EXPERIENCE GAINED IN SIMPLE DEFECATION DURING THE 1955 SEASON

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Introduction

In 1936 the Australian and British West Indian sugar industries experienced great difficulty clarifying the juices from recently introduced P.O.J. varieties. Their research institutes came to the simultaneous conclusion that the traditional cold liming technique of clarification would have to be replaced by a fractional liming double heating system, when clarifying these cane juices.

South Africa at this time relied on sulphitation to handle her refractory juices. The Tongaat factory used a type of Harloff process where the juice was heated to 170°F., most of the lime added, and then sulphited to a pH of 7.0 at the tower exit. Secondary lime was added to the sulphited juice and the alkalinity corrected with phosphoric acid to give a clear juice of 7.5 pH. The treated juice was heated to 220°F. and allowed to settle in subsiders. In effect, a double liming double heating process was being used with sulphitation super-imposed. Following the successful experiments at the Illovo sugar mill, Tongaat introduced defecation in August, 1954, by merely discontinuing the use of sulphur and reducing the quantity of phosphoric added.

The method employed at the beginning of the 1955 season was to use phosphoric paste at the rate of 0.35 lbs. per ton sugar made, lime the primary juice to 6.5 pH, heat to 150°F., then add secondary lime to pH of 8.0—8.4 and heat to 220°F., giving a final clear juice of 7.4 pH. Later in the year, towards the end of September, phosphoric acid was dispensed with when available P_2O_5 in raw juice varied between 320—450 ppm.

During the year a number of experiments were carried out to improve clarification and these form the subject of this paper.

Addition of Lime to the Subsider Muds prior to Filtration

Filter operation throughout the year was poor, due partly to blockage of the filter screens, but mostly to lack of bagacillo. Both clear and cloudy filtrates passed quantities of mud and the resultant clear juice from the subsiders appeared very cloudy.

Determinations of available P_2O_5 (see Table 1) showed that the filtrate in the subsider muds contained a much lower P_2O_5 content than the clear filtrate from the Olivers.

TABLE I

Available Phosphate Contents of Juices

Method.	Test.	Primary Juice from Mills. ppm. P_2O_5	Clear Filtrate from Olivers. ppm. P_2O_5	Juice after Primary Heating. ppm. P_2O_5	Juice after Secondary Liming and Phos. Addition. ppm. P_2O_5	Clear Juice. ppm. P_2O_5	Juice in Subsider Mud. ppm. P_2O_5
Before continuous liming of mud ...	1	382	122	—	61	26	35
	2	322	113	200	61	43	61
	3	332	139	288	104	43	70
	4	334	148	224	100	76	100
After continuous liming of mud	1	220	10	100	—	36	52
	2	322	10	181	47	19	29
	3	400	14	350	—	—	76
	4	410	24	350	57	47	67

This lead to the conclusion that phosphate was going back into solution at the filters. It is possible that di-calcium phosphate formed in the acid stage of the process is prevented by a layer of freshly formed tricalcium phosphate from dissociation. Dissociation then takes place with aqueous dilution at the filter station. We were able to demonstrate that by continuously liming the mud we were able to prevent this solution. The average P_2O_5 of clear filtrate was 130 ppm. before and only 14 ppm. after liming.

Liming the mud produced a denser cake free of slime, which peeled off the filters. Retention of the mud was on the whole good, but at times was extremely poor (see Table II). With good retention and low recirculation of mud in clear and cloudy filtrates, a sparkling clarified juice was obtained. The significance of bagacillo ratio on retention will be discussed later.

TABLE II

Filtration with Continuous Liming of Mud

	Feed to Oliver Filters.			Solids in Filtrates.		Press Cake.			
Test.	Solids Percentage.	Bagacillo Percentage.	Bagacillo Ratio.	Cloudy.	Clear.	Mud Percentage.	Bagacillo Percentage.	Bagacillo Ratio.	Overall Retention of Mud Percentage.
1	9.80	4.47	45.7	0.78	0.25	18.4	9.7	52.6	85.0
2	7.97	2.71	33.9	1.32	0.90	18.2	10.4	57.2	59.4
3	5.90	1.19	20.2	2.13	0.70	19.4	10.3	53.0	38.0
4	6.30	0.84	13.3	3.09	0.60	21.4	9.8	46.0	29.0
5	8.00	3.26	40.7	1.28	0.26	19.4	10.3	53.0	76.8
6	6.50	2.38	34.8	1.42	0.60	18.1	11.4	62.0	59.0
7	6.38	2.84	47.0	0.89	0.04	19.6	10.1	51.5	92.0
8	6.28	4.15	66.0	1.12	0.17	14.8	10.6	72.0	91.5

Adding lime to the mud must be controlled to prevent excessive alkalinity at temperatures of about 200°F., causing total destruction of invert sugars. At Tongaat the pH of clear filtrate was kept between 8.0–8.5, but even this could result in some invert destruction. Before liming of the mud can be accepted a study of invert losses involving the effect of pH, time and temperature will have to be made.

Fractional Liming with Double Heating and Simple Cold Liming

The British West Indies and Australia were quite satisfied to use simple cold liming for clarification, until refractory P.O.J. juices had to be processed.

Smith[1] experimented with clarification techniques using cold liming, hot liming, fractional double hot liming and fractional double hot liming with superphosphate. He concluded that for P.O.J. juices fractional double hot liming was the best technique. One technique was deemed better than another by its ability to remove in the most practical and economic way those impurities in the raw juice which had a deleterious effect on:

(*a*) Percentage of sugar that can be removed from the juice.

(*b*) The ease of recovering this sugar.

(*c*) The refining and other qualities of the recovered sugar.

He therefore analysed the raw and clear juices to ascertain the removal of reducing sugars, organic non-sugars, ash, P_2O_5, gums, waxes and insoluble matter. These analyses were coupled with plant scale trials to study the analyses of molasses and sugars, together with the refining quality and recovery of sugars. The juice analyses gave fluctuating and inclusive results, due to the ever-changing initial quality of the raw juice. Process data however showed that fractional liming with double heating was the superior method.

Behne[2] determined pH values giving optimum overall purity rises. His conclusions are summarised below:

TABLE III

Optimum pH Values for Clarification

Factory.	Method of Clarification.	Juice prior to Final Heating and Settling.	Clear Juice.
Invicta	Cold	8.0	6.8
	Fractional liming double heating	7.1—7.4	6.2—6.5
Isis	Fractional liming double heating	8.1	7.4—7.7

He observed that optimum conditions would have to be determined for each mill. At Tongaat we are more interested in obtaining a clear juice having a steady pH at the optimum value. The rise in purity between raw and clear juice is influenced by the condition of the Oliver filtrate, which mixes with raw juice directly after weighing. For this reason purity rise was studied for hot and cold liming on a small scale. The apparatus was a small square tank with a conical bottom, holding approximately 1 cu. ft. of raw juice, fitted with vertical sightglass, heating coils and stirrer. A known quantity of lime was added to the juice either cold or at 140°C., followed by rapid heating to the boiling point, allowing the precipitate to settle for two hours before sampling for purity. Comparative results for three series are given in Fig. I. The optimum pH for clear juice is about 7.4 for Hot Liming and 7.6 for Cold. Larger purity rises were obtained than those in practice, the difference being partly attributed to the recirculation of mud in filtrates. In practice we have found that a clear juice pH of 7.4 is obtained by liming to 8.0–8.4 before final heating and settling.

Davies compared the refractive juices from P.O.J. 2878 variety with the high claribility juices of B.H.10/12 cane variety. An exhaustive study of each cane during maturity revealed that the P.O.J. variety differed by having high total P_2O_5 (mean 350 ppm.) and organic silica contents. It was thought that part of the P_2O_5 was not available. Honig[4] in a study of phosphates in clarified juice concluded that of a total content of 25–80 ppm. only 10–40 ppm. were inorganic. Davies decided that organic silica was a major contributing factor to claribility. It is interesting to note that Uba cane with 0.128 per cent. organic silica on total solids was almost as high as P.O.J. 2878 0.142 per cent as compared to 0.056 per cent for B.H.10/12.

Davies, Duncan & Yearwood[5] varied the liming procedure on a lab. scale and concluded that for P.O.J.2878 additional phosphoric resulted in voluminous muds and cut it out altogether. The best results were obtained by double liming double heating. This was confirmed at later trials in the College Sugar Factory. There was an all-round improvement in removal of non-sugars and a remarkable increase in gravity purity rise of 1.72 over cold liming for the P.O.J. cane.

TABLE IV

Comparative Operating Data for Fractional Liming Double Heating

	College Sugar Factory,[5] 1936.	Guanica Puerto Rico,[6] 1938.	Tongaat, 1955.
Primary lime pH	6.4	6.2—6.4	6.5
Primary heating temperature ...	180—212°F.	200°F.	150°F.
Secondary lime pH	7.6	8.0—8.4	8.0—8.4
Secondary Juice temperature ...	212°F.	216—218°F.	220°F.
Clear Juice pH	—	7.2—7.4	7.4

Table IV compares factory results using fractional liming with double heating and indicates that the primary heating temperature at Tongaat may be too low.

Having stressed the reason for Tongaat's adoption of the F.L.D.H. process and the fact that this technique was introduced to handle refractory juices, it seems only fit to compare this process with cold liming.

Plant Scale Trials
Fractional Double Liming vs. Cold Liming

The factory experimented with cold liming from Friday, 14th October to Saturday, 29th, thereby giving a two weeks' trial. 5°Be. lime was added to the cold juice and heating to 220°C. was accomplished in two stages—primary heating to 150°F. and secondary heating to 220°F. The following observations were made:—

Liming

Davies[5] showed that one of the advantages of the fractional liming method was a saving in lime of 36.6 per cent. over cold liming. This was not borne out at Tongaat, where during the period of test the reverse was found (see Table VIII).

The lime requirements for a given clear juice pH will depend amongst other things on the amount of phosphate removed. Thus if less lime is required for fractional liming the ratio $\frac{\text{CaO added}}{P_2O_5 \text{ removed}}$ will be less than in cold liming for the same P_2O_5 removal and final pH. Thirty-one determinations were made using the laboratory apparatus previously mentioned and results showed no definite tendency, although on the average corresponding $\frac{CaO}{P_2O_5}$ ratios were higher for liming hot (see Fig. II). In these laboratory experiments true fractional liming and double heating was not followed, and all the lime was added and digested at a temperature of 140°F. before the secondary heating to 212°F.

pH Control

The changing quality and flow rate of the mixed juice, together with the high drop in pH from cold limed juice to clarified made manual pH control very difficult for cold liming. Over and under liming took place continuously about a mean of 8.6 pH, giving clear juice pH's varying from 7.1–7.6. Under these conditions syrups and sugars darkened in colour, due to colour formation in the evaporators. There was an immediate improvement in sugar colour when the factory returned to fractional liming.

Filtration of Muds

During cold liming a series of tests were made to determine the retention of mud (see Table V).

TABLE V

Filtration During Cold Liming

	Feed to Oliver Filters.			Solids in Filtrates.		Press Cake.			
Test No.	Solids Percentage.	Bagacillo Percentage.	Bagacillo Ratio.	Clear.	Cloudy.	Mud Percentage.	Bagacillo Percentage.	Bagacillo Ratio.	Overall Retention of Mud Percentage.
1	9.8	2.3	23.4	.50	—	18.5	10.0	54.0	43.3
2	8.6	2.3	26.7	.45	—	23.6	9.4	40.0	67.0
3	11.9	2.3	19.3	.55	3.35	18.8	8.8	46.5	42.5

Performance was extremely poor and large quantities of mud were recirculated. Comparing the results of Table V with Table II it will be seen that cold liming produced heavy muds of 10 per cent. solids compared to 6–7 per cent. with fractional liming. The quantity of bagacillo at Tongaat was inadequate for those high mud solids.

Foster[7] showed that with increasing $\frac{\text{bagacillo}}{\text{solids}}$ ratios in the feed, mud retention improved at a practically linear rate for constant mud solids. By increasing mud solids (i.e. thicker muds) and maintaining the bagacillo ratio, better retention was obtained. However, Foster only investigated mud solids up to 5.4 per cent. and in this paper results are given in the range 5.9 per cent.—11.9 per cent. It can be shown in Fig. III that retention does not improve materially by increasing mud solids above 6.0 to 6.5 per cent. However increasing the bagcillo ratio from 20 to 40 per cent. will increase the retention 50 per cent. Davies[8] in a recent paper shows that the optimum conditions for maintaining 90 per cent. retention is to keep mud solids between 5.0—7.0 with a bagacillo ratio of 50 per cent.

Mud solids during fractional liming were satisfactory and when the bagacillo ratio was above 40 per cent. excellent retention was obtained. During these periods the clarity of the clear juice was brilliant. Muds produced by cold liming were too thick and with inadequate bagacillo poor retention caused recirulation resulting in very muddy filtrates. For this reason purity rises could not be compared satisfactorily and no figures are given.

Factory Data Comparison

A further comparison between fractional and cold liming is obtained from weekly factory data giving the analysis of molasses, the pol. of sugars produced and sugar recovery for the period preceding and following the cold liming tests.

During the two weeks of cold liming sucrose losses increased, giving higher true molasses purities. Only when the factory reverted to fractional liming did the sucrose return to normal.

Sugar pol. remained normal during cold liming. Sugar colour visibly darkened and only improved on reversion to fractional liming.

B.H.R. dropped during cold liming. In the second week all losses were high. Increase in undetermined losses was probably due to fermentation at week-ends.

Deterioration at Week-ends

During week-ends deterioration appeared to be greatest for cold liming (see Table IX). On the week-end of 15th October fermentation was so bad that one subsider bubbled and frothed. It was noticed that the juice in contact with the mud was always acid for cold liming, being 6.8—6.9 pH, that for fractional liming was 7.1—7.4 pH. Possibly the precipitate formed in the mud is more basic for

TABLE VI

Analysis of Final Molasses

Week Ending.	Liming Method.	Brix.	Gravity Purity.	Dry Substance.	Clerget Sucrose.	Invert Sugars.	Organic Matter.	Sulphated Ash.	True Purity.
15.10.55	Fractional liming double heating	86.0	41.6	77.9	35.8	13.0	18.4	11.9	45.8
22.10.55	Cold liming	87.2	42.4	78.6	37.0	12.0	18.3	12.5	47.2
29.10.55	Cold liming	86.6	44.4	76.2	38.5	11.0	16.2	11.7	50.5
5.11.55	Fractional liming double heating	86.5	42.6	77.5	36.8	10.8	19.5	11.6	47.5
12.11.55	Fractional liming double heating	87.0	42.2	79.1	36.6	10.4	20.4	13.0	46.3

TABLE VII

Analysis of Sugars

Week Ending.	Liming Method.	Refinery Pol.	II Grade Pol.	Export Pol.
15.10.55	Fractional liming double heating	98.26	98.46	98.12
22.10.55	Cold liming	98.33	98.20	98.27
29.10.55	Cold liming	98.40	98.40	98.32
5.11.55	Fractional liming double heating	98.33	98.46	—
12.11.55	Fractional liming double heating	98.44	98.31	—

TABLE VIII

Recovery of Sugar

Week Ending.	Liming Method.	T.C.H.	Press Cake Percent. Cane.	Lime lbs./ton Sugar Made.	Sugar Losses Percent. Sucrose in Cane: Press Cake.	Molasses.	Undetermined.	B.H.R.
15.10.55	Fractional liming double heating	184.2	4.78	8.70	0.20	6.47	1.87	91.01
22.10.55	Cold liming	154.0	4.48	8.35	0.18	7.22	0.79	91.37
29.10.55	Cold liming	181.6	4.61	8.18	0.24	7.58	2.06	89.61
5.11.55	Fractional liming double heating	167.9	4.77	8.34	0.21	6.20	1.47	91.71
12.11.55	Fractional liming double heating	186.7	3.77	8.45	0.18	7.23	0.45	91.72

fractional liming than cold liming. Three precipitates of phosphate are known to exist[9]:—

Dicalcium phosphate	CaHPO4
Tricalcium phosphate	$Ca_3(PO4)_2$
Hydroxyapatite	$3Ca_3(PO4)_2Ca(OH)_2$

The ratio of CaO : P_2O_5 is theoretically 1.31 for hydroxyapatite but precipates having a more basic character have been observed. Possibly a precipitate having the hydroxyapatite composition is formed during fractional liming, while cold liming produces the tricalcium phosphate precipitate predominantly.

TABLE IX

Deterioration of Juices During Week-ends

Week Ending.	Process.	Clear Juice Purity. Shut Down Saturday.	Start Up Sunday.	Difference.	Clear Filtrate Purity. Shut Down Saturday.	Start Up Sunday.	Difference.
8 Oct.	... Fractional liming	89.6	87.7	—1.8	86.0	86.1	+0.1
15 Oct.	... Cold liming	90.0	83.5	—6.5	85.7	82.0	—3.7
22 Oct.	... Cold liming	89.2	85.6	—3.6	85.6	81.3	—4.3
29 Oct.	... Cold liming	88.6	87.9	—1.7	87.0	82.8	—4.2
5 Nov.	... Fractional liming	89.7	89.4	—0.3	83.1	82.9	—0.2
12 Nov.	... Fractional liming	89.1	86.6	—2.5	83.0	83.4	+0.4

Summary

Tongaat has applied the Double Liming Double Heating technique of defecation since August, 1954. With available P_2O_5 contents of 320—450 ppm. it has been found unnecessary to add phosphoric acid paste.

Phosphate appeared to be redissolving in the clear filtrates of the Oliver Filters. To counteract this and displace the dissociation equilibrium, muds were continuously limed. It was found that a dense cake, free of slime, which peeled off the filters, was produced. When mud retention was satisfactory brilliant clear juices were obtained. pH control was essential to prevent invert destruction and before liming can be accepted, this disadvantage must be investigated.

Cold liming was found to be unsatisfactory under prevailing conditions at Tongaat. Manual control of the limed juice pH was difficult, resulting in over and under liming. This caused darkening of syrups and sugars. Very thick muds were produced and an inadequate bagacillo supply resulted in poor retention. Recirculation of mud caused very cloudy juices and this was thought to be associated with the increased sucrose loss in molasses with accompanying increase in True Purity and decrease in B.H.R. Cold limed juices appeared to ferment more easily than fractionally limed juices and it was noted that the juice in contact with subsider mud was always acidic for the former (6.8—6.9) and alkaline (7.1—7.4) for the latter.

I will conclude by saying that these results show the importance of having a filter station which is efficient and having pH control which is reliable. When these two conditions are realised it is then possible to make an accurate assessment between two techniques of clarification. All figures given are for purely local conditions at Tongaat and may not be representative of the industry.

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(*For joint discussion on this and the following paper turn to page* 88)

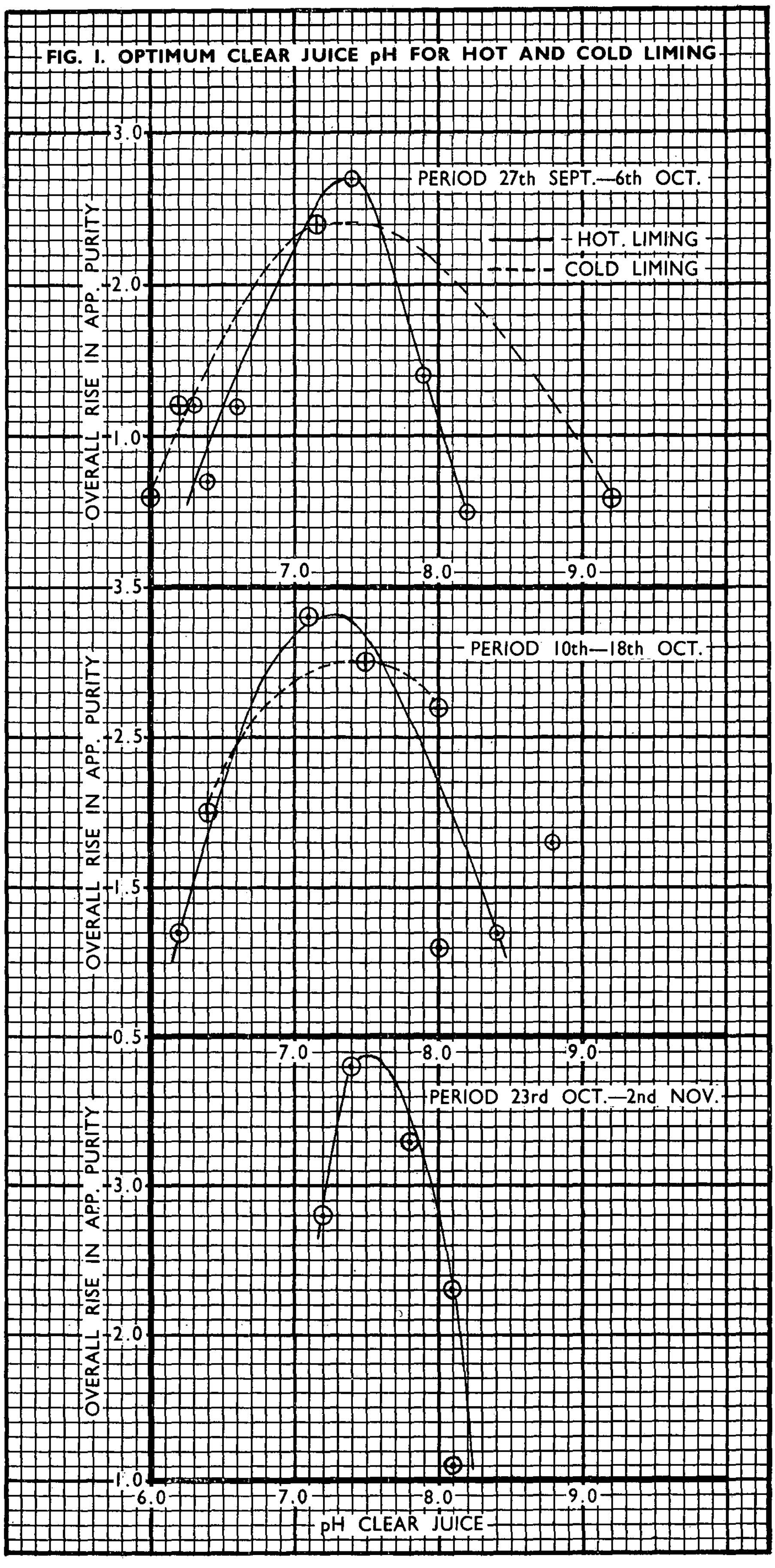
FIG. I. OPTIMUM CLEAR JUICE pH FOR HOT AND COLD LIMING
PERIOD 27th SEPT.—6th OCT.
HOT. LIMING
COLD LIMING
OVERALL RISE IN APP. PURITY
3.0
2.0
1.0
7.0
8.0
9.0
PERIOD 10th—18th OCT.
3.5
2.5
1.5
0.5
PERIOD 23rd OCT.—2nd NOV.
3.0
2.0
1.0
6.0
pH CLEAR JUICE

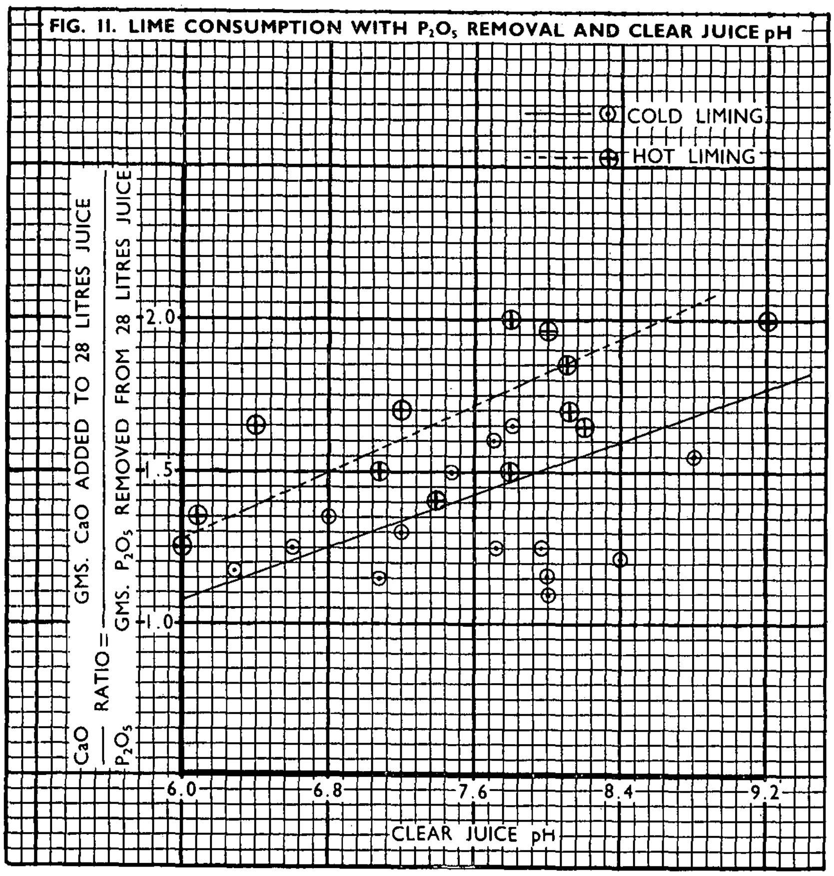
FIG. II. LIME CONSUMPTION WITH P_2O_5 REMOVAL AND CLEAR JUICE pH
COLD LIMING
HOT LIMING
$\frac{CaO}{P_2O_5}$ RATIO = $\frac{\text{GMS. CaO ADDED TO 28 LITRES JUICE}}{\text{GMS. } P_2O_5 \text{ REMOVED FROM 28 LITRES JUICE}}$
2.0
1.5
1.0
6.0
6.8
7.6
8.4
9.2
CLEAR JUICE pH

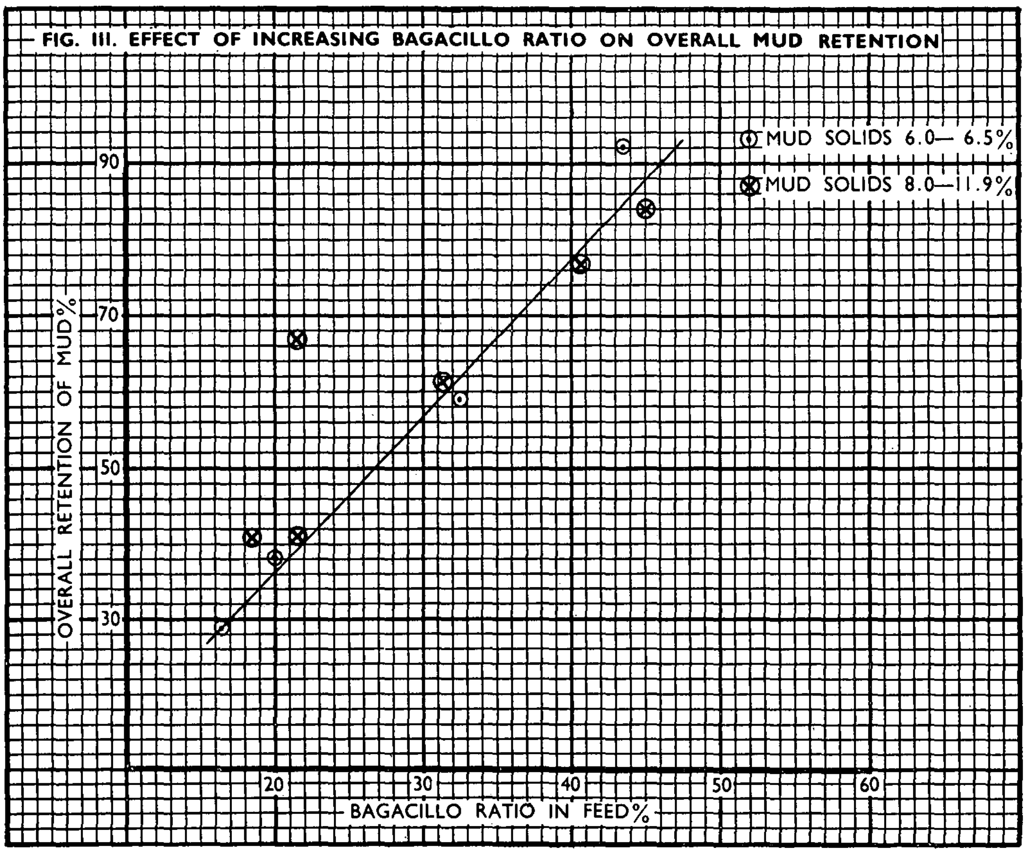
FIG. III. EFFECT OF INCREASING BAGACILLO RATIO ON OVERALL MUD RETENTION
MUD SOLIDS 6.0— 6.5%
MUD SOLIDS 8.0—11.9%
OVERALL RETENTION OF MUD%
90
70
50
30
20
30
40
50
60
BAGACILLO RATIO IN FEED%