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## SYRUP CLARIFICATION FOR PLANTATION WHITE SUGAR TO MEET NEW QUALITY STANDARDS

By

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### **Abstract**

There is significant interest in many sugar producing countries towards producing a higher quality plantation white or direct consumption sugar with lower colour and lower residual sulfur dioxide levels. National standards for residual sulfur dioxide and colour have been lowered to reflect the growing demand for improved quality and concerns for health risks.

Investigations at SRI have focused on identifying optimum conditions for clarification options to remove scale components, colour and other impurities by clarifying intermediate and final effect syrups. Trials in two Australian factories demonstrated the benefits for improved sugar quality and productivity improvements for the pan and centrifugal stages.

Recent investigations in four overseas countries have focused on the application of syrup clarification with or without syrup sulfitation to achieve compliance with the changing standards.

Results show that syrup clarification technology can be used to achieve lower sugar colours and reduced residual sulfur dioxide. In one case where syrup clarification was used in conjunction with syrup sulfitation, the colour of the product sugar was reduced from about 150 IU to below 100 IU. In another case, the colour of the sugar was reduced from 150 IU down to 120 IU by replacing the syrup sulfitation process with syrup clarification. In the latter case, residual sulfur dioxide values were reduced from about 25 ppm down to about 14 ppm. There are other advantages to be derived from clarification of the raw syrup, including reduced turbidity and lower ash. A reduction in viscosity has provided productivity gains for the pan and centrifugal stations.

### **Introduction**

In many cane sugar producing countries, the standard sugar product has a very low colour in the range 80 to 250 IU. This sugar is commonly referred to as plantation white sugar although the precise quality standards vary from country to country. The methods employed to produce this sugar usually involve sulfitation of the juice and/or the syrup and, in a few cases, clarification of the evaporator syrup.

There has been significant interest from many factories in these countries towards producing a higher quality plantation white sugar with lower colour and lower residual sulfur dioxide levels. National standards for residual sulfur dioxide and colour are being lowered to reflect the growing demand for improved quality and concerns of health risks. For example, in 2004, China lowered by up to 40% the maximum limits of sulfur dioxide to between 6 and 30 mg/kg depending on the grade of sugar produced.

Sulfitation reduces the concentration of colour compounds including plant pigments (phenolics and flavonoids), melanoidins and some colour precursors such as organic acids and other decomposition products that can contribute to colour development during later processing. Sulfitation of syrup acts to prevent further colour development during the crystallisation process. One option to replace the second sulfitation process is to use syrup clarification based on the phosphatation process.

The phosphatation process can reduce colour compounds that are produced during the factory processes. Such colour and colour precursor groups include melanoidins, caramels and alkaline degradation products of fructose and glucose.

SRI investigations into syrup clarification date back to 1960 when laboratory and factory trials were undertaken to assess the benefits of phosphatation clarification of syrup to improve the filterability of raw sugar (SRI, 1961; Foster *et al.*, 1962). It was demonstrated that a process of heating syrup to 76–86°C and the addition of phosphoric acid and lime would cause precipitation of a calcium phosphate scum that included fats, waxes, proteins and ash constituents. It was concluded that the performance of the juice clarification stage had a major effect on the improvement achieved with syrup clarification. The factor having most influence on the performance of the syrup clarifier was the amount of aeration treatment given to the syrup before it entered the clarifier.

Bennett (1971) undertook a major research and development programme to investigate the use of speciality chemicals as part of the phosphatation clarification process for refinery melt liquor. Two chemicals; Talofloc (cationic colour removal agent) and Taloflote (polyacrylamide flocculant) resulted from the work. A similar flocculant, Talodura, was subsequently developed for use in syrup clarification in raw sugar factories.

There have been a number of articles in the literature reporting outcomes from more recent investigations into the use of syrup clarification including Rein and Cox (1987), Fok Hon-Jun (1989), and Smith *et al.* (2000).

Very little is known about the clarification of intermediate syrups (i.e. from 3<sup>rd</sup> and 4<sup>th</sup> effects of a quintuple evaporator set). Precipitation and removal of polysaccharides, colourants and colour precursors, ash and scale-forming constituents from intermediate syrup should result in reduced scaling in the final effect as well as provide sugar of improved quality.

SRI has recently undertaken extensive investigations into the benefits of clarification of intermediate and final effect syrups for Australian factories and some overseas factories seeking to improve their sugar quality and reduce residual sulfur dioxide levels.

### **Laboratory investigations**

Experiments conducted in the laboratories at SRI involved both intermediate and final effect syrups and were designed to determine:

- the level of removal of polysaccharides, colour, ash and turbidity for a range of chemical additions;

- crystal growth rates in both raw and clarified syrups collected during the factory trials; and
- chemical and X-ray powder diffraction analyses of the components removed in the clarification process.

Individual experiments involved the addition of phosphoric acid (250 or 500 mg/kg solids) to syrup samples at 80°C before liming. The limed syrup was aerated using a high speed, high shear mixer prior to the addition of the flocculant. The flocculants used for the study (and their average hydrolysis level) were A2100 (5%), LT27AG (27%), A2120 (30%) and A2125 (34%). These anionic flocculants had average molecular weights of  $\sim 23 \times 10^6$ .

### Results of clarification of intermediate syrups

The composition of the main inorganic components, turbidity and colour of the clarified syrups are given in Table 1 and indicate:

- Syrup clarification achieved a reduction of 86% in turbidity for the 4<sup>th</sup> effect syrup but only 54% for the 3<sup>rd</sup> effect syrup.
- There was only a marginal reduction in colour for the 4<sup>th</sup> effect syrup.
- Clarification at pH 7.8 gave the maximum reduction in residual phosphate (P<sub>2</sub>O<sub>5</sub>), but gave the largest increase in calcium oxide (CaO).
- A significant increase in silica (SiO<sub>2</sub>) content occurred during clarification of the 3<sup>rd</sup> effect syrup.

Table 1 – Clarification experiments with intermediate (3<sup>rd</sup> and 4<sup>th</sup> effect) syrups at pH levels of 6.5 and 7.8 using A2120 flocculant at 10 mg/kg solids.

Sample	Brix	pH	Turbidity @ 900 nm	Colour (IU)	CaO (mg/kg solids)	MgO (mg/kg solids)	SiO <sub>2</sub> (mg/kg solids)	P <sub>2</sub> O <sub>5</sub> (mg/kg solids)
Raw 3 <sup>rd</sup> effect syrup	34.1	6.1	13	16000	2207	2163	257	55
Clarified 3 <sup>rd</sup> effect syrup	34.5	6.5	6	15700	2422	2178	689	137
Clarified 3 <sup>rd</sup> effect syrup	34.4	7.8	8	16500	3080	2107	689	11
Raw 4 <sup>th</sup> effect syrup	45.2	5.9	61	16800	2400	2235	241	53
Clarified 4 <sup>th</sup> effect syrup	45.0	6.5	8	16300	2435	2189	442	65
Clarified 4 <sup>th</sup> effect syrup	45.2	7.8	9	15800	3214	2237	252	8

## Results of the effects of additives on intermediate syrup clarification

The effect of the addition of chemicals to precipitate scale-forming components and other constituents during intermediate syrup clarification was investigated. In addition, the effect of other chemicals to reduce colour and polysaccharide content of intermediate syrup was also investigated.

### ***Removal of scale-forming components***

Most of the oxalate and aconitate ions present in the juice and sugar process streams are highly associated and hence are bound within the syrup matrix. As a result, they are not readily accessible for spontaneous chemical reactions. Adding free oxalic or aconitic acid should readily initiate precipitation with available free calcium ions and impact on the residual contents of these acids. Clarification of 3<sup>rd</sup> and 4<sup>th</sup> effect syrup was carried out in the presence of additional oxalic acid and aconitic acid. The results shown in Table 2 indicate that:

- The addition of oxalic acid to intermediate syrup affects SiO<sub>2</sub> solubility. This work, to a certain degree, validates recent work on the composite fouling of calcium oxalate and amorphous silica in which calcium oxalate solutions either promoted or inhibited SiO<sub>2</sub> polymerisation, depending on the initial calcium oxalate and sucrose concentrations (Yu *et al.*, 2002; Yu *et al.*, 2003). Their studies showed that the amount of composite scale deposited on a heat exchange surface and the scale texture are dependent on sucrose concentration, the initial calcium oxalate and SiO<sub>2</sub> concentrations and calcium oxalate and SiO<sub>2</sub> supersaturation ratios.
- Clarification of 3<sup>rd</sup> effect syrup in the presence of aconitic acid resulted in a significant increase in the SiO<sub>2</sub> content of the clarified syrup while clarification of 4<sup>th</sup> effect syrup resulted in a significant decrease in the SiO<sub>2</sub> content.

The results of the organic acid analysis of selected clarified syrups are presented in Table 3. They show a reduction in the total organic acid contents of the treated 3<sup>rd</sup> effect clarified syrup samples but an increase in the total organic acid contents of the treated 4<sup>th</sup> effect syrups. The increase may be due to sucrose degradation of the higher brix material by the chemical additive.

When oxalic acid at 50 mg/kg solids was added, the oxalic acid content of the 3<sup>rd</sup> effect clarified syrup was ~50% lower than the level in the clarified syrup without additive. Further intermediate syrup clarification trials were conducted to verify the results. Four intermediate syrup samples were treated with oxalic acid at 20, 50 and 100 mg/kg solids. Although there was a drop in the oxalic acid content of the 3<sup>rd</sup> effect syrup when 50 mg/kg solids of oxalic acid was used, the drop (relative to the control) varied between 3 and 27% as compared to ~50% reported for the previous series. It is speculated that the differences in the composition of the unclarified syrups influenced the amounts of residual oxalic acid remaining after the clarification process.

Table 2 Effect of additives on the SiO<sub>2</sub> concentration during clarification of intermediate syrups.

<b>Syrup</b>	<b>Additive</b>	<b>Additive conc., mg./L</b>	<b>SiO<sub>2</sub> conc., mg/kg solids</b>
3 <sup>rd</sup> effect (2000 work)	None	None	320
	Oxalic acid	20	577
		50	256
		100	468
3 <sup>rd</sup> effect (2001 work)	None	None	607
	Oxalic acid	20	783
		50	665
		100	816
	Aconitic acid	100	n.d.
		200	737
		400	n.d.
4 <sup>th</sup> effect (2000 work)	None	None	130
	Oxalic acid	20	245
		50	222
		100	282
4 <sup>th</sup> effect (2001 work)	None	None	677
	Oxalic acid	20	431
		50	487
		100	538
	Aconitic acid	100	204
		200	194
400		97	

n.d. = not determined

Table 3 – Concentration of organic acids in clarified intermediate syrups.

Additive	Oxalic (mg/L)	Aconitic (cis & trans) (mg/L)	Citric (mg/L)	Gluconic (mg/L)	Malic (mg/L)	Succinic (mg/L)	Glycolic (mg/L)	Total organic acid (mg/L)
<b>3<sup>rd</sup> effect</b>								
None	80	2188	626	424	790	135	116	4359
50 mg/kg solids oxalic acid	39	1919	628	376	691	96	55	3804
200 mg/kg solids aconitic acid	72	1975	396	296	493	85	99	3416
150 mg/kg solids sulphite acid	63	1915	837	509	549	80	58	4011
<b>4<sup>th</sup> effect</b>								
None	56	1672	417	260	375	61	34	2875
50 mg/kg solids oxalic acid	62	2168	207	167	392	66	35	3097
200 mg/kg solids aconitic acid	82	2543	260	238	436	82	80	3721
150 mg/kg solids sulphite acid	84	2532	431	316	477	80	69	4058

#### ***X-ray fluorescence (XRF) and X-ray powder diffraction (XRD) analyses of scum samples***

XRF and XRD studies were carried out on six samples of precipitates obtained in the presence of oxalic acid and aconitic acid during intermediate syrup clarification studies.

XRF analyses indicated CaO, SiO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, Fe<sub>2</sub>O<sub>3</sub> and MgO were the main oxides in all the scum samples.

Interpretation of the XRD data shows that all the scum samples are microcrystalline with calcium oxalate, calcite and hydroxyapatite being the major crystalline compounds in all the scum samples.

#### ***Colour and total polysaccharide of intermediate syrup***

The performance of a number of compounds to reduce the colour and polysaccharide contents of intermediate syrups was investigated.

Table 4 shows the results of the effect of added cationic polymer (polyamine (C573) and polyformaldehyde (WTS PS 5110)), and diethyl amino ethane (DEAE) bagasse on the colour and polysaccharide content of clarified intermediate syrup. Greater reductions were generally achieved with the 4<sup>th</sup> effect syrup.

Table 4 – Effect of additives on intermediate syrup colour and polysaccharide content.

Additive	Additive conc. (% v/v)	Colour (IU) (Reduction, %)		Total polysaccharide (mg/kg solids) (Reduction, %)	
		3 <sup>rd</sup> effect syrup	4 <sup>th</sup> effect syrup	3 <sup>rd</sup> effect syrup	4 <sup>th</sup> effect syrup
None (i.e. control)	None	14 300	15 500	3890	4664
DEAE bagasse (% w/v)	1.00	13 800 (3.5)	13 400 (13.5)	n.d.	n.d.
DEAE bagasse (% w/v)	2.00	12 200 (14.7)	13 400 (13.5)	3853 (1.0)	4466 (4.3)
C573 (polyamine)	0.06	10 200 (28.7)	11 200 (27.7)	n.d	n.d.
C573 (polyamine)	0.08	9120 (36.2)	8380 (45.9)	3686 (5.2)	4185 (10.3)
WTS PS 5110 (polyformaldehyde)	2.00	9320 (34.2)	9520 (38.6)	3096 (20.4)	3934 (15.7)

n.d. = not determined

The results provide the following information:

- DEAE bagasse reduced the colour of clarified syrup by up to 15% compared to the control sample. It had little impact on the polysaccharide content.
- C573 reduced the colour (up to 46%) and polysaccharide content (up to 10%).
- WTS PS 5110 reduced colour (up to 40%) and polysaccharide content (up to 20%).

The results have shown that, at high addition rates, WTS PS 5110 is an effective coagulant for reducing both colour and polysaccharide content while C573 effectively reduces the colour of intermediate syrup. Additions at lower rates had little effect. The maximum permissible dose (U.S. FDA Code of Federal Regulations) for polyamines is 300 ppm. The limit on the use of polyformaldehydes is not known.

### Results of clarification of final effect syrup

The greatest reduction in turbidity was achieved at a pH of 6.5 with a medium to high hydrolysis flocculant at 10 mg/kg. As the pH increased, the turbidity reduction decreased.

The composition of the inorganic components and the colour of the syrups clarified with LT27AG and A2120 were determined. The results are given in Tables 5 and 6 and are similar for the two flocculants. Clarification of syrup resulted in ~50% reduction in SiO<sub>2</sub> but an increase by a similar amount for CaO at the higher pH level. There was no benefit by increasing the amount of P<sub>2</sub>O<sub>5</sub> (as phosphoric acid) added to syrup from 250 to 500 mg/kg solids. The residual MgO and P<sub>2</sub>O<sub>5</sub> values were lower with the samples limed at pH 7.8 compared to those samples limed at pH of 6.5. However, higher levels of residual CaO were obtained for the samples limed at pH of 7.8.



Table 5 – Effect of phosphoric acid and limed syrup pH on syrup constituents after clarification with LT27AG flocculant.

Constituents	Concentration in unclarified syrup (mg/kg solids)	Concentration in clarified syrup (mg/kg solids)			
		P <sub>2</sub> O <sub>5</sub> at 250 mg/kg solids		P <sub>2</sub> O <sub>5</sub> at 500 mg/kg solids	
		pH = 6.5	pH = 7.8	pH = 6.5	pH = 7.8
CaO	1897	2034	2850	2038	2596
MgO	1955	1894	1761	1814	1805
SiO <sub>2</sub>	438	232	235	295	230
P <sub>2</sub> O <sub>5</sub>	115	82	46	217	54
Colour (IU)	10 800	12 100	9300	11 100	8750

Table 6 – Effect of phosphoric acid and limed syrup pH on syrup constituents after clarification with A2120 flocculant.

Constituents	Concentration in unclarified syrup (mg/kg solids)	Concentration in clarified syrup (mg/kg solids)			
		P <sub>2</sub> O <sub>5</sub> at 250 mg/kg solids		P <sub>2</sub> O <sub>5</sub> at 500 mg/kg solids	
		pH = 6.5	pH = 7.8	pH = 6.5	pH = 7.8
CaO	1897	2240	2876	2178	2647
MgO	1955	1870	1804	1873	1153
SiO <sub>2</sub>	438	311	122	232	246
P <sub>2</sub> O <sub>5</sub>	115	235	36	245	60
Colour (IU)	10 800	12 500	9450	11 400	9590

The results show that the colour of the clarified syrup was reduced by about 14% at a pH of 7.8 while a pH of 6.5 resulted in a slight increase in colour.

#### Effects of other additives during clarification of final effect syrup

A number of colour precipitants were tested in a program to identify any commercially available chemicals that would enhance the colour reduction potential of the syrup clarification process. The additives tested are listed in Table 7.

Table 7 – Details of additives used in the clarification of final effect syrup.

Name	Description	Recommended dosage
Qemitreat SEP	Poly-DMDAC / epiamine blend, decolourising agent	20-40 mg/kg
Qemitreat SE	Cationic polymer blend, decolourising agent	20-40 mg/kg
Qemibiosurf 7150	Cationic polyelectrolyte blend, decolourising agent	20-40 mg/kg
Chitosan	Cationic coagulating agent	Not known
Foodpro SA9855	Substitute for phosphoric acid in syrup clarification. The active ingredient is believed to be a soluble phosphate.	80-250 mg/kg syrup
Sodium polyphosphate		Not known
Polyaluminium chloride		Not known

Superfloc A2125 performed slightly better than either LT27AG or A2120 (based on turbidity removal) and was used for this series of tests. A further test was conducted at pH 6.8. At this pH, the colour reduction was 7% greater than at pH 7.5.

Additives were assessed at addition rates of 40 and 150 mg/kg solids and solution pH values of 6.8 and 7.5. The results indicate that there was no extra turbidity or colour reduction obtained from the addition of any additive at either concentration.

The addition of Foodpro SA9855 at concentrations of 80 and 250 mg/kg syrup resulted in 7% less colour removal than the addition of a similar concentration of phosphoric acid. The addition of sodium polyphosphate produced identical results to Foodpro SA9855.

### Summary of the laboratory results

A summary of the optimum conditions to achieve the maximum benefit from clarification of intermediate or final effect syrup is given in Table 8.

Table 8 – Optimum conditions for clarification of syrup.

Impurity component	Syrup stream	Optimum conditions
Turbidity	4 <sup>th</sup> and final	pH 6.5, 250 mg/kg P <sub>2</sub> O <sub>5</sub>
Colour	4 <sup>th</sup> and final	pH 7.8, 250 mg/kg P <sub>2</sub> O <sub>5</sub>
CaO	Final	pH 6.5
P <sub>2</sub> O <sub>5</sub>	Any	pH 7.8
SiO <sub>2</sub>	Final	pH 7.8

### Factory trials in Australia

Factory trials were undertaken at two factories using two different designs of flotation clarification equipment capable of clarifying up to 30 m<sup>3</sup>/h of final effect syrup. In both cases, flotation of the scum was achieved using dissolved air flotation technology. Sufficient syrup was clarified to enable batches of sugar to be produced in 80 t and 100 t strike pans. Separate strikes using raw syrup were boiled in the same pans to allow the benefits of syrup clarification for the pan and fugal stages to be quantified.

#### Trials at factory #1

The final effect syrup was heated to 85°C and then treated with 250 mg/kg of P<sub>2</sub>O<sub>5</sub> on solids as phosphoric acid and limed to a pH of 8.0 using lime saccharate. Superfloc A2120 was added at a rate of 10 mg/kg solids.

A strike using untreated syrup was conducted in the fully controlled pan. Crystal was initiated using slurry and the pan drop massecuite was fugalged in batch fugals. The same pan and procedure was followed for the clarified syrup trial. Shipment sugar of approximately 99.6 pol was produced.

The following average benefits were achieved across the syrup clarifier:

- 92% reduction in turbidity;
- 1.6 units increase in syrup purity; and

- Reductions of colour (10%), ash (3.6%) and polysaccharide (marginal) in the clarified syrup.

Analyses of the sugars produced during the trial are given in Table 9. Significantly, there were reductions in the colour%impurities (21%) and ash%impurities (63%) in the sugar produced which suggest that colour and ash were selectively removed during syrup clarification.

Table 9 – Analysis of the sugar produced during a single trial at factory #1.

Sample	Pol	Moisture (%)	Purity (%)	Colour, (IU)	Ash (%)	M.A. (mm)	CV
Sugar from untreated syrup	99.68	0.08	99.76	639 (257)	0.08 (0.02)	0.83	0.22
Sugar from clarified syrup	99.62	0.08	99.70	620 (215)	0.04 (0.02)	0.66	0.28

Note: The values in parentheses are for the colour and ash in the affined crystal

The 16% reduction in crystal colour is indicative of the improved crystal growth rate and circulation in the strike pan arising from the reduction in viscosity of the clarified syrup.

Despite the smaller crystal size and wider CV of the sugar produced from the clarified syrup, the fugal settings for the wash and spin times were similar. It was expected that reduced times would be possible for crystals of similar size characteristics to those produced from untreated syrup.

Crystal growth rate measurements indicate that the crystal growth rate increased by an average of 26% following clarification. The viscosities were measured at 65EC on the saturated syrups used for the growth rate measurements, and showed a decrease of 5% following clarification. The methods used were described by Broadfoot and Steindl (1980).

### **Trials at factory #2**

For the trials at factory #2, a small clarifier with a maximum capacity of about 6 m<sup>3</sup>/h was used. Each strike consisted of 40 t of C magma seed, 50 t of either treated or untreated syrup and then 10 t of A molasses was boiled back at the end of the strike. The measured benefits were due to just 50% of the feed material to the strike. During the fugging process, wash and spin times were adjusted to provide a sugar product that satisfied the Brand 1 specification (~99.0 pol). Details of the conditions for the trials are given in Table 10. A summary of the results are given in Tables 11, 12 and 13.

Table 10 – Operating data for the syrup clarification trials at factory #2.

Operating parameter	Average	Range
<i>Flotation clarifier</i>		
Feed concentration	66.9 brix	63.8 – 69.9 brix
Final concentration	62.6 brix	59.3 – 65.9 brix
Feed temperature	81.7 °C	75.7 – 84.2 °C
Feed pH	7.3	6.9 – 7.5
Phosphoric acid addition	300 mg/kg P <sub>2</sub> O <sub>5</sub> on solids	250 – 350 mg/kg P <sub>2</sub> O <sub>5</sub> on solids
Flocculant	Superfloc A2120 at 10 mg/kg on syrup solids	
Air flow	0.5 L/min	
<i>Vacuum pan</i>		
Steam flow	14.7 t/h	10.8 – 15.8 t/h
Vacuum	-85.5 kPa (g)	
Balance water		0 – 6 t/h

Table 11 – Summary of the changes in the syrup following clarification.

Parameter	Outcome
Turbidity	Reduced by 90%
Purity	Increased by 0.3 units
Colour	No change
Colour%Impurities	Increased by 3%
Ash	Reduced by 10.6%
Ash%Impurities	Reduced by 7.9%
Total polysaccharides	Reduced by 3%

Table 12 – Summary of the main changes in the quality of the raw sugar.

Parameter	Outcome
Colour	Reduced by 39%
Colour%impurity	Increased by 9.7%
Ash	Reduced by 47%
Ash%impurity	Reduced by 6.6%
Reducing sugars	Reduced by 24%

Table 13 – Centrifugal settings to process the A massecuites from the untreated and clarified syrup strikes.

Massecuite feed	Timer settings		
	Purge, s	Wash, s	Dwell, s
Massecuite from untreated syrup	35	2.0	25
Massecuite from clarified syrup	35	1.4	18

The two major benefits across the flotation clarifier were the reductions in turbidity and ash. The lack of any reduction in the colour of the syrup cannot be explained, although the initial colour was relatively low. As seen in Tables 12 and 13, both the colour and ash of the sugar are reduced significantly and the pol increased ( $-0.25$ ) with less wash and spin times at the centrifugals.

Laboratory growth rate studies determined that the average growth rate increase in clarified syrup over untreated syrup was 5%. The average reduction in viscosity was 6%.

### **Discussion on the factory trials**

There are significant differences in the results obtained from the clarification trials at the two factories. Three times more ash was removed from the syrup in the clarifier at factory #2. The only possible explanation for the variance was the difference in pH of the syrup (8.0 at factory #1, 7.3 at factory #2). The ash reduction in the sugar from the factory #1 trial was 10 times greater (expressed as ash%impurity) than the reduction achieved at factory #2. This has most likely resulted because of the boiling scheme used by factory #2 where 50% of the A massecuite strike was derived from untreated material. At factory #1, the strike was completed with clarified syrup footing, slurry graining and fed on clarified syrup only.

The lack of any colour reduction across the clarifier during the trials at factory #2 was disappointing and most likely due to the lower pH. The 10% reduction in colour that was achieved at factory #1 was similar to results reported by other researchers. The syrup colour at factory #2 was usually very low, typically 10 000 to 12 000 IU, whereas the factory #1 syrup had a colour value of more than 14 000 IU.

### **Results from overseas factories**

SRI has been involved with the installation of clarification equipment into a number of factories in India, China, Central and South America. In all cases the objective was to achieve improvements in sugar quality at higher throughputs. The performance of some of these installations is summarised to demonstrate that syrup clarification can replace syrup sulfitation and provide a sugar with a lower colour and lower residual sulfur dioxide levels.

### **Syrup clarification performance at an Indian factory**

The factory used double sulfitation to achieve product sugars with an average sugar colour of 140 IU. The syrup sulfitation step was achieving an average colour reduction of 9%.

Initial operation with the syrup clarification plant replicated the optimum conditions determined for Australian syrups. However, it became apparent that no colour reduction was achieved for pH levels above 7.0 and, in some cases, there was a colour increase in both the clarified syrup and the sugar. Analysis of the raw syrup indicated that the residual calcium (expressed as mg/kg CaO per 100 brix) in raw syrup ranged from 10 000 to 12 000. This is about five to ten times the level in Australian syrups. The standard practice for juice sulfitation in India is to slug dose the juice entering the sulfitor with lime to a pH greater than 9.0.

Results from trials at the factory when only phosphoric acid was added to the syrup prior to clarification indicated that about 12% reduction in calcium (as calcium oxide) was achieved.

Trials were also conducted where lime saccharate was added to raise the pH to just 6.0. The resulting reduction in residual CaO was measured at 9.1%. The 24 hour average difference in residual CaO across the clarifier between added lime and no added lime was 400 mg/kg CaO on

brix. This indicates that the added phosphoric acid is preferentially reacting with the added CaO. However, there is another 9% of CaO being removed from the syrup in one or more forms that is independent of whether or not lime is added to the syrup.

Results achieved following the installation of syrup clarification equipment are given in Table 14. The final effect syrup was passed through the normal sulfitation process and then through the syrup clarification system. The product sugar colour ranged between 80 and 100 IU and the residual sulfur dioxide content reduced from 20-25 to 9-12 mg/kg. The results were achieved in spite of the very wide range of syrup feed properties.

Table 14 – Summary of results obtained during a two week period at the Indian factory.

	pH	Brix	Colour IU	CaO mg/kg solids
Raw syrup	6.5 (5.9 – 7.3)	55.9 (45.0 – 64.0)	13 190 (7300 – 23 480)	10 470 (8600 – 12 330)
Clarified syrup	6.2 (4.9 – 6.6)	54.6 (47.0 – 62.0)	10 780 (6570 – 18 100)	9280 (7800 – 11 200)

Note: Values in brackets represent the range of values measured.

Laboratory trials conducted on site determined that the optimum pH for clarification was between 6.2 and 6.8. Within this pH range, the colour reduction averaged 18% and the turbidity reduction averaged 95%.

#### Syrup clarification performance at a Chinese factory

A syrup clarifier installed at one factory was capable of processing about 72% of the syrup flow. When the clarifier was in operation, the remainder of the syrup (~28%) was passed through the sulfitation system. Average results for periods before and during operation of the clarifier are given in Table 15. The operating conditions during the data collection period were:

Syrup flow rate	25 m <sup>3</sup> /h.
Syrup temperature	82.5 °C
Raw syrup pH	6.8
Lime saccharate addition	Nil
Phosphoric acid addition	240 mg/kg syrup solids
Flocculant addition rate	9 mg/kg syrup solids
Boiling scheme	C-B-A
Wash times at fugals	7 to 10 seconds

Table 15 – Summary of the quality results for the plantation white sugar produced at one Chinese factory with a syrup clarifier.

Process	Pol	Reducing sugars %	Ash %	Colour IU	SO <sub>2</sub>
100% Syrup sulfitation	99.76	0.072	0.065	153	25
72% Clarification & 28% syrup sulfitation	99.77	0.064	0.055	120	14

Subsequent to this data collection period, a high molecular weight polyaluminium chloride was being added at a rate of 1500 mg/kg syrup solids. The factory reported a colour reduction across

the clarifier of 40% as well as an improvement in the turbidity. This is a significant improvement on the result achieved at SRI for similar addition rates of polyaluminium chloride. No information on any change to the final sugar quality is available.

## Conclusions

The scale removal experiments conducted in the SRI laboratories have indicated a small reduction in the oxalic acid content of the 3<sup>rd</sup> effect syrup when oxalic acid at 50 mg/kg syrup solids was added before clarification. The reduction achieved was variable and was sensitive to the quantity of oxalic acid added to initiate precipitation. Investigations of other options for scale reduction were not successful.

The factory trials demonstrated that clarification of final effect syrup can assist to improve the quality of raw sugar and provide additional benefits of pan and fugal productivity improvements. For Australian factories producing raw sugar, these benefits appear variable and most benefit arises from the reduction in viscosity of the syrup and reductions in ash in the sugar.

The overseas investigations have demonstrated that syrup sulfitation can be successfully replaced by syrup clarification to achieve improvements in plantation white sugar quality. As well, the level of residual sulfur dioxide is reduced significantly. These investigations have clearly shown that the optimum conditions for Australian syrups are not appropriate for syrups resulting from sulfited juice as is common in many overseas factories.

The benefits of clarifying intermediate effect syrup appear to be limited. However, it is proposed to undertake factory trials in the future to further assess the benefits as issues such as steam economy become important to factories contemplating cogeneration.

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## References

- Bennett, M.C.** (1971). Preliminary results on the use of 'Talofloc' in sugar refining. Proc. Sugar Ind. Technol., 30: 52-59.
- Broadfoot, R. and Steindl, R.J.** (1980). Solubility-crystallisation characteristics of Queensland molasses. Proc. Int. Soc. Sugar Cane Technol., 17: 2557-2581.
- Fok Hon-Jun** (1989). Flotation-clarification in sugar refining – Part 1. Int. Sugar J, 91:1088, 145-147.
- Foster, D.H., Sockhill, B.D. and Wright, P.G.** (1962). Performance of factory flotation plant for the improvement of syrup filterability. SRI Technical Report No. 70.
- Rein, P.W. and Cox, G.S.** (1987). Syrup clarification in raw sugar mills. Proc. S. Afr. Sugar Technol. Ass., 61: 22-31.
- Smith, I.A., Schumann, G.T. and Walthew, D.C.** (2000). Some developments in flotation clarification. Proc. S. Afr. Sugar Technol. Ass., 74: 265-266.

**Sugar Research Institute** (1961). Filterability of raw sugars and syrups. Special report to member mills. April.

**Yu, H., Sheikholeslami, R. and Doherty, W.O.S.** (2002). Mechanisms, thermodynamics and kinetics of composite fouling of calcium oxalate and amorphous silica in sugar mill evaporators – A preliminary study. *Chem. Eng. Sci.*, 57(11): 1969-1978.

**Yu, H., Sheikholeslami, R. and Doherty, W.O.S.** (2003). Studies on the interactions of calcium oxalate monohydrate and silica scale components. *Proc. Aust. Soc. Sugar Cane Technol.*, 25 (CD-ROM).