A PILOT SCALE BATCH PAN

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Abstract

The existing small scale batch pans used for research in the South African sugar industry have limited capabilities for simulating industrial operations. In particular, these pans have been designed and used primarily for determining equilibrium conditions and have not been designed to achieve the rates of crystallization which are achieved in full scale pans. Operating an existing laboratory scale pan under closely controlled conditions also produced much wider crystal size distributions than are obtained in industrial scale pan boiling. To address these limitations and provide a facility to study crystallisation under closely controlled conditions which simulate industrial scale pan boiling, a new pilot scale batch pan has been designed and constructed. Features included in the design of the pan are, a steam heated calandria, the use of standard diameter pan tubes (100mm), a variable speed mechanical stirrer and a high level of instrumentation and control. The design of the pan is described in terms of the experience gained on the small laboratory scale pan. Some performance data from the pan are presented.

Keywords: Batch pan, massecuite, automation, boiling, mechanical circulation

Introduction

As part of research into determining the optimum operating conditions for a continuous A-pan (Love 2002) there was a need to have a quantitative measure of the extent to which impurities reduce the maximum crystallisation rate that can be achieved during pan boiling.

Unfortunately good quality crystal growth rate data which are directly applicable to industrial scale pan boiling are scarce. This lack of data in the literature appears to be more a consequence of experimental difficulties rather than as a result of a lack of interest in the subject.

Given the experimental difficulties of measuring crystal growth rates under closely controlled conditions that simulate industrial crystallisation, a number of specialised laboratory techniques have been devised to measure sucrose crystal growth whilst avoiding the complexities of pan boiling (e.g. Smythe, 1959; Broadfoot and Steindl, 1980; Nicol and Parker, 1971; Guimaraes *et al*., 1995; Vaccari *et al*., 1996; Pautrat *et al*., 1996). Whilst these laboratory techniques may be precise, and give useful insights into the magnitude of specific effects on crystal growth rates (such as shear at the crystal surface), their applicability to industrial pan boiling is questionable.

It was this desire to quantify the effects of impurities on maximum crystallisation rates that was the original motivation behind attempting to simulate industrial scale pan boiling on a small scale - eventually leading to the development of a new pilot pan.

Previous work on small scale pan boiling

Whilst small scale pan boiling offers significant advantages over full scale tests for obtaining better quality data on sucrose crystallisation, there are significant problems which need to be addressed. Clark (1999) expressed concern over the use of laboratory scale crystallisation for the evaluation of emerging purification technologies, specifically the ability to simulate full scale operations adequately because of poor circulation and difficulties in control of supersaturation. Garside et al. (1990) recommends a minimum volume for an experimental evaporating crystalliser of 20 litres, stating that in smaller vessels, the bubbles influence the fluid dynamics too much. Wright and White (1974) commented that much work on crystallisation was "restricted to idealised laboratory experimentation, which is often quite unrelated to the performance of the industrial crystallisation process" and thus gathered data from a pan in a factory that was available for "off production" experimental boilings.

Laboratory scale pans have been used within the South African sugar industry for a number of years. Bruijn (1964) described the construction of two laboratory pans with strike volumes of 4.2 and 13 litres respectively. Because of the limitations of these pans for measuring the maximum exhaustion that could be achieved from low purity massecuites, a new design of experimental pan with a strike volume of only 500 to 600ml, known as the boiling down apparatus, was developed (Bruijn, 1977). This system proved to be very useful in obtaining the necessary practical equilibrium data (target purity) with a duplicate apparatus being used by Tongaat-Hulett for extending the investigation (Rein and Smith, 1981).

Whilst these pans provided the target purity data currently used for evaluating factory performance (in terms of approaching the maximum possible exhaustion of final molasses) the pans were not operated to try and simulate the operating procedures of full scale pans.

The 13 litre pan at the Sugar Milling Research Institute (SMRI) was subsequently upgraded by fitting it with extra instrumentation and computer control (Wienese *et al*., 1987) and initially used for the study of impurity transfer in A-boilings (Lionnet, 1987). The pan was also used to study impurity transfer in white pan boilings (Lionnet, 1998). These investigations also did not attempt to directly simulate full scale performance and comparisons (Lionnet, 1999) showed that the crystal growth rates in the experimental pan were considerably slower than in industrial pans. This was probably because of evaporation limited growth.

A laboratory pan based on the original 13 litre design was designed for Tongaat Hulett sugar by the SMRI in 1982.

Initial crystallisation experiments using the 13 litre laboratory scale pan

Initial attempts to investigate the effect of purity on crystallisation rate began by using the 13 litre laboratory scale pan designed by the SMRI. The pan was refurbished but the basic construction of the pan was not modified. A computerised monitoring, control and data logging system based on a multi-tasking operating system (Love, 1991) was added, incorporating feed regulation using time proportional on/off control (Love, 2001). An on-line mass balance was implemented on the control computer to estimate the pan contents throughout the boiling cycle. This was based on readings from two electronic scales, one which monitored the mass in the two feed tanks and another which monitored the mass of condensate collected in a vacuum receiving tank.

The refurbished pan was able to achieve very stable pressure control (i.e. within ± 0.1 kPa) by regulating an air bleed into the suction of the vacuum pump through a needle valve. Unfortunately an undersized vacuum pump necessitated excessive attention to vacuum tightness and pump

maintenance. Good quality feed control was able to keep variations in boiling point elevation (a good indicator of the supersaturation in the mother liquor) to within ± 0.1 °C as shown in Figure 1. The growth rate was quantified by analysing digital images of crystal samples taken throughout the boiling cycle, also shown in Figure 1.

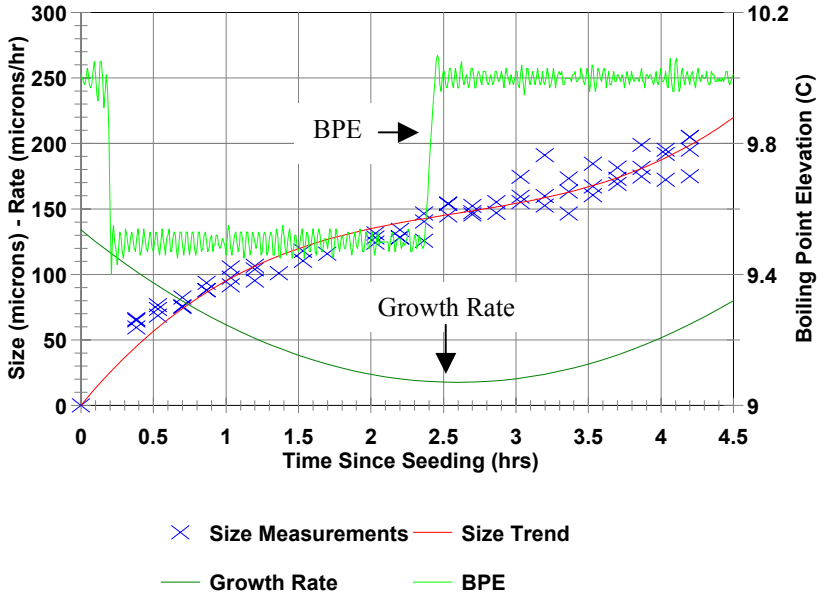

Figure 1. Growth rate of crystals in a laboratory scale (13 litre) vacuum pan boiling A-massecuite.

After close attention to instrumentation and experimental details, it was possible to show that the on-line mass balance provided an estimate of the pan contents that was accurate to within 0.25 units of brix after a three hour boiling cycle – approximating the accuracy of the brix analysis itself.

Even with the excellent level of control and monitoring achieved in the pan, it was obvious from a qualitative visual observation that the grain size distribution was inferior to that produced in full scale pans with a much wider range of crystal sizes. This can be seen quantitatively in Figure 2 where the CV of crystals in A-massecuite is seen to increase rapidly as the crystals grow rising to levels well above those achieved in full scale pans.

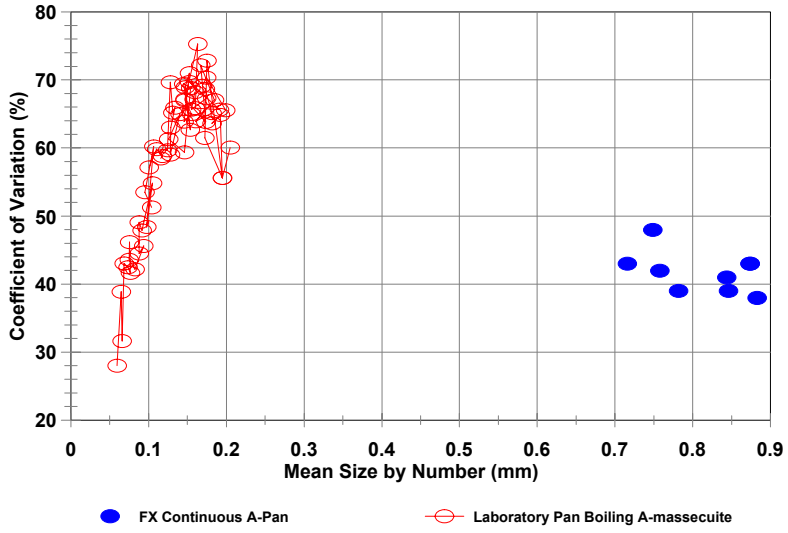

Figure 2. Comparison of spread in crystal size distribution from the laboratory scale pan with that achieved in industrial scale pan boiling.

Despite many attempts at altering boiling techniques and testing with high purity products (i.e. refined sugar boilings) no observable improvement in this aspect of the boiling could be achieved. The importance of pan design in obtaining good product sugar quality is well known (e.g. Donovan, 1988) and having both ensured good control and experimented with boiling technique, the design of the laboratory pan was deemed to be the most likely cause of the poor performance.

During 1999, the development of a new processing scheme by Tongaat Hulett Sugar (Fechter *et al.*, 2001) required experimental boilings to characterise the behaviour of the new process streams that would be encountered. This provided an opportunity to utilise the knowledge and experience gained with the laboratory pan to design and build a larger, pilot scale, pan. The selection of the type of pan and the details of its design are described in the following sections.

Requirements for a new pilot scale pan

Experience with the laboratory pan indicated that the following aspects of the design of the pilot pan should be given particular attention:

- *Massecuite Circulation*
  Good circulation is a major consideration in full-scale batch pan design and appears to be more difficult to achieve on small scale because of the relative increase in frictional drag resulting from the increased wetted perimeter to cross-sectional area of smaller pipes.

- *Heating surface temperatures*
  Insufficient heating surface and electrical heating can result in very high heating surface temperatures and the possibility of areas of high temperature massecuite where crystal dissolution takes place. Steam heated tubes, as used in full scale equipment, limit the maximum temperature possible whilst the provision of good circulation and adequate heating surface reduces the maximum temperature necessary.

- *Feed line blocking*
  The problem of the blocking of the syrup feed line whilst the pan was being fed on water needed to be addressed.

- *Adequate vacuum system*
  A vacuum system that was marginally adequate on the laboratory pan meant that both frequent refurbishment of the vacuum pump and very tight control of air leaks into the pan were essential to achieve accurate pressure control. A failure of the vacuum system during a boiling cycle on the 13 litre pan often led to the termination of the test and the discarding of the results.

The new pilot pan design needed to avoid these limitations of the laboratory pan whilst attempting to approach the design and performance of a full scale pan as closely as possible. Industrial batch pan designs have been iterated over a period of more than a hundred years and whilst considerable design experience and information was available within Tongaat Hulett Sugar, much of this information is needed to be applied with caution to the design of a small scale pilot pan. (It is an interesting reversal of conventional chemical engineering practice to be using data from full scale installations to design a pilot plant!)

Design of a new pilot scale pan

The concept behind the design of the new pilot scale pan was that it should be built using the standard diameter of pan tube (100 mm) but using multiple short tubes equally spaced around a central down-take with a circulation ratio approaching that of a full scale batch pan. This configuration requires a stirrer in the downtake to promote circulation even though for many applications a full scale pan would not require mechanical assistance to promote circulation. This is because the effect of frictional drag from the down-take wall is increasingly significant with smaller pans whilst there is no equivalent effect of pan size on frictional drag through the tubes (when using standard size pan tubes).

The schematic diagram of Figure 3 and the photographs in Figures 4 and 5 may assist in clarifying the description of the design of the new pilot scale pan which follows.

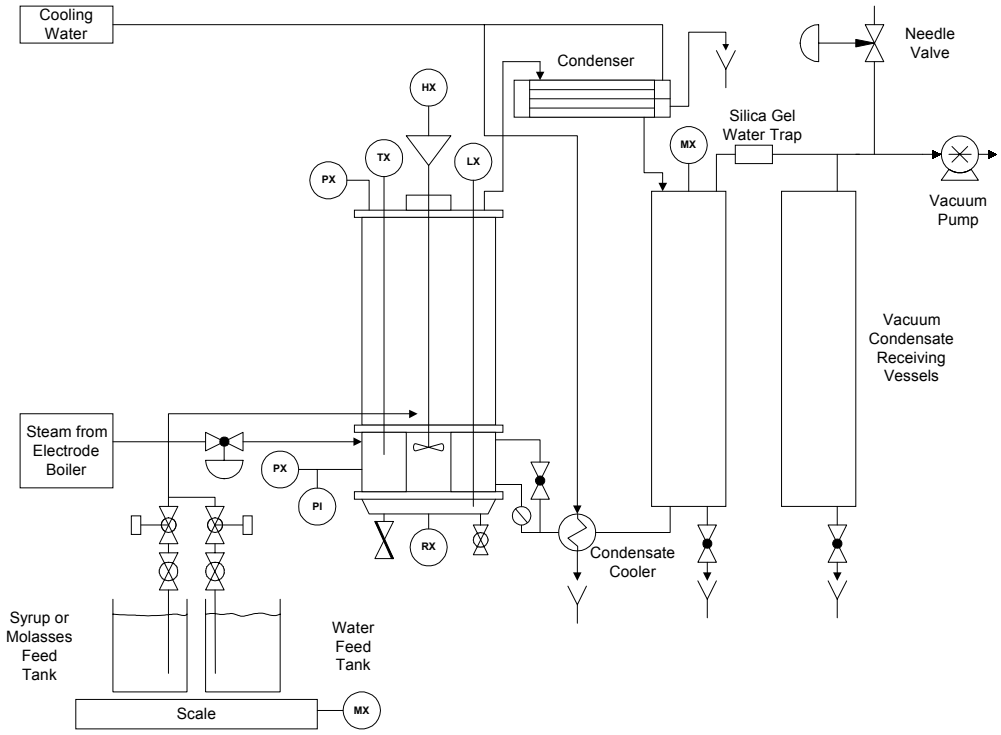

Figure 3. Schematic diagram of pilot pan and associated plant and instrumentation.

Pan body

Both the pan body and calandria are constructed from stainless steel, using standard pipe and flanges where possible and standard 100 mm stainless steel pan tubes. This approach eliminates the corrosion problems associated with intermittent pan operation and has the added benefit of shiny internal surfaces which improve lighting and thus visibility of the pan contents.

The design uses six (100 mm diameter) tubes equally spaced around a central down-take as discussed below under the section on heating surface.

The pan body is constructed from standard 450 mm pipe and with the selected down-take diameter of 140 mm (see below) provides a strike volume of 51.4 litres and a graining volume of 21.3 litres. This ratio of strike volume to graining volume ration of 2.42 is higher than the value of 1.89 for Tongaat-Hulett's present design of 85 m^3 batch pan, but is achieved with a massecuite level only 200 mm above the top tube plate. This low head will favour good circulation. Expressed differently, the graining volume is 41% of the strike volume, approximating the recommendation of Rouillard (1987) of a value of 40% as a compromise between good circulation and number of cuts required to achieve the required final crystal size.

Figure 4. General view of pilot scale pan with vacuum condensate tanks on either side.

The base (saucer) of the pan was sized to minimise holdup of massecuite and eliminate stagnant regions whilst promoting good circulation. The base includes a fitting to allow a standard industrial model RF probe (short length) to be fitted into the pan and protrude into the down-take where the forced circulation past the probe ensures a representative reading. (To achieve probe readings comparable to full scale boilings without recalibration of the probe it was found necessary to shorten the tip to compensate for the conduction path from the tip to the walls of the down-take).

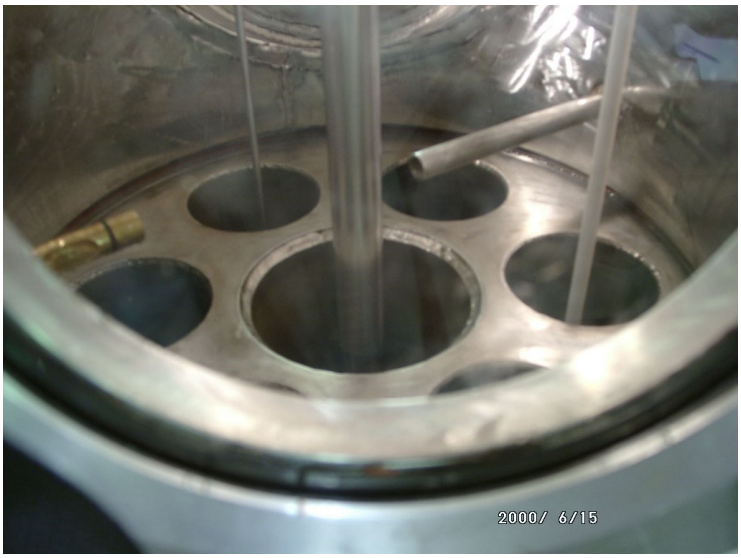

Figure 5. View of the top of the calandria of the pilot scale calandria, showing central down-take with stirrer shaft and some of the surrounding tubes (photographed through a lower sight glass).

The pan body is fitted with three (standard industrial size of 200 mm diameter) sight glasses above the calandria. Two are just above the calandria level to allow the massecuite level to be monitored during boiling and the other is positioned near the top of the pan body to provide extra light and to allow any foaming and potential carryover to be monitored. The sight glasses are not provided with any steam or water wash points as this would introduce added complexity to system for on-line mass balances. Natural condensation has proved to be sufficient to keep the glass clean in most instances but a wash system compatible with the mass balance requirements could be designed and fitted.

Heating Surface
The use of six 100 mm tubes of 200 mm length provides a heating surface area to volume ratio of 6.8 m^{-1}. For comparison, other appropriate figures are:

6.07 m^{-1}	Tongaat-Hulett design of 85 m^3 batch pan used in raw sugar factories
5 to 6 m^{-1}	quoted by Wright (1974) as normal range for batch pans
5.8 m^{-1}	mean value for batch pans in South Africa (Rouillard, 1987).

With the small size of the calandria, steam distribution was not considered to be a problem and the only special arrangement used was a tangential steam inlet to promote mixing in the steam chest and reduce the possibility of stagnant pockets where incondensible gasses can accumulate. Since the calandria is only 200 mm high, only a single incondensible gas vent is installed (rather than the conventional top and bottom vents). The vent is positioned to remove incondensibles from close to the down-take.

In the calculation of surface area for heat transfer, the down-take surface area is not included. As presently designed, the down-take is only marginally different from the heating tubes and there will undoubtedly be heat transfer through this surface. Any boiling that occurs in the down-take will impede circulation and the possibility of installing an insulating sleeve within the down-take will be investigated.

Pan Circulation
Whilst the importance of circulation in pans is well known, there is very limited data available on the actual circulation rates or velocities achieved in industrial equipment, making appropriate selection of a stirrer for the pan difficult.

In reviewing the literature on circulation in pans, it is important to take account of the error of Webre (1933) who proposed that no boiling takes place in the tubes and that natural circulation is due to the difference between the density of hotter massecuite in the tubes and colder massecuite in the down-take. The validity of this assumption was questioned by Allan (1962) and later demonstrated by the detailed measurements of Rouillard (1985) to be completely erroneous. Unfortunately circulation velocities calculated using Webre's erroneous assumption have continued to be quoted despite knowledge of the existence of vapour bubbles within the pan tubes. Bosworth (1959) measured the circulation velocity of massecuite in the volume above the calandria using an adaptation of a hot wire anemometer. The results thus do not relate directly to the flow rate through the tubes or through the down-take and the direction of flow could only be inferred.

For mechanically stirred pans Hoekstra[1] collated some data on the installed power of stirrers in the local industry. Bachan and Sanders (1987) describe the benefits of the Ekato design of impeller on the performance of refinery pans. Cox and Purdham (1989) demonstrated the benefits of a marine type of impeller over a Kaplan Turbine impeller for white massecuites. These authors do not provide any direct measurements of circulation rates.

The readily available data on pan circulation (which must obviously be treated with circumspection) can be summarised as follows:

Circulation Ratio (ratio of cross sectional area of the tubes to the down-take area):

2.8	- average for South African Batch pans (Rouillard, 1987)
2.75	- Tongaat Hulett 85 m^3 pan design.

[1] RG Hoekstra, Tongaat-Hulett Sugar Internal Report.

Rouillard (1987) used a mathematical model which predicted an optimal circulation at a circulation ratio of 3.5, although there is little variation between values of about 2.5 and 4.0. The model predicts that specific evaporation rates will drop slightly as circulation ratio increases.

Circulation velocity (inlet velocity at the bottom of the tube):

0.14 to 0.61 m/sec. (Rouillard, 1985, estimates from mathematical model)
0.03 to 0.13 m/sec (Rouillard, 1985, forced circulation tests).

Mean Circulation rate in massecuite above calandria or coil (Bosworth, 1959):

0.10 m/s	A-massecuite
0,08 m/s	AB-massecuite
0.04 m/s	B-massecuite
0.02 m/s	C-massecuite.

Installed Stirrer Power:

1.65 kW/m^3	(Wright, 1974)
0.97 to 1.57 kW/m^3	Hoekstra[1])
1.06 kW/m^3	(Bachan and Saunders, 1987)

Small pans have a proportionally larger frictional drag in the down-take than larger pans with the same circulation ratio as a result of the increased wetted perimeter to cross-sectional area of the smaller downtake. Consequentially the laboratory pan is designed with a circulation ratio of 2.98, approximating that of full scale natural circulation pan but with a stirrer to promote circulation. The design circulation velocity was selected as 0.15m/s, based on Rouillard's pumped circulation tests and taking into account the data of Bosworth (1959).

Rather than use a simple Kaplan type impeller or a conventional marine impeller design, the stirrer selected was an available commercially designed impeller - a 134 mm A310 impeller supplied by Aeromix. This is a complex aerofoil design and the suppliers provide limited information on its performance. The impeller appears to be the same as a unit described in the book by Oldshue (1983) - published by the company which designed and manufactured the impeller. The impeller has a pitch of 1.5 (implied by Oldshue and checked by measurement).

The spreadsheet calculations indicate that the design circulation velocity can be achieved with a reasonably low rotational speed of 150 rpm (assuming no slippage). To allow for slippage and the uncertainty in this aspect of design, the impeller is directly coupled to a variable speed DC motor with sufficient extra speed capability (maximum speed of 1500 rpm). The motor is rated at 0.3 kW, giving an installed power of 5.8 kW/m^3 - more than sufficient when compared with the quoted design figures.

At the design speed of 150 rpm, the tip speed of the impeller is 1.04 m/s well below figures quoted as causing crystal breakage and secondary nucleation. Cox and Purdham (1989) quote a maximum tip speed of 10 m/s whilst van der Poel *et al.* (1998) quote a maximum tip speed of 5.8 m/s.

Feed System
With a stirrer in the pan down-take, good feed distribution can be achieved by simply directing the feed line into the top of the down-take. To eliminate the problem of the syrup feed line blocking whilst water is being fed through a separate feed line (as occurred on the laboratory pan) only a single feed line is provided into the down-take - connections outside the pan allow either water or syrup/molasses to be fed through this line.

Sampling Facilities
Two options are provided for sampling from the pan. A small proof-stick above the calandria allows small samples (approx. 0.4 ml) to be taken for microscopic examination. A second sampling point is provided in the base of the pan. By connecting this to a vacuum sampling system, sufficient quantities of sample can be taken for laboratory analysis.

Control System
The control system is a modified version of the system developed for the 13 litre laboratory pan, providing the necessary control loops and an on-line mass balance to estimate the contents of the pan at all stages throughout the boiling cycle.

Ancillary Equipment
The pan sizing was also designed to be compatible with an available electrode boiler with a capacity of 39 kg/hr of steam at 100 kPa abs., produced from water at 25°C. With the pan operating at an assumed maximum specific evaporation rate of 90 kg/hr/m^2 the boiler operates at approximately 87% of capacity.

The pan is provided with a condenser, constructed from a length of 150 mm pipe mounted horizontally and fitted with copper cooling coils. The condenser is capable of condensing 34 kg/hr of vapour at 10 kPa abs.

An accurate estimate of the quantity of incondensibles to be removed from the pan and associated equipment under vacuum is difficult to obtain without extensive calculation (Ryans and Croll, 1981). For industrial installations, Tongaat Hulett normally size vacuum systems based on a practically attainable leakage rate (defined by a standardised test) and estimates of the quantity of air which will enter with input streams. Following this calculation procedure, the estimated volumetric flow of water saturated incondensibles at 30°C and 10 kPa abs. is 2.3 m^3/hr. A single stage liquid ring vacuum pump with a nominal capacity of 40 m^3/hr was installed, providing a considerable safety margin for poor sealing of air leaks and deterioration in pump performance with time.

To allow the pan calandria to be operated under vacuum, the steam condensate and incondensibles removed from the calandria are collected in a condensate receiving tank held under vacuum. A small cooler/condenser cools these streams to minimise the load on the vacuum pump which would otherwise have to condense the vapour and vapour flash from these streams.

Two identical tanks are provided for collecting the condensed vapour from the boiling massecuite and the steam condensate from the calandria. These tanks operate under vacuum and are constructed from 2 m lengths of 300 mm polypropylene pipe fitted with level glasses and drain valves. The volume of approximately 140 litres provides capacity for 9.4 hrs of operation at an average specific evaporation rate for A-massecuite of 40 kg/m^2/hr. The vapour condensate tank is hung from a load cell, providing a mass reading which is fed to the control computer via an RS232 link.

Two feed tanks (for liquor/syrup/molasses and water) are provided. These are large buckets with removable lids (to minimise evaporation) from which feed to the pan is sucked through tubes which dip into each tank. The feed tanks are mounted on an electronic scale with an RS232 connection to the control computer.

Performance of the pilot pan

The initial commissioning of the pilot pan was done using factory syrup to boil A-massecuite. After some initial problems had been rectified, the pilot pan was able to emulate the control and on-line mass balance performance of the 13 litre laboratory pan. An example of the growth trends during the boiling of an A-massecuite in the pilot pan is shown in Figure 6.

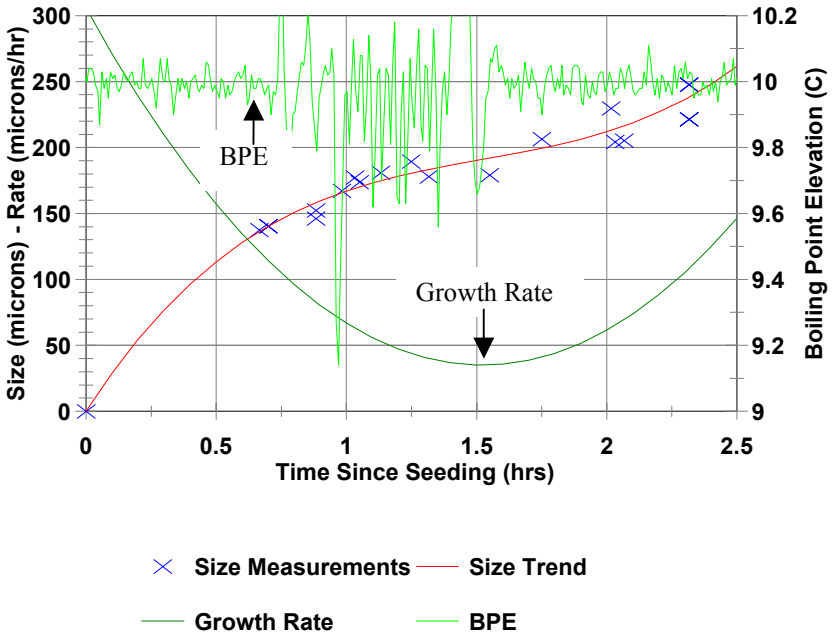

Figure 6. Crystal growth in the pilot scale pan boiling A-massecuite.

The grain size distributions achieved in the pilot appeared substantially more uniform than those from the laboratory pan and similar to those from full scale installations. This is demonstrated quantitatively in Fig 7 where the results are plotted along with previously shown in Figure 2. The CV no longer increases rapidly with an increase in crystal size

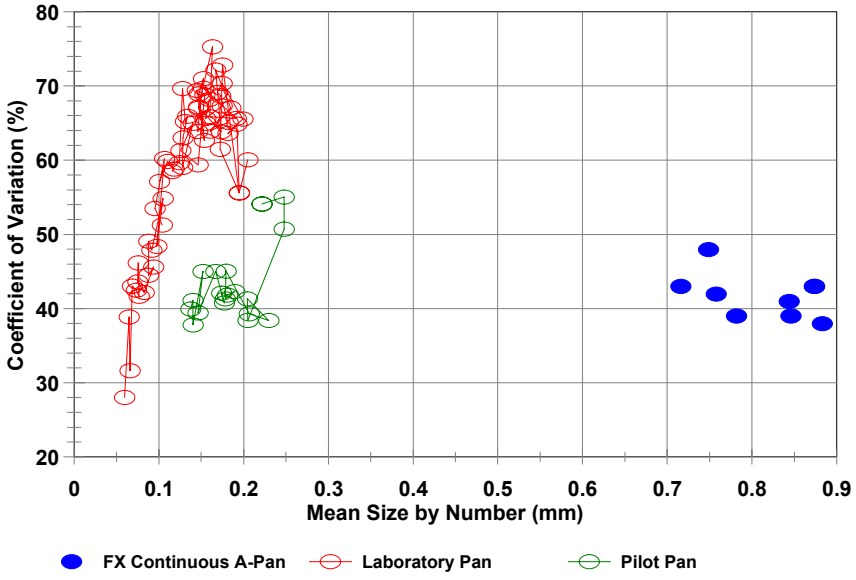

Figure 7. Comparison of pilot scale pan with laboratory scale pan and industrial scale continuous pan.

Time constraints did not allow the investigation into the effect of purity on crystal growth rate but it is intended that this will still happen. The major experimental program undertaken with the pan has been to quantify crystal growth rates and impurity transfer that can be expected the new direct white sugar process developed by Tongaat Hulett Sugar (Fechter *et al*., 2001).

To gain confidence in the ability of the pilot pan to simulate full scale operation, two sets of test boilings were undertaken, one on raw factory syrup (A-massecuite) and the other on refinery fine liquor (first boiling massecuite). The A-massecuite boilings showed that the pilot pan was reasonably representative of industrial boiling conditions, achieving an average colour transfer to affinated sugar of 2.5%. Growth rates were not limited by evaporation rate and average growth rates of 174 microns/hr were achieved over a 3 to 4 hour boiling cycle.

The refined sugar boilings were able to achieve an average colour transfer to affinated sugar of 5.1% and an average crystallisation rate over the boiling cycle of as high as 250 microns/hr.

Using the successful A-massecuite and refinery boilings as a reference, the pilot pan was used to simulate the four boiling scheme of the new direct white sugar process.

Preliminary tests with C-massecuites have shown that the pan is able to achieve a brix at strike of over 94 with a very “gummy” massecuite.

Conclusions

By learning from the limitations of a 13 litre laboratory scale pan it has been possible to design and construct a 50 litre pilot scale pan which is able to simulate industrial boiling conditions. This provides a facility for studying sugar crystallisation under closely controlled conditions. The small scale of the pan also allows operating conditions to be varied over a much wider range than could be contemplated with experimentation on a full industrial scale.

The pan performance has been checked by boiling refinery fine liquor (1st boiling massecuite) and raw sugar factory syrup (A-massecuite) and found to give performance comparable to full scale operation. Preliminary tests indicate that the pan is able to process high viscosity C-massecuites although it may not be possible to reach the highest brixes obtained at the end of the strike in full scale pans.

The pan has proved particularly useful for measuring the crystallisation rates and colour transfer that can be achieved with the process streams in a new, direct white sugar manufacturing process.

Although the original objective of quantifying the effect of impurities on crystal growth rate has not yet been achieved, the pan has demonstrated the potential to generating the necessary data.

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