

# SOME DEVELOPMENTS IN FLOTATION CLARIFICATION

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## Abstract

Flotation clarifiers are used on syrup in some southern African mills to improve raw sugar quality. Air bubble size is a critical performance factor, and sintered pipe sections have replaced mechanical aerators for the dispersion of air in the clarifier feed. The process is particularly effective in eliminating suspended material from direct consumption raws. Data are provided on removal of bagacillo and increase in filterability. The results of plant trials on the use of syrup sulphitation in conjunction with flotation are summarised. A purification process for raw melt has been developed using the same design of flotation unit as for raw syrup. This involves liming of the melt followed by a light carbonation. The resultant precipitate is floated off and disposed of by returning to a syrup flotation clarifier in the raw house. The requirement for filters is reduced to a "check filtration" duty on clarified melt, eliminating the cost and complexity of the two-stage filtration and desweetening of carbonation muds. Laboratory and preliminary plant results for this process are mentioned.

## Introduction

Flotation clarification of syrup has been in operation at a few South African mills over the past 20 years. The original motivation was to enable production of VHP quality sugar at mills that received poor quality cane (Rein *et al.*, 1987; Rein, 1988). Use of flotation clarification processes has subsequently been extended to meet the following needs:

- Reduction of insoluble matter in direct consumption brown sugars
- Further reduction of VHP sugar colour (trial basis only)
- Low-cost purification of melt in a back-end refinery.

Following some comments on operation of the flotation process in general, this paper discusses applications of each of the above.

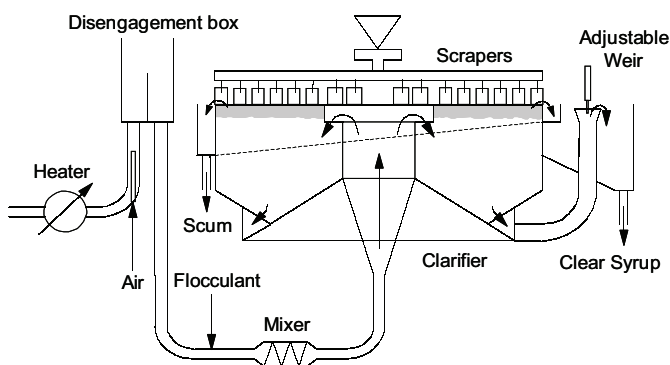


Figure 1. A typical flotation clarifier vessel.

## Operation of Flotation Clarification.

### Importance of Air Addition

The formation of a scum that is stable for several hours and is easily removed from the surface of the syrup depends on having air bubbles ranging in size from 5 to 20 microns, that are incorporated inside the floc formed by the flocculant. Bubbles that are too large break out of the floc after a while and the particle will sink.

Early methods of aeration used oversized centrifugal pumps with the impellers machined down so that they were inefficient and produced high turbulence (Rein *et al.*, 1987). These pumps gave erratic performance and proved troublesome to maintain. They have since been replaced by sintered sparge tubes which have generally proved successful provided that they are of the correct construction and pore size, and that velocity of the syrup flow across the surface of the sparger is high enough to strip off sufficiently small bubbles.

### Syrup Heating and Cooling

Raw syrup requires heating to 85°C prior to flotation. This should be a relatively simple operation but problems have been experienced with blockage of plate heat exchanger channels in one case, and with steam hammer in a direct contact heater at another installation. Cooling of syrup after flotation clarification is necessary to avoid sugar loss and colour formation in subsequent storage. This is achieved simply and effectively using a small flash vessel whose vapour space is connected to the evaporator final effect.

### Scum Depth

The flotation clarifier is provided with an adjustable weir that controls the level of the syrup (and therefore the scum) in the clarifier. The consistency and depth of the scum is examined by the operator from time to time and the level of the syrup set by adjusting the weir height if necessary. Automation of this process has been patented (Schumann, 1995) using infrared measurement of scum level, but life of the sensors immersed in the hot syrup has been a problem. The use of fibre optics may provide the solution here.

## Reduction in Insoluble Matter by Flotation Clarification

Insoluble matter such as bagacillo can detract from the appearance of direct consumption brown sugars, particularly when used in beverages such as coffee. A flotation clarifier was installed at Mount Edgecombe mill (ME), which packed a large proportion of its raw sugar production for the retail market. Analysis for both insoluble matter in total and for bagacillo was carried out in 1994 on weekly samples of raws from Mount Edgecombe and the Maidstone mill (MS), then a producer of bulk raws only. Results appear in Table 1.

Insoluble material and bagacillo at Mount Edgecombe were only a fraction of the levels found at Maidstone. It is possible that Maidstone may have suffered a greater degree of post-contamination of wind-blown bagacillo into uncovered A crystallisers. Nevertheless the flotation clarifier at Mount Edgecombe clearly played a major role in reducing the insolubles content of their product sugar.

At the end of 1994 the Mount Edgecombe mill was closed permanently. The flotation clarifier was moved to Maidstone, which took over the packing of brown sugar for Tongaat-Hulett Sugar, and has operated there ever since. Early in the 1999 season, problems were experienced with the air sparging system at times, which resulted in sub-optimum performance of the flotation clarifier. These problems were rectified during the mid-season operating period (June to September). Later in the season poor clarification was again experienced, this time due to blockage of the in-line mixer. The effects of these differences in flotation clarifier efficiency are reflected graphically in Figure 2 and can be summarised as follows.

- Turbidity removal is the main measure of flotation clarifier performance, with an aim of 75% or more. This was achieved from June to August.
- Bagacillo in treated syrup is essentially a mirror image of turbidity removal, dropping sharply from May to June and then rising again in September.

Table 1.

Analysis	Insolubles (ppm)		Bagacillo (ppm)	
	MS	ME	MS	ME
Flotation Clarifier?	No	Yes	No	Yes
Week Ending :				
23-Jul-94	120	23	91	7
30-Jul-94	114	50	88	32
06-Aug-94	99	62	81	16
13-Aug-94	216	35	172	17
20-Aug-94	171	23	132	7
27-Aug-94	105	24	87	10
03-Sep-94	189	27	161	12
10-Sep-94	174	26	139	17
17-Sep-94	216	36	154	10
24-Sep-94	216	22	150	11
01-Oct-94	193	41	148	21
08-Oct-94	197	32	89	9
15-Oct-94	231	37	133	24
Average	172	34	125	15
Difference		-80%		-88%

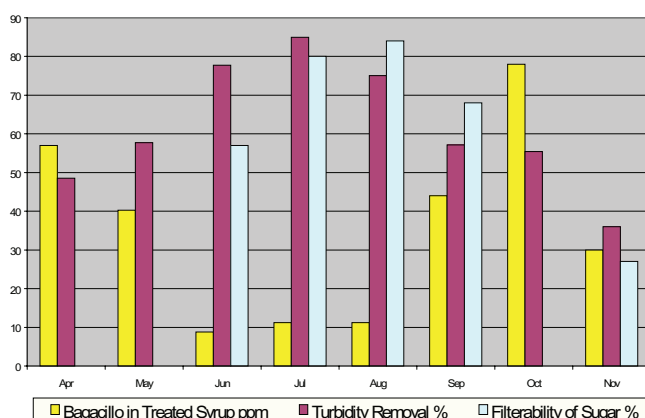


Figure 2. MS Syrup and Sugar Analyses 1999.

- Due to complaints from international customers about poor filtration of some South African raws, filterability analysis of export sugar was started during 1999 with a specification of 45 minimum. Filterability for that proportion of Maidstone's production that went to export correlates well with turbidity removal across the syrup flotation clarifier, and the two values of 80 plus were the highest recorded from any of the 13 mills supplying sugar to the Terminal.

#### Sulflotation Process for Low-Colour Raws

Premium markets may exist at certain times and places for raw sugar with a colour below the normal range for VHP sugar of

1200 to 1500 ICUMSA units. Plant trials were carried out at Felixton in 1991 on a process aimed at producing sugar of 850 colour.

Felixton already had a syrup clarifier and the trials involved addition of sulphur dioxide, plus lime slurry for neutralisation, to the clarifier feed. The process was operated for a few days at a time, alternating with conventional flotation involving addition of flocculant only, over a period of 14 weeks. Average colour and turbidity reduction performance is compared for the two processes in Table 2.

Earlier laboratory work had shown 23% colour reduction for sulflotation versus 6% for simple flotation (Rein 1988). The factory trials gave a similar result (7%) for simple flotation but a somewhat lower figure of 15% for sulflotation. The reduction in sugar colour, relative to the average for simple flotation, was larger at 24%. It has been found previously that the beneficial effect of flotation clarification on sugar colour is greater than indicated by the improvement in syrup colour, and it appears that a similar enhancement may apply for sulflotation compared to simple flotation. Nevertheless, the aim of 850 colour was not achieved at the average SO<sub>2</sub> dosage rate of 0,2% on brix and further work on the process was shelved.

### Carbflotation Process for Melt

Carbonatation is an efficient process for purifying raw melt. As conventionally applied, it requires expensive two-stage filtration plant for separation of calcium carbonate precipitate. Rein (1988) reported preliminary laboratory work on a process that would greatly reduce the filtration requirement, by using flotation to separate the precipitate, which could then be disposed of by recycling to the raw house clarification stage. Hon-Jun (1989, 1990) subsequently reported commercialisation in China of carbonatation followed by flotation of the precipitate, but in that process the scums are combined with phosflotation

Table 2.

Treatment	Simple Flotation	Sulflotation
SO <sub>2</sub> on Brix (%)	0	0,2
<b>Change over Flotation:</b>		
Feed Syrup Colour	24700	23300
Treated Syrup Colour	22900	19900
Colour Reduction	7%	15%
Turbidity Reduction	77%	77%
<b>Change over Crystallisation:</b>		
Pan Feed Colour	24600	23000
Sugar Colour	1210	920
Colour Reduction	95%	96%
<b>Change over Affination</b>		
Whole Sugar Colour	1210	920
Affinated Sugar Colour	590	430
Colour Reduction	51%	53%

scums and treated using dedicated filters instead of by recycling.

When the need arose for melt purification at the Triangle (TR) factory in Zimbabwe, investigation of the carbflotation process was resumed as follows.

### Background and Laboratory Work

The use of flotation using the formation of calcium carbonate as the solid phase to absorb impurities is attractive for the following reasons:

- It has been established that in the carbonatation refining process only a small fraction of the calcium carbonate formed is used to remove the impurities (mainly colour), the remainder being formed as a filter aid. If flotation is used then only as much calcium carbonate is formed as is required to remove colour.
- Operation of the carbonatation process requires that steps be taken to ensure that the precipitate formed is filterable (i.e. that sufficient carbonate is formed as filter aid). For this reason the normal carbonatation operation is carried out quite slowly to produce a filterable bulky precipitate. Furthermore, impurities such as starch and phosphate, which act as crystal habit modifiers, must be removed from raw sugar to maintain acceptable filtration rates. A flotation process, by comparison, will require a fine precipitate with maximum surface area and filterability concerns are not important.
- A flotation clarifier is cheaper to build and operate than the filtration equipment required for a conventional carbonatation refinery.

The process was particularly suited to TR for the following reasons:

- TR has an ethanol plant and the fermenters would provide an adequate supply of CO<sub>2</sub> at a high concentration (a distinct advantage over flue gas) to the refinery.
- Being a back-end refinery, the scum from the carbflotation could be returned to the raw house syrup clarifier, whose scum in turn is returned ahead of the juice clarifiers.
- The syrup clarifier has been installed for some years and the staff is therefore familiar with the operation of flotation clarification equipment.

The possibility of building and operating a flotation clarifier using carbonatation as a basic process was investigated on a pilot (laboratory) scale. Based on this laboratory work the following was established:

- Good mixing of the solution/slurry during the process is critical, and the precipitation reaction (i.e. the formation of the solid phase) must take place as rapidly as possible. This requirement is less critical if suitable crystal habit modifiers (CHMs) are present or added.
- For the work undertaken in our laboratory, phosphate was found to be a satisfactory CHM and gave good results. Even though only a small quantity is needed, it is relatively cheap and easily available.

- A pre-flocculation with cationic flocculant (alum) improved flocculation.
- The best anionic flocculant tested was Magnafloc LT-27.
- Up to 40% colour removal was achieved in the laboratory trials, i.e. slightly less than the indication from limited tests several years before of 50% removal.

*Plant description*

The process, shown in Figure 3, is as follows:

- A-sugar is melted to produce melt at about 30 t/hr at 70° brix and 85°C.
- Phosphate (as phosphoric acid) is added. Alum can be added if required.
- The melted sugar solution is limed according to the setpoint determined from the laboratory work, which is approximately 0,2% lime (as CaO) on brix.
- The melt is gassed to a final pH of between 7,5 and 8 with CO<sub>2</sub> to form a fine calcium carbonate precipitate.
- Air is sparged into the mixture and the solution passes through an air disengagement box to remove large bubbles.
- Flocculant is added and the melt is allowed to separate from the scum in a standard Tongaat-Hulett design flotation clarifier.
- The melt from the clarifier (underflow) is polish filtered through a Gaudfrin filter.
- The scum is returned to the raw house syrup clarifier.

*Commissioning*

Prior to the installation of the carbflotation process the TR refinery remelted A-sugar which was then filtered and

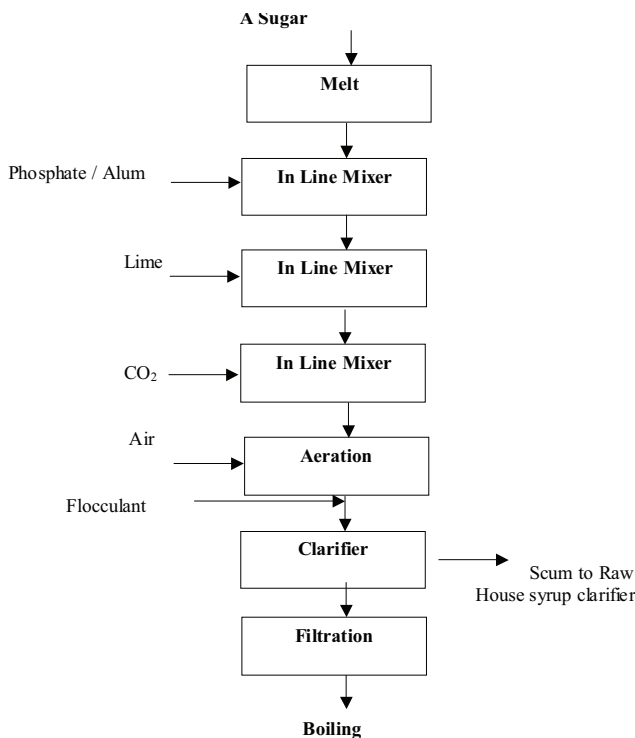


Figure 3.

recrystallised to produce sugar with a colour of about 80 to 100 ICUMSA. The carbflotation process was installed with the goal of producing sugar of less than 40 colour. The plant was installed in such a way that the refinery could be run with and without the carbflotation process.

The plant was commissioned during the last few weeks of the 1999 season. During the initial trials it was established that the air sparging is critical to the success of the process and further adjustments were made to the design to ensure fine air dispersion into the carbonated melt. The Gaudfrin filter was not able to be cleaned as the normal acid cleaning facility had not been installed, and thus did not operate optimally. In spite of this, with carbflotation in operation sugar of 40 colour was produced.

Further trials are planned for the 2000 season and this will give a better indication of the long-term benefits of the carbflotation process.

**Conclusions**

Flotation clarification of syrup has previously been shown to be an effective technique for increasing raw sugar pol and reducing molasses viscosity. Its usefulness for reducing suspended matter content, and increasing filterability, of raw sugars has now also been proven. The only additives required in this application are anionic polyelectrolyte, and dispersed air with a sufficiently small bubble size.

The production of low colour raw sugar by combining syrup sulphitation with flotation has been demonstrated on a plant scale but not commercialised.

A plant for purification of melt using light carbonation in conjunction with flotation is currently being optimised. By returning the carbflotation scum upstream of a raw syrup clarifier, whose scums in turn are disposed of via mixed juice ultimately to raw house filter cake, the requirements for filter plant to eliminate impurities from the refinery are minimised.

**Acknowledgements**

A number of people have been involved in these developments over the years. Particular mention needs to be made of Philip Ingham who undertook the sulflotation work at Felixton, Rhona Mohabir and Lorraine Makhanya who continued the laboratory carbflotation investigations, and Aaron Mugadhi, Patrick Smith and Steve Paver who are handling the carbflotation plant optimisation at Triangle.

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