A CARBONATATION PERFORMANCE TEST

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Abstract

A practical method for assessing the carbonation qualities of unaffinated raw sugar was developed. A correlation between laboratory tests and factory performance is shown and some applications of the method are outlined.

Introduction

Over the past 10 years much has been written on the subject of filterability and filterability tests. In the main, these tests centred around the use of filter aid and affined raw sugar.

With the advent of very high pol raw sugar (>99,3°S), the need for affining raw sugar fell away and attention was focused on carbonating quality as being the main factor affecting filter station throughput.

A rapid practical carbonation test

This test was developed in order to determine whether there was any difference between the carbonation performances of various V.H.P. raw sugars.

Experimental

The basic details of the test are as follows:

500 ml of 60° brix solution of unaffinated raw sugar in distilled water is placed in a beaker maintained at 55°C ± 2°.

To this solution is added a quantity of freshly prepared slaked lime slurry of S.G. 1,090 so as to give a 1% by weight CaO/solids ratio.

This mixture is gassed down to pH 9,0 with scrubbed factory flue gas containing ± 11% CO_2, using a 4 mm bore perforated spiral-wound copper tube gassing ring.

The resulting carbonated liquor is heated to 80°C and filtered through a No. 1 Whatman filter paper using the modified filterability apparatus described by Jennings[2].

The first two minute runnings are discarded and the volume of filtrate collected after a further seven minutes is recorded as the result.

Results and discussion

The results of tests carried out on raw sugars received at the refinery throughout the season are summarised in Table I. When it is considered that all the sugars, except those from Glendale, were classed as "high pol" (>99,0°S), it is apparent that there are marked differences in quality which are not reflected in the chemical analysis.

Most of the sugars received were processed directly, the balance being stored. In Figure 1 the weighted averages of the test results are plotted against the tons of melt per filter actually recorded in the refinery. The correlation between the two sets of data is significant at greater than the 99% level. When making the comparisons the considerations mentioned by Jennings[2] were taken into account.

There appears to be a definite peak in the carbonation performance of raw sugar in July-August. The reason for the seasonal fluctuation is not apparent.

Applications

Having established a meaningful test, it was decided to ascertain what effect varying conditions had upon the factory performance.

A quantity of standard factory slaked lime slurry

TABLE I

Summary of data. — Volume of Filtrate.

Mill	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Avg.
FX	62				40	33	15	31	36
DL				63	46	69	22	18	43
EM	20	26	39	83	51	33	24	13	36
MV	34	24	37			20	11		25
TSC	53	62	113	63	60	62	27	22	58
JB	46	44	44	51	42				47
GD	9	19	20	29	16	10		8	16
Weighted Avg.*	43	37	52	63	49	38	22	22	
Factory tons/filt.	15	18	23	29	24	16	12	12	

Correlation coefficient for 8 sets of data 0,94. Significance>99%.

*The weighted averages are based on tons of sugar received and not tons sugar melted. It is not possible to record exactly the tons of sugar from each source actually melted. The error involved in these comparisons will be small since most of the incoming sugar was processed directly.

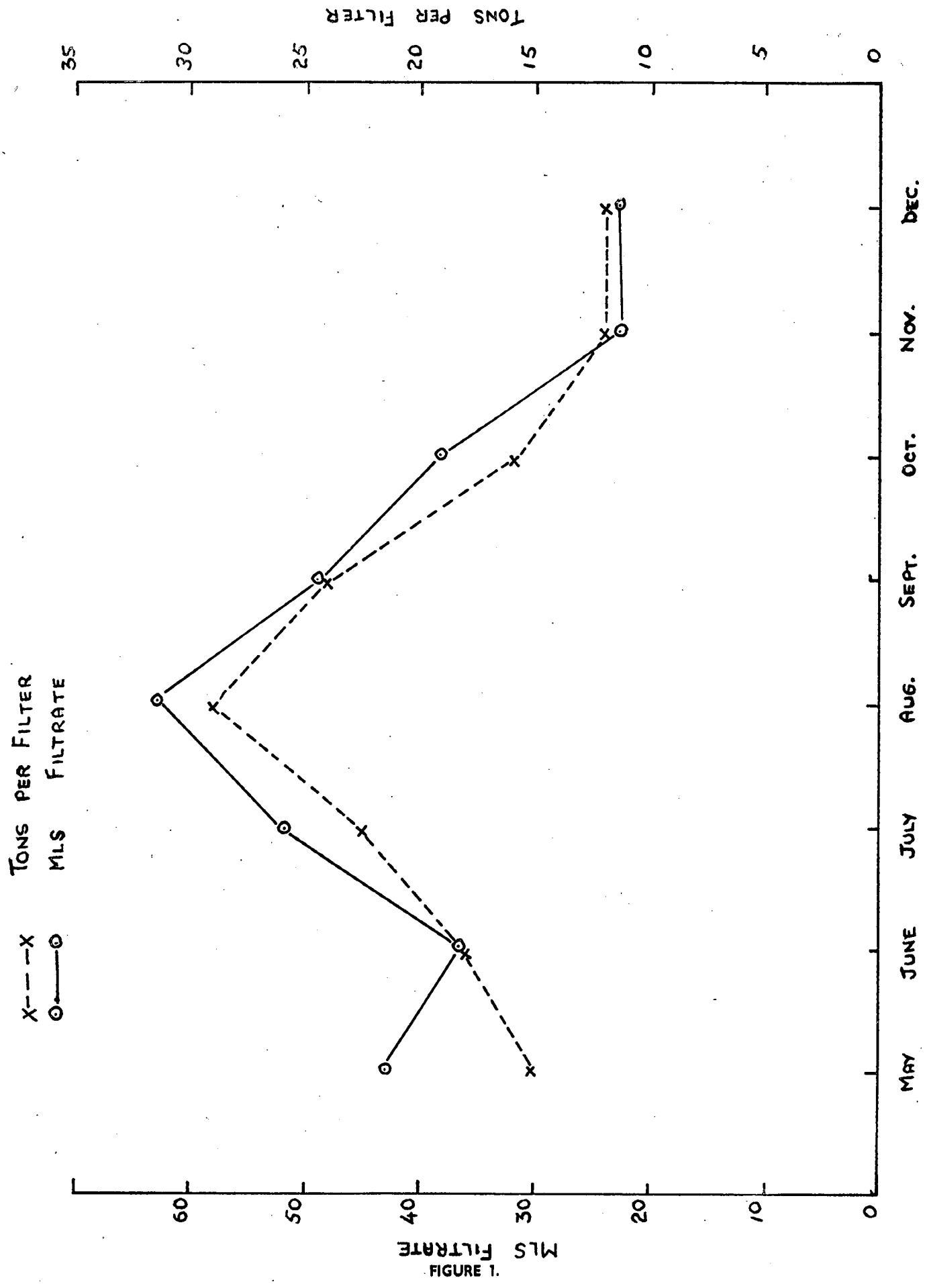
TONS PER FILTER
35
30
25
20
15
10
5
0
MLS FILTRATE
60
50
40
30
20
10
0
MAY
JUNE
JULY
AUG.
SEPT.
OCT.
NOV.
DEC.
X---X TONS PER FILTER
O——O MLS FILTRATE

FIGURE 1.

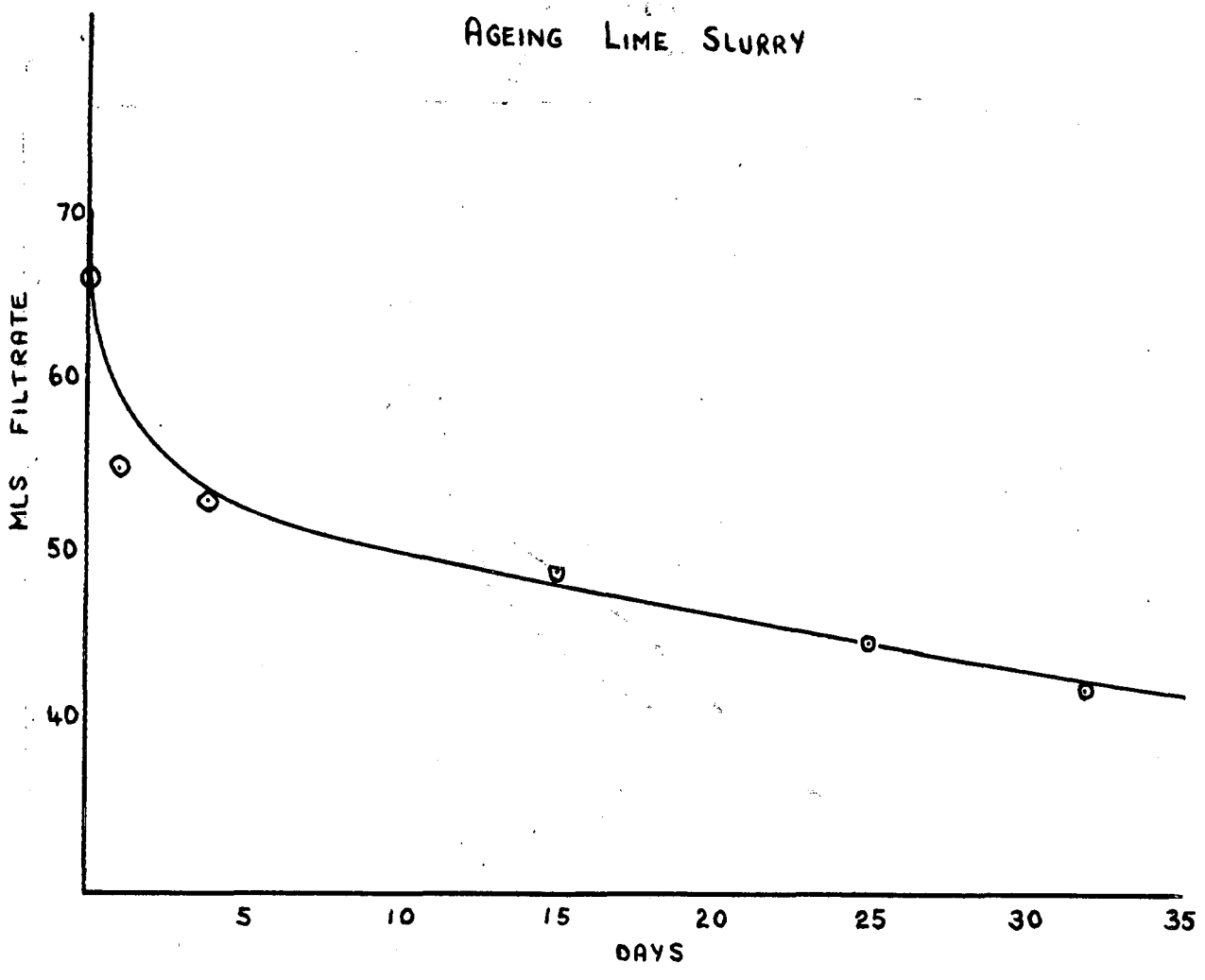

FIGURE 2.

S.G. 1,090 was taken and stored in a closed container for a total of 35 days. Sufficient for the test was taken and test results on standard sugar were plotted against time. To avoid the effect of ageing, previously aged raw sugar was used.

When examining the resultant curve, it can be seen that lime slurry deteriorates with time and that the greatest deterioration occurs within the first 48 hours after slaking.

The effect of increasing lime concentration was then studied. In this instance standard sugar and freshly prepared lime slurry were used.

From the plot of the results it can be seen that the effect of increasing the lime begins to diminish at a maximum value of about 1,2% CaO/solids. This observation is in keeping with the results obtained by Bennett[1], when comparing South African raw sugar and West Indian raw sugar.

Arising out of the fact that the refinery stores raw sugar for periods of up to 10 months before processing and that there is a definite deterioration in factory filter performance towards the end of the season when melting stored sugar, it was decided to study the effect of ageing raw sugar.

50 kg of raw sugar was taken and stored in the laboratory at 25°C ± 2° for seven months. Sufficient sample was extracted each month to perform the test.

Plotting volume filtrate against time, it can be seen that there is a definite deterioration in test performance. The greatest decrease occurring during the first month. This is in keeping with observations made at the refinery.

Summary and conclusions

The highly significant correlation coefficient found between tons/filter and tests results as shown in Table I and Figure 1 provide a strong indication that the test as outlined, can be considered to be of practical value. The relatively short time taken for the test (approximately 1 hour 20 minutes) lends itself to routine analysis.

The results of the applications have shown that some of the observations made at the refinery concerning seasonal fluctuations in carbonation performance, can be attributed in part, to the behaviour of the raw sugar in storage.

The full importance of these preliminary findings should be verified by further investigations. This method is specific to Huletts Refinery, however, it could readily be applied by other carbonation refiners adapting for local conditions.

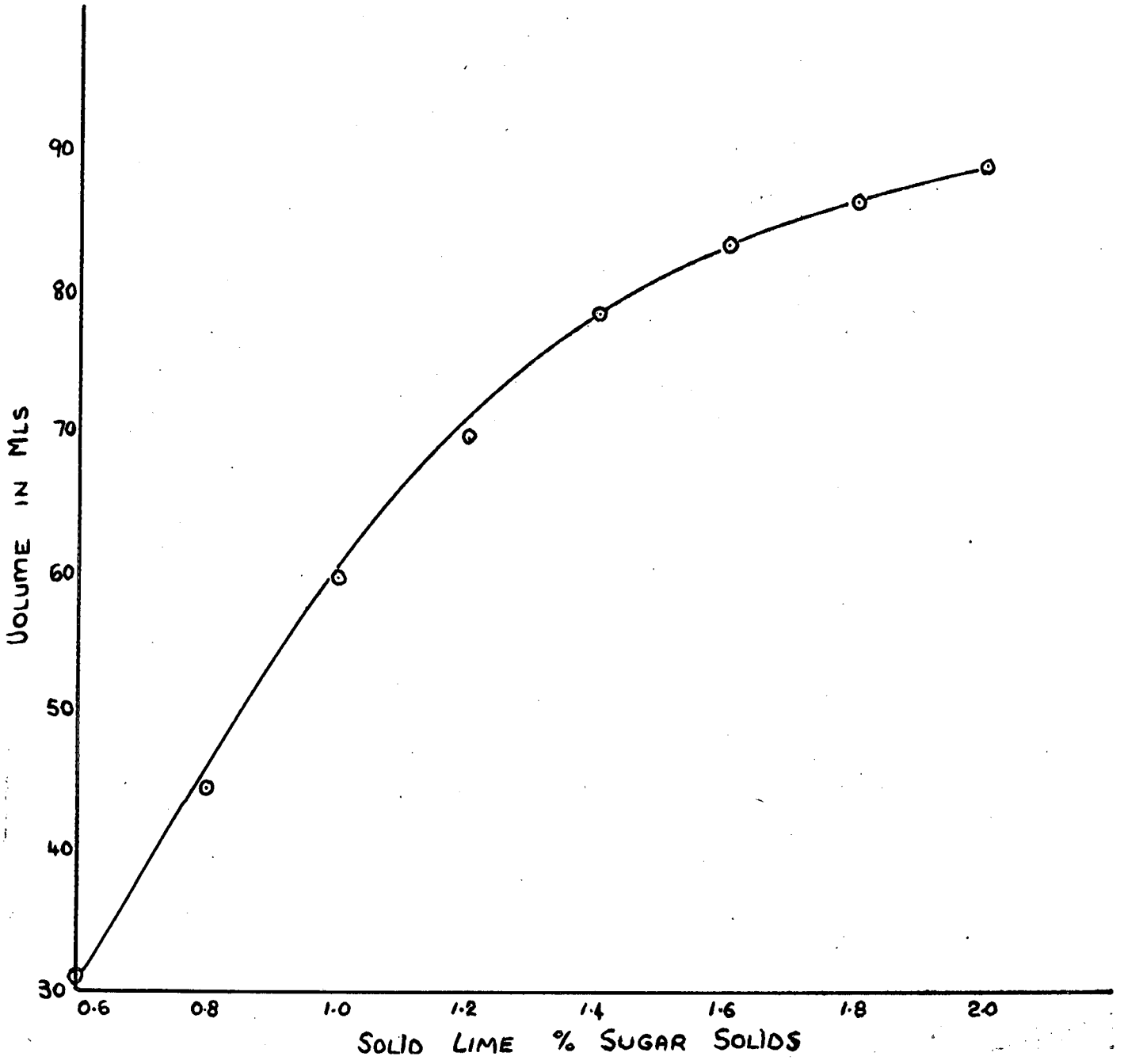
VOLUME IN MLS
90
80
70
60
50
40
30
0·6
0·8
1·0
1·2
1·4
1·6
1·8
2·0
SOLID LIME % SUGAR SOLIDS

FIGURE 3.

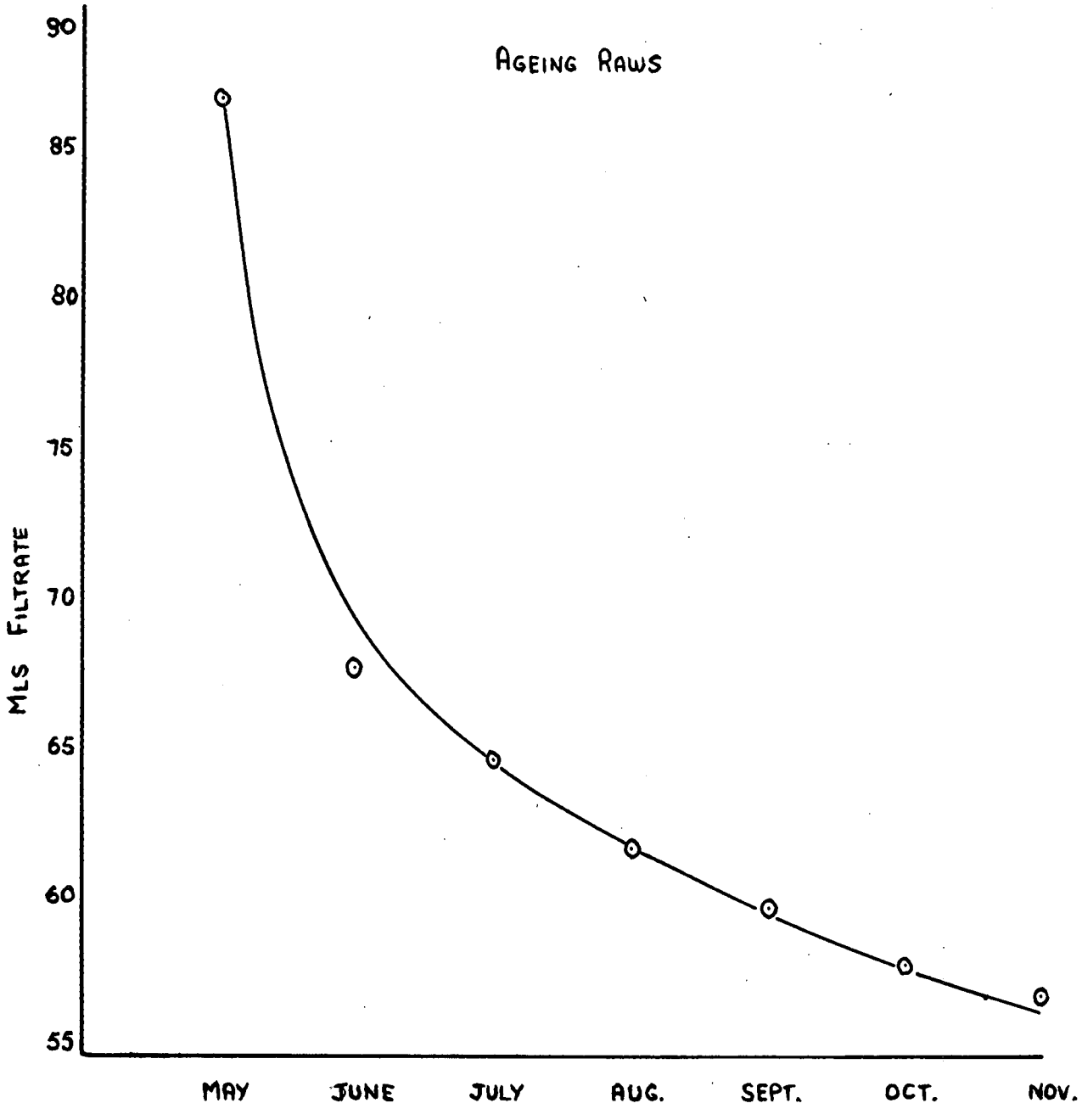

FIGURE 4.

ANNEXURE

Results of carbonatation test conducted in triplicate on three samples of unaffinated raw sugar.

Time in Mins.	Vol. in ml.			Vol. in ml.			Vol. in ml.		
3	12	12	12	12	11	11	17	17	17
6	41	41	41	39	38	38	54	53	54
9	65	66	66	63	62	62	86	85	85
12	85	88	89	84	82	82	111	110	111
15	100	103	108	101	98	98	130	131	131

Acknowledgements

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REFERENCES

1. Bennett, M. C., 1967. Liquor Carbonation, Part 1, Impurity Effects on Filterability. Int. Sugar J. 69, (1967), 101-104.
2. Jennings, R. P., 1966. A modified method for Determining Filterability. Proc. S. Afr. Sug. Technol. Ass., 40 (1966), 199-204.

Discussion

Dr. Matic: The S.M.R.I. tried for a long time to develop a laboratory test that would accurately reflect the behaviour of sugar in a refinery. Thought was given to a carbonatation test but difficulties arose as it is necessary that parameters be controlled extremely carefully in order to get controlled results. It is therefore surprising that this very simple method gives such good results.

Mr. Wilkes: The success seems to be due to the use of flue gas instead of pure CO_2, which cannot be used. The flue gas was piped to the laboratory from the factory and was then scrubbed.

Mr. Rault: For good results it is essential that carbonatation be carried out slowly.

Mr. Wilkes: Carbonatation takes about an hour and the filtration a further twenty minutes. In the factory the retention time is about an hour and a half.

Dr. Murray: The permeability of the carbonate bed depends on two things, namely, the viscosity of the solution and the leakage diameter of the crystals being formed. The former is very dependent on temperature and presumably this is controlled very accurately. The latter is a function of many factors, e.g. protective colloid action, false graining, etc. I would like to ask if you have examined the nature of the carbonate crystals formed to see if variations occur in size according to preparation conditions?

Mr. Wilkes: This test was originally intended merely to advise process managers how much lime to add to various sugars so the mechanics of the process have not been investigated.

Dr. Matis: If the trend shown in Figure 4 regarding ageing of sugars is correct then this could have serious implications.

Mr. Wilkes: The tests reflected in Figure 4 were done in triplicate but it is intended to investigate this further.

Mr. Cargill (in the chair): Is this test a better guide to the process manager than a filterability test?

Mr. Alexander: For a carbonatation refinery such as Hulsar this test should be better.

As regards age of sugar, the test should be applied to the sugar before and after affination because as far as we know most of the changes that take place in a raw sugar are in the surface layer around the crystal.

Mr. Jennings: The correlation between this test and Hulsar is definitely better than that between the C.S.R. test and Hulsar.

Dr. Tilbury: There is a possibility that microbiological deterioration of the raw sugar might be responsible too for the decrease in filterability after storage. From studies elsewhere it has become evident that the ERH of raw sugar is a critical factor governing its microbiological deterioration and if the ERH is below 0,75, microbiological organisms will not multiply dring storage except for a certain type of osmophilic yeast which is not regarded as a polysaccharide producer.

Mr. Alexander: The ERH of HP raws is about 0,7.

Mr. MacGillivray: We had figures of between 0,7 and 0,75 for V.H.P. sugar but there were only a limited number of measurements.