SAYE,* DENNIS J. and XIAOJUN DANG, Separations and Commercial Processes Research, Nalco Company, 1601 W. Diehl Road, Naperville IL, 60563. **Improved Beet Sugar Purification from "Conditioned" Diffusion Juice: Field Studies During the 2003-2004 Campaign.**

ABSTRACT

A diffusion juice conditioning technology was designed to remove dissolved gasses such as CO_2. Degassing of the diffusion juice resulted in a product juice stream of consistently higher pH, thus decreasing lime demand during juice purification and associated costs. That ability was substantiated with a pilot unit operated at a beet sugar factory in the Midwest United States. In addition to decreased lime demand, pilot testing revealed the potential to significantly improve thin juice color and purity. Subsequently, full-scale juice conditioning systems were installed in two Midwest beet sugar factories. By the end of the 2003-2004 campaign, these systems were capable of treating the entire flow of raw diffusion juice at the respective factories. Field studies have shown that treatment of diffusion juice prior to pre-liming significantly impacted the quality of the resulting thin juice. This talk describes the results of field studies and related research performed during the 2003-2004 campaign.

INTRODUCTION

In the extraction of beet sugar, relatively high concentrations of carbon dioxide are generated in the diffuser. When the diffusion juice cools, much of the carbon dioxide remains dissolved. In tower diffusion systems and enclosed slope diffusers, excessive CO_2 remains dissolved and the raw juice going to preliming is more acidic. Consequently, more lime is needed to neutralize the acids and thus, increases the lime demand in the prelimer.

At Nalco, we became aware of a patented technology that was shown in early testing to be capable of removing dissolved CO_2 from sugar beet diffusion juice. This juice conditioning system has been described in a previous presentation[1]. Initial results supporting the concept had been obtained with a small test system treating a 10gpm flow of diffusion juice. Evidence for CO_2 removal was based on an observed increase in pH when the diffusion juice was degassed.

In 2003, Nalco entered into an agreement with the inventor to assist in the development of this technology for the sugar industry. For the first field trial with Nalco, a pilot-scale test unit was installed at a Northern-Midwest beet sugar factory (Factory 1). This unit consisted of three sections and could operate at diffusion juice flows up to 70 gpm. Diffusion juice first passed through a venturi where air could be aspirated into the juice prior to going through a centrifugal separator that permitted release of some of the dissolved gases. The diffusion juice was then pumped through nozzles at the top of the second stage forming droplets, which fell counter-current through a stream of forced air. The small droplets of diffusion juice created a liquid air interface of very high surface area that permitted gas exchange. Juice collected at the bottom of the second stage was pumped through another set of nozzles in the third stage where the droplets were exposed to a vacuum of up to 20 inches (Hg) and ultimately to the prelimer.

At a second Northern-Midwest beet sugar factory (Factory 2), two full-scale juice-conditioning systems were installed. These units consisted of two stages, with the elimination of the first stage described for the pilot unit, above. Each of these units could handle juice flow at 800gpm/unit.

[1] **The Development and Application of a Juice Conditioning System for Increased Sugar Quality and Recovery.** Sanders, D., Nalco Company, 1601 W. Diehl Road, Naperville IL, 60563-1198

Combined, these units could accommodate the entire flow of diffusion juice at this site. The fieldwork described in this paper was conducted at these two sites.

The initial objective of the field trials was to validate the capability of this degasification system to remove excess dissolved carbon dioxide from the diffusion juice. The results obtained in the initial testing suggested that this juice conditioning technology may impact the purity and color of downstream process juices. This represents work in progress and is an update on continuing research to optimize the application of this technology to the beet sugar process.

PROCEDURE

MATERIALS AND METHODS

Determination of Lime Demand. The initial pH of the treated and untreated Diffusion Juice was determined for each sample and the demand for alkalinity (i.e., lime demand) was determined by simple titration with 0.1N NaOH to a pH 11.0 endpoint. The titration volume represented the amount of lime that would be applied in the prelimer to neutralize acids in the diffusion juice. This includes the acidity contributed by dissolved CO_2.

Synthetic Thin Juice. Synthetic Thin Juice (STJ) was prepared using the procedure of the Factory 1 laboratory.

Purity Determinations (Apparent Purity) Apparent Purity was determined using an Autopol 880 Polarimeter with integrated J-57 Refractometer (Rudolph Research Analytical, Flanders, NJ)

Color Determinations Color of Synthetic Thin Juice was determined by pH-adjusting the Synthetic Thin Juice to pH 7.0 (or as described), measuring absorbance at 420nm in a 1cm cuvette, and calculating absorbency index using pH-adjusted Brix (by refractometer).

Protein Assay Protein was determined by a modified Bradford Method. (Coomassie Plus-The Better Bradford™ Assay, Pierce Biotechnology, Rockford, IL). The Micro-Assay procedure (LDL=1mg/l) was used to for STJ samples.

Analyses performed at the Nalco Technical Center, Naperville, IL

- SEM/EDS
- Particle-size-distribution
- Solids Analysis
- GC/MS

SAMPLE COLLECTION

Samples of diffusion juice were collected before and after passage through the pilot unit. Determinations of lime demand were performed immediately after sample collection. Synthetic Thin Juice (STJ) was prepared from each of the paired samples of diffusion juice. Purity and Color of the STJ samples were determined. Splits were immediately frozen and then shipped to Nalco's Technical Center in Naperville, IL for further chemical analysis.[2]

RESULTS AND DISCUSSION

Carbon Dioxide Removal Removal of dissolved CO2 was quantified in terms of pH increase and titratable acidity. The results from this field trial clearly demonstrated that that the degasification of the diffusion juice prior to the prelimer decreased lime demand. Average

[2] **Analytical Characterization of Conditioned Diffusion Juices and Related Process Samples** McGinnis*, T. P., Water and Core Technologies Research, Nalco Company, 1601 W. Diehl Road, Naperville IL, 60563-1198. .

diffusion juice pH in treated samples was 0.2 ± 0.03 pH units higher than in untreated samples. The mean titration volume in treated samples was 13.2%± 2.1% less than in the respective untreated juice. This result was consistently demonstrated in subsequent field trials, including a second Northern Midwest factory

During the 2003/2004 campaign, field studies were conducted at Factory 2. Paired inlet and outlet samples were obtained from the two full-scale units. Samples were collected over a one-month period. Lime demand was measured in the diffusion juice. Average pH following conditioning was 0.19±0.02 pH units higher than the inlet diffusion juice. Mean titration volume of treated juice decreased 4.9%±1.0% all results were statistically significant when evaluated by t-Test.

Purity and Color The initial objective of this investigation was to demonstrate that lime demand could be decreased with the degasification equipment. To demonstrate that this equipment would not adversely affect the purification process, we wanted to ensure that treatment would not result in downstream problems and negatively impact sucrose yields. Synthetic thin juice (STJ) Purity and Color were monitored. No adverse effects were observed. In fact, the results at Factory 1 indicated that both juice purity and color improved increased across the system. In paired samples the average STJ color from treated diffusion juice decreased by 6.1% ± 3.6% compared to STJ prepared from untreated diffusion juice. The average purity of STJ from treated samples was observed increase by nearly 0.4% over that of the respective untreated samples. Statistically, a t-Test of the data showed that the change in mean STJ color was significant (N=18, P=0.001), as well as the difference in the average STJ purity (N=18, P=0.0002).

During the study at Factory 2, STJ was prepared immediately from each diffusion juice sample and purity and color were determined. Overall, STJ color showed a decrease similar to that observed at Factory 1 (average decrease = 6.4% ± 2.7%, N=63, P=0.00001). An overall difference in the average STJ purity was not statistically significant (Average difference <0.01%, N=63, P=0.091)

Effect of Temperature During this trial, the temperature of the diffusion juice being treated by degasification equipment ranged from 39°C to 70°C. There was no independent control of inlet juice temperature. Performance of the degasification system is likely to be affected by juice temperature. It follows that the temperature of the juice may also be important with respect to purity and color, so results were sorted by inlet temperature. Despite scatter in the data, both color and purity showed trends with increasing temperature. STJ color and STJ purity both improved when diffusion juice was treated at elevated temperatures. When the inlet temperature of the diffusion juice was at the lower end of the temperature range, the average decrease in STJ color was 4.9% ± 3.0% (N=43, P=0.0004). At higher temperatures, the STJ color had an average decrease of 9.6% ± 5.8% (N=20, P=0.0012).

Likewise, mean STJ purity showed a statistically significant increase when prepared from diffusion juice at higher inlet temperatures. In contrast, there was no apparent difference in the average STJ purities when inlet temperatures were low.

What is being removed? Nalco Research has determined that a number of volatile organic compounds (VOC) are present in beet diffusion juice.[2] Many of these volatile organics have been identified and have been described in sugar industry literature as participating in color-forming reactions. We are continuing to investigate the relationship between these volatiles and thin juice color and have attempted to quantify the amount of these volatiles removed during degasification. Based on the observed changes in synthetic thin juice color, we believe that sufficient quantities of VOC may be removed to impact downstream juice color. However, the differences in VOC that we determined to date, are not of sufficient mass to account for observed changes in apparent purity. Therefore, we have directed our attention to the question, "What else is being removed?"

Splits of synthetic juice samples from Factory 2 were analyzed at the Nalco Technical Center (NTC) by Gel Permeation Chromatography (GPC). Samples prepared from treated diffusion juice contained less polymeric material than in the respective untreated samples. Recognizing that the non-sugars present in diffusion juice include colloidal materials from the beet itself, we have begun to look for differences in high molecular weight contaminants. Residual proteins in the STJ samples were quantified and the results indicated that there was less protein in the STJ samples from treated diffusion juice. Decreases in protein were more apparent in the STJ from juices treated at higher temperatures, the trend being similar to that observed for color and purity. Quantification of other macromolecules is underway or being planned.

These results suggest that less high molecular weight contaminants may be carried over into thin juice. This would infer that juice conditioning might be impacting the ability to remove these contaminants during preliming and carbonation. When the conditioning units were ON, there were visual differences in the 1st Carbonation Underflow compared to when the units are OFF. At Factory 2, the underflow became a darker color when the units were in operation. The underflows also showed differences in particle-size-distribution. Scanning electron micrographs (SEM) of the underflow show some differences in the precipitate morphology. Gravimetric analysis of underflow samples showed equal levels of total solids, yet there was as much as 5% greater loss on ignition (i.e., loss at 925°C). This suggests that a greater quantity of organic non-sugars were bound to the $CaCO_3$ when the units were ON.

CONCLUSION

Initially, we set out to show that the degasification/conditioning system could decrease the amount of dissolved CO_2 in the diffusion juice and consequently decrease the factory's lime demand. This was consistently observed. However, the observed effects on process juice purity and color have become the focus of this research. Based on our field observations and related data, we believe that there are multiple mechanisms at work in this juice conditioning system. We have begun to consider a number of hypotheses to explain our observations. We are committed continuing our investigation into how this equipment could provide value to the beet sugar purification process.

REFERENCES

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