SEPARATION AND REMOVAL OF PRELIMER SUSPENDED SOLIDS

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Abstract:

In preliming, a substantial portion of non-sugars is aggregated as the juice passes through a pH gradient. In a typical process, these aggregated non-sugars pass through subsequent liming and carbonation steps, where some of these solids are returned to the prelimer and may re-dissolve into the juice. Consequently, this may decrease the purity of the clarified "thin juice." The cost of downstream processes increase with the amount of contaminating non-sugars that need to be removed. Previous research conducted by the beet sugar industry has indicated that the purity of thin juice may increase by 0.5% to 0.6% if the solids generated during preliming are removed prior to carbonation. There have been numerous attempts to adopt this practice, and there has been general agreement that this practice could be extremely beneficial.

Primarily, solids handling issues have hampered successful implementation. Among these was the fragile nature of prelimer solids and sugar loss resulting from problems in filtering these solids. Previous studies by other investigators have indicated that the physical separation of prelimer solids could be greatly improved by cationic polymer application. In the present research, we have identified and tested two effective anionic flocculants that permit the removal of suspended prelimer solids.

During the 2007-2008 campaign, polymer was introduced to the influent of an Enviroclear rapid settler (clarifier) that received a portion of the prelimer effluent. The flocculants that were tested included a food grade anionic flocculent and a new anionic flocculent of very high molecular weight and high mole charge. Both polymers were effective in the capture of prelimer solids in the side stream system receiving approximately 10% to 25% of the factory flow. The resulting floc was found to be robust and amenable to co-filtration with the first carbonation underflow. Filtration of prelimer solids mixed with first carbonation solids did not impact the performance of the Putsch filter presses.

Introduction:

The product of diffusion (i.e., the raw juice) is comprised of approximately 15% sucrose along with proteins, polysaccharides and other soluble or colloidal materials extracted from the beets. Purification of sucrose from the raw juice is accomplished by precipitating the non-sugars from the extracted juice through a number of unit processes on the beet side including preliming, cold liming and hot liming, followed by two carbonation processes.

In preliming, the extracted beet juice is heated and the pH is gradually increased by the controlled addition of lime to a final pH of about 11.5. A substantial portion of non-sugars[1] aggregate as the juice passes through specific pH ranges. In the typical

[1] In this context, sugar refers to sucrose only. Non-sugars include any dissolved or colloidal material other than sucrose

process, these aggregated non-sugars pass through subsequent liming and carbonation steps, where some of these solids may re-dissolve into the juice and decrease the purity of the clarified "thin juice." The costs of downstream unit processes are increased with the amount of contaminating non-sugars that need to be removed. Previous research conducted by the beet sugar industry has indicated that the purity of thin juice can be increased by 0.5% to 0.6% if the solids generated during preliming were removed prior to carbonation. Depending on slice rate, an increase of 0.5% could represent substantial savings over the course of a campaign.[2]

There have been numerous attempts to adopt this practice, including the TASCO (1966) and Novi Sad (1964) juice purification processes (Beet Sugar Technology, 2nd Edition). In the early 1990's, this process was also investigated by most of the domestic US beet sugar producers. There was general agreement that this practice could be extremely beneficial. However, successful implementation was hampered primarily by solids handling issues. Among these was the fragile nature of prelimer solids and sugar loss resulting from problems in filtering these solids.

The study by Lloyd, *et al* conducted at Imperial Sugar (now Wyoming Sugar) in 1991 (Proceedings of the Biennial ASSBT General Meeting) showed that the physical separation of prelimer solids could be greatly improved by cationic polymer application. In bench testing, several Nalco anionic flocculants were identified as having the ability to flocculate the suspended solids in the prelimer effluent. The resulting floc was found to be robust and amenable to filtration.

At the SMBSC factory, an Enviroclear rapid settler (clarifier) was installed to do some pilot testing on a slipstream of the process. In January 2008, a polymer feeder (provided by Nalco R&D) for the process was hooked up to the influent of the Enviroclear rapid settler. Beginning February 21, 2008 a field trial was conducted that focused on the removal of solids generated in the prelimer. Two polymers were tested in this application, TX12621 and N7415SC. The N7415SC polymer is a food grade anionic flocculent, 29% mole % charge, acrylamide/acrylic acid copolymer. The second polymer tested, TX12621, was a very high molecular weight, 40% mole % charge anionic acrylamide/acrylic acid copolymer flocculent.

Purpose:

The factory wanted to investigate the implementation of a solids/liquids separation step to remove solids generated in the prelimer as part of their process. The objective was to remove agglomerated non-sugars from the prelimer effluent such that less contaminating non-sugars proceed to the first carbonation process.

If these non-sugars are not removed, they pass into carbonation and are co-precipitated with calcium carbonate in the carbonation process. The solids formed in carbonation (i.e., underflow) are returned to the prelimer at a rate of approximately 10% of the total raw juice flow. Consequently, precipitated non-sugars in the underflow may get resolubilized and ultimately decrease the purity of the thin juice. Successful implementation of a prelimer solids removal process could potentially decrease beet sugar production costs.

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[2] Savings will be greater in factories where there is no desugarization of molasses

Procedure:

In January 2008, a polymer inversion/makedown unit and feed system for the process was delivered to SMBSC and hooked up to the influent of the Enviroclear rapid settler (i.e., clarifier). Due to extremely cold temperatures at that time, the clarifier could not be operated. When the weather warmed sufficiently we were able to thaw any liquid that had remained in the clarifier and necessary modifications were made by the factory operators. Beginning February 21, a field trial focused on the removal of solids generated in the prelimer was conducted on a side stream flow of prelimer effluent.

Two polymers were tested. Laboratory studies that had been conducted by Nalco had shown these polymers to be acceptable candidates for settling prelimer solids. These polymers had allowed acceptable filtration of the settled solids with good cake formation. Prior to starting on-site application, jar tests were conducted to verify effective polymer dosage. These jar tests indicated that both polymers would optimally settle the prelimer solids at a concentration of 40ppm, as product and provide a relatively clear supernatant.

Phase 1. Application of TX12621 to Process Side stream

Flow of prelimer effluent to the side stream clarifier was originally intended to be about 100gpm. However, the factory was unable to maintain that low flow rate due to plugging at the gate-valve. SMBSC does not use juice screens to remove beet pulp that passes through diffusion and this pulp is carried through the process and was found to accumulate at the control valve. To remedy this situation, flow was increased to over 200gpm. At the higher flow rate, the juice flow was sufficient to carry most to the pulp in the juice stream and minimize episodes of plugging. However, flow would occasionally drop off to less than 100gpm and the operators would need to open the valve briefly to regain the higher flow rate.

Initially, samples of the influent to the clarifier (i.e., prelimed raw juice) and of the overflow were collected while no polymer was applied to establish a baseline. Even without polymer application, some solids settle from the prelimed juice but it was apparent that a lot of suspended solids were contained in the overflow samples. Even slight changes in inlet flow rate caused substantial quantities of solids to “burp” over into the overflow.

After establishing the baseline, we began applying the TX12621 at a low dose. Factory personnel were concerned that the polymer addition might cause problems at the Putsch filter presses. Should the filter presses become blinded when filtering mixtures of prelimer solids and Dorr tank solids, it could shut down the factory until the filter fabrics were changed out on the Putsch filters. For this reason the decision was made to start low and slowly ramp up the polymer dosage.

Over the course of the testing, TX 12621 was applied at concentrations up to 40ppm. Maintaining flows near 200gpm proved to be problematic and the factory increased flow rate to over 300gpm.

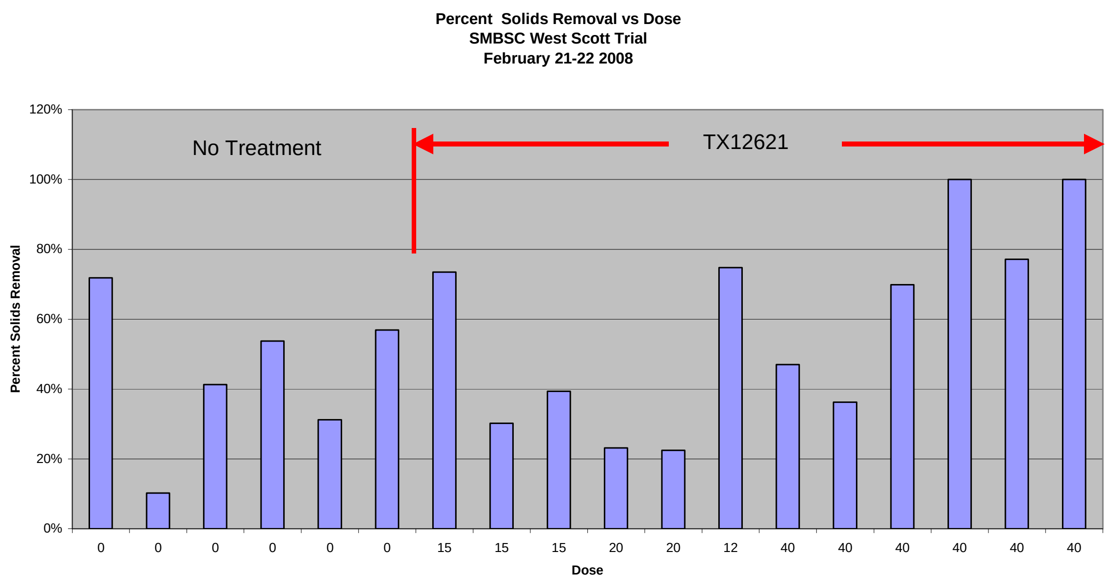
Phase 2. Application of Nalco 7415SC to Process Side stream

On February 26, we continued the testing at SMBSC but switched from the TX12621 to Nalco 7415SC. Jar testing had shown N7415SC to be effective in agglomerating the prelimer solids. SMBSC was using this polymer in their wastewater treatment and had sufficient quantities on-site to allow us to hook up a tote and continue this trial.

<u>Results:</u>

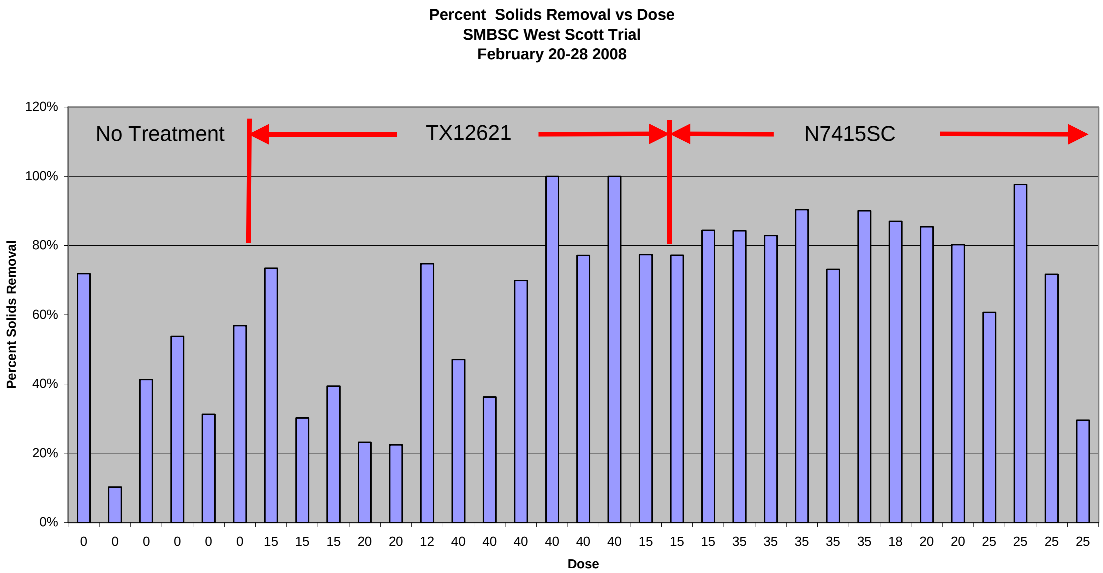
Phase 1. Solids Capture with TX12621 to Process Side-stream

TSS was measured in the influent, overflow, and also in the underflow from the Enviroclear. The data shown below clearly shows that we effectively increased the solids content of the underflow and consistently decreased the solids across the clarifier near 80% and approached 100% TSS removal at doses of 40ppm.



Phase 2. Solids Capture with Nalco 7415SC to Process Side stream

Even at flows consistently over 300gpm, the N7415SC provided good settling of the prelimed solids in a dose range of 15 to 35 ppm. The percent TSS removal across the clarifier was near 80%. For the two final samples, the flow was increase to 450gpm. At the high flow rate, more solids went into the overflow.



The factory continued to run the N7415SC polymer up until the last week of the 2008-2009 campaign. They had planned to use the polymer until the beet slicing was completed, but while conducting anti-foam tests, they experienced a sever infection in the Enviroclear and discontinued the application.

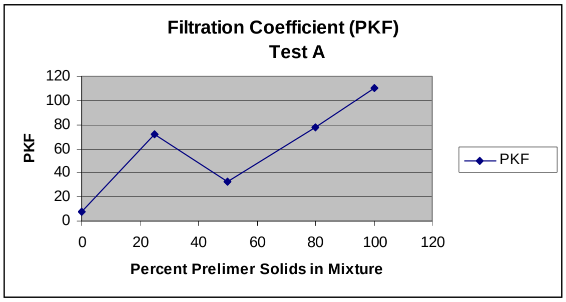
Effect on Solids Filterability

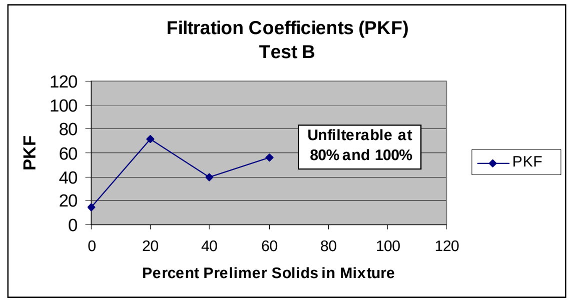
Settling in the Enviroclear continued to be satisfactory as long as polymer was applied. The underflow from this clarifier was sent to the putsch filters along with the 1st carbonation solids and showed no adverse effect on the Putsch filter performance. It had been feared that sending the solids generated in the prelimer to the filter presses would blind the fabric. Solids from the prelimer tend to be more gelatinous and sticky than the 1st carbonation solids, which contain primarily calcium carbonate. The prelimer solids have been difficult to handle in the past, though in this field trial, there were no adverse results at the filter presses.

The fact that there had been no noticeable effect on the filterability at the Putsch filter presses during the polymer application has important implications with respect to the total sugar process. There may be a limit to the amount of prelimer solids that can be mixed into the Dorr tank solids going to the Putsch filter presses, but so far this has not become problematic.

Preliminary bench work had been conducted at SMBSC with an apparatus obtained from Putsch, the manufacturer of SMBSC's filter presses. This work determined the filtration coefficients (PKF) for mixtures of Dorr Tank solids and Prelimer solids. A low PKF would indicate better filtration with a consequently drier cake. Results of these experiments suggested that the best filtration occurred in mixtures that are roughly 40% to 50% prelimer solids.

Dorr Tank solids alone, which were comprised primarily of $CaCO_3$, gave the lowest PKF values. Prelimer solids, which consisted mostly of aggregated colloids were more fragile and very hydrated. Addition of prelimer solids to the Dorr Tank solids tended to increase the PKF. The drop in PKF that was observed in 50% mixtures was unexpected.





Our hypothesis was that PKF would increase proportionally with the increasing percentage of prelimer solids in the mixture. These results, however, indicate that filtration might be optimized by keeping the mixtures near 50:50.

Impact on Thin Juice Purity

To prove the value of removing the prelimer solids ahead of carbonation, The SMBSC Factory Chemist obtained influent to the Enviroclear and made two synthetic thin juice (STJ) preparations in the laboratory. For one he used a well-mixed sample of the prelimed juice and for the other, he let the solids settle and prepared the thin juice from the supernatant. He observed increases in STJ purity from 0.5% to as great as 1.5% when the solids were removed. Previous work in the industry had suggested that removal of solids after preliming could increase thin juice purity by up to 0.6%. Only a few sets of samples were analyzed. In future studies, a greater number of samples will need to be analyzed to determine whether the substantial purity improvements in this study are representative.

Preliminary estimates of ROI

SMBSC processes 2MM ton of sugar beet per campaign. If we assume that we could save an estimated $275K in downstream production costs for each 0.1% increase in thin juice purity, then a potential improvement of thin juice purity by 0.5% would provide nearly $1.4MM in savings for the factory.

The cost can be estimated on the lowest effective dosage we observed. N7415SC applied at an application rate of 15 ppm, on beet, would require 60,000 pounds of polymer over the course of a campaign. Using the cost of this polymer at the time of this trial, the additional polymer cost would be approximately $210,000 per campaign.

Conclusions:

Both Nalco 7415SC and TX12621 polymers are effective in capture of prelimer solids in the side stream system receiving 200gpm to 450 gpm (approximately 10 to 25% of the factory flow). Filtration of the underflows comprised of prelimer solids mixed with 1st carbonation solids does not impact the performance of the Putsch filter presses.

Discussion:

Flows through the Enviroclear during this study were generally between 300 and 350 GPM. When higher flows were attempted, carryover was excessive. Flows lower than 300GPM tended to result in plugging in the piping. This plugging problem was thought to be due to incorrect sizing of valves and possibly configuration of the piping. Plans for next campaign included the installation of automated control valves of appropriate size.

Settling performance of the Enviroclear and limitation of flow rates were also hampered by the configuration of the overflow. The outlet piping was on one side of the clarifier and consequently, the overflow tended to occur over weirs on one side of the clarifier (i.e. short-circuiting). Modifications to the piping to correct this situation were considered before the 2008-2009 campaign.

An unexpected result was the improved settling of the 1st carbonation solids whenever polymer was being applied to the prelimed juice going to the Enviroclear. A mechanism to explain this observation will be investigated in future studies.

A few possible explanations were considered. The underflow from the Enviroclear clarifier was being mixed with 1st carbonation solids downstream from where some of the 1st carbonation underflow was sent back to the prelimer. Therefore, all of the prelimer solids removed in the Enviroclear were going directly to the Putsch filters. Assuming that all of the polymer stayed with the solids, the only plausible explanations are that

- Carryover of polymer treated solids from the Enviroclear enhances settling during 1st carbonation.
- By removing a portion of the prelimer solids, the quality of the 1st carbonation underflow that is returned to the prelimer is improved and enhances 1st carbonation settling.
- The filtrate from the Putsch filters, which is returned to the Dorr tank somehow affects settling in the Dorr tank
- A decrease in the amount of difficult-to-settle solids generated in the prelimer causes improvement in 1st carbonation settling.

There are likely to be other possible explanations and that may be an area of future investigation. This factory is very interested in continuing implementation, though they will be proceeding cautiously and continue efforts to quantify the value of this process to their operation.

References:

1. Beet Sugar Technology, 2nd Edition.
2. Lloyd, Norman, Gary Garcia, Vince Salzman, Fabien Cabrera, Thomas Charboneau and Kenneth Stewart. 1991. Prelimer Solids can be Separated. Proceedings of the Biennial ASSBT General Meeting.