

NOTES ON EXPERIMENTAL RESEARCH TO DETERMINE METHODS OF IMPROVING SUGAR QUALITY AT ILLOVO

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This paper includes a brief description of some tests undertaken at Illovo during the latter part of the milling season. The tests were undertaken primarily to obtain an indication of the filterability improvement obtained from a remelt sugar boiled from A sugar melt and furthermore to investigate the feasibility of Phospho-Lime defecation of B and C melt or possibly raw syrup.

A number of different methods were utilised to boil the remelt sugar, these all being described in the procedure which follows. Improvement in the filterability of the remelt sugar compared with that of the original A sugar varies from an increase of 17 per cent to as much as 300 per cent depending on the method used. The data obtained comprises the results of 7 tests only and in this respect, serves only as an indication of the effect of remelt on sugar filterability.

The Phospho-Lime experiments on B and C sugar melt were initially undertaken utilising a Williamson type flotation clarifier to remove the calcium phosphate floc. Very unsatisfactory results were obtained on this low purity material although a great many tests were carried out altering all the variables possible. It was then suggested that an Alfa-Laval separator should be tried to remove the calcium phosphate floc from the treated melt. This proved very successful on a laboratory scale and a plant scale test on raw syrup was then planned and undertaken. This test, although very brief, gave an indication that a satisfactory removal of undesirable impurities could possibly be obtained by utilising this method. A larger scale experiment will be undertaken at Illovo during the coming crop.

Remelt Sugar Experiment

The first three experiments consisted of remelting a sugar which was grained on normal syrup and run up on normal syrup (syrup containing approximately 70 per cent raw syrup and 30 per cent B and C sugar melt). This melt was then utilised to grain and run up a remelt sugar strike.

For the next experiment, the A massecuite was boiled from a B magma footing and run up on normal syrup. The A sugar was then melted, the melt being used for graining and running up a remelt sugar strike. For experiment five the A massecuite was boiled from a C magma footing which was run up on normal syrup. This A sugar was then melted and utilised to grain and run up a remelt sugar strike. For experiment numbers 6 and 7 the A massecuite was grained and run up on normal syrup. The A sugar was then melted and utilised to grain the remelt sugar strike. After graining, however, the remelt sugar strikes in these two experiments were run up on normal syrup.

In all experiments, both the original A sugar and the remelt sugar were sampled and analysed by S.M.R.I. for filterability. These results are tabulated

in Table I. Experiments 1, 2 and 3 which were identical in procedure resulted in remelt sugar filterabilities ranging from 41 to 61 with increase in filterability compared with the original A sugar of 80 per cent to 141 per cent. Experiment 4 resulted in the greatest improvement in filterability. The increase from A sugar to remelt sugar being 300 per cent. Experiment 5 resulted in an increase of filterability from A sugars to remelt sugar of 67 per cent indicating the effect of low filterability C sugar nucleus utilised to boil the A massecuite. Experiments 6 and 7 both identical in procedure resulted in an increase of filterability from A sugar to remelt sugar of 17 per cent and 61 per cent which was as expected, the lowest increase for all seven tests.

All the preceding experiments were undertaken over a period of approximately one week and during this period a very considerable variation in the cane quality processed was experienced. This is indicated to a very large extent by the variation in the original A sugar filterability, which dropped to a low value during the latter part of the milling season. The remelt sugar was boiled and cured in plant utilised for A sugar and thus the possibility of contamination with A massecuite resulted to a certain degree. These results, therefore, can only serve as an indication or guide as to the improvement of filterability when boiling remelt sugar.

Phospho-Lime Defecation Experiments

This experiment was undertaken to investigate the feasibility of utilising an existing Williamson type of flotation clarifier to remove the calcium phosphate floc from B and C sugar melts which had been treated with mono-calcium phosphate and lime respectively. The tests extending over several weeks proved very unsatisfactory because although the flotation removed a considerable amount of the floc complete removal even with considerably reduced throughput, could not be obtained. The resultant floc carry-over in the supernatant liquor resulted in filterabilities which proved to be lower than that of the original B and C sugar melt. Hence, because no measure of success could be obtained utilising the Williamson clarifier on B and C sugar melt, no results are tabulated in this paper. After observing the results of this experimental work it is considered that the Williamson clarifier, as designed to cope with A sugar melt for refining purposes, will not adequately clarify low grade material such as B and C sugar melt. This is probably due to the higher viscosity of this material which prevents adequate separation of the floc in the flotation process.

After the removal of floc from B and C melts by flotation had proved unsuccessful, the separation of floc from B and C melts by Alfa-Laval separator was suggested by R. Neilson, research worker on the experiment. A test batch of B and C melt treated with

TABLE I
Comparison of a Sugar Filterability with Remelt Sugar Filterability

Exp. No.	Method used to Boil "A" Sugar	Method Used to Boil Remelt Sugar	Filterability A Sugar	Filterability Remelt Sugar	% Increase Filterability
1	Grained and Run upon "Normal" (See note) Syrup	Grained and Run up on "A" Sugar Melt	34	61	80%
2	Grained and Run up on "Normal" Syrup	Grained and Run up on "A" Sugar Melt	18	47	161%
3	Grained and Run up on "Normal" Syrup	Grained and Run up on "A" Sugar Melt	17	41	141%
4	"B" magma Footing Run up on Normal Syrup	Grained and Run up on "A" Sugar Melt	12	48	300%
5	"C" Magma Footing Run up on Normal Syrup	Grained and Run up on "A" Sugar Melt	24	40	67%
6	Grained and Run up on "Normal" Syrup	Grained on "A" Sugar Melt Run up on Normal Syrup	24	28	17%
7	Grained and Run up on "Normal" Syrup	Grained on "A" Sugar melt Run up on Normal Syrup	23	37	61%

Note: "Normal" syrup is syrup containing approximately 70% incoming raw Syrup and approximately 30% B and C sugar melts.

TABLE 2
Results of Alfa Laval-Laboratory Separation Test on B and C Melt subsequent to Phospho-Lime treatment

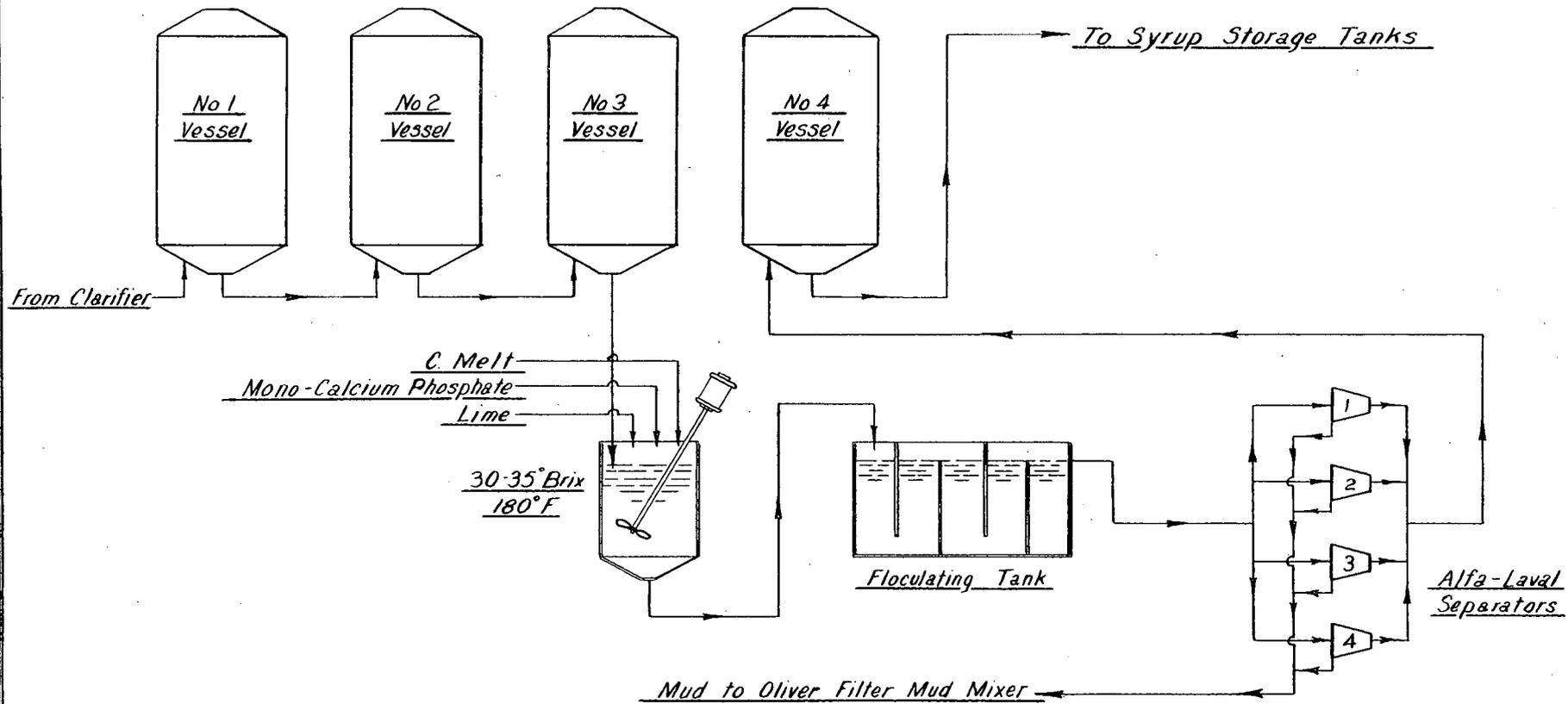
	Before Separating	After Separating
Filterability	6%	60%
Starch	780 ppm	115 ppm
Gums	2450 ppm	1300 ppm

TABLE 3
Results of Alfa Laval-Separator Test on approximately 30° Brix Raw Syrup subsequent to Phospho-Lime treatment

Batch	STARCH P.P.M.			GUMS P.P.M.			P ₁ O ₅ P.P.M.		
	Before Treatment	After Treatment	% Remaining	Before Treatment	After Treatment	% Remaining	Before Treatment	After Treatment	% Remaining
1	1,830	1,560	85%	9,850	9,050	92%	1,260	770	61%
2	1,800	1,210	67%	11,600	8,600	74%	1,160	990	85%
3	1,840	1,130	62%	9,750	8,650	89%	940	750	80%
4	1,770	940	53%	10,200	7,450	73%	1,340	540	40%
5	2,160	1,740	81%	11,600	10,600	91%	2,000	1,070	54%
6	1,410	1,130	80%	10,100	8,500	84%	1,130	1,100	97%
7	1,070	690	64%	8,250	6,800	82%	1,040	530	51%
8	1,590	1,090	68%	9,650	8,750	91%	610	470	77%

NORMAL: "A" Sugar Filterability at the time of experiment 17.0
"A" Sugar boiled from treated syrup 27.0

FIGURE 1
Proposed Suspended Solids Removal
With Alfa-Laval Separator.



mono-calcium phosphate and lime was tested in a laboratory Alfa-Laval separator, the results of which are tabulated in Table II. These results indicated a very considerable increase in filterability and a comparable decrease in the starch and gum content of the supernatant liquor.

After due consideration it was decided to conduct a larger scale experiment at the Illovo factory but in order to obtain maximum benefit in the boiling house, it was considered preferable to utilise raw syrup rather than B and C melt. Furthermore, in order to improve separation of the suspended solids in the Alfa-Laval separator better results of separation were thought to be possible if dilute raw syrup could be utilised rather than more concentrated syrup. With this in view the experiment conducted on the larger model Alfa-Laval separator was undertaken by diluting raw syrup from the 4th vessel of the quad evaporator to 30 brix corrected in order to simulate the assumed brix of syrup in the 3rd vessel of the quad evaporator. The optimum phospho-lime treatment for adequate flocculation and clarification of B and C melt was found to be approximately .2 to .3 per cent on brix. In this experiment, therefore, besides two batches of syrup all the batches were treated similarly with .3 per cent mono-calcium phosphate on brix.

The experiment was conducted by filling batch tanks of approximately 2,000 gallon capacity fitted with a stirrer, with raw syrup which had been diluted to approximately 30 brix corrected. The syrup was then heated to 170—175°F to simulate third vessel conditions. A mono-calcium phosphate solution was then added to the tank, allowed to mix in to the syrup and then the syrup pH was adjusted by means of lime slurry solution, to approximately 7.3 pH. After allowing a flocculation period of approximately $\frac{1}{2}$ hour, $\frac{1}{2}$ a pound of separant per 2,000 gallons of syrup was added and after mixing in the syrup, was then passed through an Alfa Laval separator. In all, eight 2,000 gallon batches of syrup were utilised in the experiment. Batch 1 was treated with .15 per cent mono-calcium phosphate on brix, batches 2, 3, 4, 5, 6 and 7 were treated with approximately .3 per cent mono-calcium phosphate on brix and batch 8 was treated with .6 per cent mono-calcium phosphate on brix. In all cases the final pH was adjusted to 7.3 with lime slurry. The quantity of lime utilised was batch 1 — 3 lbs., batches 2, 3, 4, 5, 6 and 7 — 6 lbs., batch 8 — 12 lbs. Samples of the original syrup and the syrup after the separator were collected in all cases and analysed by S.M.R.I. for starch, gums and P205 in parts per million. The treated syrup after the Alfa-Laval separator was collected and boiled into a single A massecuite strike, this sugar being analysed for filterability and compared with a composite A sugar filterability collected over the same period of time as the experiment.

The results of the analyses on the various syrup batches is tabulated in Table III. This table shows the starch in parts per million, gums in parts per million and P205 in parts of a million for each of the syrup batches before and after treatment. Also tabulated is the quantity of each of these constituents remaining as a percentage of the original concentration in each batch. This table also tabulates the filterability of the

normal A sugar collected during this period, compared with the filterability of the A sugar boiled from the experimental treated syrup. Table III indicates that in all cases there is a decrease in starch, gums and P205 after phospho-lime treatment. No explanation can be given as to the variability of the results except that the raw material utilised in the experiment was probably varying throughout the experiment and also other variables such as throughput were not at all times properly maintained. The filterability of the sugar boiled from the treated syrup was approximately 60 per cent higher than that of the normal A sugar during the period of the experiment. The conditions under which this sugar was boiled were also very unsatisfactory.

During the coming crop it is intended to continue this experiment on a larger scale utilising an Alfa-Laval separator, type No. NMBRTX 213-00S and treating the entire syrup throughput from one of the existing quadruple effect evaporators at Illovo which will constitute approximately one third of the total syrup throughput. A schematic flow diagram of the proposed system utilising the Alfa-Laval separators after phospho-lime treatment of third vessel syrup is presented in figure I.

Acknowledgment

Acknowledgment is due to Illovo Sugar Estates Managing Director, Mr. O. W. M. Pearce, for initiating these experiments, Illovo staff for the experimental work and Sugar Milling Research Institute for all the analyses.

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Dr. Douwes Dekker (in the chair): It has been established, both in Australia and America (Louisiana) that there is a significant relationship between the filterability of a syrup and of the sugar boiled from it. Louisiana particularly, has also tried to find out whether there is a relationship between the filterability of clarified juice and syrup. This has not yet been established, but there is definitely in these two countries, a correlation between the filterability of a syrup and its sugar. One is tempted to draw a conclusion from this that any thing we can do to our syrups to remove suspended matter, should improve the filterability of the sugar.

Mr. Pole has given us results of experiments in which the phosphitation system was applied. We could also apply the carbonatation system to the juice from the third vessel, which would cost less than mixed juice carbonatation, and would give a considerably better syrup. A third experiment which can be carried out in this country is sulphitation of syrup. I refer now to Umfolozi as being particularly suitable for an experiment of this nature, because they still have their melt sulphitation plant available. The treatment of the syrup is very important in our attempts to improve filterability of our sugar. Simple filtration over kieselguhr would probably not be possible because syrup is one of the substances which filters very badly and we have to do something more

than adding a mechanical filter aid. Mr. Pole has carried out tests with the phosphitation system, and we have the results. He has shown us that he removes starch and gums. Not considerable amounts of starch or gums but P_2O_5 to a very large extent. The filterability of this sugar improved from 17 to 27, and that was certainly worthwhile. I am very glad that Mr. Pole has decided to continue these experiments in the 1964 season.

Mr. Pole: We are making arrangements with Alfa-Laval to install one of their separators, a larger model than we normally use, to handle up to 6,000 gallons an hour of our secondary quad throughput. They have subsequently been advised by their principals that this type of separator is used in refineries in Canada and the States, probably in removing calcium phosphate floc, either combined with the Williamson process or the Jacobs process.

Mr. Kramer: What are the power requirements of these centrifugals?

Mr. Pole: The one we were using required 20 h.p. and the bigger machine which we shall acquire will use 25 h.p. We will need three or four for our full capacity should they prove successful.

Mr. Howes: Here are some points which may be of future use to you. We have done some work on purely refining molasses using a similar machine. I am not fully aware of the Williamson conditions, but pH seems to be significant. You have mentioned 7.3. In our process of refining molasses for an edible treacle, the condition we ensured was a pH of 4.8, and we found a very good separation and flocculation at that pH. We used phosphoric acid to drop the pH, and calcium acetate to finally precipitate.

Mr. Pole: We have not even touched on the aspects of what are the optimum conditions for chemical treatment. We have done a fair number of laboratory tests, but we have not reached any final conclusions. Your low pH would probably cause us a lot of losses in inversion which would have to be considered. So there may be an optimum pH, at probably a slightly lower value than we have established. The only parameter that we used in our laboratory tests was the apparent formation of the floc, and what we gauged to be the settling characteristics, although the settling of calcium phosphate floc is almost negligible.

Mr. Boyes: Might it not be advantageous to use the syrup? I appreciate Dr. Douwes Dekker's remarks about the difficulty of filtering syrup. What about putting in a reheating stage to try and reduce viscosity. You might get a better removal of gums. The sludge, that separates out of the syrup fairly rapidly, might be removed by centrifuging. I know you can remove a lot of silica, although that apparently is not so important. I think perhaps the improvement might be greater.

Mr. Pole: When we put in this separator and plant we are going to introduce the flexibility of being able to use 4th vessel syrup as well. We shall also have on loan from Alfa-Laval a plate heat exchanger. I do not know what heating surface we will have, or whether we will have sufficient to heat say, from 3rd vessel temperatures up to any desired temperature, possibly 190° F. or 200° F. to reduce viscosity. But we will have the means for heating and we will be able to use 4th vessel syrup as a comparison with 3rd vessel, if this is preferable.

Mr. Phipson: Did Mr. Pole notice any evidence of scaling of the separator during his experiments? Syrup is a rather awkward material and does form scale at high Brix.

Mr. Pole: To my knowledge there was no scaling at all. I must mention that this test was only carried out for a period of about 24-30 hours.

Mr. Dedekind: At Sezela, a Stellar filter was used on syrup, and it filtered it quite nicely. It had a brilliant colour, but it was not analysed for gum removal.

Mr. Alexander: At the refinery, as you can see from our paper, we have filtered all our syrup over char, recrystallised the sugar out and still some of the filterabilities are lower than the Cuban sugars that we hear about. In fact, our 4th sugar would not be classified in some other countries as a good filtering sugar. The S.M.R.I. carried out some tests on the filterability of the fine liquor and the brown liquor which we use for boiling the sugars. The filterability of this liquor appeared to be better than the filterability of the sugar. This rather confuses the issue, unless one accepts the fact that there has been some sort of polymerisation of the filter-impeding compounds, which were passed through filtration in the first stage, but at a later stage, when they are boiled into sugars, they have a different effect.