

THE EFFECT OF SOME OPERATIONAL FACTORS ON COLOUR TRANSFER DURING PAN BOILING

By G. R. E. LIONNET

Sugar Milling Research Institute

Abstract

The SMRI pilot batch pan was used to investigate the effects of a number of operational factors on the transfer of colour from mother-liquor to crystal during A-masseccuite boiling. The factors investigated include stirring, supersaturation, crystal size and crystallisation rate. Colour transfer was found to increase with supersaturation, crystallisation rate and crystal size but to decrease with stirring. Although far from complete, this work indicates that the effects of seasonal factors and cane quality on colour transfer are much larger than those of the operational factors investigated.

Introduction

The amount of impurity trapped into the sucrose crystal during crystallisation is controlled by two main factors. The first is the quality of the feed material from which the crystals are grown while the second is an operational factor which includes the conditions such as temperature, crystallisation rate, stirring etc., under which crystallisation takes place.

The first factor, namely the effect of feed quality, has been investigated¹ recently. Time of the year and mill of origin were found to have significant effects on the quality of the sucrose crystal, other boiling conditions being kept constant, the former having by far the larger effect.

Moritsugu & Payne,² using a laboratory pan and cane syrups, showed that the effect of temperature cannot be viewed in isolation since temperature affects viscosity, supersaturation and other factors. Crystallisation rate has been found^{2,3,4,5} to be an overriding factor with high rates increasing the impurity transfer. Moritsugu & Payne² showed that, other conditions being the same, stirring increases the crystallisation rate without increasing the degree of inclusions. Similar effects were found⁶ in pans boiling white sugar from beet syrups. A number of authors^{7,8,9} found that the amount of impurities in the crystal increases with crystal size. Moritsugu,² working with cane syrups, found less inorganic inclusions in the crystals as size decreased, at otherwise similar laboratory crystallisation conditions. The trends were linear.

Experimental Procedures

Objectives

The main objective of the investigation was to investigate the effect of a number of operational factors on the transfer of colour during the boiling of A-masseccuites.

When required, feed syrup quality was kept constant by preparing an artificial syrup, using water, sugar and final molasses in the required proportions. A large sample of well mixed final molasses was kept for that purpose.

Pilot-plant

The Sugar Milling Research Institute pilot batch pan was used for all the boilings. This pan and its ancillary equipment have been described in detail elsewhere.^{1,10} The only modification was the addition of a sampling device which allows

massecuite to be sampled at any time during the boiling, without interfering with the crystallisation process.

Control of the operational factors was as follows:

- Stirring: either on, at maximum speed, or off. No attempt was made to try and quantify stirring or mixing efficiency.
- Supersaturation: controlled through the use of the boiling point elevation (BPE) measurement.
- Crystallisation rate: controlled by the heat input into the pan and through the supersaturation.
- Crystal size: the effect of crystal size was investigated by sampling the massecuite and nutsching at various intervals during the boiling. Larger crystal sizes were achieved by keeping part of a final massecuite as footing and boiling this up again to pan full.
- Temperature: controlled by the absolute pressure used for the boiling.

The precision with which the various factors could be controlled and reproduced from run to run will be discussed later. It can, however, be said that the control was satisfactory, as far as the requirements of the investigations are concerned.

Analytical procedures

- (a) Affination of the sugar: This critical step has been described in detail in a previous paper.¹ All the sugar analyses reported here were done on affinated sugar.
- (b) Absorbances: Again, these procedures have been described in detail previously.¹ It should be noted that all the absorbances were read on solutions which had been filtered through a 0,45 μm membrane.
- (c) Crystallisation rate: A formula given by Morel du Boil¹¹ has been used. This gives the crystallisation rate as kilogram of crystal per square metre surface area per second. This formula is used here only to compare crystallisation rates under fairly similar conditions. It requires the initial mass of seed crystal (weighed), the final mass of crystal in the massecuite (obtained by mass balance across the pan and by using massecuite and mother-liquor purities) and the boiling time. A shape factor is also required.

Results and Discussion

Factorial experiment

A factorial design was used for a preliminary experiment. Apart from quantifying the effects of the factors selected, this approach includes the investigation of experimental errors. This last point would be useful in the planning of further tests.

Stirring, supersaturation and heat input were selected as the main factors. Temperature and syrup quality were kept constant as described earlier in the paper.

One combination was repeated 4 times to obtain the experimental error.

Colour transfer, given by the ratio of affinated sugar colour to syrup colour, and crystallisation rate ($\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) were used as responses.

The following results were obtained:

- Only supersaturation showed a statistically significant effect on colour transfer. Increasing the BPE from 8,5 to 9,4°C resulted in the colour transfer increasing from 0,015 to 0,017 (13%). This change in BPE corresponds to the massecuite brix increasing from 84,3 to 88,6, that is about 4 brix units. It should be noted that the usual final brining up was not done in this investigation.
- Supersaturation had a statistically significant effect on crystallisation rate. The increase from 8,5 to 9,4°C BPE caused the crystallisation rate to increase from $6,3 \times 10^{-3}$ to $9,2 \times 10^{-3} \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$ (46%).
- Heat input showed the expected positive effect on crystallisation rate. Changing from low to high input caused the crystallisation rate to increase from $7,0 \times 10^{-3}$ to $8,5 \times 10^{-3} \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$. This increase was, however, not statistically significant, as far as the factorial experiment is concerned.
- It was also possible to use a multilinear regression to relate crystallisation rate to the selected factors. Equation 1 was obtained.

$$\begin{aligned} \text{Crystallisation rate} = & -0,0283 + 2,98 \times 10^{-3} \cdot \text{BPE} \\ & + 4,52 \times 10^{-5} \cdot \text{Heat input} \\ & + 1,05 \times 10^{-3} \cdot \text{Stirring} \end{aligned} \quad (1)$$

(n = 20; r = 0,87)

where BPE is in °C, heat input is represented by the voltage used (175 or 210 volts) and stirring is either 0 (off) or 1 (on). All 3 independents in equation 1 were statistically significant at least at 5%.

These results which show that colour transfer increases with supersaturation and thus with crystallisation rate are summarised in Table 1.

Table 1

| Relation between supersaturation, crystallisation rate and colour transfer | | | |
|--|----------------------|----------------------|----------|
| | | | % change |
| Supersaturation (BPE, °C) | 8,5 | 9,4 | |
| Crystallisation rate ($\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) | $6,3 \times 10^{-3}$ | $9,2 \times 10^{-3}$ | + 46 |
| Colour transfer | 0,015 | 0,017 | + 13 |

It is evident that a large change in crystallisation rate results in a relatively small change in colour transfer. This indicates that the use of crystallisation rate to control colour transfer on the industrial scale would be very costly in terms of pan capacity and exhaustion.

Stirring was found to increase crystallisation rate by 15% for a change from the stirrer being on to the stirrer being off. The effect of stirring on colour transfer was masked by the experimental error and can thus be assumed to be fairly small, if present at all.

Effect of crystal size

Sugar, water and final molasses were mixed to obtain two lots of syrups of different purities and colours. The syrups were boiled under standard conditions to yield massecuites which were sampled at half and full pan volume. A footing of final massecuite was left in the pan, and the procedure was repeated.

Colour transfer has been plotted against average crystal width in μm in Figure 1.

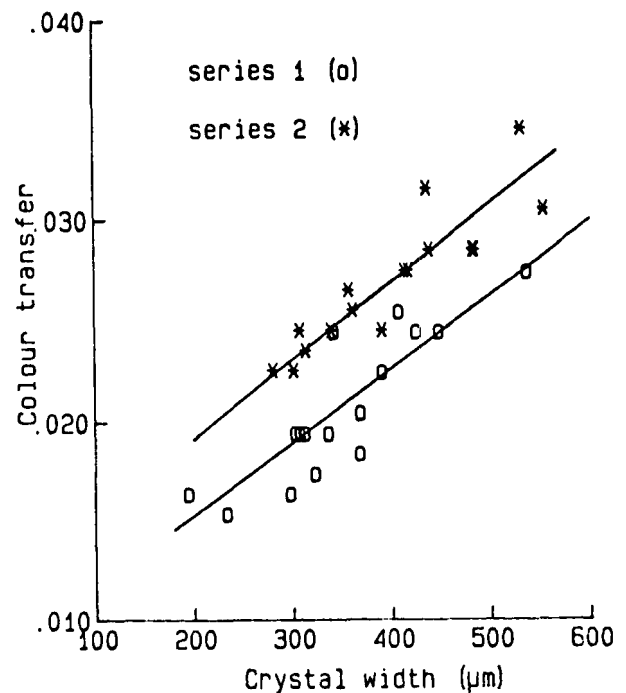


FIGURE 1 Colour transfer versus crystal width.

These results show that colour transfer increases linearly with crystal width (W) over the range investigated, as quantified by equations 2 and 3:

Series 1:

$$\text{Colour transfer} = 0,00810 + 3,810 \times 10^{-5} \cdot W \quad \dots (2)$$

(n = 16; r = 0,87)

Series 2:

$$\text{Colour transfer} = 0,0132 + 3,590 \times 10^{-5} \cdot W \quad \dots (3)$$

(n = 16; r = 0,91)

It is also evident that, for the same crystal size, the transfer is higher in series 2. This is due to the crystallisation rate for series 2 ($0,010 \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) being greater than that for series 1 ($0,0065 \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$).

It was also possible to obtain colour transfers for similar crystal sizes but at different syrup colours. This has been plotted in Figure 2, using results obtained from the series 2 runs.

Colour transfer does not change much with syrup colour but the effect of crystal size is again evident.

For a 10% drop in crystal size, colour transfer drops by about 5%. It thus appears that the control of crystal size would be more useful industrially than that of crystallisation rate in attempts to reduce sugar colour.

The use of the boiling point elevation (BPE)

Supersaturation in impure sucrose solutions is not easily measured. In this work, BPE has been used as an indirect measure of supersaturation. It can be expected that supersaturation will be dependent on temperature, boiling point elevation and purity. Under normal boiling conditions, supersaturation should vary directly with mother-liquor brix which is easily measured.

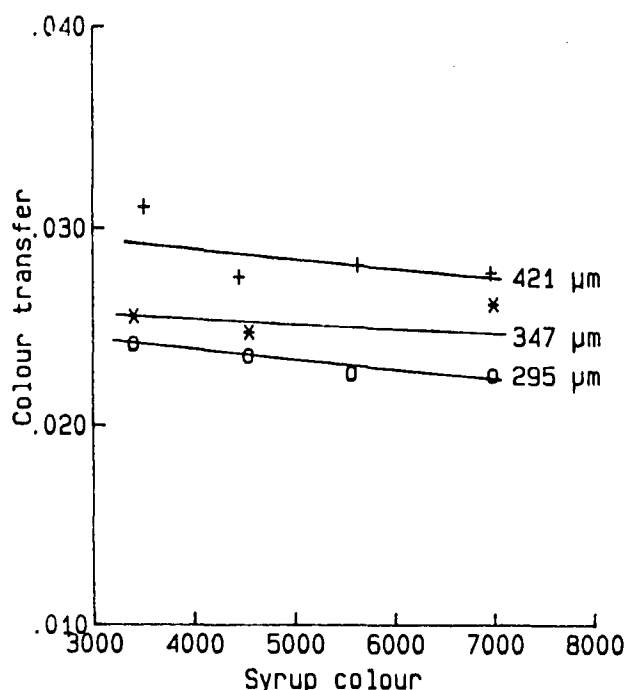


FIGURE 2 Colour transfer versus syrup colour, at the same crystal size.

These relationships were tested by regressing nutsch molasses brix against BPE, temperature and feed syrup purity. The following result was obtained:

$$\text{Nutsch molasses brix} = 99,6 + 0,091 \times \text{Temp.} + 0,542 \times \text{BPE} - 0,351 \times \text{Syr. pur.} \quad (4)$$

(n = 26; r = 0,96)

where Temp. is the massecuite temperature (°C), BPE is the boiling point elevation (°C) and Syr. pur. is the feed syrup purity. The ranges for these variables were 55 to 70°C, 8 to 14°C and 75 to 100 respectively.

It is evident from these results that BPE can be used as a measure of supersaturation.

A large number of boilings was carried out to try and relate colour transfer to crystal width, temperature, BPE and syrup purity, the last three factors being used to represent supersaturation. The results obtained were disappointing. Although the correlation coefficient was high, the independents were not all statistically significant, with crystal width accounting for most of the correlation.

Although this work is far from complete, brixing for example has not yet been investigated, it would be worthwhile to compare the effects of those factors that have been investigated. This has been done in Table 2, which contains data from this and previous work.¹

Table 2

Comparing the effects of a number of factors on colour or colour transfer

| Factor | Change in factor | Effect on | | |
|---|------------------|--------------|-------------------|-----------------|
| | | Syrup colour | Aff. sugar colour | Colour transfer |
| Mill | 4 mills | 36% | 38% | — |
| Time of year | May to December | 51% | 103% | 49% |
| Supersaturation (as BPE) | + 10% | — | — | + 13% |
| Crystallisation rate (kg·m ⁻² ·s ⁻¹) | + 45% | — | — | + 13% |
| Crystal width (μm) | + 10% | — | — | + 5% |
| Stirring | off vs. on | — | — | < 10% |

The results concerning the mills and the time of the year were obtained¹ in 1986 and the percentages given represent the change between lowest and highest values.

It is clearly evident that the seasonal effect is by far the most significant one, showing a doubling of affinated sugar colour. This increase was found to be at the beginning and end of the season (ie summer) compared to the middle of the season (ie sucrose peak).

Four mills were also compared and the maximum average difference between the lowest and highest colour was 38%, again a fairly substantial value. This can be attributed mainly to cane quality.

Mann¹² has shown that operational factors can be used successfully to control sugar colour on the industrial scale. This control was mainly through crystal size and reduced brixing.

Of the factors investigated here, only crystal size appears to be useful industrially. Mann¹² dropped specific grain size by about 15% which would reduce colour transfer by approximately 8%. The effect of crystallisation rate during the boiling (as opposed to brixing) appears far too small to be useful industrially. As stated previously, brixing has not yet been investigated.

Conclusion

Although this work is far from complete, the results obtained at this stage indicate clearly that time of the year and cane cleanliness have a much more pronounced effect on sugar colour than operational factors at the crystallisation stage. There are definite limitations to the amounts by which operational procedures can be changed in existing crystallisation stations for the production of VHP sugar. It can therefore be expected that operational factors will, at best, only provide a limited degree of control of sugar colour. This conclusion indicates that investigations into the use of chemical means for the reduction of colour in syrup or remelt might be worthwhile.

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REFERENCES

- Lionnet, GRE (1987). Impurity transfer during A-massecuite boiling. *Proc S Afr Sug Technol Ass* 61: 70-75.
- Moritsugu, T and Payne, JH (1965). Effect of some variables on the quality of crystalline sucrose. *Proc Int Soc Sug Cane Technol* 12: 166-169.
- Mantovani, G; Vaccari, G; Sgualdino, G; Aquilano, D and Rubbo, M (1986). Colouring matter inclusions in sucrose crystals. *Proc Int Soc Sug Cane Technol* 14: 663-669.
- Mantovani, G; Vaccari, G; Sgualdino, G; Aquilano, D and Rubbo, M (1985). Sucrose crystal colour as a function of some industrial crystallisation parameters. *Ind Sacc Ital* 78: 79-86.
- MacKintosh, DL and White, EJ (1969). Enclave inclusions in sugar crystals. *Proc Aust Soc Sug Cane Technol* 36: 291-298.
- Van der Poel, PW (1980). Influence of operation with stirrers on the quality of sugar crystals. *Experience at CSM Zucker* 105: 237-240.
- Singh, S and Delavier, HJ (1974). Distribution of non-sucrose substances in sucrose crystals. *Zuckerind* 24: 575-582, 639-651.
- Delavier, HJ and Hirschmuller, H (1968). Distribution of non-sucrose in sucrose crystals. *Proc Int Soc Sug Cane Technol* 13: 342-350.
- Guo, SY and White, ET (1984). The distribution of impurities in inclusions in sugar crystals. *Proc Aust Soc Sug Cane Technol* 6: 333-342.
- Bruijn, J (1964). The construction of two laboratory pans. *Proc S Afr Sug Technol Ass* 38: 102-105.
- Morel du Boil, PG (1985). Sucrose crystal habit in a refinery. *Proc S Afr Sug Technol Ass* 59: 33-38.
- Mann, GF (1987). Modifications to the boiling procedures at Sezela in an attempt to reduce VHP sugar colour. *Proc S Afr Sug Technol Ass* 61: 83-85.