

ASPECTS OF FLOTATION CLARIFICATION OF MIXED JUICE

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Abstract

The influence of several parameters on removal of various impurities during vacuum flotation clarification of mixed juice has been investigated, and possible ways of dealing with the secondary precipitate obtained in this clarification process have been examined.

Dispersed air flotation clarification is described and the experience gained with this separation technique is recorded.

Introduction

The vacuum flotation process of juice clarification was introduced to South Africa in 1966 by Rabe¹. The efficiency of this process for starch removal was immediately recognised but there were, at the same time, several aspects of the process, both operational and fundamental, which required further investigation.

A literature survey revealed that a clarification process using phosphoric acid and lime, with the removal of phosphate precipitate by air flotation, was patented by Williamson in 1918². Instead of vacuum, heat was used in this process for release of dissolved air. The Williamson clarifier and particularly various modifications of it introduced by Jacobs³, and Bulkley-Dunton and Sveen Peterson⁴ became firmly established in sugar refineries for treatment of raw sugar melt. A thorough study of various parameters influencing performance of one of these clarifiers has been made by Saranin⁵.

In raw sugar mills the application of a flotation technique to the treatment of cloudy filtrate obtained from rotary vacuum filters was described by Higginbotham⁶, and Foster *et al*⁷ studied the application of this technique to syrup. A preliminary report by Sloane⁸ appears to be the only publication dealing with the application of flotation to the clarification of mixed juice.

The additional information required in connection

with the Rabe process was therefore not available. In an attempt to fill this gap the S.M.R.I. investigated several aspects of the flotation process and a part of this work, to various stages of which E. J. Buchanan, A. Jullienne, R. M. Morris and A. J. MacRitchie, particularly contributed, is summarised in this paper.

Influence of pH, flocculant level and freshness of juice on removal of impurities

Normally, the operating conditions during vacuum clarification are as follows: mixed juice is heated to 60°C and limed to pH 8.1 or slightly higher. The juice is then treated with mono-calcium phosphate or phosphoric acids to reduce pH to 7.4 and, after the addition of a coagulant, introduced into the vacuum clarifier⁸.

It was of some interest to investigate other pH levels at which precipitate could be floated and the influence of these pH levels on removal of various impurities.

A series of experiments were carried out with juices obtained from several mills. In each run one litre aliquots of juice were heated to 60°C and the required amount of calcium saccharate (about 20 mg CaO per ml) was added in order to obtain the desired pH. After the addition of 10 ppm of flocculant (0.05% solution of Nc 1273 - Dow Chemical) the juice was transferred to a conical vacuum flask and the precipitate floated at 15" vacuum. From the heating stage to flocculant addition the juice was stirred at constant speed. For comparison purposes a portion of the same juice was defecated at pH 7.5.

The clear juice thus obtained was analysed for starch, P₂O₅, SiO₂, protein and Ca + Mg using standard S.M.R.I. methods. Although absolute values varied with the origin of the juice, the trend was always the same and a set of typical results is reproduced in Table 1.

TABLE 1
Influence of pH on removal of impurities by vacuum flotation

	Mixed Juice	Defecation pH 7.5	Vacuum flotation at pH				
			7	8	9	10	11
Po1 % juice	10.96					12.09	
Ref. Brix	13.94	14.24	13.58	13.46	13.34	13.44	14.04
Purity	78.62					89.96	
Starch ppm/Bx	2254	1110	230	210	230	165	115
P ₂ O ₅ mg/l	160	12	40	12	5.4	3.9	1.6
SiO ₂ mg/l	180	72	50	48	47	21	23
Protein % on Bx	3.239	1.079	1.187	1.347	1.258	1.150	0.918
Ca + Mg mg/l	383	542	498	524	661	719	1552

There appears to be a gradual improvement in removal of impurities by vacuum flotation as the pH increases from 7 to 11. As far as starch, phosphate, silica and to a certain extent protein are concerned, the improvement is marked when approximately pH 9 is reached. However, there is also a sharp increase in Ca + Mg content of the clarified juice under those conditions. Furthermore, a much more voluminous precipitate is produced at these high pH's and this may be of considerable importance in both subsequent mud treatments and the expected sucrose losses in filter cake.

An advantage of vacuum flotation at any pH over simple defecation as far as starch and to a lesser degree silica removal is concerned is clearly evident from Table 1. The removal of other impurities appears to be of a similar order.

The influence of flocculant level on the efficiency of vacuum clarification at pH 9 was assessed by measuring the turbidity of the clear floated juice. It was previously established⁹ that a significant correlation exists between starch content and turbidity of the juice measured at 900 m μ .

It is apparent from Table 2 that as the amount of flocculant decreased the turbidity of the juice increased. In this experiment, however, the juice clarity was very good even at the 3 ppm level. In the absence of flocculant no, or only partial, flotation occurred. There were exceptions to this, particularly when flotation at high pH was carried out, and it appears that this behaviour was a consequence of juice quality. For example, juice obtained from N:Co.376 cane variety usually floated easily whilst that obtained from Co.331 was often very difficult to float.

TABLE 2
Effect of flocculant level on juice turbidity

Flocculant (Nc. 1273) ppm on juice	Turbidity at 900 m μ 2 cm cell
20	0.053
7	0.067
5	0.068
3	0.074

The effect of freshness of juice on efficiency of flotation was investigated in a series of tests performed over six hours on two batches of the same juice, one of which contained a preservative, Hg Cl₂ (Table 3). The turbidity of the clear juice after flotation of the preserved juice decreased with time, whereas that of the unpreserved juice increased. The latter could possibly be explained by the fact that a partial hydrolysis of starch present in the juice occurred and, as only insoluble starch is removed by flotation, the amount of "soluble" starch¹⁰ in the clear juice, and therefore juice turbidity, increased. No explanation could be offered for the decrease of turbidity found in preserved juice.

Secondary precipitation

A formation of small amounts of precipitate in the clear juice obtained by vacuum flotation on

TABLE 3
Effect of juice freshness on turbidity of
clear juice after flotation

Time hr	Turbidity at 900 m μ	
	Juice preserved with Hg Cl ₂	No preservative
0	0.138	0.111
1	0.074	0.113
3	0.048	0.156
5	0.024	0.175

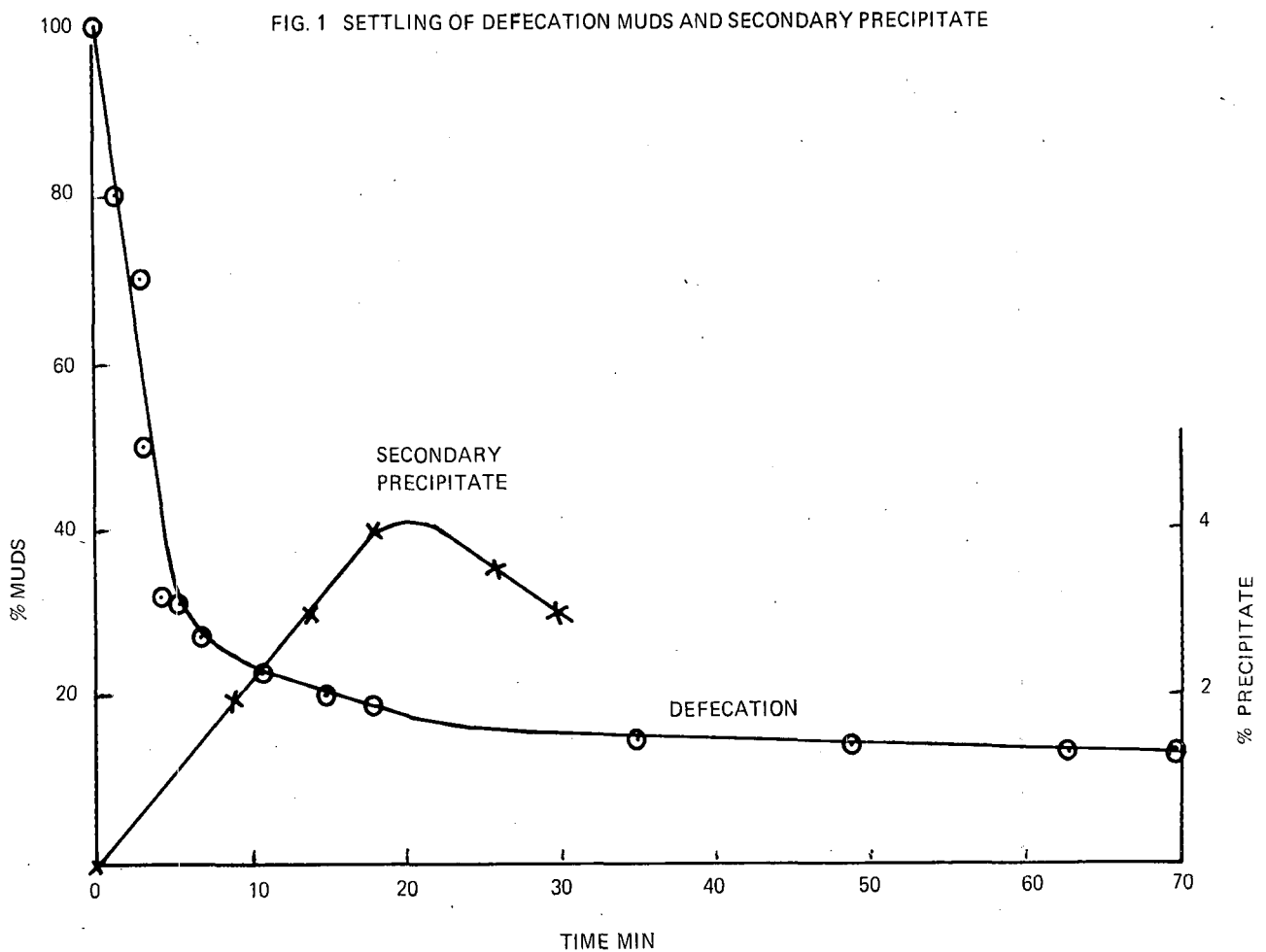
standing, and particularly after heating, was noticed in early laboratory experiments. The full implications of this secondary precipitation was, however, realised only after the full scale start of the Rabe process at Darnall, where after a few days of operation heavy scale deposited in heaters and evaporators.

The analysis of this scale (Table 4) revealed that two-thirds of the heater scale and half of the evaporator scale was made up of organic material, most of which was protein. The main inorganic constituents were calcium and phosphate. Clearly, a temperature higher than that used in vacuum flotation was required for precipitation of protein contained in the juice. The clear juice obtained after flotation had therefore to be heated to boiling and then settled in a conventional way.

TABLE 4
Analysis of scale ex Darnall

	Heater scale % dry sample	Evaporator scale % dry sample
Loss on ignition	78.3	55.5
SiO ₂	0.8	0.6
R ₂ O ₃	4.0	4.6
Ca ⁺⁺	5.3	12.1
P ₂ O ₅	7.0	10.8
Protein	34.7	21.3

Normally four zones can be distinguished in laboratory settling tests with defecation muds which are usually performed in a tall cylinder immersed in a constant temperature bath: (a) clear liquid on the top followed by (b) a zone of uniform solid concentration, (c) a zone of increasing solids concentration and finally (d) a zone of compaction. In contrast with this, the secondary precipitate from the flotation process gave only zones (b) and (d), i.e. one of a uniform concentration of precipitate and another of the concentrated precipitate. The latter gradually increased from zero to a maximum of 4% after 18 minutes (see Figure 1) and thereafter began to compact. However, even after one hour a small amount of very fine precipitate remained floating in the juice. It appears, therefore, that different settling mechanisms are operative during settling of defecation muds and secondary precipitate.



It was suggested at the time¹¹ that both evaporator scaling and the ash content of sugar manufactured by the Rabe process could be reduced by flotation at high pH's and subsequent carbonation of the clear juice obtained.

In order to test this suggestion juices from several factories and juices expressed in the S.M.R.I. mill from N:Co.376 and a P.O.J. cane variety were used in a series of experiments. The following clarification techniques were used always on aliquots of the same batch of juice:

- (a) defeccation by liming to pH 7.5, heating to boiling and settling the precipitate;
- (b) Rabe vacuum flotation at 60°C after liming to pH 8.4 and phosphating to pH 7.4. The clear juice was heated to boiling and settled;
- (c) flotation at 60°C after liming to pH 10 or 11;
- (d) clear juices obtained in (c) were carbonated using 30% CO₂/air mixture to pH 8.5, heated to 75°C and filtered;
- (e) carbonation according to de Haan. The juice was limed to a pH of 10.5 at 55°C, gassing using a 30% CO₂/air mixture was started and the pH held constant for 15 minutes by continuous addition of milk of lime. The juice was filtered and the filtrate carbonated to pH 8.5, heated to 70°C and filtered.

The clear juices obtained by these various techniques were analysed and some typical results are

presented in Table 5. Except for defeccation, starch removal in all cases was very good but residual calcium even after carbonation was high. The level of remaining impurities was comparable.

In order to establish the manner in which the residual Ca was retained in the clear juice a further series of experiments was carried out and the results indicated that the formation of organic acids at the high alkalinity levels used was mainly responsible for this. In carbonation, total alkalinity is of greater importance than pH¹² and when de Haan carbonation was carried out, maintaining the alkalinity at 250 mg CaO/litre, an amount of calcium and magnesium in clear juice as low as 217 ppm was obtained. However, when the same technique was applied to juice floated at pH 9.8 (this would correspond with an alkalinity of about 250mg CaO/l) the level of Ca + Mg obtained in clear juice was of the order of 500 mg/l. This is at the best only a slight improvement on the results obtained by defeccation or normal Rabe clarification and, in view of this, it was concluded that no real advantage would be obtained if the suggested modification was adopted.

Dispersed air flotation

The temperature at which the Rabe flotation process can be operated is limited by the boiling point of juice at the particular vacuum used. At these relatively low temperatures only partial clarification

TABLE 5
Analyses of clear juices obtained by various clarification techniques

Clarification technique	Ref. Brix	Purity	Starch ppm/Bx	P ₂ O ₅ mg/l	SiO ₂ mg/l	Protein % on Bx	Ca + Mg mg/l
Defecation pH 7.5	12.65	84.76	700	17	192	1.235	707
Rabe vacuum flotation ..	12.65		50	142	73	1.124	605
Flotation at pH 10	11.88		50	1.5	21	1.014	1200
Flotation at pH 11	11.60		85	1.6	19	1.227	1162
Flotation pH 10 + carbon- atation	11.50		165	Nil	21	1.217	739
Flotation pH 11 + carbon- atation			25	Nil	21	1.133	733
de Hahn carbonatation: 1st filtrate	11.20		25	1	19	1.267	926
2nd filtrate	11.50	25	1	22	1.272	637	
Mixed juice	12.00	76.25	1200	252	258	3.987	680

of juice is achieved as evidenced by the formation of secondary precipitate. Furthermore, the amount of air available for formation of bubbles which effect the flotation is limited by the solubility of air at the particular temperature. A clarification technique which could combine the efficiency of high temperature phospho-defecation with low retention times achieved by vacuum flotation would have many advantages. It would eliminate the subsiders and with them the exposure of clarified juice to high temperature for prolonged periods. As a result the always present danger of inversion of sucrose and of destruction of reducing sugars would be considerably reduced and the undesirable formation of colour substantially decreased.

Possible ways to achieve this were therefore investigated at the S.M.R.I. It was obvious from the beginning that the fragile floc produced on phospho defecation could not be treated in the same harsh way as solid particles in mineral flotation. Recently, however, Baarson and Ray¹³ described a new flotation technique in which several metal hydroxides, which are gelatinous and flocculant materials, were floated by means of dispersed air introduced at low rates in order to prevent breaking of the floc and its redispersion. Various parameters influencing this "precipitate flotation" technique were studied by Rubin *et al*^{14, 15}.

Preliminary experiments with cane juice were carried out using sintered glass funnels as flotation cells. Defecated juice was poured into the funnel and air under pressure introduced through the stem. By forcing air through the sintered glass disc fine dispersion of air bubbles was obtained. After about one minute of sparging precipitate was completely floated and clarified juice of exceptional clarity was obtained. When the clarification and flotation were carried out at 60°C starch removal was comparable to that achieved by the Rabe process. Analysis of sugar boiled in the laboratory from clarified juice obtained in this way showed a starch content of 70 ppm and a gum content of 635 ppm. The behaviour of the syrup during boiling in the laboratory pan was quite normal. Flotation of the juice defecated in the normal way and heated to boiling was also completely satisfactory.

It was soon realised, however, that bubble size

and type of floc are of utmost importance in this process. For example, only if sintered glass of porosity 2 was used was the floc floated. Similarly, only the floc obtained after the addition of flocculant was always amenable to flotation. The point of addition of flocculant was also of importance. When the flocculant was added after aeration of the juice more rapid rise of the floc and better flotation was achieved. This was attributed to entrapment of bubbles inside the floc when the latter coagulated in the aerated medium. In the absence of flocculant, the flotation was erratic and appeared to depend on the type and quality of juice processed.

A photomicrograph study of the bubble-particle attachment indicated that bubbles playing an active part in the flotation process have a mean diameter of 82 microns and a standard deviation of 1.9. Size distributions were determined using a Patterson and Cawood eyepiece graticule. Photographs of the aerated and floated floc indicated that bubbles were attached to the floc by a combination of flocculation and collision. Bubbles were found to be fairly evenly distributed inside and on the surface of the floc particles. It was noted that surface attachment occurred frequently in small cavities in the irregular surface of the floc (Figure 2).



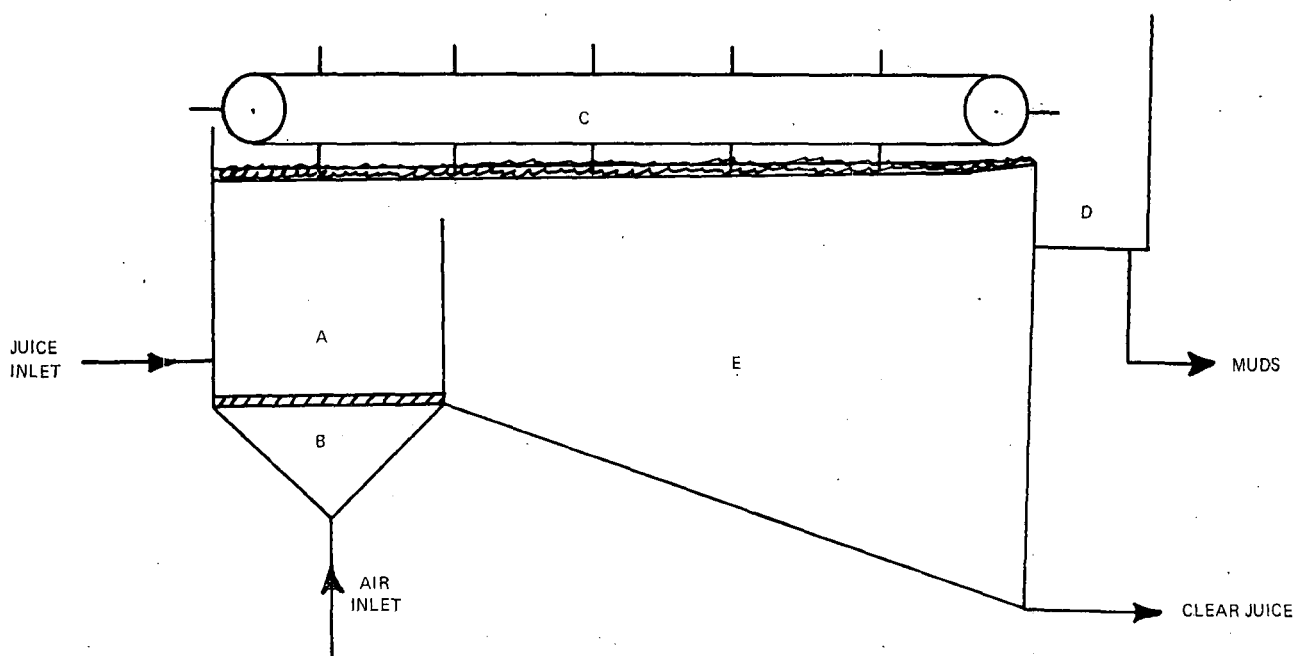
FIGURE 2. Photomicrograph of bubbles affecting flotation of cane juice floc.

For further experiments a continuous laboratory model of 21/min capacity was designed and built. The apparatus consisted of two compartments (Figure 3) into the first of which (A) the limed juice was introduced above a sparger (B). A layer of scum forms on the surface of the juice and is continuously scraped by baffles of an endless belt (C) into overflow (D). The clear juice is removed from the bottom of the second compartment (E). Difficulties were experienced with this model because of turbulence which could not be completely prevented in the compartment (E). A part of the floated floc was carried downwards and redispersed, finally finding its way into the clear juice.

were considered. It was concluded that difficulty would be experienced with the limited availability of suitably fine spargers. The fine dispersion of air by surface tension depression was examined using a wide range of frothing agents. The most suitable additives were found to be iso-amyl alcohol and ethyl alcohol. However, even the most economical additive, ethanol, at a rate of 2ml/l would be too expensive for commercial application.

As an alternative method of air dispersion, the juice was introduced into a clarifier by means of a centrifugal pump with an air bleed on the suction pipe. Although this was fairly successful the operation was somewhat unstable. Slight improvement was

FIG. 3 CONTINUOUS DISPERSED AIR CLARIFIER (MARK 1)



In an attempt to overcome this difficulty a new continuous model was built (Figure 4). The sparging column used in batch flotation experiments was fitted with a glass cylinder at the top in order to provide a compartment for collection of clear juice. Two inlets, one for the juice and the other for flocculant, were drilled in the Perspex column. A gutter was placed around the top of the glass cylinder for floc removal. A ring-shaped juice outlet was placed at the bottom of the clear juice compartment. The level inside the clarifier was controlled by means of a level controller connected to the clear juice outlet. The mud formed at the top of the clarifier was removed by overflowing into the gutter.

achieved by the addition of an air injector. However, by introducing a positive displacement pump ahead of the centrifugal pump a very steady aeration was obtained.

An improvement in performance, compared to the previous model, was achieved, but again the turbulence at the top of the column, caused mainly by a considerable number of rising large air bubbles, affected the floated floc layer. Consequently some of the floc sank and was carried away in the clear juice.

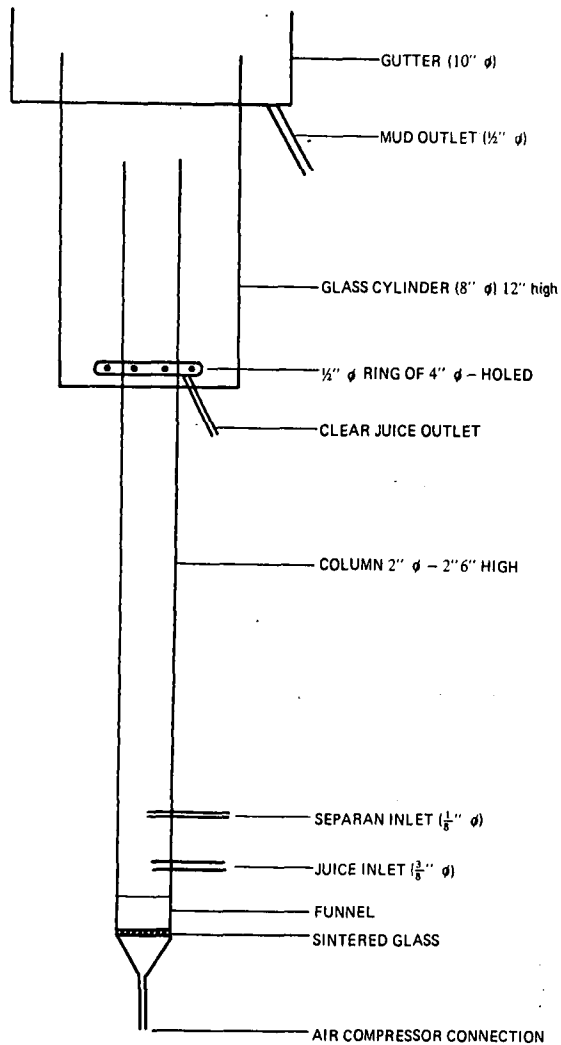
A third continuous model, designed for a juice rate of one-third gal/min was built (Figure 5). This was an adaptation of the clarifier used for water pollution studies by Stander *et al*¹⁶. The juice and flocculant were introduced at the top of tube A, the latter in order to compensate for the shearing action of the pump on the floc. The velocity down tube A was 4 ft/min which was sufficient to carry the floc downwards but allowed all bigger bubbles introduced with the juice to escape up the tube A. The upward velocity in B and downward velocity in C were both 15 ft/hr. The residence time of juice in this clarifier was 15 min.

At this stage the practical implications of introducing air by sparging in a commercial scale unit

The experiments with this model were conducted at Melville using the juice flowing to the factory Rabe clarifier. The juice was used as such or after it had been heated to 95°C in an electrically heated tank before it was fed to the laboratory clarifier. In both cases clear juice of a quality comparable to

FIG. 4.

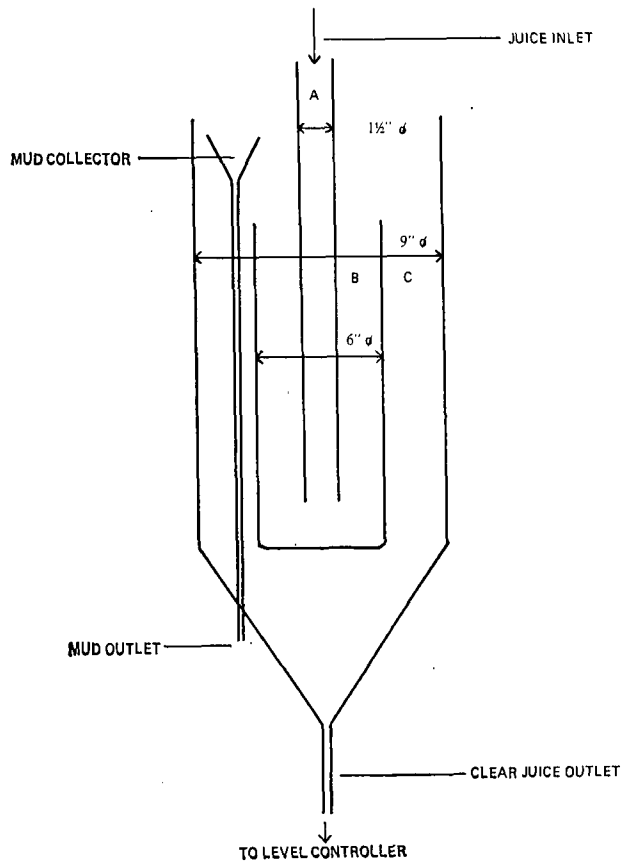
CONTINUOUS DISPERSED AIR CLARIFIER (MARK 2)



the factory clarified juice was obtained in parallel tests. However, a small amount of very fine precipitate always remained in the clear juice of the laboratory clarifier. Insufficient mixing of juice and flocculant at the top of tube A, the detrimental effect of the centrifugal pump on floc, and turbulence at the top of the clarifier may be the reasons for the imperfect clarification and improvements are possible. Nevertheless the fact that apparently an additional settling of the precipitate would still be required at this stage in order to obtain perfectly clarified juice defeats, at present, the whole purpose of high temperature flotation clarification.

FIG. 5.

CONTINUOUS DISPERSED AIR CLARIFIER (MARK 3)



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Discussion

Mr. Boyes: There appear to be two objectives for research. One is to develop, as described here, a flotation unit to work quickly and effectively in removing mud. The other is a sequestering agent to prevent build up of calcium on the replacement of magnesia.

This is aimed at the ideal, namely, to remove physical impurities from the juice.

At present for the starch problem the enzyme process seems the cheapest and most effective but it is a degradation process, not a removal process, and probably carries down sugar in final molasses.

Mr. van Hengel: I have suggested that the carbonation process should be added to the flotation process, not only to reduce the calcium and magnesium content of the clear juice, but also to give crystal-clear juice and to cut out the use of P_2O_5 , which is expensive. A disadvantage would be the introduction of filtration.

The de Haan process is described on page 3 of this paper where juice is heated to $55^\circ C$. Now this temperature is critical and yet in the flotation process the tests during carbonation have been done at $60^\circ C$ with poor results as regards calcium and magnesium, up to 500 ppm.

Mr. Rault mentioned the large quantity of molasses when Natal Estates was operating the carbonation process but I must point out that it was carried out at a temperature of $70^\circ C$.

The de Haan process requires a retention time of about 15 minutes for application of gas and lime. The Rabe tank requires 6 minutes. I therefore cannot see why the 217 ppm calcium and magnesium claimed for the de Haan process at $55^\circ C$ in fifteen minutes at a dangerously high level of pH cannot be repeated at 9.8 pH for 250 ppm at a

retention time of 6 minutes. The only reason I can think of is the temperature of $60^\circ C$.

Mr. Jennings: Has the idea been tried of sparging CO_2 at the same time as air?

Dr. Matic: No, this has not been tried but it is an interesting suggestion.

Mr. Alexander: These tests were apparently discontinued because there was always a certain amount of material in the clear juice that would not float properly and I would like to know if any analyses were done to determine why this would not float like the rest of the precipitate.

Dr. Matic: The material was not analysed but we were of the opinion that the centrifugal pump was damaging the floc.

I think the last type of clarifier could be improved by introducing air in such a way as to prevent some of the turbulence that is still occurring and by a more efficient mixing of the flocculant that is added.

I think mechanical alteration of the equipment is more important than the chemical aspect in order to get clear juice.

Mr. Buchanan: I do not think that there is a chemical or physico-chemical reason for the carry over of part of the suspended matter. It is purely a mechanical limitation. The juice cannot be slowed down sufficiently to avoid disrupting the bubble solid contact.

Mr. Alexander: I presume batch tests were done on the ordinary juice and the juice preserved with mercuric chloride, and yet the differences in turbidity are attributed to starch.

Dr. Matic: Yes, because the juices were completely clear in respect of suspended matter and starch is colloiddally dispersed. In static tests the juice is absolutely clear but, as soon as the continuous process is attempted, we cannot get rid of suspended matter.