

LABORATORY CARBONATATION

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The shortcomings of the current filterability tests in predicting the filter performance of sugars in Hulett's Refinery have recently been shown (3, 4). It would seem logical for a carbonatation refinery to use laboratory carbonatation to predict the filterability of incoming sugars. Although very little has been published, work into carbonatation techniques has been done by Dr. T. Yamane (5) as well as Tate & Lyle (1).

It has been found that the flow rate of factory carbonatated liquor through filter cloth in a C.S.R. filterability apparatus, modified to operate at 80°C, gives a good reflection of actual factory performance (3). Laboratory carbonatation should therefore be able to predict factory performance, provided that a satisfactory, reproducible method, based on factory techniques, is used.

During preliminary work using a carbonatation tank borrowed from the Sugar Milling Research Institute, it was found that filterability results comparable with those of factory carbonatated liquor were obtainable only with a much higher lime/solids concentration than that used in the factory. The large amount of precipitate so formed, however, caused even the poorer sugars to behave well after carbonatation—in some cases better than factory carbonatated liquor. Since Yamane (5) has found a very good correlation between his laboratory carbonatation data and factory performance, even though the flow rate which results from laboratory carbonation is much lower than in the factory, it was decided to fix the lime at 0.8% on solids and collect sufficient data to compare results with factory carbonatation.

To enable several methods to be compared, a carbonatation apparatus was designed and built to allow six batches of liquor to be carbonatated separately. The design is based on that of the S.M.R.I. tank, but each unit has been scaled down to contain only 500 grams of liquor—sufficient for the filterability test. The units are cylindrical with a conical bottom into the apex of which gas may be introduced through a sintered metal disc. An outlet valve is provided for each. The six units are enclosed in a common water tank through which water at 80°C is circulated by means of a constant temperature water bath. Separate needle valves and flow meters control the flow of gas to each unit from a common supply. (See Fig. 1.)

Results achieved with this apparatus were found to be reproducible and comparable with those obtained using the S.M.R.I. tank.

In addition to fixing temperature and lime, gas flow rates were established to give a duration of gassing of 90 minutes as in the factory. A programme was then devised to compare filterabilities of labora-

tory carbonatated liquors with the filterability of corresponding samples of factory carbonatated liquor.

Melt liquor samples were taken in the factory at 15 minute intervals over four hours and composited. The brix was adjusted to 65° and the liquor divided into 500 gram aliquots. The samples were heated in the water bath to 80°C and lime slurry corresponding to 0.8% on solids added. The limed aliquots were transferred to the carbonatation tank at 15 minute intervals, gassed to pH 9.0 and filtered immediately.

Pure CO₂ was used in preference to an air-CO₂ mixture, as less frothing occurs with no apparent difference in results. Four different methods of gassing were used in the examination with duplication of each:

1. Constant rate gassing with slow stirring by means of a mechanical stirrer.
2. Constant rate of gassing but no stirring.
3. Two stage gassing, the second half at a slower rate.
4. Two stage gassing, the second half at a faster rate.

Filterabilities (2) of the carbonatated melt liquor obtained in this way were compared with the filterability of factory carbonatated liquor collected over a corresponding period of time.

Results (See Fig. 2)

In each case, a higher filterability was obtained when slow stirring was employed. A faster rate had been found to reduce filterability. In addition, the filtrates obtained were clearer than those from the unstirred batches.

In contrast, the best correlation coefficient, 0.78, resulted from unstirred, constant rate gassing, with 0.63 for the stirred carbonatation. The other two methods gave correlation coefficients of 0.64 and 0.66 respectively. The trends as shown in the graph are similar. Taking into consideration the fact that over the period during which these tests were run, difficulties were being encountered in the carbonatation process in the refinery, these results seem sufficiently significant to warrant further investigation. Testing will be continued during the year ahead to accumulate sufficient data for a full evaluation.

Acknowledgements

In conclusion, the author would like to express his appreciation to Messrs. Bowes and Grenfell of the Sugar Milling Research Institute for their assistance in the construction of the new carbonatation tank, and to Miss Laughton of Hulett's Research Laboratory who assisted with a lot of the testing. Thanks are also due to the Director of the Sugar Milling Research Institute for permission to use the facilities of the Institute, in particular the carbonatation tank.

Summary

Laboratory carbonatation as a means of evaluating the filterability of raw sugars received at Hulett's refinery is investigated. The design of a laboratory carbonatation apparatus is discussed and results of the first tests given.

References

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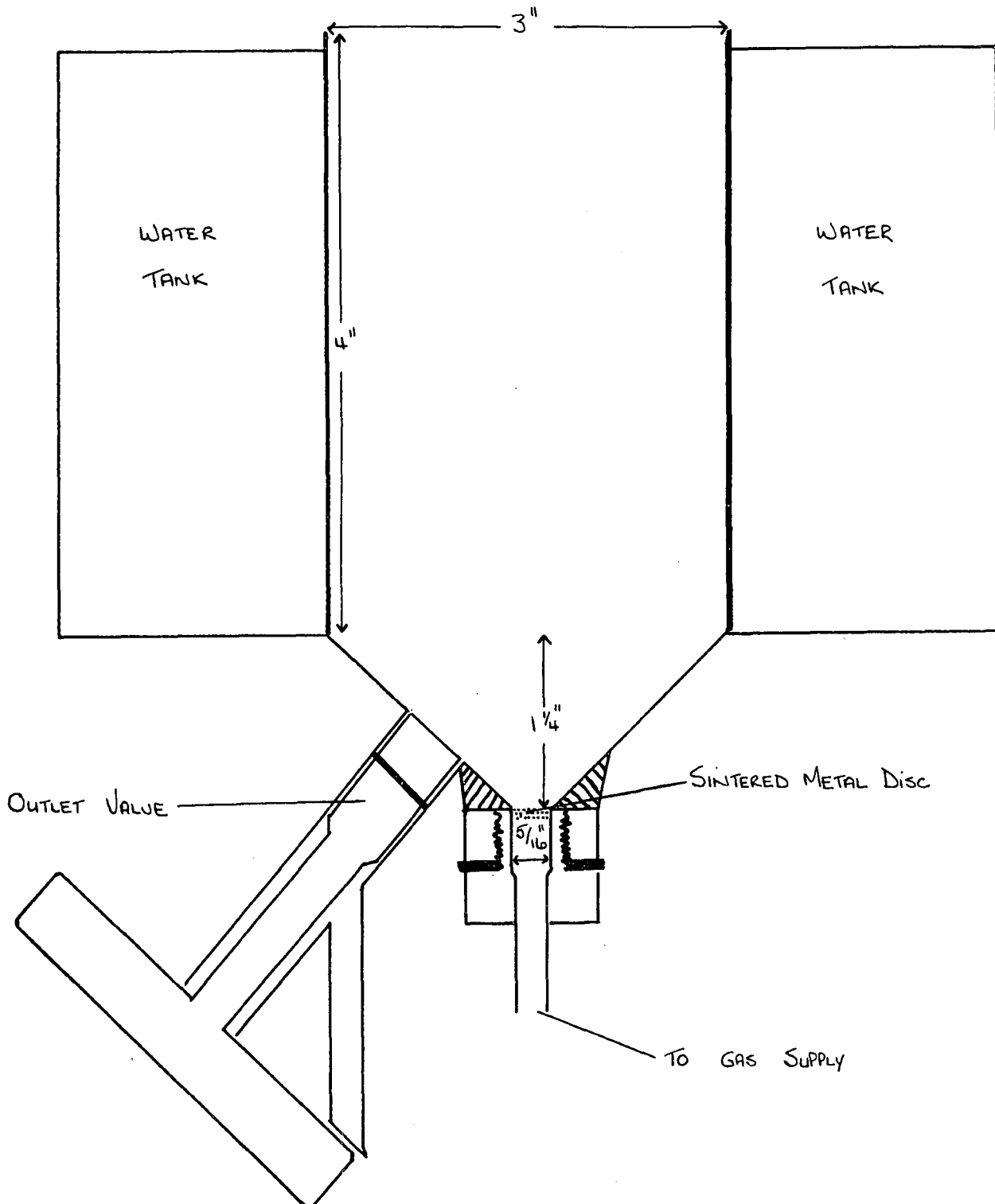


FIGURE 1

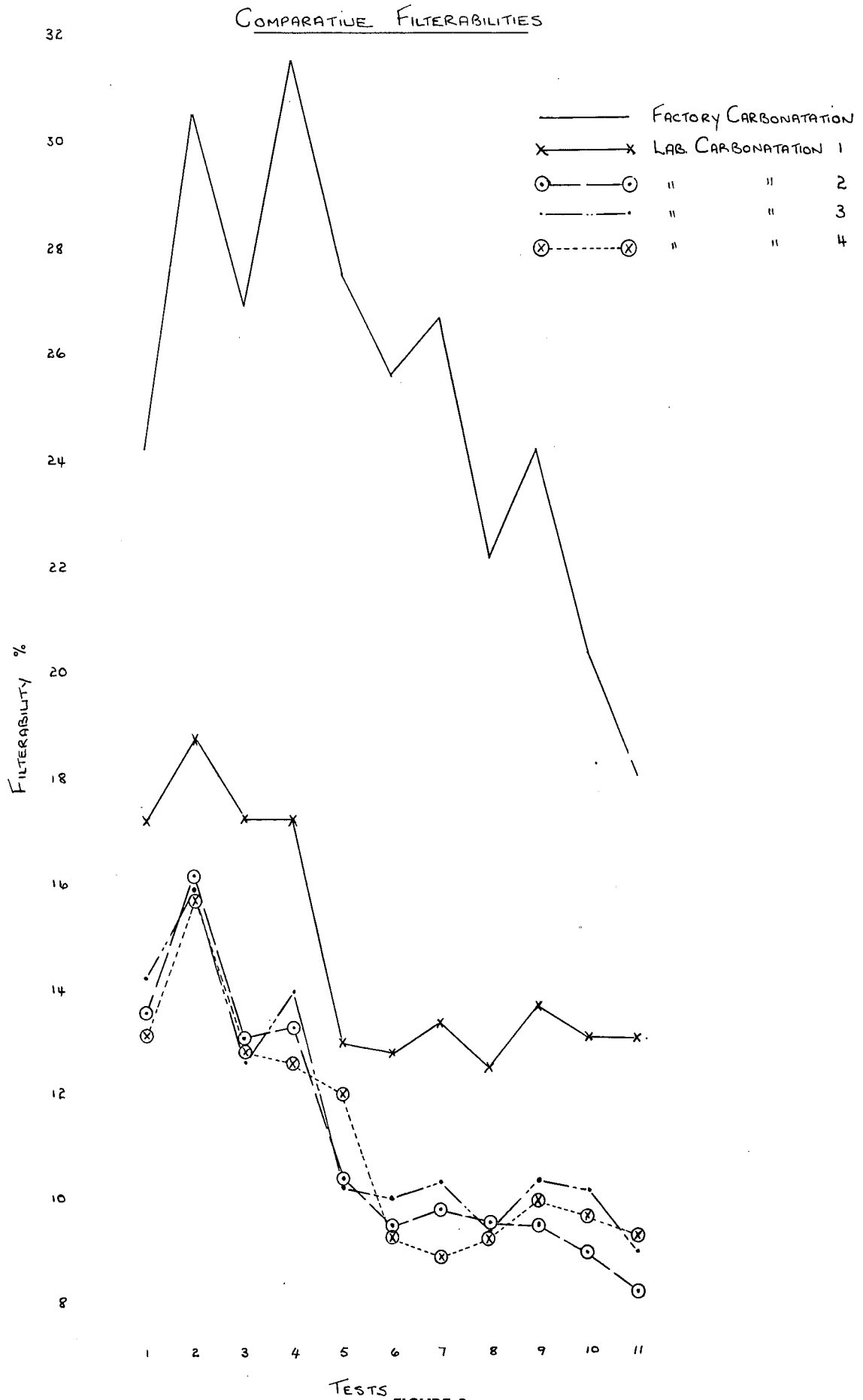


FIGURE 2

Dr. Matic: Yamane, using a rough method of carbonatation, obtained as good, if not better, correlation with filterability than Mr. Dawes. It would be interesting to know why different figures were obtained when other methods of carbonatating were used. Has any effort been made to determine the impurities arising from the different methods e.g. have filter impeding impurities like starch been determined? The correlation appears to be better at high filterabilities than low filterabilities.

Mr. Dawes: The factory carbonatated liquors used in the refinery were not always typical of the liquors produced, but work is continuing and we hope next year to get better correlation. No work has been done at this stage on correlating impurities with filterability.

Mr. Chiazzari: Why was filtration done at 80°C and why was gassing not done at the normally accepted temperature of 50°C? I note also that a pH of 9 was chosen instead of 8 for gassing down.

Mr. Dawes: The modified C.S.R. test was designed for 80°C and carbonatation is done at the same temperature, as below that we run into trouble with our filters. The average pH of the carbonatated liquors we used from the refinery was 9, although

sometimes it was as low as 8.4.

Mr. Alexander: Most filterability tests are carried out at a pH of 9 because this is the average figure for carbonatation refineries. Similarly, 80°C is the average temperature used for gassing in carbonatation refineries although we know that Tate and Lyle has gone as high as 90° with certain sugars. In South Africa the quality of the lime we use is important, with particular regard to the effect of manganese, and also the effect of oxygen on the reaction.

Mr. Chiazzari: Is there not some confusion between gassing temperature and filterability temperature.

Mr. Dawes: In the laboratory the test has been kept as simple as possible so as not to introduce other complications.

Mr. Buchanan: It appears that agitation rate affects filterability and it would be interesting to know what agitation rate was used.

Mr. Dawes: When a high rate of stirring was used results were poor but they improved when the rate was reduced. The average rate was 100 r.p.m. using a small stirrer. We tried to get a series of results at one constant rate of stirring—at this stage various rates have not been investigated.