysaccharide content is reflected also in the superior filterability of the enzyme sugars. The possibility of additional benefits to process in the reduction of viscosity and improved molasses exhaustion has still to be investigated.

Finally, the colours of both raw and affinated sugars produced using defecation plus enzymes are lower, possibly because of a lower pH during clarification.

Note: The differences in pol of the sugars from different weeks should be remembered when making comparisons. For this reason Table V, which relates impurities to non-pol solids is a more useful guide than Table III.

Conclusions

In the absence of tangible side benefits such as a reduction in ash content of raw sugar, the choice of a starch-reducing clarification process depends upon cost, and ease of operation.

The trials at Darnall have not indicated any important side benefits from processes incorporating flotation.

Defecation followed by the addition of enzyme to syrup presents an attractively economical system for reducing starch levels efficiently. The process is far easier to control than vacuum flotation, which appears in turn easier to control than either Split Stream process. The enzyme process has additional advantages in that the level of starch in sugar may be controlled by varying the enzyme dosage, and that the process is not influenced by normal fluctuations in juice quality.

Chemical costs of the split stream processes were considerably lower than for vacuum flotation. The consumption of coagulant in the Tongaat process could probably be reduced, but the decrease in costs would not be sufficient to offer a realistic challenge to simple defecation plus enzymes.

It can be said with confidence that the enzyme process represents the most efficient, economical and controllable method for overcoming the starch problem in Natal raw sugars. Attention to the simple defecation process may go some way towards improving other quality aspects.

Acknowledgements

This exercise is an example of what can be achieved by close co-operation and interchange of ideas between factory and research personnel of different milling companies.

The help of all the many persons involved is gratefully acknowledged, with special thanks to Glen Carter of Tongaat, David Tayfield of Darnall, Arthur Dixon and Marion Laughton of Hulett's Central Laboratory for their several contributions.

The Directors of Tongaat Sugar Company are thanked for the permission to publish details of the twin stream process and for providing the services of Mr. Carter, while thanks are also given to the Directors of Hulett's S.A. Sugar Mills and Estates Ltd., for permission to publish process records and other data used in this paper.

A final word of thanks to the management and staff at Darnall for the work put in during alterations to the plant.

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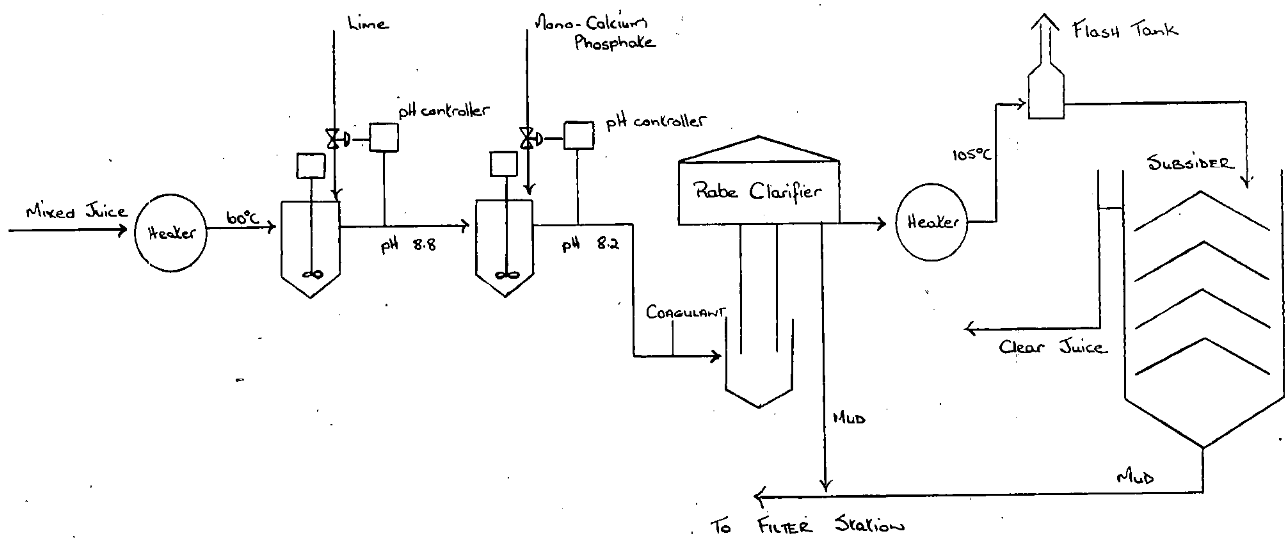

FIGURE 1: Rabe vacuum flotation.

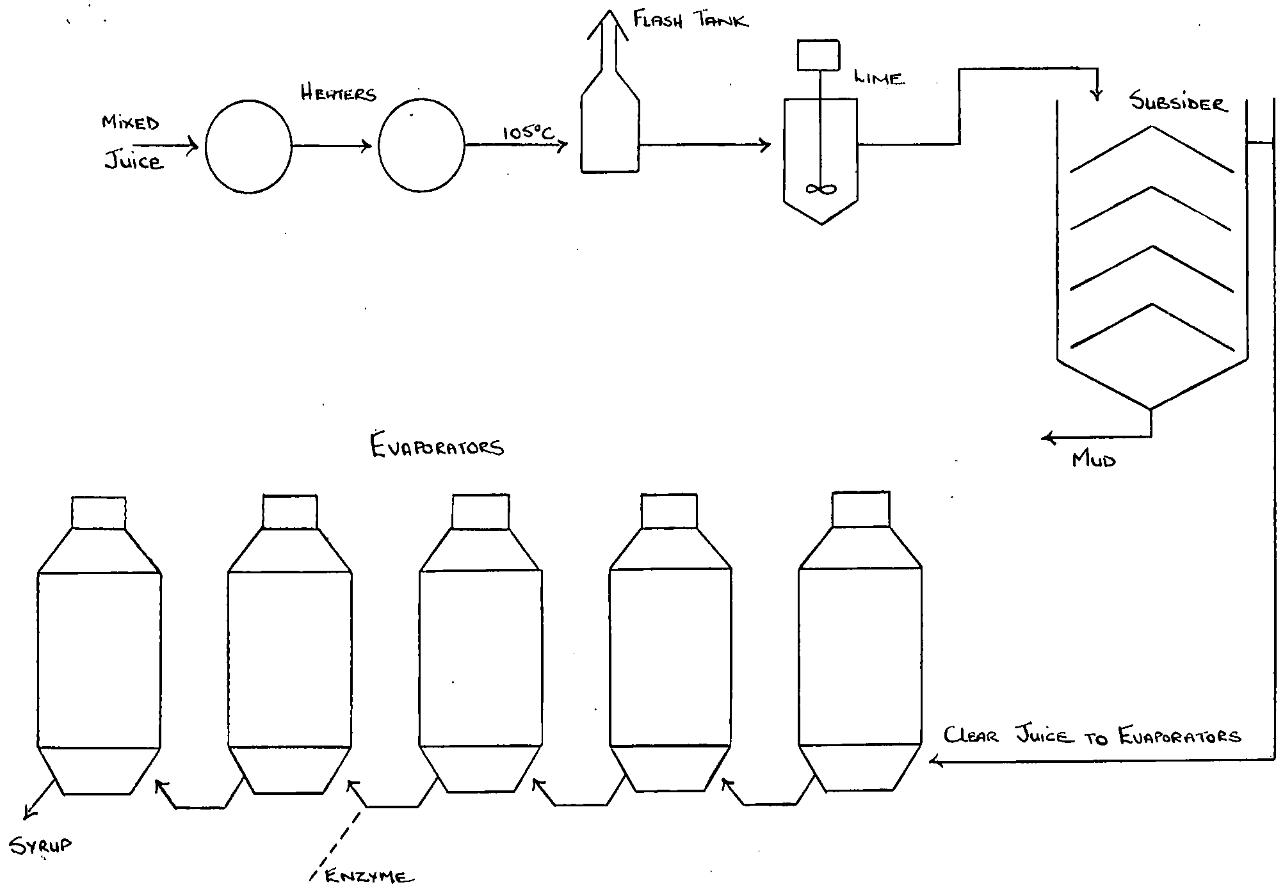

FIGURE 2: Enzyme and defecation.

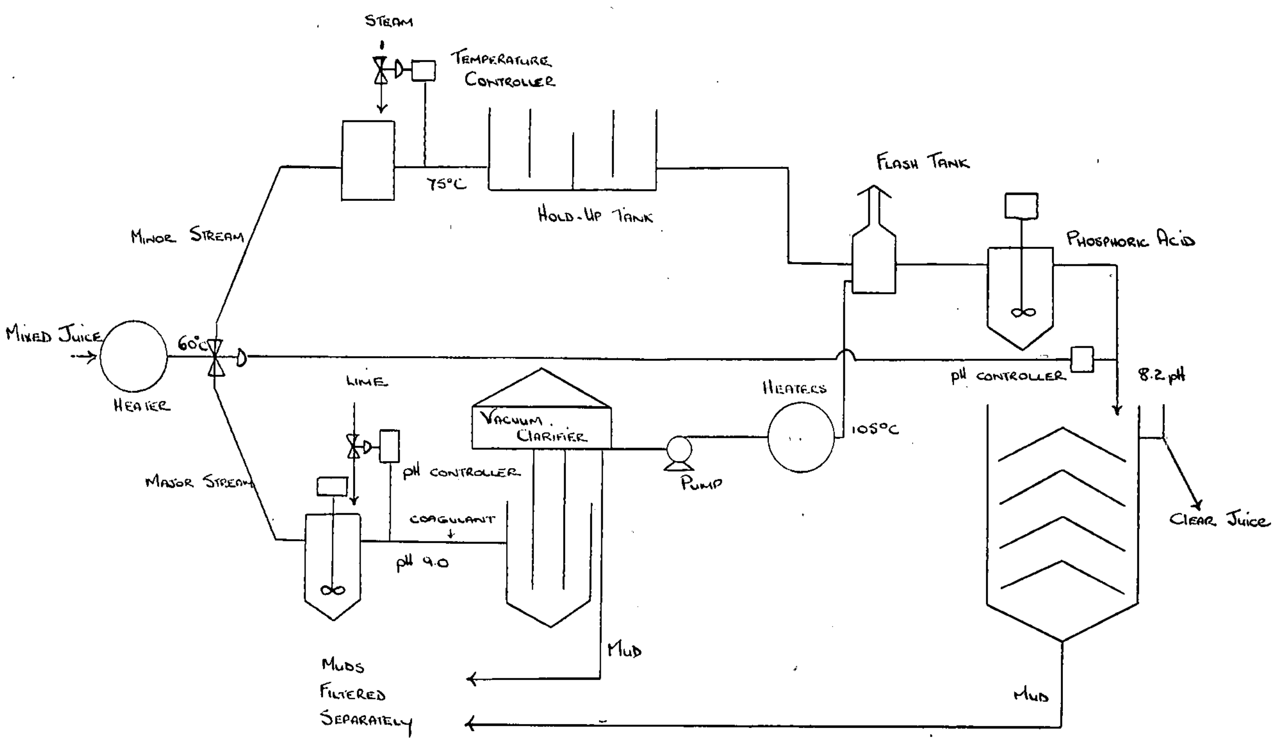

FIGURE 3: 90% split stream process.

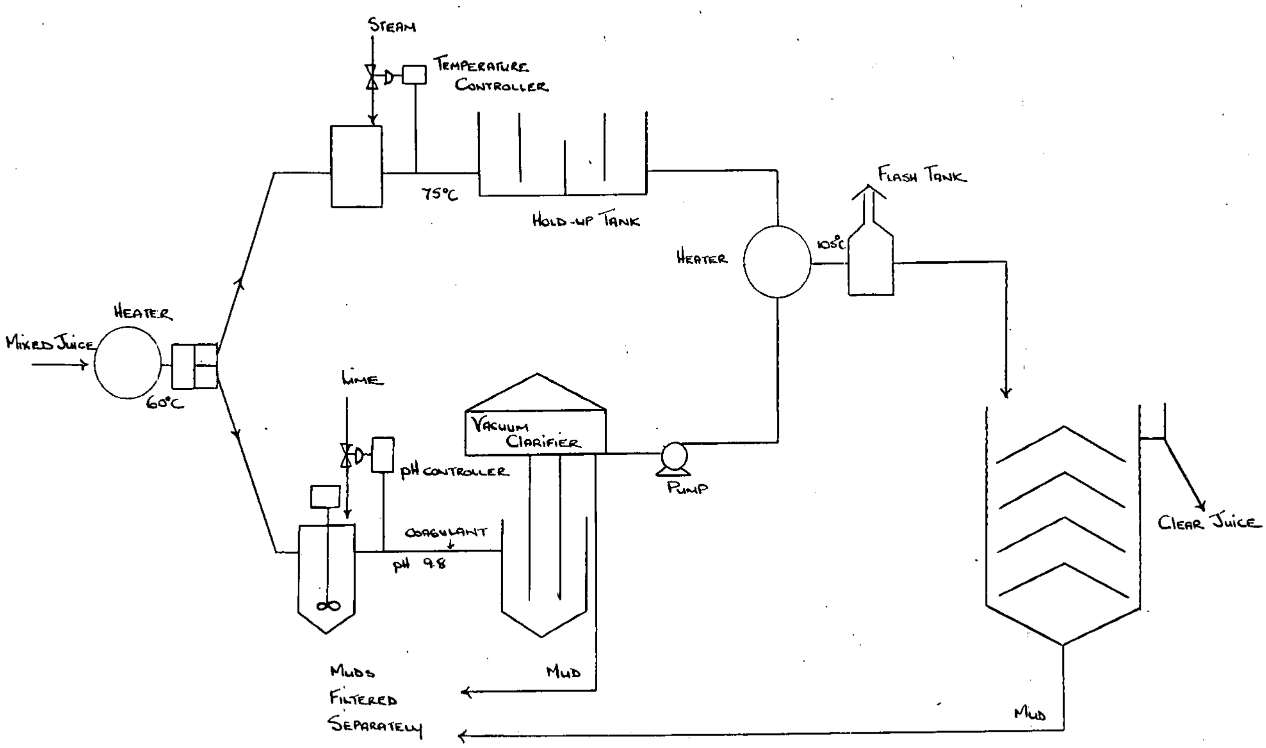

FIGURE 4: Tongaat process.

Discussion

Mr. Gunn (in the chair): At present there seems to be no doubt that enzymes are the cheapest way of producing low starch sugar and this will probably deter research into any other processes such as the flotation process.

The Tongaat process appears to be the next cheapest method.

Mr. van Duyker: The chemical costs for the Rabe process at Umzimkulu include lime and our lowest cost in a week was 18c per ton of sugar. The average cost last year was 25c per ton.

Mr. Carter: Figures published by Huletts Research and Development reflect that while enzymes are being used in a factory, the viscosity of molasses is reduced.

When we used enzymes at Tongaat we were fortunate to have three quadruple effect evaporators, to two of which enzymes were added. The syrups were sampled, the brixes adjusted to a common factor of sixty and the viscosity was determined. There was a marked reduction in the viscosity of the syrup where enzymes had been used.

I have no doubt the enzyme method is the cheapest available at present.

The trial of the Tongaat process at Darnall was not carried out under favourable conditions as there was trouble with pH control. The stream was adjusted to get an acid bias and then limed to pH 7.8. The syrup would be 7.0. If the pH was reduced slightly the syrup would drop to 6.3 pH.

I think more attention should be paid to reduction of ash. Although figures for silica and magnesia removal are not given in these tables there is a reduction of these constituents in juice that has passed through a vacuum clarifier at pH 10.0.

The operation of the Rabe process without the addition of phosphate to the tank is difficult and not worth the trouble.

Mr. Rabe: My happiest time in the industry was when we were operating the defecation process, owing to its wonderful simplicity.

We get the simplicity of the defecation process when we use enzymes and as the costs are so reasonable then we must use them.

Dr. Matic: One of the important things in this paper is the cost comparisons that are given, all for one factory.

An earlier attempt was made to assess the Tongaat process from pilot plant data and laboratory work. The conclusions reached do not differ much from those of the full trials, which shows that in some cases processes can be assessed on a small scale.

In Tables 4, 5 and 6 the high ash content is rather surprising.

Mr. Robinson: When we are operating at 200 tch at Darnall our clarifier capacity is barely sufficient and may be partly responsible for the high ash figures.

Mr. Jennings: We have produced some sugar at Amatikulu using the same process and the ash figure was quite satisfactory.

Mr. Carter: I think, in answer to Dr. Matic, regarding ash, that this is connected with chloride content. Where chloride is high, ash is also high.

Mr. MacGillivray: Would any reduction of costs be made in the enzyme process and the split stream process if the starch levels could be monitored, maybe with auto-analysers?

Mr. Jennings: It is mentioned in the text that if starch levels could be monitored in the incoming juice it would be possible to control the amount of enzyme.

I agree with Mr. MacGilivray that we could save money if we could predict the amount of starch in incoming juice and adjust the dosage level accordingly.

It might also be possible to control the operation of the Tongaat process but control of the Rabe process appears more difficult.

Mr. Jullienne: For cheapness, should not the natural enzyme in the juice be used, with an eight minute retention time. The destruction could be completed by adding enzymes to the evaporators. On average, in South Africa, about 60% destruction could be achieved by using the natural enzymes in the juice. The balance of 40% could be destroyed in the evaporators.

Mr. Robinson: Simple defecation appears to remove 30% of starch. The danger with natural enzymes in a retention tank is that of sugar destruction, which is difficult to detect.

Mr. Jennings: The problem with natural enzymes is that the results achieved differ among the factories. Some get very good results, whereas at Darnall for instance, there was only 30% removal. It is all a question of the relative quantities of invertase and amylase present in the juice.

The exhaustibility of syrups produced when enzymes have been used has been investigated by the S.M.R.I. and I hope that this work will continue.