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M 12. EVALUATION OF MUD FILTRATE CLARIFICATION TO IMPROVE FACTORY PERFORMANCE

By

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Abstract

Clarification performance and flocculant dosage is strongly linked to the mud solids loading in the feed entering the clarifier. The recycle of filtrate can represent an extra ~10-15% mud solids loading on the clarifier, thereby reducing its effective capacity. Filtrate recycling may cause significant increase in turbidity, complexed calcium ion formation, phosphate, proteins and polysaccharides in mixed juice that impact on evaporator scale formation and molasses exhaustion. The paper details the results obtained from laboratory, pilot scale and factory trials of filtrate clarification using both sedimentation and flotation methods. Clarified filtrate could be produced of similar quality to ESJ. Filtrate clarification was able to significantly remove insoluble solids, turbidity, phosphate, and polysaccharides content with slight reductions in minerals content of the filtrate. On the basis of improved filtrate quality, the clarified filtrate could be directed to ESJ, instead of the normal practice of directing the mud filtrate to mixed juice. The potential impacts of implementing filtrate clarification are discussed in respect to improved performance and throughput of the clarification station.

Introduction

Clarification performance and flocculant dosage is strongly linked to the mud solids loading in the feed entering the clarifier. Carryover of mud solids into the clarified juice is likely to reduce sugar filtrability and other quality parameters, as well as increasing scale formation in the evaporators and pans. The recycle of filtrate can represent an extra ~10-15% mud solids loading on the clarifier, thereby reducing its effective capacity. The impurities recycled with filtrate (i.e. proteins, organic acids and mineral ions) can also impact on downstream processing operations. Large amounts of organic acids in the primary juice will not only result in the use of increased lime usage, but will hold some of the calcium in a combined complex state. The calcium-organic acid complex will slowly breakdown during subsequent processing resulting in the formation of scale and other impurities. The complex that is not broken will mostly end up in molasses and may impact on exhaustion.

The recommended dosage of flocculant added to a clarifier is 2 to 4 ppm. Previous studies (Crees *et al.*, 1977) have shown that even when as low as 2 ppm of flocculant is used, carryover of flocculant still occurs. Clarifying the filtrate may reduce the amount of flocculant reintroduced back to mixed juice. As flocculants are of very high molecular weight, their presence even in minute quantities can promote precipitation of scale-forming compounds.

There are alternative ways to reduce mud solids recycle, such as improving filter retention. While there are many types of filter equipment available, rotary vacuum filters are predominantly used to process the clarifier underflow in Australian mills. Improved filter retention can be achieved through operating slower filter drum speeds to achieve thicker cakes, recycling a proportion of the filtrate as mud dilution and adding lime. These measures are partly effective in the short term and also rely on the capacity of the filter stage. However, adding lime is essentially adding more scale-forming components (calcium) to the process and also has the effect of blinding filter screens. This can increase maintenance time, and reduce throughput and recovery of the filter station. Improving filter retention does not have a significant impact on reducing soluble impurities that are mainly responsible for scale formation, and other processing and sugar quality problems.

The adoption of filtrate clarification technology overseas (Singhal *et al.*, 2005) is largely based on the need to improve sugar quality and recovery. Benefits realised include increased purity across clarification, reduced colour, turbidity and calcium content. In Australia, the technology would also target these improvements but additional benefits would be achieved through the reduction in impurities and their impact on scaling in evaporators as the cost of evaporator cleaning is significantly higher in Australia compared to overseas factories. For sugar factories like Tully Mill where the clarifier is close to capacity, filtrate technology would also allow greater utilisation of existing equipment and prevent the need to install additional capacity.

The paper examines laboratory, pilot scale and factory investigations that aimed to determine the impacts of filtrate clarification on filtrate quality and factory performance.

Laboratory filtrate clarification trials

Laboratory trials were conducted to establish suitable processing conditions for clarification of mud filtrate. Filtrate quality and clarification performance were assessed from settling rates, mud volume (after 30 min), and analyses of turbidity, protein and polysaccharide content and residual metal ion levels.

Turbidity of the juice was obtained by measuring the absorbance (A) at 900 nm in 1 cm cuvette cells against Milli-Q water (BSES, 2001). The turbidity was calculated as 100A and reported in nephelometric turbidity units (NTU).

Total polysaccharides were determined using the Sugar Processing Research Institute (SPRI) procedure (Roberts, 1981).

The protein content was determined using the bicinchoninic acid (BCA) Protein Assay Kit (Pierce, Bonn, Germany). Assays were prepared on microplates by mixing 25 µL of protein extract with 200 µL of BCA and incubated at 37 °C for 30 min. The microplates were cooled to ambient temperature and the absorbencies measured at 562 nm. A stock of bovine serum albumin (BSA) was prepared and used to generate a calibration curve.

The inorganic ion concentrations were analysed using an inductively coupled argon plasma optical emission spectrometer (ICP-OES). To reduce the interference of the organic sugar matrix, samples were diluted to a sucrose concentration of 2 wt%.

Tully Mill

To understand the benefits of filtrate clarification, base data were obtained at Tully Mill (September, 2011) by collecting mixed juice and filtrate to produce a 'synthetic' primary juice with and without filtrate (prior to liming). The primary juice with filtrate contained higher proportions of polysaccharides (7%), proteins (30%) and calcium (12%), though with a lower phosphorus content (5%).

Batch settling (sedimentation) and dissolved air flotation (DAF) experiments were carried out on-site at Tully Mill (September, 2011) on filtrate with pH adjustment (to pH ~7.2) using lime saccharate. In the sedimentation experiments, the juice was settled in the SRI batch settling kit. DAF experiments were undertaken using a customised stainless steel pressure vessel (22.5 cm diameter, volume 2 L) where compressed air was supplied for 5 min at an operating pressure of ~70 psi before allowing the filtrate to flocculate and separate by flotation. Clarification was undertaken for the following conditions:

- liming (with and without phosphoric acid) at 76 °C, then boiling the filtrate and settling/floating the flocs using a flocculant (5 mg/L Magnafloc LT27)
- liming at the filtrate temperature (~57 °C) followed by boiling and settling/floating the flocs using a flocculant
- clarification at lower temperatures. The benefit of clarification at lower temperatures would mean that little or no extra heating would be required in factory applications.

Table 1 presents the results of the initial filtrate clarification experiments. The turbidities of the clarified filtrate obtained by sedimentation are in the range 6.7 to 12.2 NTU, whereas the values obtained by the flotation process are in the range 21.2 to 26.1 NTU. The values obtained by the normal clarification process are comparable to those of clarified juice (5-14 NTU) typically obtained at Tully Mill. There was no noticeable difference between the filtrate clarified with or without phosphoric acid irrespective of the clarification process.

Table 1—Initial laboratory filtrate clarification tests

Mode	Sedimentation				DAF			
H_3PO_4 addition	Yes	No	Yes	No	Yes	No	Yes	No
Clarification temperature, °C	100	100	76	76	100	100	76	76
Filtrate turbidity, NTU	9.8	8.3	6.7	12.2	23.9	24.9	21.2	31.3
Mud height, %	15.5	16.5	23	20				
Mineral ion concentration								
CaO, mg/L	420	427	434	420	427	448	462	462
MgO, mg/L	158	151	154	159	164	162	159	166
P_2O_5, mg/L	27	29	28	28	39	43	44	46
SiO_2, mg/L	132	130	131	122	128	130	124	122

The levels of phosphorus and calcium are higher in the filtrates treated by the DAF process. Not boiling the filtrate prior to clarification had no effect on the proportion of the inorganic ions in the clarified juice, as similar results were obtained with boiling.

A set of trials was conducted with the DAF process at a shorter aeration time of 2 min rather than 5 min because of the extremely dark clarified filtrates obtained at the longer time. The injection of air contributes to oxidation reactions causing an increase in colour. The amount of flocculant used in previous trials was reduced from 5 mg/L to 2 mg/L and the filtrate was limed at 57 °C. The limed filtrate was boiled and air injected to float the mud and impurities. Under these conditions (results not shown) there were significant reductions in polysaccharides (up to 50%), proteins (up to 50%), magnesium (49%), and phosphorus (up to 83%) in the clarified filtrate. However, the turbidities of the clarified filtrates were unacceptably very high (>30 NTU).

The initial laboratory trials showed that clarification by sedimentation is a better process than clarification by DAF to effectively reduce the amounts of non-sucrose impurities in the filtrate. Good clarification, at 76 °C, was achieved without boiling the limed filtrate (Table 1). Typically, heating primary juices to 76 °C or higher is expected to improve precipitation of calcium and removal of polysaccharides (Doherty, 2011). Further trials with limited or no heating, boiling or the addition of phosphoric acid were carried out. The effect of the pH of the limed filtrate on the clarification process was also examined. These trials (Table 2) showed clarification achieved significant removal of polysaccharides (~40%), proteins (20-60%) and phosphate (50-65%).

Table 2—Additional laboratory filtrate sedimentation clarification tests

Description	Filtrate	No phosphate addition	Phosphate addition	No phosphate addition, and no heating or boiling		
Clarification temperature, °C	57	76	76	57	57	57
pH	5.9	7.2	7.2	7.2	7.6	7.8
Filtrate turbidity, NTU	-	16.9	17.2	17.1	15.8	14.4
Polysaccharides, mg/kg solids	163 460	96 970	101 550	98 100	95 430	94 910
Proteins, mg/kg solids	45 770	53 030	32 310	-	36 900	17 830
Mineral ion concentration						
CaO, mg/L	336	350	336	336	336	350
MgO, mg/L	149	114	111	113	114	113
P_2O_5, mg/L	92	34	37	44	34	32
SiO_2, mg/L	129	118	114	107	114	114

There was no difference worth noting between clarification at the lower temperatures or with phosphoric acid addition, except a slightly lower phosphorus content was obtained by clarification at 76 °C. For the no heating, boiling or phosphoric acid addition, increasing the liming pH from 7.2 to 7.8 reduced clarified juice turbidity as well as phosphorus and protein content.

Isis and Pioneer Mills

Batch laboratory filtrate clarification tests were also conducted during the 2012 season at Isis Mill and Pioneer Mill (Table 3) in order to evaluate if similar benefits could be achieved with different juice/filtrate properties. One important processing variation employed at Isis Mill is the addition of lime saccharate to the mud mixer to condition the mud prior to filtration. This assists with mud cake formation allowing improved cake washing to reduce mud pols. The practice adopted by Isis Mill is to dose lime saccharate to achieve a filtrate pH of 7.8-8.0. This practice was once employed at Tully Mill but was since discontinued as the process increased calcium levels in ESJ through recycling and Tully Mill also experienced excessive scaling of filter screen surfaces causing bald spots and reduced throughput and pol recovery. This required filters to be taken off line for pressure or chemical cleaning. These problems are generally not experienced at Isis Mill most likely due to differences in mud and impurity loadings.

The temperature of the factory filtrates at Isis and Pioneer Mills was ~70 °C and the filtrate was typically >10 brix. The filtrates also contained <50% of the amount of insoluble solids compared to Tully Mill. No lime saccharate was used in the tests at Isis Mill as the filtrate pH was ~7.8 (Pioneer Mill filtrate pH was ~6.6). The filtrate clarification tests experienced very fast settling rates and clarified filtrate turbidities of

<10 NTU were easily achieved. The clarified filtrates produced were of similar quality to ESJ.

In the Isis Mill trials (July 2012), boiling and the addition of small amounts of phosphate (<20 mg/L as P_2O_5) reduced the clarified filtrate turbidity from 8-9 NTU to 4-5 NTU with a corresponding increase in mud volume. Reducing the flocculant dosage to 1 mg/L was also able to produce clarified filtrate turbidity of <10 NTU with no phosphate addition when the juice was heated to 76 °C. Interestingly the minerals content of the Isis Mill filtrates were significantly lower than those obtained at Tully Mill even though the filtrate brix at Isis Mill was 50-60% higher than that at Tully Mill. Calcium levels were 3-4 times lower and other minerals were 6-8 times lower indicating the superior quality of Isis Mill filtrate. The quality of the filtrate is linked to cane quality and variety, weather conditions and filter station processing (bagacillo addition and wash water application).

The good quality filtrate at Pioneer Mill (November 2012) also achieved low turbidities of <5 NTU with a low dosage of flocculant (1-2 mg/L). Boiling the filtrate had a marginal impact on clarified filtrate turbidity but increased the amount of polysaccharides and proteins removed. The mineral contents of the Pioneer Mill filtrates were similar to the ESJ at that Mill and to filtrate obtained at Tully Mill.

Initial mud settling rates of >50 cm/min were measured at each of the factories (even though high turbidity clarified filtrate was produced at Tully Mill). These settling rates are substantially higher than that of primary juice, although the filtrate brix and solids loading is ~40-60% of that of primary juice. Lower mud solid loading, can increase the settling rate in the clarifier by reducing the effects of hindered settling (Crees *et al.*, 1978). Dilution of primary juice to reduce solids concentration has also previously been shown to more than double settling rates in juice clarification (Steindl, 1998) which confirms the high settling rates observed for filtrate clarification. Clarifiers are generally sized based on the settling rate, so importantly the high filtrate settling rate means a smaller clarifier is required for a given throughput of juice/filtrate.

Table 3— Isis Mill and Pioneer Mill filtrate clarification tests

Mode	Isis Mill				Pioneer Mill				ESJ (Pioneer)
H_3PO_4 addition	No	No	Yes	No	No	No	No	No	
Flocculant dosage, mg/L	5	5	5	1	2	2	1	1	
Clarification temp., °C	>100	70	76	76	76	100	76	100	
Turbidity, NTU	5.2	8.5	4.6	7	4.1	3.5	4.6	3.5	2.8
Mud height, %	5 - 6	5 - 6	8	5 - 6	13	12	13	8	
Polysaccharides, mg/kg solids					54 100	48 100	55 800	45 300	43 000
Proteins, mg/kg solids					1600	1370	1430	1280	1320
Mineral ion concentration									
CaO, mg/L	102	99	83	101	392	392	392	392	350
MgO, mg/L	14	14	12	14	282	299	299	299	315
P_2O_5, mg/L	6	5	5	5	55	53	57	53	41
SiO_2, mg/L	11	11	9	11	69	71	73	71	90

Pilot filtrate clarification trials

The laboratory trials showed that clarification by sedimentation was the preferred option for treating filtrate and that heating of the filtrate was not required. A pilot filtrate clarification circuit (including phosphoric acid, flocculant and lime saccharate addition) was designed for implementation at Tully Mill (Figures 1 and 2) using a spare settling tank (~6 m^3) that was converted into a sedimentation clarifier.

The conversion included installation of an external flocculant addition and mixing chamber; feed entrance pipe and nozzle; perforated plate and take off launders; and sight windows. A filtrate feed rate of ~250 L/min (10-15% of the factory filtrate stream) was used in most of the trials providing a residence time in the clarifier of ~20 min. While steady filtrate flow rates were achieved in the trials, the metering pump used to dose lime saccharate created a pulsing erratic flow which may not have been beneficial to clarification performance. Trials were conducted during the 2012 season with the pilot rig to examine various filtrate flow rates, pH set points, and flocculant dosage rates. Composite samples of feed and clarified filtrate samples were collected and analysed.

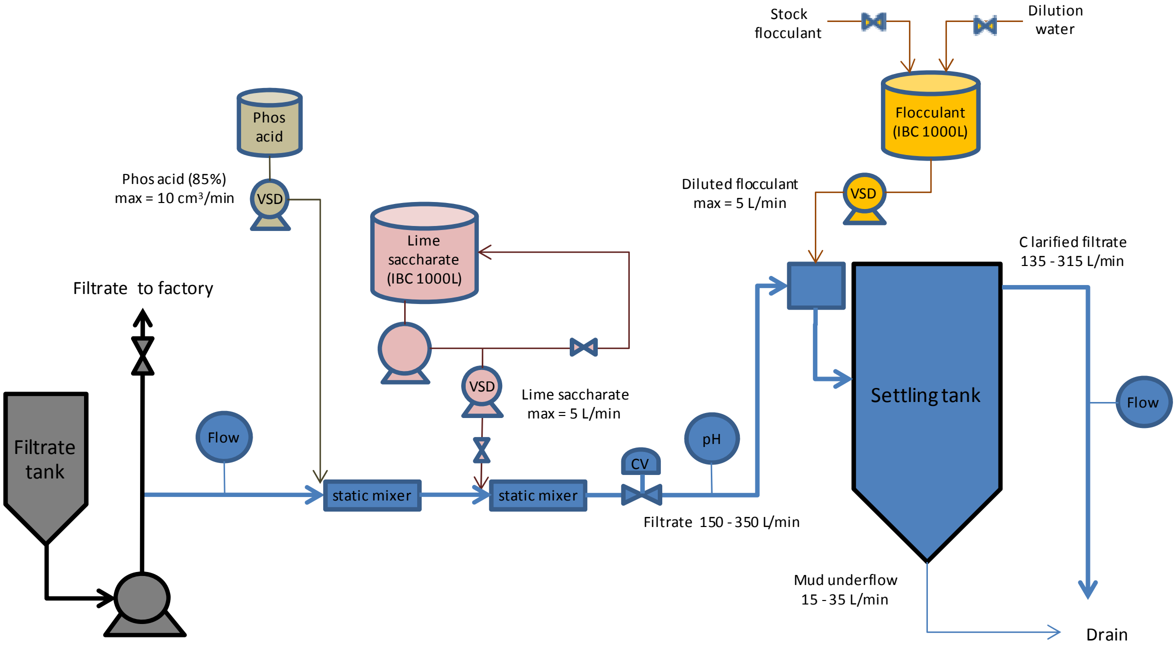

Fig 1.— Process flow diagram of the pilot clarifier

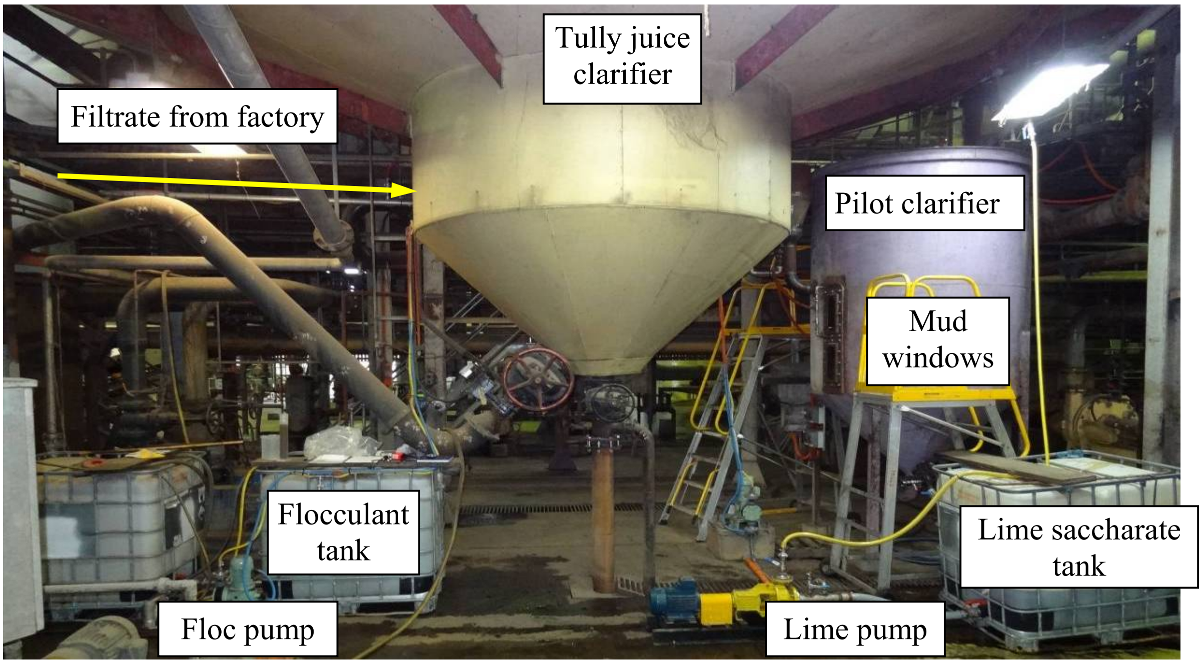

Fig 2.—Pilot filtrate clarifier installation at Tully Mill

Table 4 details the results of the pilot trials. The clarification process reduced the turbidity of the filtrate considerably from a value of >200 NTU to between 15 NTU and 30 NTU. This value is slightly greater than that achieved in previous laboratory filtrate clarification trials and that of clarified juice (5-10 NTU). The clarified filtrate samples had significantly lower phosphorus and magnesium concentrations but slightly higher calcium and silica levels than the filtrate feed. Improved turbidity reduction was achieved with higher pH set points and increasing the flocculant dosage. However, increasing the pH set point above 8.2 led to a significant increase in the residual calcium level.

Filtrate samples were also taken at the pilot clarifier inlet (after flocculant addition) and clarifier outlet and were allowed to settle in tubes, to determine if the lower performance was linked with the pilot clarifier design or operation (i.e. pulsing flows caused by the metering pumps). The filtrate sample taken from the mixing chamber prior to entry into the clarifier produced a lower turbidity juice (~14 NTU) than that exiting the clarifier (~19 NTU). The slight drop in performance may indicate poor feed distribution or non-ideal flow patterns and mixing within the clarifier. It is expected that a larger clarifier would allow a more evenly distributed feed system to be incorporated which would improve the clarification performance.

Table 4—Tully Mill pilot filtrate clarification tests

Description	Feed	1	Feed	2	Feed	3a	3b	3c	3d
pH	6.2	8.2	6.6	8.0	6.1-6.3	7.2	7.5	8.5	9.1
Feed temp., °C	58-60	-	58-60	-	60	-	-	-	-
Filtrate brix, %	6.6	6.9	5.6	6.0	6.2	6.1	6.5	6.7	7
Feed flow, L/min	-	230	-	250	-	200	200	200	200
Flocculant dosage, mg/L	-	4.0	-	5.0	-	7.5	7.5	7.5	10
Turbidity, NTU	450	25.8	430	27.1	217	20	16	15.4	12.7
Polysaccharides, mg/kg solids	173 000	76 700	-	-	270 000	160 000	-	-	-
Proteins, mg/kg solids	9190	4810	-	-	11 400	8620	-	-	-
Mineral ion concentration									
CaO, mg/L	273	336	294	322	308	336	294	392	476
MgO, mg/L	116	105	116	106	108	106	91	93	91
P_2O_5, mg/L	126	36	115	57	140	67	60	50	50
SiO_2, mg/L	54	72	69	97	81	79	88	99	96

Juice processed in the clarifier generally contains 1-2 wt% mud solids but this contributes to mud volumes of 10-15% which are processed by the filter station. Analysed samples of Tully Mill filtrate feed were found to contain ~0.5-0.6 wt% insoluble solids with the filtrate clarification process able to remove >95% of these solids. If the clarified filtrate was returned to mixed juice (15-20% of the total juice flow) for subsequent processing this would result in a reduction in insoluble solids loading to the clarifier of 5-10%. However the reduction in soluble solids will have a greater impact on factory performance.

Factory filtrate clarification trials

In recent years it has been the practise at Tully Mill when liquidating clarifiers, to bring any filtrate from the mud filters back to the incubator, and process through the clarifiers as is done for mixed juice (liming, heating and flocculant addition). Clarified filtrate is then processed through the evaporators. Table 5 details the results of a number of short duration factory trials and shows the clarified filtrate was of similar quality to ESJ or better quality than the ESJ (turbidity). The calcium levels were higher than the ESJ (possibly due to excess lime addition) whereas the remaining mineral ions were lower than those in the ESJ. In some of the trials, the ESJ turbidity was quite high at the time (period immediately before factory shutdown or the poorer quality juice prevalent at the end of season).

Table 5—Tully Mill factory filtrate clarification tests

	2009			2010			2012	
	Filtrate	Clarified filtrate	ESJ	Filtrate	Clarified filtrate	ESJ	Clarified filtrate	ESJ
Turbidity, NTU	1142	10.1	11.6	>2000	22.8	21.1	25.4	26.5
Polysaccharides, mg/kg solids	-	-	-	-	-	-	79 300	62 700
Proteins, mg/kg solids	-	-	-	-	-	-	1620	2820
Mineral ion concentration								
CaO, mg/L	574	420	406	363	458	363	854	588
MgO, mg/L	200	87	184	130	96	113	71	139
P_2O_5, mg/L	198	16	39	149	35	35	28	37
SiO_2, mg/L	75	49	75	45	58	44	39	60
K_2O, mg/L	856	590	1181	691	852	1012	699	964

Discussion and conclusions

The project has examined the clarification of filtrate under a range of conditions. Both flotation and sedimentation clarification were assessed in laboratory scale testing. Both the DAF and the sedimentation clarification processes resulted in significant removal of turbidity, polysaccharides and phosphorus from the filtrate, but the sedimentation process generally produced a better quality filtrate in terms of lower turbidity and colour. Phosphate addition was found to provide only marginal improvement in clarification performance. It was noted that some heating of filtrate may be required from a practical point of view to avoid sucrose degradation from microorganisms. The laboratory trials conducted at different mills found the impurity loading of the filtrate had the greatest impact on clarification performance and optimum processing conditions. Under high impurity loadings flocculant dosage rates were increased to 5 mg/L to achieve reasonable turbidity juices. This is similar to typical practice to increase flocculant addition when processing juices of high mud solids loading (Steindl, 1998). When processing good quality filtrates much lower flocculant dosages of 1-2 mg/L were sufficient to produce acceptable settling rates and turbidities.

A pilot scale clarification process based on sedimentation was designed, constructed and operated by processing ~10-15% of the factory filtrate stream (without heating) at Tully Mill. Trials were conducted to identify some of the practical issues associated with filtrate clarification. A number of short duration factory trials were also conducted at Tully Mill where the filtrate was collected and processed through the clarification circuit (with heating) prior to a factory shutdown. These trials showed that clarified filtrate can be produced of similar quality to ESJ.

Filtrate clarification was able to remove >95 wt% of the insoluble solids and turbidity, 30-40% polysaccharides content and slight reductions (10-15%) in minerals content of the filtrate. Phosphate was also reduced by up to 60-70% in the pilot and factory trials. Based on the improved filtrate quality, the clarified filtrate could then be directed to the ESJ, instead of the normal practice of directing the filtrate to mixed juice, thereby increasing the capacity of the clarifier station. Other potential flow-on benefits from improving clarification include:

- maintaining throughput and reducing carryover into the clarified juice when cane quality deteriorates or when processing high mud solids loading due to improved clarifier capacity
- reducing the propensity of scale formation in heaters and evaporators due to lower ash and impurities leading to reduced factory cleaning requirements
- improving molasses exhaustion (sugar recovery) through the reduction of the proportion of metal-organic acid complexes and polysaccharide content.

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