CLARIFICATION WITH EMPHASIS ON STARCH REMOVAL

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The removal of starch has, with the advent of the Rabe process, sprung into great prominence and this paper attempts to cover briefly the range of work done in this connection at Tongaat.

(1) The Enzymatic Process

Tongaat has, for some years, due to work done by P. N. Boyes,[1] been actively engaged in starch removal by the enzymatic process.

From the graph of time vs. starch removed it is evident that the removal slows down after ten minutes and the possible loss of sucrose does not warrant further retention times in the tanks.

The sucrose loss in these tanks is given in Table II.

It is seen that the brix is higher in the exit Juice

TABLE I

Starch content of juice in ppm and removal % in brackets

	Original	Tank 1	Tank 2	Tank 3	Tank 4
No. 1	350ppm 100%	200ppm (43%)	160ppm (54%)	145ppm (59%)	155ppm (56%)
2	450ppm 100%	230ppm (49%)	290ppm (36%)	270ppm (40%)	230ppm (49%)
3	230ppm 100%	150ppm (35%)	100ppm (23%)	90ppm (61%)	90ppm (61%)
4	270ppm 100%	200ppm (26%)	153ppm (43%)	110ppm (59%)	127ppm (53%)
5	370ppm 100%	183ppm (50%)	210ppm (43%)	180ppm (51%)	220ppm (41%)
6	440ppm 100%	275ppm (37%)	261ppm (40%)	260ppm (40%)	250ppm (43%)
7	170ppm 100%	120ppm (30%)	130ppm (24%)	170ppm (0%)	70ppm (59%)
8	255ppm 100%	205ppm (20%)	140ppm (45%)	100ppm 60%	135ppm (47%)
Av.	316ppm (100%)	203ppm (36%)	181ppm 43%	166ppm 47%	157ppm 50.32

GRAPH I

THE REMOVAL % OF STARCH VS. TIME
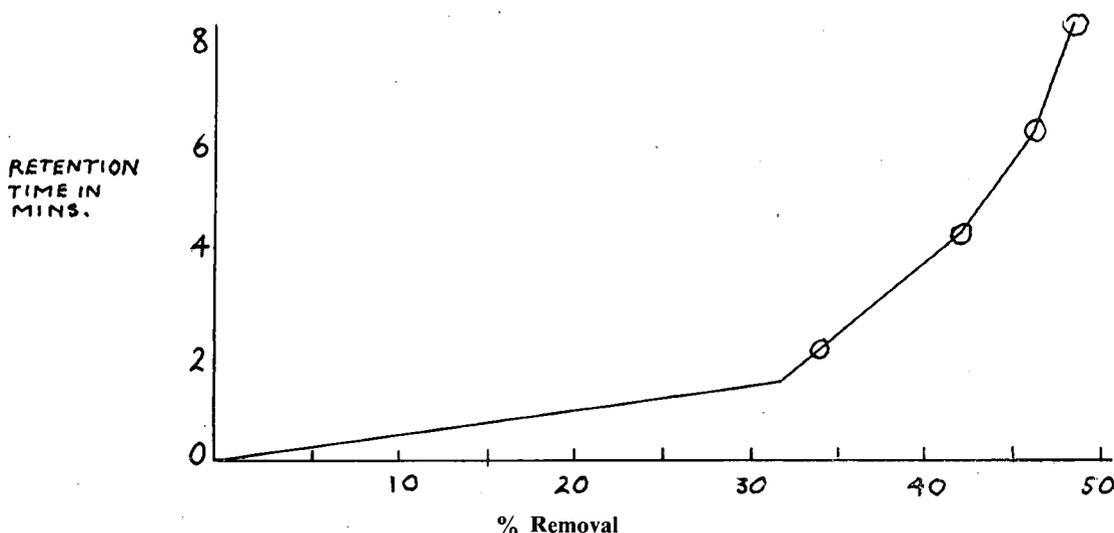
Briefly, this process involves the heating of Juice at pH 6.5 to 165°F (at which temperature the starch gelatinises) and then passing the Juice through destarching tanks where the starch, during a specified time interval, is removed by enzymatic action.

A typical analysis of the starch removal in these tanks is seen in Table I, Graph I.

due to evaporation along the starch tanks. Thus the sucrose in the exit Juice when converted back to its original brix gives a loss of sucrose of 0.15%.

On the season's production this loss of sucrose is estimated to be R10,000. The loss of this sucrose results in non sugars which are detrimental to the Boiling House, but on the other hand there could be

TABLE II

Study of sucrose and reducing sugars in juice before and after starch tanks

(A) Raw juice and O.F. to heaters:

	Brix (corr.)	Sucrose	Purity	Glucose %	Glucose Ratio
1	13.66	11.20	82.0	0.86	7.90
2	13.54	11.09	81.9	0.71	6.40
3	13.74	11.23	81.7	0.82	7.30
4	13.96	11.45	82.0	0.73	6.38
5	14.19	11.29	79.6	0.71	6.29
6	14.23	11.64	81.8	0.69	5.93
7	12.92	10.59	82.0	0.63	5.94
8	13.92	11.50	82.6	0.56	4.87
9	14.76	12.10	82.0	0.77	6.36
10	14.51	12.30	82.9	0.73	5.93
Average ..	13.94	11.43	81.99	0.72	6.33

(B) Juice after heating and digestion in starch tanks:

Brix (corr.)	Sucrose	Purity	Glucose %	Glucose Ratio
13.63	11.10	81.4	0.86	7.75
13.63	11.14	81.7	0.69	6.19
13.93	11.19	80.3	0.82	7.33
13.97	11.47	82.1	0.71	6.19
14.09	11.27	80.0	0.71	6.30
14.43	11.60	80.4	0.71	6.12
12.97	10.61	81.8	0.69	6.50
14.01	11.50	82.1	0.56	4.86
14.90	12.60	84.6	0.73	5.79
14.58	12.26	81.85	0.71	5.79

Average .. 14.02   11.48   81.85   0.72   6.27

benefits to the boiling scheme by having lower viscosity massecuites.

From investigations during this season into sucrose losses in the clarification station, the present defecation system with its long retention time was estimated to lose ± R25,000 worth of sucrose per season.

Thus the present system of clarification and starch removal costs an estimated R35,000 per season or 18 cents a ton, based on 200,000 tons, in sucrose lost and removes 50% to 60% of the starch.

(2) Middle Juice Carbonation

Due to Natal's geographical position in an area of deficient rainfall sugar manufacture is made more difficult and additional techniques are required to produce world class raw sugars.

The most promising clarification process derived when this work started in 1965 seemed to be the Carbonation process. There was one point governing its use however, namely that the cost was considerably greater than simple defecation.

It was decided that the most promising process was the Middle Juice carbonation process developed extensively in Taiwan by T. Y. Chou,[2] but instead of a full scale double carbonation, we would merely "polish" the defecation liquors in a single carbonation stage thus reducing costs.

After some time suitable batch type operations were performed using laboratory apparatus, kindly lent us by Hulett's, and for many months the process seemed to work best on a syrup of 40 Brix at a carbonation temperature of 60°C and maximum pH of 10.0.

Under these conditions the removal of impurities was expected to be:

(a) Phosphates 97%
(b) Starch 55%
(c) Gum 55%
(d) Silica 63%

The cost of the process was estimated to be 59 cents per ton for chemicals and 84 cents per ton including depreciation on equipment required for the process.

(3) *The Rabe Process*

At the time of the work on carbonation the industry began to rumble with the news of the Rabe process at Umzimkulu and by March 1966 we had the news from R. Jennings[3] of Hulett's Refineries that this sugar had processed with excellent results through the Rossburgh refinery.

Not being in a position to assess the process under factory conditions until July, it was nevertheless felt that some indication of its clarification potential was necessary on a laboratory scale.

Thus a series of comparisons were made with the developed carbonation process and the existing factory conditions.

For comparative purposes the Rabe process was done on Mixed Juice and the resultant clear Juice concentrated to 60 Brix. Three and a half hours later the Factory syrup was collected and part analysed, part subjected to the carbonation process.

After a series of twelve runs, the analyses of the known filter impeding compounds in these syrups were averaged and are presented in Table III.

nation might be covered by an increase in B.H.R., but this seems dubious.) So from the results obtained the evidence seemed to be very much in favour of the Rabe process.

(4) *Starch Removal Based on Sedimentation*

The Rabe process bases its starch removal on the trapping of the starch sack in a floc which is carried up by a flotation process and the scum decanted off to the filter station.

The reverse of the flotation process is sedimentation as in the existing clarification process. The author wondered whether this process could not be used successfully as a single stage clarification process without the high cost of the Flotation set up since it would mean no new equipment and a cheaper chemical bill. On the debit side however, were the facts that:

(i) The floc was likely to float due to incompleted deairation when clarifying at 65°C.

(ii) There could be considerable microbiological destruction of sucrose at the temperature at

TABLE III

Analysis of filter impeding impurities in three processes

		Starch ppm on Brix	Starch free Gum ppm	Silica ppm	Phosphate ppm	Ash %
(1)	Tongaat Process	960	15,770	376	114	1.31
(2)	Rabe Process	115 (82.5)	13,875 (12)	192 (49)	80 (30)	1.27 (3)
(3)	Carbonation Process	600 42.0	6,500 (59)	84 (77)	10 (92)	0.96 (17)

The figures in brackets represent the % Removed by Processes 2 and 3 above the present Factory Process which was taken as the standard.

From the results it is seen that carbonation (with the exception of starch removal) is superior to both defecation and the Rabe process in removing filter impeding compounds. However, in order to attempt to evaluate the Rabe process against carbonation as envisaged, it was decided by the author to arbitrarily multiply each impurity percentage removal over and above the standard process by an appropriate correlation factor given by T. Yamane et al.[4]

This factor is a correlation coefficient between the impurity and its effect on filterability through a carbonation slurry.

The total of these percentages thus modified will give an indication of the performance of the three processes. See Table IV.

Now if the process was perfect all impurity removal would be 100% and the total index would be 217.

From the results obtained the Rabe process appeared to cost 53 cents per ton including depreciation on capital equipment required and from the index of performance seemed to be but 10% poorer in overall performance compared to carbonation, but at only 2/3 of the cost. (The extra 30 cents a ton for carbo-

which the operation is carried out.

(iii) The muds could well be weak, meaning a higher sucrose loss in filter cake.

From work in the laboratory the removal appeared to be 85% when the Juice was heated to 65°C, limed up to 8.4 pH and settled for $1\frac{1}{2}$ hrs. During this time laboratory tests showed no increase in the Reducing Sugar Ratio, indicating no loss of sucrose and in settling tests at these high pH's the Juice actually settled faster than the process feed.

On the 9th February 1967 a Dorr of 24' diameter was made available for the test and the lime was added by means of a $\frac{1}{2}''$ pipe as the Juice flowed into the mud thickening chamber.

The first eight hours after filling the Dorr were spent in trying to get some control over the pH which fluctuated between 10.0 and 6.7 as the flow of Juice was very intermittent and the lime cock kept blocking.

Finally however, a working understanding was reached and from 6.00 p.m. until 12.00 p.m. the clarifier behaved well producing Juice of clarity

TABLE IV

Performance index of two processes

Impurity	Coefficient	Rabe process		Carbonation	
		% Removal	Index	% Removal	Index
Starch	0.95	83	79	42	40 ·
Gum	0.66	12	8	59	39
Silica	0:14	49	7	77	11
Phosphate	0.42	30	13	92	39
Possible Total	217		107		129

TABLE V

Control of Dorr No. 6 under cold clarification

P.H. of Juice		Reducing Sugars/Pol		Starch content ppm on Brix		Mud vol. of Juice	
In	Out	In	Out	In	Out	In	Out
8.5	8.0	4.09	3.87	2300	510	30	83
7.4	8.1	3.61	3.34	3100	350	34	95
9.3	7.6	4.63	3.98	2000	440	29	98
8.4	8.3	4.51	3.78	1750	390	29	97
7.8	8.0					32	98
6.7	7.7					30	98
9.4	7.3					28	98
						29	98
Average 8.21	7.86	4.21	3.74	2288	423	30	96

equal to its twin next door, which was operating under the normal defecation process.

During this period the following control figures observed. See Table V.

From 12.00 p.m. until 6.00 a.m. the following morning the flow of Juice once more became erratic and the Juice became turbulent with a drop in starch removal to 67% — no better than the enzymatic process.

At 8.00 a.m. the process was stopped and the following points had emerged:

(1) Sedimentation can remove starch and the maximum seems to be ± 85%.

(2) During the 24 hours under observation no apparent loss in sucrose occurred but this is not sufficient proof that during a season's work there would not be bacterial build up and sucrose destruction.

(3) No flotation of the mud occurred but the settling rate of the mud was slower than expected and the Dorr's capacity did not reach its designed 100 tons per hour.

(4) The mud volume had compacted down to 98% showing that the actual retention time in the clarifier must have been ± 3 hrs.

Acknowledgements

References

1. Boyes, P. N. 34th Conf. S.A.S.T.A. p. 91 (60).
2. Chou, T. Y. *Middle Juice* Carbonation Honig Vol. 1. pp. 711-725.
3. Jennings, R. 40th Conf. S.A.S.T.A. p. 199 (66).
4. Yamane, T. *et al.* I.S.J. 67 pp. 333-337. 68 pp. 3-6 (65-66).

Discussion

Mr. Buchanan: When any process involving the destruction of starch is being used it must not be forgotten that starch consists of two fractions, amylose and amylopectin, and as amylopectin is less soluble there is always the danger that amylose might be selectively destroyed and the amylopectin end up in the sugar, where it apparently causes retardation of filtration rates.

Dr. Matic: According to figures in Table III the ash content in the carbonatation process is considerably less than in the Tongaat or Rabe process, using a pH of 10. At the S.M.R.I. our results were different as residual calcium was always extremely high.

Mr. Carter: The ash content of carbonatation liquors will depend to a large extent on the process used.

Dr. Matic: We tried various processes without success to try and reduce the figure. Mr. Carter was simply carbonatating defecated liquor from the factory and when we did this our results were even worse.

Mr. Carter: At the time, Tongaat was producing a sugar with a filterability of 26 and with a high ash content, possibly due to carry-over.

The Rabe process was carried out on a laboratory scale and the clarification could have been improved.

The carbonatation juice had been filtered before the ash determination was made and this would have decreased the ash figure.

Dr. Graham: At the S.M.R.I. we were not measuring total ash but calcium and calcium plus magnesium and these were the figures we could not reduce. The table shows a reduction in silica also, which would constitute part of the ash, and therefore we are discussing different figures.

Mr. Alexander: Were you not possibly doing two totally different carbonatations? Mr. Carter carbonatating at 40° Brix and the S.M.R.I. at 15° Brix? This would alter the solubility of silica and of many other salts. Where Glucose is mentioned in the paper it would be better to use Reducing Sugars and when the removal of starch by the enzymatic process is discussed the breakdown should be given—starch is not removed as such.

Mr. Rault: In a paper I read at the 1960 Congress on the carbonatation process it was shown that the ash figure for M.E., which had used this process for many years, was lower than for any other Natal factory. We found that magnesia decreased but that calcium increased correspondingly.