SOME NOTES ON FILTERABILITY TESTS ON A, B AND C RAW SUGARS AT SEZELA: 1964/65

by E. DEDEKIND

Introduction

The Sezela factory is equipped to refine the whole of its raw sugar output consisting of single cured A and B, and double cured C sugars.

Graining is done with virgin syrup and the volume of footings for B and C massecuites is regulated to suit the purities. The crystallizers for all grades of massecuites are equipped with cooling coils.

The 1000 r.p.m. 42 in. x 24 in. A centrifugals, 1500 r.p.m. 42 in. x 24 in. B set and the 1500 r.p.m. 42 in. x 24 in. C afterworkers each receive 1.25 gallons of superheated water per cycle. Classification of molasses is practised according to the purities.

General

During the 1964/65 season numerous filterability tests on single cured A and B and double cured C sugars were carried out in our laboratory. The equipment used was the “C.S.R.” type apparatus. Snatch samples were taken from the respective centrifugals, and these fresh sugar samples were analysed for Pol and the filterability determined. The purities of the massecuites from which the samples were taken were noted, and a summary of these analyses is tabulated in Table 1.

Monthly composite samples of raw sugars were analysed by the S.M.R.I. and their results were published under “High Polarizing Raw Sugars” in their monthly report.

Table II shows the S.M.R.I. results in comparison with the Sezela tests.

Table III reflects the results of the S.M.R.I. three-monthly analysis for impurities.

<table>
<thead>
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<th>TABLE I</th>
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<tr>
<td>MONTH</td>
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<tr>
<td></td>
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<tr>
<td>April</td>
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<tr>
<td>May</td>
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<td>November</td>
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N.B.—Samples shown for December represent Export Sugars only, the Refinery was closed on the 28th November.

On perusal of Table I it will be observed that the filterabilities of all sugars show a drop from July onwards, and no explanation can be given further than that throughput was increased, consequently the centrifugals were set on a shorter spinning cycle. Clarifier capacity was strained, and the pans were required to boil a larger volume of massecuites. The individual results of B and C sugars during November show a remarkable variation. B sugars varied from 56% down to 7%, and C’s from 46% to 7%.

It is interesting to observe that after a prolonged factory shut-down from Friday 26th October to Monday 29th October for shortage of cane due to heavy rainfall with resultant flood damage to railway lines, B sugars filterability dropped as low as 8.8% and that of C sugars 5.5%. This is proof that deterioration of thin juices, syrup and treacle has an adverse effect on the filterability of sugars.
Proceedings of the South African Sugar Technologists' Association—March 1965

TABLE II
Monthly Analysis

<table>
<thead>
<tr>
<th></th>
<th>SEZELA</th>
<th>S.M.R.I.</th>
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<tbody>
<tr>
<td></td>
<td>FILTERABILITY %</td>
<td>FILTERABILITY %</td>
</tr>
<tr>
<td>April</td>
<td>72</td>
<td>53</td>
</tr>
<tr>
<td>May</td>
<td>69</td>
<td>58</td>
</tr>
<tr>
<td>June</td>
<td>60</td>
<td>51</td>
</tr>
<tr>
<td>July</td>
<td>56</td>
<td>48</td>
</tr>
<tr>
<td>August</td>
<td>38</td>
<td>43</td>
</tr>
<tr>
<td>September</td>
<td>48</td>
<td>50</td>
</tr>
<tr>
<td>October</td>
<td>54</td>
<td>49</td>
</tr>
<tr>
<td>November</td>
<td>36</td>
<td>49</td>
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<tr>
<td>December</td>
<td>49</td>
<td>58</td>
</tr>
<tr>
<td>January, 1965</td>
<td>55</td>
<td>50</td>
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The August sample shows high starch content and low filterability, whereas in Table I the individual results of A, B and C show a fairly good result. It must be assumed that the composite sample deteriorated in storage. The S.M.R.I. reported that the December sample had apparently deteriorated, but the sample, nevertheless, showed a good filterability of 58%.

TABLE III
S.M.R.I. Results of Three-Monthly Composite Samples

<table>
<thead>
<tr>
<th>Month</th>
<th>Wax p.p.m.</th>
<th>Gums p.p.m.</th>
<th>Starch p.p.m.</th>
<th>SiO₂ p.p.m.</th>
<th>P₂O₅ p.p.m.</th>
</tr>
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<tbody>
<tr>
<td>May/June/July</td>
<td>20</td>
<td>600</td>
<td>275</td>
<td>65</td>
<td>5</td>
</tr>
<tr>
<td>Aug./Sep./Oct.</td>
<td>30</td>
<td>700</td>
<td>340</td>
<td>60</td>
<td>10</td>
</tr>
<tr>
<td>Nov./Dec./Jan</td>
<td>10</td>
<td>610</td>
<td>205</td>
<td>65</td>
<td>5</td>
</tr>
</tbody>
</table>

It is an acknowledged fact that the cane reaches its peak both in purity and maturity about September/October. On perusal of Table III, it will be noticed that the sugars for this period showed high wax, gums, starch and P₂O₅ content. This in itself is remarkable, and one can only conclude that this was the result of the severe winter conditions experienced during the 1964 winter season.

Acknowledgments

Acknowledgments are due to the Sugar Milling Research Institute for the assistance given in the analysis of monthly samples sent to them, and to the laboratory staff at Sezela for the efficient manner in which they have carried out these tests.

Dr. Douwes Dekker (in the chair): We have heard three papers on the impurities present in our raw sugars and on filterability.

Probably the most vexing problem of our industry is the quality of our raw export sugars.

There is some slight danger in these papers in so far as confusion might arise about the word filterability. We have various methods for determining filterability, one — the method used at the S.M.R.I., which is a method based on the original way in which the C.S.R. Co. determined filterability, two — a shorter method also developed by the C.S.R. Co. and which gives us results which are higher, three — the shorter C.S.R. Co. method carried out at a higher temperature. There is also the danger that we are comparing filterabilities of sugars which have been affiliated with those that have not been affiliated. It is necessary that people working on this subject should decide what method will in future be used, because at present one can be confused and possibly draw wrong conclusions from figures that are published. The method we now use in the laboratory has been standardised and is supposed to give an indication of how a sugar will behave in the refinery.

Mr. Boyes: Some years ago the S.M.R.I. published figures showing very good gum removal at Natal Estates using the carbonatation process. Mr. Jennings mentions in a table in his paper that gum percentage in liquor going to the filters was .222 per cent and coming away .182 per cent. We see also that the starch was .082 per cent and in the filtered liquor .047 per cent. The carbonatation process seems to have removed 50 per cent of the starch which ties in with a similar reduction in the gums. Does the carbonatation process remove starch free gums?

Mr. Jennings: Carbonatation appears to remove starch in preference to starch free gums; in fact negligible quantities of starch free gums are removed.

Dr. Douwes Dekker: We are talking about the effect of carbonatation on the removal of starch and gums. Confusion is again likely to be caused here because carbonatation processes differ, particularly between a raw sugar factory and a refinery. The conditions under which carbonatation is carried out in a refinery determines how much starch and gums are removed. Dr. Bennett of Tate & Lyle visited the S.M.R.I. recently and he said that by modifying the conditions of the carbonatation he could influence quite considerably the amount of impurities removed. Great importance is attached to the size of the calcium carbonate particle.

Mr. Alexander: In a carbonatation factory, where the process is carried out at 15° Brix, a considerable amount of starch and gums are in insoluble form and are therefore more easily removed than in a refinery where the Brix is 60°.

Dr. Douwes Dekker: Mr. Jennings indicated that gums can be both laevo- and dextro-rotatory.

Mr. Bruijn: We have had experience of gums formed in deteriorated cane which are different from gums in fresh cane. In raw sugar a mixture is found. If gums from fresh cane are hydrolysed fructose, glucose, galactose and some unknowns are found. The presence of fructose indicates that some of the gums will be laevo-rotatory. Fractionation by alcohol tends to fractionate into molecular sizes and not into different types of gums.
Mr. Pole: Work should be done on the effect of gums in raw juice on the total sucrose balance. It may be necessary to remove gums before a sucrose analysis is made of mixed juice.

Mr. Brujin: If gums are added to sucrose you get a polarisation of about 2.8 times the polarisation of sucrose and this is not removed by the lead clarification method followed for raw sugar. If double polarisation is used, gums are not inverted and are thus corrected for. It is extremely difficult to hydrolise gums.

Dr. Douwes Dekker: If we keep to the Clerget method then the data is not affected by the amount of gums present in mixed juice. As we now know more about the effect of gums on direct polarisation, this can be used as an argument in favour of retaining our present method of determining sucrose in mixed juice.

Mr. Alexander: It should be remembered, however, that refinery payments for raw sugar are based on direct polarisation and not double polarisation methods. The quantity of gums in the sugar thus affects the refinery considerably.

Mr. du Toit: The matter of filterability, gums and starches is very complex and it is easy to draw the wrong conclusions. Mr. Dedekind indicated when he was working with stale cane that there are important factors other than starch and gums which affect processing. At the agricultural session yesterday Mr. Johnson stated that in India it had been found that when old cane was harvested it deteriorated more rapidly than young cane.

Referring to Mr. Dedekind’s paper, Table 1, very good filterabilities appear to have been obtained using the sulphitation method.

It seems from a graph in Mr. Jenning’s paper that starch has a much greater effect on filterability than gums. But when these impurities are low, then do not gums have a greater effect than starch?

Mr. Jennings: The starch used in the particular experiment to which the graph refers was not cane starch.

Dr. Douwes Dekker: Mr. Brujin, in his work on deteriorated cane, has found two immediate effects appearing, namely, inversion of sucrose and disappearance of starch, probably due to enzymes in the juice. At a later stage the effect of micro-organisms is felt and gums are formed.

Regarding sulphitation, experience shows that this is a better method than defecation, mainly because it removes more P2O5 and wax. At present we prefer remelting to sulphitation as it is cheaper.

Mr. Main: Difficulty in getting adequate coal supplies is one reason why remelting is not always possible.

Illovo crushes cane grown at a long distance from the mill and a marked deterioration has been noted as the season progresses.

The remelting of A sugars was considered, but the cost was prohibitive.

Should we not stop considering only remelting as regards filterability and rather investigate new clarification methods.

Mr. van Hengel: It has been calculated that if B and C sugars are remelted, using coal it would cost 32 cents per ton against 75 cents per ton for sulphitation. If part of the A sugar was also remelted, the sugar having first been double cured, it would cost 52c per ton to remelt.

The average brix of syrup in Natal this year was 58.77°. If this is increased to 65° in quadruple effect then the evaporation of the amount of water in single effect necessary for the remelt of the sugar is almost covered. Of course strict control of the pans is necessary.

Regarding the filterability of sulphitation sugars at Sezela, which looks so good, it must be pointed out that these figures refer to sugars boiled from a sulphitated mixed juice mixed with about 20 per cent of the return of the refinery, which is remelted A sugar.

At Natal Estates B and C sugars are melted in addition to sulphitation and yet the results are not much better than Melville where just remelting of C and half B sugars is carried out.

Dr. Douwes Dekker: Mr. Main can rest assured that the question of clarification is being studied from many angles. It must be stressed, however, that clarification processes should be carried out correctly, e.g., correct temperatures and pH, etc.

P2O5 content of our juice is low and in an experiment at Umfolozi the addition of P2O5 gave better filterability. The S.M.R.I. has asked factories to report regularly the P2O5 contents of their juices.

Filterability problems differ in each country. In Louisiana for instance, the problem is gums and a Committee has been formed to study gums. Peru has trouble with a high ash content.

Mr. Main: I believe that some years ago a consignment of raw Cuban sugar went through Hulett’s refinery without giving any trouble at all.

A little later a refined sugar which had hardened during storage was reprocessed and caused terrible trouble with the filters.

Mr. Young: Difficulty was found in filtering this sugar and our assumption was that there were insufficient impurities to form a suitable floc for filtration.

Mr. Eddings: If a large amount of reject refined sugar is remelted we usually have difficulties, probably for the reason given by Mr. Young.

We have also noticed that raw sugars that are known to process badly do improve a little if left in storage for some months before refining.

Mr. Dedekind: With reference to Mr. van Hengel’s remarks, I must point out that the proportion of return from the refinery that is mixed with the sulphitated mixed juice is only 10 per cent to 12 per cent, not 20 per cent. In table 1 in my paper it will be seen
that in December the filterabilities were still good despite the fact that the refinery was closed. C massecuites are not boiled with the return from a refinery with a purity of 93 per cent.

At Sezela all graining is done on virgin syrup, even for C massecuites, which possibly accounts for the good filterability. If we did not use the sulphitation method with our raw sugars we might have to use activated carbon in the refinery, with a consequent increase in costs.

Mr. Perk: Umfolozi produced the best refined sugar in the country from a defecation process without using activated carbon in the refinery or sulphur in the mixed juice.

Mr. Rault: In the carbonatation process the more lime that is used the more decrease there is in gums. Starch tests unfortunately were not carried out at Natal Estates, but we do know that even while we were getting good filterability there was starch present. The more lime that was used, the better was the filterability, but of course the cost factor enters into this.

Mr. Johnson: We found in India some years ago that there was a definite connection between filterability and age of cane, variety of cane and time between harvesting and milling. Co. 281 was the early season cane at the time and it was being replaced with Co. 527, the juice from which filtered badly and was extremely low in \( P_2O_5 \). Cold liming was used and at the same time the addition was made of six pounds of 45 per cent triple super phosphate which gave a heavy precipitate which presumably carried down a certain amount of gums in the juice. I am referring now to a double carbonatation process producing plantation white sugar. If Co. 527 and Co. 281 were mixed in the proportion of 40 to 60 before milling, then there was no trouble with processing.

In the extremely hot humid climate in India deterioration of cane after harvesting was very rapid. Twelve to fourteen months cane, properly topped, did not deteriorate too badly, but eighteen to twenty months cane, which had probably lodged anyway, deteriorated very rapidly and if it could not be milled quickly it paid us to burn it instead.

The problem of varieties was most important and the agricultural programme was adjusted accordingly.

Mr. du Toit: Regarding phosphate in juice, if nitrogen is added to cane the phosphate content of the cane stalk is reduced drastically. In the last ten years nitrogen consumption per unit area has increased enormously in the sugar belt and at the same time the amount of phosphate applied has decreased. Therefore the nitrogen to phosphate ratio has increased sharply and consequently the phosphate content of the juice has been lowered. It is interesting therefore to note that by adding phosphate to the juice, a better filterability is obtained.

Mr. Bruijn: The gums precipitated by Mr. Jennings were later added to raw sugar. Has he an analysis of the purity of the gums — were they mainly carbohydrate or were there other impurities? I have not been able to make a completely pure gum, there are usually traces of nitrogen and ash.

Mr. Jennings: The gums did contain a certain amount of impurities. Mr. Bruijn mentioned that gums polarised at 2.8 times the polarisation of sucrose — were the gums from deteriorated cane?

Mr. Bruijn: Yes.

Dr. Dick: Is there any correlation between the amount of gums and the filterability of the juice and the corresponding figures for the raw sugar?

Dr. Douwes Dekker: In Queensland the relationship between filterability of clarified juice and of the resultant raw sugar has been investigated, but no correlation was found as too many other factors were involved.

To what extent can the presence of gums in our juice be attributed to damage to cane by insects?

Dr. Dick: Many years ago Mr. Dymond wrote a paper in which he blamed the poor filterability of juices on mealie bugs.

If he was right there should have been an improvement over the years because the numbers of mealie bugs have decreased.

Of course, evidence for this may not be available because so many other factors have also changed.

Dr. Douwes Dekker: There have been too many other changes for a firm conclusion to be drawn.

Mr. Young: Has filter station performance been related to laboratory filterability? Work done per filter cycle can be measured, and this depends on filterability and on degree of blinding of the cloth and other factors. The method of carbonatation also has an effect.

Mr. Dedekind: We have to use a shorter cycle when filterability goes down. Last season, using a light cloth, blinding did occur, but throughput was also increased.

Mr. Jennings: A considerable number of tests have been carried out at the refinery to relate the three methods for determining filterability and also in determining the filtration rates of carbonated liquor using the C.S.R. apparatus. Comparing the S.M.R.I. method, the two C.S.R. methods and the filtration of the carbonated liquor the correlation is very good between the four filtering rates. However, it has proved practically impossible to relate these figures to refinery performance.

Mr. Chiazzari: At Gledhow we have not been able to relate laboratory figures to refinery figures. The refinery has never been held up, however, due to filterability troubles. We have found it very difficult to actually measure a rate with our six filters. We do, however, invariably have some trouble in September which we try and counteract by increasing the CaO and also the temperature of the final liquor. We may also decrease the cycle.

In September we also get foaming in the saturators.
At the beginning of the season our recovery is good, it deteriorates in winter and picks up again in September after spring rains. I can only think the reason is deterioration of cane. In winter there is a stock pile of cane and deterioration sets in.

In connection with the refining of Indonesian sugar at Huletts refinery, was any alteration in recovery noticed over that period?

Mr. Alexander: The purity of final molasses did go down, despite extra impurities in the Indonesian sugar owing to its low pol.

Dr. Douwes Dekker: I do not like the word "recovery", and would prefer to know what the losses are — undetermined, in filter cake and in final molasses.

Mr. Dedekind: Sezela has also, like Gledhow, suffered from frothing in September and used glycerol monostearate to stop it.

You will see from Table 3 in my paper that in September when the cane is in its best condition, wax, gums and starch are up. Our losses in final molasses and our undetermined losses go up at this time of the year.

Mr. Perk: It is noticeable that all factories have good Boiling House Performances at the beginning of the season, but in the middle of the season they start falling off.

Dr. Dodds: Is it possible to classify cane varieties according to starch and gums content? When Uba formed a large proportion of the crop it was considered to have a very high starch content.

Dr. Douwes Dekker: It is proposed to start an investigation into the starch and gum contents of juices of various varieties and to then go a step further and boil sugars from these juices in a laboratory pan and compare the filterabilities.

Mr. du Toit: Starch figures have been given for the different varieties.

Dr. Douwes Dekker: The papers have provoked an extremely long and interesting discussion and have provided us with some very important data.