

INVESTIGATIONS INTO VACUUM CLARIFICATION AT DARNALL MILL

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Hulett's Darnall Mill

Vacuum clarification was introduced at Darnall in October, 1966. A series of experiments aimed at obtaining optimum operating conditions was commenced immediately. Unfortunately, progress of the experiments was governed by the difficulty of undertaking comparisons using only one tank.

It was therefore decided in the early part of 1967 that Darnall should be provided with a second clarifier for development purposes. This clarifier, constructed by the engineering staff at the mill, has been in operation since October, 1967.

Fed from a common feed tank, the prepared juice can pass through one, or both, clarifiers in parallel, as required. The basic differences between the two tanks can be seen in Figure 1, a more detailed description appears in Table 1.

TABLE 1

	Old Clarifier	New Clarifier
Diameter	17' 1"	20' 0"
Capacity	1042 cu. ft.	1404 cu. ft.
Ratio: Diam. of inlet to diam. of tank	0.234	0.625
Retention time at 300 tons mixed juice per hour	6.1 mins.	8.8 mins.
Coagulator	Vertical stationary baffles	Horizontal slow mechanical mixing
Flocculant addition	4 points	8 possible points, four used
Mud removal	Centre take off	Outer take off
Juice Outlet	Outer take off	Outer take off
Level controller	<i>mud weir</i>	<i>juice weir</i>

A serious problem was encountered at Darnall when the process was first introduced, namely, scaling of tubes in the pre-evaporator¹. The scaling was so bad that the mill had to stop for "factory full" as there was insufficient steam pressure from the pre-evaporator to boil the pans. The first mill stop occurred after the mill had been running for only 105 hours.

To overcome this problem the juice flow was altered so that after the vacuum clarifier, juice was heated to 105°C and sent to the subsiders, before going to the pre-evaporators. The volume of mud formed by secondary precipitation was so small that it was only necessary to flush the mud thickening cone of the subsider for about five minutes every hour. Due to the increased retention time of the juice at the bottom of the subsider, inversion frequently occurred and it became necessary to install four extra trays to reduce this hold-up time.

Preparation of Juice

At present mixed juice from the mill passes through a twelve pass liquid/liquid heater, the exit temperature being approximately 45-50°C, and then through a four pass vapour heater, automatically controlled to give a juice of 60°C, above which temperature starch begins to become soluble. After heating, the juice is limed to 8.7 pH in a Perry tank, from which it gravitates to a second Perry tank where mono calcium phosphate is added to adjust the pH to 8.2. Both pH's are automatically controlled.

During the development of the present arrangement of liming and phosphating, several methods were tried. These are listed below together with the reasons they were discarded. At this stage only the old tank was in use.

1. Proportional dosing of phosphate at the mixed juice tanks, heating to 60°C and liming to 8.1 pH.

The starch content of the raw sugars was disappointing and the sugars received by Hulett's Refinery during November and December 1966 showed little improvement over sugars received earlier in the season, when normal defecation and full remelt system were employed. The concentration of phosphate added seemed to be critical, and on several occasions it was found that an increase in phosphate strength reduced the starch content of the juice from the vacuum clarifier.

2. Proportional dosing of lime at the scale tanks, heating to 60°C and phosphating in the Perry tank.

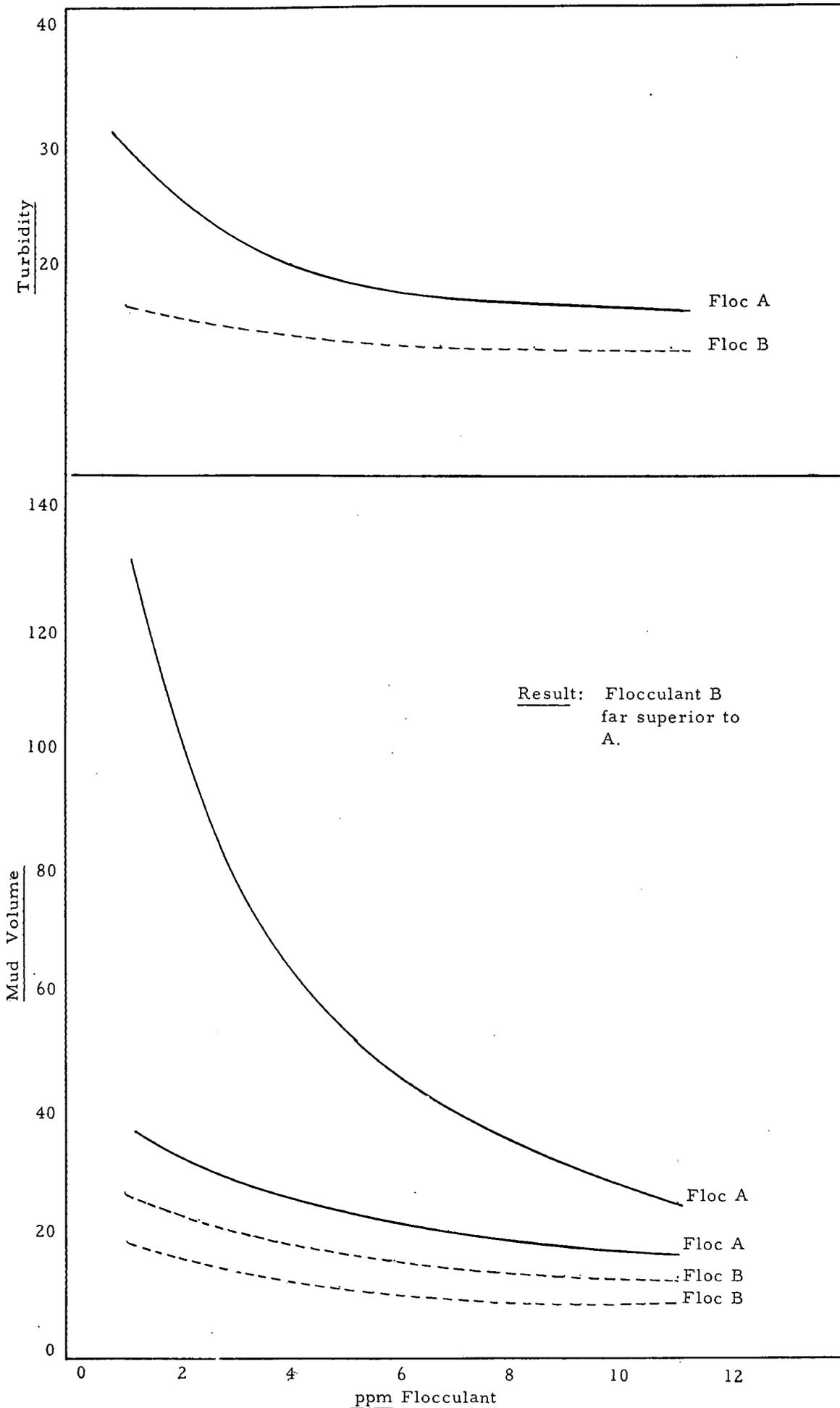
This method proved unsuccessful as no control of the pH of limed juice was possible. It was subsequently shown by potentiometric titrations of two samples of mixed juice, taken one hour apart, that one required 40 per cent more lime than the other to attain the same pH. Another problem was that the pipes of the dosing apparatus became choked. This had also occurred with proportional dosing of phosphate.

3. Heating to 60°C, liming in a Perry tank to 8.7 pH and "in line" phosphating in the sluice box immediately after the Perry tank, both pH's automatically controlled.

Following this change, the starch in vacuum clarified juice dropped considerably. The average for five days after the change was 230 ppm compared with 820 ppm for the six day period prior to the change. It was thought, however, that as another Perry tank was available, this should be used to provide more efficient mixing of the phosphate solution.

4. Heating to 60°C, phosphating in the Perry tank and "in line" liming in the sluice box.

The juice clarity was not as good as that given



Floc A 1 min. mud volume
 Floc A 5 min. mud volume
 Floc B 1 min. mud volume
 Floc B 5 min. mud volume

Figure 2

by method (3), and more often than not a considerable amount of carry-over was present.

Several other methods were tried, but yielded few significant results. It was shown, however, that any system which included pumping of a juice containing floc particles was ill-advised as pumping tends to break up the floc, making re-coagulation by means of a flocculant more difficult.

Juice clarity and economics were the major deciding factors in the choice of pH settings. The settings, liming to 8.7 followed by phosphating to 8.2, yield syrup with a pH of 6.7 to 6.9.

Present Investigations

1. Flocculants

Manufacturers were invited to submit samples of their flocculants, which were tested on a laboratory scale. Although a suitable laboratory method for carrying out these tests was not immediately available, it is considered that the method employed yields significant results.

Method

- (a) An 0.1 per cent solution of flocculant was prepared.
- (b) Equivalents of 1, 3, 5, 7, 9 and 11 ppm of the flocculant were pipetted into six 250 ml graduated measuring cylinders.
- (c) 250 ml aliquots of factory prepared juice (limed/phosphated) were poured into the cylinders, shaken and 14 inch vacuum applied.
- (d) The mud volume was recorded after one minute and again after five minutes.
- (e) The optical density (turbidity) of the resulting clear juice was determined.

Earlier investigations² showed a correlation coefficient of 0.87 from a series of over fifty comparisons of turbidity and starch content of clarified juice. It might be beneficial for a factory using the vacuum clarification process to consider the use of an in-line turbidity meter for control purposes³.

The most promising flocculants were compared in parallel, using the same juice sample, with Separan AP 30, the flocculant in use in the factory at present.

Graph 2 is a typical result obtained, and the conclusions which may be drawn are:

- (i) The difference in the one and five minute mud volumes for the various flocculants is an indication of the amount of compacting of the mud particles. i.e. The smaller the difference at lower concentrations of flocculants, the more effective the flocculant is.
- (ii) The lower the turbidity reading the better the flotation. In some cases it was found that although clarification appeared to be good, the resultant juice was turbid due to minute particles which had not risen.

It must be added that the method is not without limitations, and that the results were always viewed with some reservations. Tests on less promising flocculants were repeated several times before the sample was shelved.

Those flocculants which proved to be most efficient

have been tested on a factory scale. Equipped with two vacuum clarifiers, coupled with a 600 gallon storage tank and a 200 gallon make-up tank for flocculants, it is possible to run a meaningful comparison in the factory.

Flocculant "A" is pumped from one tank into the old clarifier, and flocculant "B" into the new clarifier. After two hours "A" is redirected to the new clarifier and "B" to the old, thus minimising the efficiency of the clarifiers as a variable. Throughout the experiment the starch content in the juice from both clarifiers is recorded.

Until all the promising flocculants have been compared and results carefully considered, no "merit list" can be drawn up.

2. Phosphoric Acid

Five 44 gallon drums of commercial phosphoric acid were obtained for experimental purposes. Three trial runs using acid instead of mono calcium phosphate were carried out between October 1967 and January 1968.

Since it was not feasible to empty the existing 1,200 gallon storage tank for mono calcium phosphate, 10 per cent solutions of phosphoric acid were made up in 45 gallon drums, and pumped by means of a mono-pump to a constant header tank supplying the required amount to the juice. In the first trial the acid was on stream for just over thirteen hours, samples of mixed juice and clarified juice were taken every hour and analysed for starch and turbidity. The results of this experiment appear in Table 2.

There appears to be little to choose between phosphoric acid and mono calcium phosphate as far as starch removal is concerned. From an economic viewpoint however, phosphoric acid is far more attractive. In the three tests conducted the average usage of phosphoric acid was approximately 0.30 lb. per ton of juice compared with 0.51 lb. per ton of juice for mono calcium phosphate.

While a fixed price for the acid is still under negotiation, it would appear that a saving in the region of one cent per ton of juice could be expected from the use of phosphoric acid, instead of mono calcium phosphate. This represents a saving of more than R15,000 per annum.

There are several additional advantages favouring the use of phosphoric acid. It would eliminate choking of pumps and pipes by mono calcium phosphate sediment, an occurrence not infrequent at Darnall, as well as the large sediment deposits in the storage and header tanks and the wearing of rotors and stators of the mono pumps. Another advantage is the comparative ease in making the solution strength required. In addition, it seemed that at the time of the trial a better pH control was possible.

3. Flocculant Addition

Using a Nalco Disperser, a 0.5 per cent solution of flocculant was made up. This was pumped by means of a mono pump to the new vacuum clarifier, through an 18 x metric series rotameter and diluted to 0.5 per cent with condensate water, the flow of which was regulated through a 47 x rotameter.

TABLE II
Phosphoric Acid

Vacuum Clarified Juice				Mixed Juice		
Brix	pH	D°	Starch	Brix	Tons	Starch
11.5	8.3	17.0	170	13.3	196	1630
13.0	8.1	21.0	190	14.3	254	1600
12.5	8.1	27.0	150	14.7	238	1670
12.5	8.1	26.0	250	13.3	194	1710
13.0	8.2	21.0	190	14.0	223	2070
12.0	8.1	27.0	210	14.5	253	1470
12.4	8.0	21.2	150	13.3	237	1550
12.7	8.1	19.6	180	13.5	196	1820
12.0	8.0	20.0	190	13.7	232	1950
12.7	7.9	16.0	140	14.0	266	1660
14.0	8.0	16.3	120	14.6	287	900
12.2	8.1	18.5	150	14.2	256	1020
11.0	8.1	17.4	140	13.7	250	1290
11.0	8.0	17.0	140	13.5	216	1610

Mono Calcium Phosphate

12.2	8.2	18.0	190	13.7	228	1970
12.0	8.1	23.9	270	14.3	223	1710
13.2	8.2	21.2	220	13.9	231	1320
13.0	8.0	20.3	180	14.1	248	1280
12.1	7.9	19.2	260	13.0	176	1820
12.0	7.9	16.3	160	13.3	122	1580
12.7	8.1	18.5	140	13.2	192	2280
14.5	8.3	22.9	160	13.7	209	1880
13.0	8.0	15.0	210	13.8	223	—
12.6	7.9	17.0	180	14.3	238	1390
11.9	8.0	16.0	130	13.6	235	1515
12.5	8.2	19.0	140	13.5	232	1690
12.2	8.2	21.0	190	14.0	226	1690

In contrast, when using the old clarifier, the 0.5 per cent solution can only be diluted to about 0.15 per cent, dilution water flow rate being metered through a rotameter only slightly larger than that used for flocculants. This is to be changed, as it has been found that a more dilute solution gives better results, probably due to more efficient dispersion of the flocculant in the juice.

It was shown, during laboratory experiments, that occasional bad flotation under vacuum could be attributed to a shortage of air in the juice. During a bout of poor flotation with a vacuum setting of 14.6 in. in the new clarifier, a manometer, connected to one of the flocc addition points of the coagulator, showed a reading of 2.0 in. vacuum.

With this in mind, a sample of juice was taken part of which was subjected to a 2 in. vacuum for about 45 seconds. To this, and to the original juice, 6 ppm of flocculant were added, and 14.0 in. vacuum applied. The results were drastically different, in that the juice which had been under vacuum showed little or no flotation while that which had not, floated exceptionally well. This test was repeated several times with the same results. The vacuum in the clarifier was subsequently dropped to about 13.5 in. giving a slight positive pressure in the coagulator. On each occasion that this was tried, the clarity of the juice improved.

In practice, however, the problem could not be overcome by simply reducing the vacuum in the

clarifier, as this frequently led to overflowing of the feed tank due to the fluctuations in the flow of mixed juice from the mill. Recently the coagulator has been lowered by two feet relative to the vacuum tank. This has resulted in juice which is more consistently clear.

Summary

The development of vacuum clarification at Darnall has been speeded up by the installation of a second vacuum tank. Experiments with juice preparation have shown that pH controlled addition of lime and phosphate, in that order, yields the best clarification results.

Phosphoric acid can be substituted for mono calcium phosphate with economic and other benefits. Investigations in progress to compare the efficiencies of various flocculants are outlined.

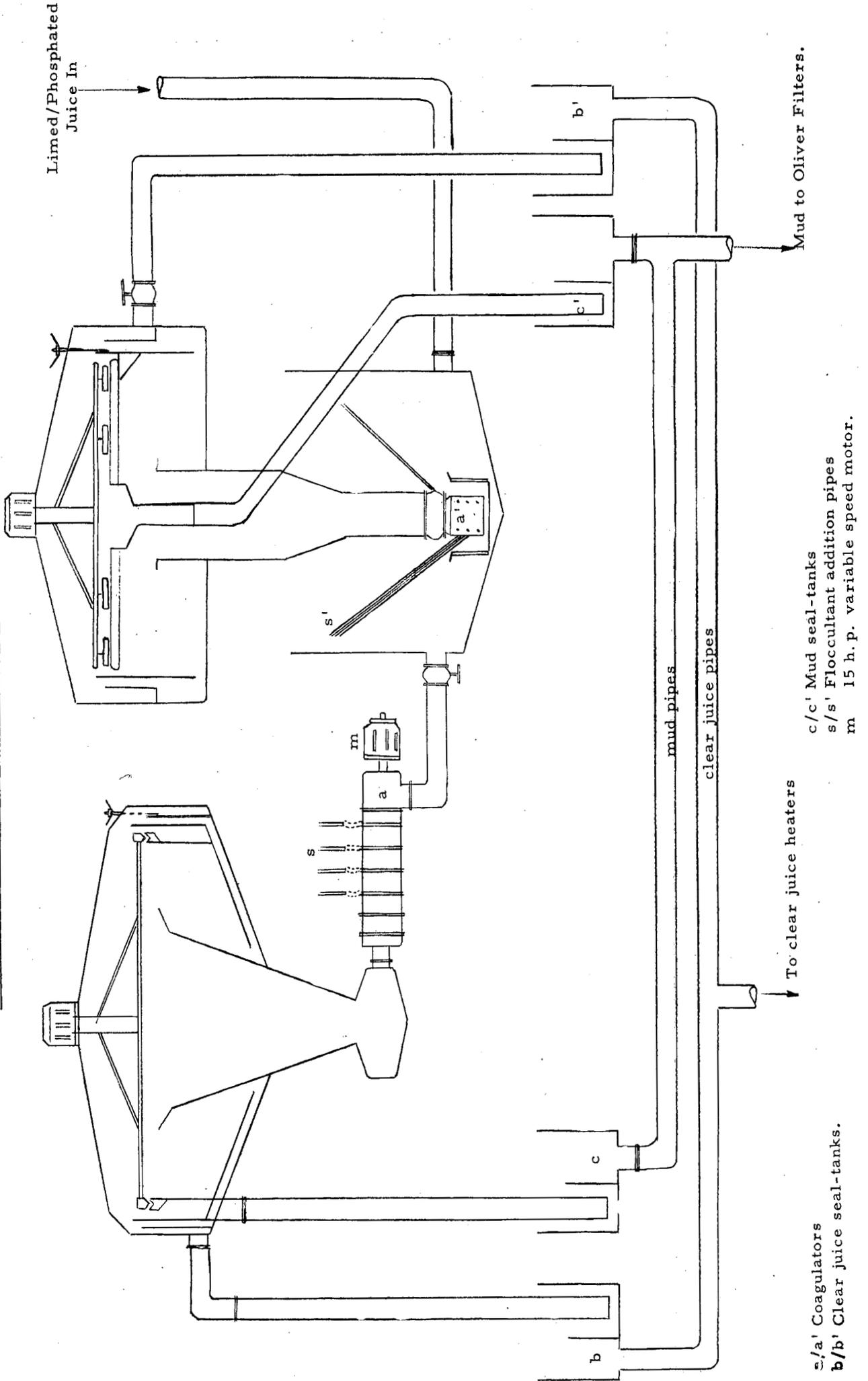
Acknowledgement

The author wishes to thank the Directors of Hulett's South African Sugar Mills and Estates, Limited, for permission to publish this paper.

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2. R. D. Archibald and R. P. Jennings. A rapid method for estimating starch in sugar products. S.A. Sug. Journal, 51 (August 1967). 8, 671.
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Darnall Vacuum Flotation Process Layout.



a/a' Coagulators

b/b' Clear juice seal-tanks.

c/c' Mud seal-tanks

s/s' Flocculant addition pipes

m 15 h.p. variable speed motor.

Discussion

Mr. Alexander (in the chair): Some of us who have just returned from Taiwan noted the great interest in clarification, especially in Mauritius and Australia.

There is interest in phosphate content, polysaccharide content, colour and oligosaccharides, so that now that we are nearing a solution to the starch problem we will have to deal with these other factors.

Dr. Matic: A word of warning is necessary about the use of flocculants. A flocculant that will be the correct one for a particular juice may not be as effective with another juice.

The order in which lime and phosphate are added would appear to be very important, and I wonder whether any conclusions have been drawn in this respect.

Two vacuum tanks were used, which differed for example in shape and retention times. What were their relative performances?

Mr. Archibald: I believe that liming before phosphating is common practice and we found it gave the best results.

In the old tank there is more interference with the flow of the juice. The juice from the new clarifier has been much clearer.

Mr. Robinson: Darnall tried adding phosphate first but was unable to get the process right. I think that the size and stability of the floc is increased by adding phosphate after lime and when vacuum is applied air bubbles are more easily trapped in the large floc particles.

Dr. Matic: At the S.M.R.I. we believe that a conical design approaches laminar flow and so cuts the breakdown of the floc which has been formed.

Mr. Alexander: I agree with Dr. Matic that flocculants must be used according to prevailing conditions and we must try and find the cheapest flocculant that will be effective for the longest period.

Mr. Jennings: Mr. Robinson's remarks about the size of floc necessary for efficient flotation appear to be contradictory to views expressed in a paper in the

South African Sugar Journal last year on "The dynamics of vacuum flotation" in which it was affirmed that a small size floc was necessary for efficient vacuum flotation. I think the larger size floc would give better flotation.

Mr. Archibald: The paper you refer to deals more with the flotation of an ore where a xanthate is used and the ore attaches itself to the air bubble. The position is quite different when dealing with a light particle as compared to a heavy ore particle.

Mr. Robinson: We are bringing out the air bubbles at low vacuum and if we inject the coagulant after the air bubbles have been made we do not get satisfactory results. I contend that it is necessary to make the floc and then make the air bubble inside the floc.

Dr. Matic: From observations of bubble formation with the aid of a microscope there is little doubt that when vacuum is applied the bubble originates by nucleating on the floc surface. Therefore to achieve flotation the floc must be formed first. As regards the size of the floc I think that Dr. Morris was correct in his paper that relatively speaking smaller particles of floc would be better than larger ones.

Mr. Rabe: I support Mr. Robinson's contention that large floc particles are required so that air bubbles form inside the floc when vacuum is applied. If mud is not properly coagulated a white foam forms on top so that flotation is not complete and the clarity of the juice is impaired. If the particle is loosely coagulated the air bubbles escape and appear on the surface as white foam.

Mr. Alexander: I think this offers a fruitful field for the S.M.R.I. to do some investigation into the mechanism of flotation in the Rabe process.

Mr. Buchanan: May I point out that Dr. Morris, the author of the other paper mentioned, was working in practice with diffused air flotation and not dissolved air flotation. For this reason he was mainly concerned with surface conditions and the stability of bubbles forming on the particle surface.