SOME NOTES ON THE PRINCIPLES OF OUR MANUFACTURING PROCESSES.

By K. DOUWES-DEKKER.

Sugar manufacture has grown through the ages from a small hand-worked process into a modern large-scale industry working on principles developed to a stage of great reliability by long experience and by constant scientific and industrial research.

But if we compare sugar manufacture with some other modern chemical industries, e.g. oil refining, plastics, vitamins and antibiotics and others, which make wide use of the techniques of high pressure and vacua, extreme temperature limits and plant constructed of the best and newest materials, we may conclude that the scientific principles of these industries are even more extensively studied and better known than those of our good old sugar industry.

There seem to be two explanations for this divergence. In the first place, the raw material to be worked in the mills is of an extremely complicated structure which varies from seasonal effects and from place of growth. This complexity of the juice makes it often difficult to generalise and to systematise our knowledge of the behaviour under different conditions of alkalinity and temperature.

In the second place, we have to take into consideration that many of our manufacturing processes have gradually and empirically developed to a stage which seem to yield satisfactory results. Granting this, the question “Why conduct expensive investigations?” may be fairly asked.

It is the aim of this paper to discuss a few principles underlying the different manufacturing processes and to see how well defined modifications of these principles can lead to new ideas and new applications.

We all know that the normal course of the manufacturing process is: juice clarification; concentration; crystallization; and we are inclined to accept this sequence as logical and therefore correct. But if, however, we observe the heavy scaling of some evaporator tubes, or the excessive degree of turbidity of syrup and first and second molasses, or the stickiness of the last boilings, some doubt is apt to arise about the correctness of the application of the juice clarification method when this juice is at its maximum dilution. We are inclined to ask: “Can this flocculation of non-sugars in evaporators and vacuum pans be prevented by a clarification at a higher brix?”

Such considerations, based on the simple principle that, with less water, less non-sugars can be dissolved, have led to experiments with the aim of finding a clarification process at a higher density. From these experiments a process has evolved, known in Java under the name of midsap clarification, and it is used in one of the larger mills. As originally applied, the mixed juice was limed at the boiling point to pH 7.2 and subsequently settled in the usual way. In order to eliminate the filter presses the sludge was returned to the mills and spread over the bagasse apron from the second mill. The clear juice was, as usual, pumped into the quadruple and concentrated, with this difference, however, that the juice from the third body, at a brix of 40°to 45°, was taken from the evaporator and, after being cooled to 130°F., subjected to the double carbonatation process.

The pH of the double-filtered juice, being about 8.7, was then reduced with SO₂ to about 6.9 and the juice was further concentrated in the fourth body to normal syrup density. By this method, based on the principle that at higher concentration more non-sugars are removed per unit weight of brix, with about two-thirds of the amount of lime usually required for the carbonatation of mixed juice, a higher rise in purity and a corresponding increase in recovery was attained. In this respect the midsap carbonatation process was certainly satisfactory.

There was one difficulty, however; the colour of the sugar was somewhat disappointing. This was believed to be due to the long time (1 to 1½ hours) that the hot limed juice was allowed to settle and an improvement of the process was suggested by omitting the settling of the neutralised mixed juice. In this modification, the limed, unsettled, and therefore very turbid mixed juice was to be concentrated to 40° to 45° brix, carbonated and filtered. The obvious objections were the anticipated heavy scaling of the evaporator tubes and the possibility of very difficult filtration of the mixed defecation and carbonatation precipitate from the concentrated and therefore somewhat viscous midsap.

Pilot plant tests showed, however, that the turbidities of the juice, instead of scaling the evaporator tubes, kept them clean. The speed of filtration of the mid-sap also appeared to be reasonable. As a consequence of these results, the new process was about to be applied on factory scale when the war broke out.

From these results it may be concluded that clarification at a higher brix deserves to be con-
A more important discovery, however, was that, even if a completely brilliant syrup was worked, the amount of non-sugar precipitated during the crystallization process in the first, second and third boilings exceeded many times the weight of non-sugars which had previously been removed by separation from this syrup. For example, in a carbonatation mill, the following figures were found:

\[
\begin{array}{|c|c|}
\hline
\text{Material} & \text{Precipitated non-sugar/} \\
 & \text{dissolved non-sugar.} \\
\hline
\text{Syrup} & 89.4 \times 10^{-6} \text{ g} \\
\text{First molasses} & 73.9 \times 10^{-6} \text{ g} \\
\text{Second molasses} & 53.8 \times 10^{-6} \text{ g} \\
\text{Third molasses} & 46.1 \times 10^{-6} \text{ g} \\
\text{Final molasses} & 32.9 \times 10^{-6} \text{ g} \\
\hline
\end{array}
\]

indicating an enormous flocculation during the second boiling.

Sulphitation syrup usually contains more turbidities than carbonatation syrup; but, while the increase in suspended matter is considerable, it is relatively not as well marked as in carbonatation syrup, as is shown by the following figures:

\[
\begin{array}{|c|c|}
\hline
\text{Material} & \text{Precipitated non-sugar/} \\
 & \text{dissolved non-sugar.} \\
\hline
\text{Syrup} & 83.8 \times 10^{-6} \text{ g} \\
\text{First molasses} & 63.2 \times 10^{-6} \text{ g} \\
\text{Second molasses} & 54.5 \times 10^{-6} \text{ g} \\
\text{Final molasses} & 34.4 \times 10^{-6} \text{ g} \\
\hline
\end{array}
\]

Improvement of the properties of the lower-grade boilings being the aim of centrifugal separation, it appears to be much more profitable to apply this process to the second and first molasses than to syrup. This means a considerable reduction in the capacity of the apparatus needed to obtain the same effect.

It seems obvious that the increase of precipitate in the lower-grade molasses cannot be ascribed only to the reduction of the amount of water in which they were dissolved. The process probably should be considered as a flocculation of suspended colloidal matter, which is governed by other rules than the simple decrease of solubility. Of the composition of the precipitate, typical analytical figures from sulphited syrup of two sulphitation mills are:

<table>
<thead>
<tr>
<th>Per 100 carbonated ash</th>
<th>Per cont.</th>
<th>Per cent.</th>
<th>Insoluble in cont.</th>
<th>Insoluble in cent.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture ...</td>
<td>35.48</td>
<td>24.56</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbonated ash</td>
<td>8.97</td>
<td>10.18</td>
<td>SiO₂</td>
<td>0.93</td>
</tr>
<tr>
<td>Ignition loss</td>
<td>55.55</td>
<td>55.76</td>
<td>Fe₂O₃ + Al₂O₃</td>
<td>5.23</td>
</tr>
<tr>
<td>Polarization</td>
<td>41.2</td>
<td>34.8</td>
<td>CaO</td>
<td>5.13</td>
</tr>
<tr>
<td>Wax ...</td>
<td>0.44</td>
<td>1.44</td>
<td>MgO</td>
<td>traces 0.97</td>
</tr>
<tr>
<td>Pentosans</td>
<td>0.72</td>
<td>1.01</td>
<td>SO₃</td>
<td>4.57</td>
</tr>
<tr>
<td>Grade albumen</td>
<td>1.52</td>
<td>1.93</td>
<td>PO₄</td>
<td>2.34</td>
</tr>
</tbody>
</table>

From the results of the analysis of the suspended matter in first and second sulphitation molasses it would further be concluded that the precipitation...
of SiO₂ and CaO tended to increase during the crystallization process. The percentage of organic lime salts also increased and the organic acid was probably aconitic acid.

Removal of suspended matter not only facilitates the boiling process, but also improves centrifugal efficiency, as was shown by a simple experiment.

Two portions of dry refined sugar were mixed with (a) an untreated final molasses; (b) with the same final molasses, from which, however, turbidities had been removed by separation. Next both artificial massecuites were centrifuged under similar conditions, resulting in sugars with polarizations of 85.5° and 91.0° respectively.

Summarizing, it was proved by the discussed investigation that the removal of suspended matter by centrifugal separation from first and second molasses resulted in a smoother boiling, in a more regular grain and in a better centrifugal efficiency. Whether or not these advantages are sufficiently important to warrant the installation of the rather expensive and rather powerful separators should be decided on the merits of each case. The separation of syrup appeared to be of low efficiency compared with the separation of second and first molasses, since proportionally large amounts of non-sugars are precipitated during the crystallization process.

Having demonstrated that there are some advantages in changing the normal manufacturing sequence of clarification—concentration—crystallization into concentration—clarification—crystallization, or into clarification—concentration—second clarification—crystallization, we may also ask if it is necessary to keep the crystallization as the final process. The answer seems obvious: since we want a crystallized product, the crystallization process must be the last. But this answer does not cover the whole field. We all know that it is extremely difficult to manufacture, directly from mixed juice, a mill white sugar as pure as a refined sugar, even if such processes as ion exchange and decolorisation of syrup and molasses with vegetable carbon are applied. Because there exists a definite relationship between the purity of the material a sugar is crystallized from, and the purity of the resulting sucrose crystals, the considerable rise in purity of the mixed juice required to boil a sugar of refined purity is difficult to realise.

Our questions can therefore be better asked in this form: "Is it practically possible to raise the purity of mixed juice in a single clarification operation to the required value?" Up to the present the answer is in the negative. To obtain sufficiently pure solutions we must resort to a different sequence of our manufacturing processes by the introduction of a second crystallization. The manufacturing sequence therefore becomes: clarification—concentration—crystallization—remelting—second clarification—second crystallization.

To obtain the sucrose solutions of the required purity we have to apply the principle of purification by recrystallization—a principle well known in the chemical industry. But we also see this second clarification applied at high densities, which method we have seen to give superior results. In fact, the refining industry employed superior results before it was applied in mill white manufacture. The combination of two clarifications gives rise to the following question: "Should the first clarification be made as thorough as possible, or should more stress be laid upon the second one?"

Although it is not possible to answer this question in a few words, we may conclude from the excellent results of high density clarification that the second method is likely to be more economical. This view is confirmed by the following practical results. If we compare

(a) a process in which mixed juice is clarified by double carbonatation and the resulting white sugar is refined by remelting without any purification of the melt, with

(b) a process in which mixed juice is clarified by the ordinary defecation process without SO₂ or CO₂ and the resulting raw sugar is refined, the melt, however, now to be clarified by the carbonatation process,

we find that the quality of the refined sugars is about the same, but in the first case about 30 per mille limestone on cane is required, and in the second case only 10 to 12 per mille.

The aim of the above is to demonstrate that the usual sequence, clarification—concentration—crystallization, can be modified in many ways. Each separate case brings the introduction of new principles which, correctly understood and applied, should result in valuable technical and economical improvements. A preliminary investigation, however, should yield the data required on which to base the technical application.

Next, I come to the introduction of a pure technological principle in the sugar industry. I mean the principle that continuous processes should be preferred to batch-type processes, because continuously conducted processes allow a better and more consistent adjustment of variables such as temperature, pressure, vacuum, reaction, velocity, etc. and therefore lead to better results. Moreover, the apparatus for this kind of processes are often smaller, require less floor space and are apt to save labour.
Originally this process was conducted as a batch process and controlled with Dupont paper. Depending on the construction of the intermittent tanks, the circulation was sometimes insufficient, resulting in local excessive alkalinities. Especially the application of perforated baffles, with the aim of improving the absorption of the gas, often prevented adequate circulation. The introduction of continuous carbonatators allowed the process completely to comply with the above-mentioned specifications of alkalinity.

![Figure 1](image)

The first type of continuous carbonatator, as sketched in Fig. 1, was provided with an external circulation pump. The purpose of the circulation is to prevent excessive alkalinity. If the proper volume of milk of lime is added to the mixed juice in the required proportion (100 gals. of 15° Bé per 1,000 gals. of mixed juice), the alkalinity is very high, i.e. about 14,000 mgrms. of CaO per litre. To reduce this high alkalinity to the required value of 800 mgrms., an excess of circulation juice, the alkalinity of which is kept at 400 mgrms. of CaO, is simultaneously added to the mixed juice. The required proportion of circulation juice to mixed juice (33 : 1) can easily be calculated from the given alkalinity values and is adjusted by the regulation of the flow of circulation juice. The alkalinity of this circulation juice, which is also the alkalinity of the juice leaving the tank, is regulated by the gas valve.

The required proportion of 33 : 1 it appears that the circulation pump should be of considerable importance.
capacity. This was the reason that the construction of a more simple apparatus was attempted, leading to the examination of the properties of a second type of tank, provided with an internal circulation device (Fig. 2). This circulation device consisted of an internal concentrical cylinder through which the juice was forced downwards by a big propeller. Both the first and second type of tank were, in respect of the gas which had to be adsorbed, of the counterflow type. Precautions had therefore to be taken that the speed of the downward juice did not exceed the normal speed with which the gas-bubbles tend to rise. This second type of continuous tank has, for several reasons, not been found satisfactory.

In the third type the counterflow type of gas adsorption was abandoned and the parallel flow type was introduced. Simultaneously the energy of the rising gas bubbles was used to promote the circulation of the liquid. To ensure an adequate speed of circulation, a preliminary investigation had shown the most advantageous shape of the concentric cylinder (Fig. 3), the use of which made the circulation pump superfluous. This simple type of continuous carbonatator proved to be entirely satisfactory. It can be applied in the sulphitation process and in the carbonatation and sulphitation of remelted sugar solutions too. The speed of circulation is regulated by the height of the juice level above the circulation cylinder, which also can be adjusted to various heights. It is, however, not my intention to discuss these apparatuses in detail; my purpose was to give you an example of a process the chemical requirements of which have been accurately specified and to show how it is possible, by using a simple continuous apparatus, to comply with these specifications.

References.
1 Netherlands Patent 61916.
2 Mededeelingen Proefstation voor de Java Suikerindustrie 1933, p. 33.
3 Unpublished report.
4 Honig and Alewijn, l.c.

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The President said the paper pointed to some intriguing possibilities, such as that of dispensing with mechanical apparatus for mixing chemicals with the juice. The author stated that the colour of the sugar produced in the early mid-sap carbonatation experiments was disappointing. One could expect this as the juice had been in contact with lime when hot, and the resulting decomposition of reducing sugars would lead to the formation of dark coloured products.

Dr. Douwes-Dekker, replying to questions asked by Mr. Phipson, Mr. Carter and Mr. du Toit, stated that in the mid-sap carbonation process, as actually applied in one of the Javan mills, a pre-clarification of the mixed juice by the normal defecation process was effected, and the clarified juice of the settling tanks was concentrated in the first three vessels of the quadruple before being submitted to double carbonatation. Cooling of the juice from the third vessel to 55°C. was necessary, and
the final concentration from about 45° to at least 60° Brix was done in the fourth vessel. The mud from the settling tanks of the defecation process was returned to the bagasse blanket of the mills; pH during the first step of the concentration process was somewhat above 7. The boiling house recovery of this mill was 93.7 per cent., comparable with 93.6 for the average Java carbonatation mill in the same year. For details about the removal of non-sugars, Dr. Honig’s article in *Sugar*, September, 1950, should be consulted.

Mr. Galbraith, referring to continuous sulphitation tanks, was reminded of the sulphuring tanks used in this country before sulphur towers became common. These tanks were very inefficient, and he asked what type of apparatus Dr. Douwes-Dekker considered would advantageously replace the present tower.

Dr. Douwes-Dekker said that if he had to advise upon the most suitable continuous sulphitation tank, it would be as illustrated in fig. 3 of the paper.

But it should be appreciated that results of even the best sulphitation tank are unsatisfactory if control on pH and temperature is insufficient.

Mr. Phipson asked if an acid syrup of 5.3 pH and 60° brix were produced, would this not lead to a high loss of sucrose through inversion?

Dr. Douwes-Dekker did not think this loss would be important. In Java it was customary to sulphite syrup to a pH of even at times as low as 5. The temperature of the syrup was of course not very high, and it was kept in the tanks as short a time as possible. As soon as the syrup entered the vacuum pan, the pH started to rise, due to the loss of sulphur dioxide, so that on the whole loss of sucrose through inversion was negligible.

Replying to a question by Mr. McKenna, he said that in the case of mid-sap clarification there was no special screening of the juice.

Mr. Rault said that many of the points raised in the paper had been the concern of the technical staff of the only factory that used the carbonatation process in South Africa, especially a reduction in the high consumption of limestone and also of the unduly high elimination of glucose already present in rather small quantities in South African cane juice. With this object in view, the following steps were taken at some time or other in the course of the last 30 years:—

De Haan’s modification of carbonatation (simultaneous liming and gassing at low alkalinity).

Higher temperature carbonatation than that tolerated elsewhere for cane juices, in order to improve filtration rates and cut down chemicals.

Introduction of non-Uba variety canes—supposed to be less refractory.

Action of residual muds left in CO₂ tanks (nucleus for easier filtered precipitate).

Improved design of CO₂ tanks and better distribution of gas.

Extensive studies on the factors influencing the settling and filtration rates of juices.

Different methods of lime addition and end points in carbonatation.

Sharper scientific control by instruments as well as numerous physical and chemical tests.

Continuous carbonatation methods.

Pre-liming, progressive liming, and the like.

These lines of investigation, successful in divers ways, were not very fruitful in lime economy, as other factors of equal economic importance were mitigating against them. These were the discarding of the plate and frame pressure filters in favour of continuous automatic labour-saving systems, which demanded juices of high filtrability and settling rates, and also the aim at a higher elimination of non-sugars for improved quality product and recovery.

In common with South African technologists, he had found that our juices would not respond to the mild treatment given to tropical juices. Every attempt at simple defecation had failed and mill whites as good as those of Java could not be obtained with juices treated with only 1 gram of SO₂ per litre and its lime equivalent. In the carbonatation process in Natal, even a 4 per cent. limestone on cane could never keep a factory going, and still less a 2.5 per cent. of limestone as in Java with the de Haan’s modification. Raw juice heaters are quickly fouled after a few hours of work, and it is difficult to imagine a factory owner bold enough to run his evaporator a muddy juice with an additional amount of incrustating material in the way of lime without previous subsidation, as described in the paper.

The Milling Research Institute will have done a great service to the industry if, in the near future, our factories are able to equal Javan practice where evaporators can keep running for three weeks without requiring cleaning operations.

Regarding the advantages of continuous liming and carbonatation in one tank and its claim of constant pH reaction during gassing with beneficial effect on glucose destruction, he had visited many beet factories in the U.S.A. and always found a workman controlling, by an alkalinity test every minute or so, the end point of the overflowing juice. In spite of the claims made there of uniformity of reaction due to free circulation, he had found, in the same tank, large fluctuations in the alkalinity of
juices from different levels of the juice—e.g. 600 mg.
to 900 mg. CaO per litre. He was, accordingly, very
interested in the design of the continuous carbonata-
tion tank shown in No. 3 sketch, especially if circu-
lation did not deter high efficiency of gas absorption,
which, for want of a better system, had up to now
been successfully realised by a series of perforated
baffle-plates in the path of the gas.

The PRESIDENT remarked that Mr. Rault had
called attention again to the difference between cane
juice in this country and that of tropical countries.
In tropical countries juice could be efficiently clari-
fied by the use of lime and with little or no
sulphitation. That was not possible here, and the
nature of the difference was a problem that the
Experiment Station had long wanted to investigate
by detailed analysis of juice from different varieties
grown in this country.

He noted that the author apparently did not have
a very favourable opinion of the Bach process. He
had recently visited a factory in Portuguese East
Africa that was using that process, and the operators
expressed themselves as very pleased with it.

Mr. Rault said the Bach process had been tried in
Natal, but had to be abandoned as not being suitable.

Dr. DOUWES-DEKKER assured Mr. Rault that he
was aware of the difference in the properties of Natal
juices as compared with those of tropical countries
like Java. He was not prepared to call this difference
a fundamental one, but thought it rather a difference
in degree. Certain juices are easy to clarify, others
are difficult, and this was a problem his Institute
would make a point of studying. While the results
he had given were those from Javan factories, and
he was aware that exactly the same methods could
not be applied in Natal, he thought the results
obtained in Java would be of use to indicate the
lines upon which research might be carried out here.

Mr. Rault had stated that unless large amounts
of lime were used, he found carbonation juices
difficult to filter. It should be remembered that in
Natal the Mauss vacuum filter was used with a
pressure difference of only about 10 lbs. per square
inch, whereas in Java filter-presses, with a pressure
of 40 lbs. per square inch, rendered filtration much
easier.

The Bach process was used in Java with good
results, but when certain factories stopped using this
process and clarified with the ordinary sulphitation
process, with careful control of the pH of the juice,
they obtained just as good results with the simple
process as they had with the sulphitation process
plus the Bach process.

Mr. WALSH thought the paper had introduced
some new ideas to the local industry. With regard to
centrifugal separation of precipitates from juices and
other liquors, he recalled that much work had been
done in the past in Natal on this problem. A great
difficulty lay in the cost of the machinery and its
limited capacity.

Continuous subsiders, with their considerable
saving in labour costs, would be hard to compete
against, but he had not the slightest doubt centri-
gugal separation would come. With the mid-sap
process for instance, the capacity of the separator
would be very much higher because the juice was
concentrated. Much remained to be learnt, however,
before centrifugal separators could be successfully
applied.

Dr. DOUWES-DEKKER agreed that we had not yet
sufficient information. One difficulty was the cost
and the amount of power required, but he understood
that some experiments had been carried out last
season with centrifugal separation of syrup.