DEVELOPMENTS IN LOW GRADE CRYSTALLIZATION

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ABSTRACT

In the manufacture of crystal sugar from sugarcane, the major loss of sucrose is in final molasses. The factors influencing the magnitude of this loss are many and varied. In order to prevent this loss, particular attention must be paid to design of the installation to ensure the desirable features if continuous operation of low grade crystallizers are achieved.

Changes in cane constituents are demanding that more emphasis be placed on the efficiency of low grade recovery in order to contain the amount of sucrose lost to molasses.

The paper reviews both the need and the requirements for handling low moisture content massecuites. Recent studies on design features determined from both experiment and practice are also discussed.

INTRODUCTION

In the Queensland sugar industry, some 6% of the sucrose supplied to a sugar factory is lost to final molasses. The magnitude of this loss is governed by the quantity and purity of the molasses. Exhaustion formula are available to express the lowest molasses purity expected, and the difference in performance, between this figure and the actual purity obtained can be calculated in monetary terms, after making an appropriate allowance for by-product value of molasses. For a factory crushing 750,000 tons of cane in a season, when the raw sugar return is A$200 per ton, each unit of purity difference represents revenue of approximately A$50,000 per season. With the average performance of Queensland mills, the difference between actual and expected purity represents a loss of well over A$200,000 per mill per season. This is a major loss of income to the sugar dependent community. While it is scarcely practicable for the total amount of this to be recouped, the regular performance of individual mills indicates that up to 50 per cent is recoverable with current technology.

Areas where controls aimed at minimizing this loss can be exerted are:
in the field. Impurity levels, which influence exhaustion, originate mainly in the cane supply. Freedom from trash, tops, dirt and decomposition products minimizes juice impurity content.

(ii) in clarification and physio-chemical means of reducing process impurity levels. Techniques include enzymes, ion exchange and ultrafiltration (Batterham).

(iii) in low grade exhaustion.

While item (i) deserves the highest priority by industry management and item (ii) requires ongoing evaluations, this paper is confined to an examination of the most recent studies applicable to item (iii). Particular emphasis is placed in this regard on the low grade crystallizer installation.

DISCUSSION

Changing Industry Practices

Concentrated syrup and its products have traditionally been handled batch-wise, during both the crystallization and separation stages. However, the advantages of continuous processing are well known to the chemical industry and there has, in recent years, been progressive development of this concept in the crystallization and separation stages. Kirby and Atherton noted the increasing use of continuous low grade centrifugals in the Queensland industry, where the number of these machines is now less than two-thirds of the number of batch machines in use some fifteen years ago, yet the amount of massecuite handled is more than twice that processed in the earlier period.

Most of the recent low grade installations in Queensland have been arranged to operate as continuous stations. One low grade continuous pan is in operation. The advantages of these changes have been savings in capital cost and in control procedures, with resultant reduction in manpower requirements. These changes have not increased the capacity or the potential to approach "expected" exhaustion.

Low Grade Exhaustion

Miller and Wright re-examined the Sugar Research Institute expected purity formula and demonstrated that, as well as reducing sugars and ash, dextran content also had a major influence on exhaustion. Improved transport scheduling has tended to produce a cane supply with a relatively low reducing sugar in the juice. At the same time, canes of greater maturity at harvest, resulting from intensive plant breeding have also produced a cane supply with lower reducing sugar content. In addition, the increasing use of irrigation has sometimes resulted in the use of less than ideal quality water, leading to increased ash content of the juice (Kingston and Kirby). These changes in juice have produced corresponding changes in the other process streams. Certainly, recent history in Queensland,
as illustrated in Fig. 1 has shown a progressive decrease in the reducing sugar-to-ash ratio of molasses. Thus, to achieve the standard of exhaustion considered adequate in the past, superior operation of the factory is required under prevailing conditions. This is exacerbated by the greater likelihood of dextran formation in chopper harvested cane which now comprises 100 per cent of the Queensland crop.
Most low grade exhaustion has necessarily been performed in the vacuum pans rather than in the crystallizers. Correct organization of purity drops according to the formula used enables the presentation of a feed to the low grade pan from which maximum exhaustion is attainable. Good boiling practice then requires that this be boiled to the highest impurity to water ratio possible before discharge from the pan. Recent information has cast some doubt on this statement (Jesič⁶) where very high ratios are involved. However, with the ratios attainable in the Queensland industry, the evidence (Broadfoot²) supports the opinion presented in this paper.

The main limitation to heavy boiling is that adequate circulation has to be maintained in the pan in order to keep a supersaturation as even as possible throughout the total massecuite. Crystallization rate at this stage would appear to be under diffusion control. Conditions in the pan are more favorable for crystallization than those in the crystallizer because the relatively high temperature reduces the molasses viscosity and vigorous pan circulation produces more shear in crystal boundary layers. Once the massecuite is dropped from the pan, the efficient mixing is replaced by such stirring action as is available in the crystallizer, and temperature reduction begins. Thus, compared with that which takes place in the crystallizers, pan exhaustion is a relatively rapid process. If pan exhaustion is not carried to the maximum, the remaining extra work has then to be performed by the slower mass transfer processes in the crystallizers. From this aspect alone there is obviously an advantage in ensuring that pan exhaustion is carried through to the maximum possible.

In pursuing this pan exhaustion, the major controllable variable is the total solids content of the massecuite, or inversely, the moisture content. This is illustrated in the recent modelling work of Maudarbocus and White¹¹ and confirmed in the experimental analysis of Lionnet¹⁰, who also found that the moisture content has much more influence on exhaustion than massecuite purity.

**Crystallizers**

Once the massecuite is placed in the crystallizers the principal variable available to control supersaturation is temperature. The limitation to this process of exhaustion is the viscosity of the mother liquor, assuming crystal content is controlled by selection of the feed purity. Michaeli and DeGyulay¹² and numerous subsequent workers have indicated that a minimum viscosity of saturated mother liquor can exist at a temperature in the region of 55°C or above, dependent on the nature of the impurities. The process goal is therefore to reach this condition with the maximum viscosity that the fugal — rapid preheater system can handle. It is shown in Appendix A that in general, a two unit change in massecuite moisture content influences the supersaturation to a much greater extent than a 7°C change in temperature, while producing the same viscosity change. Maudarbocus and White¹¹ in their analysis, predicted that heavier massecuite exhausted more rapidly, arriving at the same massecuite viscosity as a lighter one. It is obvious therefore, that moisture content is the most powerful influence on exhaustion and that
any significant deficiency in that aspect cannot be augmented by temperature reduction without meeting the viscosity barrier to good reheater and fugal operation.

This aspect has been dealt with in some depth because it is fundamental to continuous crystallizer design. So often in the past, the inadequacy of the continuous crystallizer has limited the ability of the pan boilers to produce low moisture content massecuites. This heavy boiling implies a high massecuite viscosity. In Queensland, it is not unusual to have such massecuite direct from the pan at 70°C showing a Brookfield viscosity of 250 Pa s, rising to 3000 Pa s and higher as the massecuite cools to 45°C (Figure 4). As a rough guide, a temperature reduction of 6°C will double the massecuite viscosity. One can therefore envisage the problems of designing a low grade station which is capable of treating massecuites cooled to below 40°C.

Handling Heavy Massecuites

A problem in handling heavy massecuites in crystallizers is that of ensuring mobility throughout the whole volume as the temperature is reduced. Foster5 in the 1950’s obtained experimental evidence that stirring rate was a significant variable on crystal growth rate and hence, exhaustion, but apparently, this has not yet been confirmed in factory operation. However, it appears reasonable that stirring maintains shear in the mother liquor boundary layer surrounding the crystal, thus, promoting the transfer of sucrose from solution, as well as ensuring even cooling throughout the mass. This requires close attention to design. Numerous innovations such as reciprocating coil types as described by Ness and Stewart14, have been developed to combine the aspects of proper mixing with adequate cooling.

With continuous crystallizers imposed on the above requirements there is a need to transport the massecuite through the installation in a regime as close to plug flow as possible. The majority of continuous installations are fed by pumping from a receiver vessel which accommodates the pan contents after pan drop. To maintain continuous flow through the crystallizers, it is recommended that the pumping rate be set so that the receiver is emptied just prior to dropping the next pan. Over this period of time, the contents of the receiver will cool somewhat, thus providing the crystallizers with a progressively cooler and more viscous feed. While it is possible to design the receivers to maintain the massecuite at or near the dropping temperature, thus ensuring a more constant feed temperature to the crystallizers, this is not usually recommended because significant decomposition may occur as a result of holding the material at elevated temperatures for extended periods of time.

Therefore, the crystallizers must be able to handle feed massecuite of varying temperature, and hence viscosity. At the start of each new strike, fresh hot massecuite is fed into the partly cooled material from the previous strike now in the first crystallizer. Strickland et al15 have demonstrated how this affects the spread
of the residence time distribution and ultimately the exhaustion obtainable in the crystallizer station. Kirby and White\textsuperscript{9} found that attention to design, specifically spacing and arrangement of baffles, coil design and operating levels, could minimize this effect.

Associated with the drop in temperature of the contents in the receiver during the pumping period, will be a change in composition of the mother liquor as some crystallization occurs in the receiver. In practice, factory tests have shown that the exhaustion which occurs in the receiver may be greater than that noted subsequently in the crystallizers. Figure 2 demonstrates a typical profile of the mother liquor purity and massecuite temperature recorded in one such case (Swindells et al\textsuperscript{16}). The massecuite was dropped at an average temperature of 70°C and was pumped from the receiver over a six hour period. The feed to the crystallizers

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure2.png}
\caption{Typical exhaustion profile.}
\end{figure}
has a temperature varying from 68°C and a viscosity of around 250 Pa s immediately after pan drop to a temperature of 55°C and a viscosity around 1500 Pa s when the receiver was almost empty. In this instance, 70% of the purity drop after pan discharge occurred in the receivers.

![Graph showing residence time distribution function with A mill results and B model.](image)

**FIGURE 3.** Comparison of model and full scale results.

It is apparent that feeding from a continuous pan would minimize these fluctuations. This is confirmed by experience gained from one unit in operation in Queensland (Broadfoot and Wright) although the production of massecuite of low moisture content would require close control to avoid fluctuations in output massecuite conditions.

**Model Tests**

BSES, in conjunction with the Chemical Engineering Department, University of Queensland, have explored the use of models in order to simulate the effects of different variables on the operation of continuous crystallizers. Factory tests were also performed and the validity of model results confirmed (Kirby and White), as shown in Fig. 3. Many of the requirements for successful operation of continuous units to gain maximum exhaustion were determined. It is now obvious that many batch crystallizer installations would be seriously deficient when adapted for continuous operation.
Receivers

As previously mentioned, some cooling in the receiver is desirable. It is considered that agitation is an item of equal importance, as the hot material after pan drop is still in a region where a relatively high rate of exhaustion can be obtained. The most economic design compromise at this stage appears to be the utilization of old crystallizers as air cooled receivers.

Pumping

Supply of massecuite in batches introduces certain hazards to the operation of continuous crystallizers. While on one hand there is the temptation to transfer the pan strike from receiver to crystallizers in the shortest possible time, the resulting rise of massecuite levels in the crystallizers will merely accelerate short circuiting or bypassing. The first equipment requirement therefore is a variable speed transfer pump and the first process supervisory requirement is strict control over this operation to ensure a constant supply of massecuite. In a similar fashion it is important to ensure that by-pass lines between vessels are not open during normal operation.

Massecuite distribution and flow

One means of reducing the extent of stagnant regions and bypassing is to have the feed and exit from each crystallizers entering and leaving at the vessel end walls rather than through side openings. The openings should be as wide as practicable. The difference in height between consecutive vessels must also be sufficient to ensure that the massecuites flow along the interconnecting chutes is satisfactory. White and Maudarboocu\textsuperscript{1,8} give expressions which allow flow rates to be calculated. If necessary, water heating of the underside of the chute will assist massecuite transfer.

Coil design

Coil design has a marked effect on the spread of retention times. Kirby and White\textsuperscript{9} commented on the concept of alternative flow paths through the crystallizer. They emphasized the need for complete mixing in the cross-sectional plane combined with plug flow in the longitudinal direction. The relationship between massecuite level and coil level is of major importance in cases where material above coil level is provided with a resistance free path as short-circuiting will be promoted. To absorb the slugs of heavy massecuite, the coil design needs to be as simple and as rugged as possible. Both coil spacing and coil-shell clearances have proved to be important parameters in the attempt to promote plug flow. When overall cooling requirements of a station are assessed, it can be readily shown that the relatively wide spacings still provide more than adequate heat transfer area.
Coil speed

Speed of the mixing arrangements is a relatively unexplored area. As mentioned before, the power absorbed by the massecuite would be expected to assist the dynamics of exhaustion. Also, an increase in speed decreased the spread of retention times on tests of the Burnett coil design. However, as it is essential with this design that the liquid level be below the coil level, air entrainment can occur at high rotational speeds, which is undesirable.

Baffles

Baffles are inserted to encourage the attainment of plug flow within individual crystallizers, but Webster et al. recently found that the use of baffles significantly increased the power absorbed by the massecuite from the mixing coils. The finding has serious implications on the use of existing batch vessels for continuous use unless drive power is increased.

Viscosity Reduction

The extent of problems created by attempting to treat 'heavy' massecuites in a continuous arrangement, can be alleviated by viscosity reduction measures. Of the two means available, which are, increasing the massecuite moisture content at dropping, and lowering the crystal content by molasses lubrication, the former is patently not acceptable. The latter means has been investigated by Broadfoot et al. who found it to be a useful method for massecuite viscosity reduction. It is in regular use in several continuous stations, but some control should be exercised on the extent and timing of its application. It is considered that any crystallizer station which has to resort to heating in the crystallizers to overcome flow problems anywhere in the low grade installation during normal operation requires design modifications.

CONCLUSIONS

When consideration is being given to the installation of a new crystallizer station, or the expansion of an old one, the necessity to decide between batch and continuous operation naturally arises. The conceptual advantage of a continuous installation is best seen where the provision of a continuous pan is envisaged in the near future.

The cardinal considerations to be observed are:

(i) a strong high grade performance is vital. A powerful low grade station cannot make up for weakness in high grade boilings.

(ii) heavy boiling of low grade massecuite must be maintained. A powerful crystallizer unit cannot make up for weakness in the low grade boiling, and;
if a continuous station is chosen the flow system must be capable of transferring heavy massecuite throughout the installation.

Good exhaustion depends on providing massecuite to the heaters and fugals at the top limit of their viscosity handling capability. A continuous station must be designed to handle, in as close to plug flow fashion as possible, the heavy massecuites necessary to achieve this aim. The temptation to boil low grade massecuites lighter to reduce the flow problems in the crystallizers must be firmly resisted. As our knowledge of the specialized design requirements of a continuous station increases, it is becoming more apparent that the theoretical advantages of continuous operation can be negated by poor crystallizer design and operation. The financial implications of a change from batch to continuous operation must consider practical difficulties which can be expected.

Appendix A

Comparison of the effects of changes in temperature and total solids on supersaturation potential.

**FIGURE 4.** Viscosity (Brookfield) – temperature relationship for a°C massecuite

- 93 % total solids $\frac{1}{W} = 4.0$
- 91 % total solids $\frac{1}{W} = 3.0$
REFERENCES

5. Foster, D. Private communication.
Fig. 4 is a typical plot of massecuite viscosity (Brookfield) against temperature at two levels of total solids, determined from a test program carried out in North Queensland (Swindells et al\textsuperscript{[16]}).

At any particular temperature it can be calculated that increasing the total solids in the massecuite by one per cent is equivalent to raising the supersaturation potential by approximately 0.18 units. Therefore the two levels of total solids illustrated in Fig. 4 represent at any particular temperature a difference of approximately 0.36 units of supersaturation.

If it is assumed that the rapid heater-lugal system imposes an upper limit such as 1500 Pa s on the massecuite viscosity, the 93 per cent total solids can be cooled to 54°C (point A) while the 91 per cent can be cooled at 47°C (point B). This 7°C difference can be shown to represent a difference of only 0.07 units of supersaturation potential, compared with the 0.36 units difference available by boiling to two per cent less moisture.

Thus, given adequate residence time, the heavy massecuite would be expected to exhaust much more than the light massecuite. It is realized that boiling philosophy centered on the impurity to water ratio dictates the relationship of total solids and purity at pan drop, but the above illustrates the advantage of boiling to the highest total solids content possible once the purity is selected.

DESARROLLOS EN CRISTALIZACION DE BAJA PUREZA

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RESUMEN

En la fabricación de azúcar granulado a partir de la caña de azúcar las mayores pérdidas de sacarosa están localizadas en la miel final. Los factores que influyen estas pérdidas son muchos y variados. Se debe poner especial atención al diseño de las instalaciones para evitar incrementar estas pérdidas, si se desea realizar una buena operación continua de los cristalizadores de masas de baja pureza.

Los cambios en los constituyentes de la caña están exigidos que se le dé más énfasis a la eficiencia de recuperación cu los cocimientos de baja pureza con el fin de evitar cantidades apreciables de sacarosa se pierden con la melaza.

En el trabajo se incluye una revisión de las necesidades y requerimientos para manejar masacocidas de bajo contenido de humedad. También se discuten los estudios recientes como por la práctica.