Experience in the Treatment of Thick Juice With Calcium Chloride in Operation at Missoula

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In a processing procedure patented by C. D. Barber and P. R. Barber of the Menominee Sugar Company (1)[2] and described in a published article by Don J. Barber (2), the addition of calcium chloride to thick juice is recommended for the control of sulfites in sugar and in molasses. Elimination of organic non-sugar matter from the juices by calcium chloride treatment is also claimed.

At the Missoula factory the control of sulfites in the sugar has been an operating problem difficult to cope with. To investigate the possibilities of calcium chloride for dealing with this situation a factory-scale test run was undertaken at Missoula during the 1945 campaign. The results from the test run were sufficiently encouraging so that at the Missoula factory this manner of treatment of the thick juice was continued throughout the balance of the campaign and has since been continued in the subsequent campaigns. The manner of applying the calcium chloride treatment and observation from the trial run in the 1945 campaign are described in the following.

Manner of Application

The addition of calcium chloride to the thick liquor was started on the trial run on the 38th day of campaign. By that time normal operations had been well established so that the effects of the calcium chloride treatment in processing operations could be observed and proper attention devoted to the test.

The test was begun with the low addition rate of 400 pounds per day, which at the prevailing slicing rate was equivalent to 0.25 pound per ton of beets. Inasmuch as but little was known from experience results, other than those at the Green Bay factory of the originators of this processing procedure, with regard to the proper amount to use for maximum economic benefits, it was purposely intended to start the test using a relatively small amount and then to build up the rate of addition gradually to what would appear to be the optimum. The increase in lime salts in the filtered standard liquor was taken as the basis for determining the optimum addition rate.

The high melter was selected as the place for the introduction of the calcium chloride. The high melt consists of high raw sugar melted in evaporator thick juice. A specially constructed reaction tank, provided with a

[1]General Chemist, American Crystal Sugar Company.
[2]The numbers in parentheses refer to literature cited.

rapid circulation agitator and a constant level overflow, is recommended by the sponsors of the processing method. It was found by experience at the Green Bay factory that unless good mixing and adequate retention for completion of the reaction in the agitator tank is provided, delayed precipitation may cause scaling in the pipe lines. Since the melter at Missoula is equipped with an efficient agitator and is of ample size it was felt that this would adequately serve for the mixing and reaction tank. No additional expense for mixing tank equipment was therefore necessary at Missoula.

The melter is also provided with steam coils and an automatic temperature controller on the steam valve by means of which the temperature of the juice in the tank was maintained at or very close to 97°. The high temperature was probably favorable for flocculation and granulation in the reaction of the calcium chloride with the non-sugars.

Flake calcium chloride, 70 to 72 percent $CaCl_2$, was dissolved in water in a small auxiliary solution tank. In later operations when the flake product was not obtainable fused calcium chloride, 77 to 80 percent $CaCl_2$, was substituted. The fused product was somewhat less convenient to handle than the flakes. It was dissolved by dropping the whole fused mass from a drum into a tank of shallow dimensions, adding water to partially immerse the mass and then agitating with direct steam injection. When the mass was dissolved the strong solution was flowed into the original solution tank provided and then diluted as desired.

A small centrifugal pump connected to the solution tank continuously circulated the calcium chloride solution by drawing it out at the bottom and returning it to the solution tank through an injector. Through a ¼-inch take off from the circulating line, with a valve set to deliver in a continuous stream a predetermined constant amount, the required solution for treatment was drawn off into the thick liquor in the melter.

The calcium chloride solution was made up of such variable concentration so that at the set flow rate through the charging valve the dosage could be adjusted for the slicing rate and the amount desired per ton of beets. This manner of preparing the solution in batches of constant volume, varying the strength to suit the requirements and leaving the valve set for a constant flow rate, was decided upon in preference to the method advocated by the sponsors of the treatment method of making up a solution of constant density and then adjusting the spigot accordingly to deliver the amount desired.

Filter aid slurry, prepared with filtered thick liquor in a separate filter aid mixing tank, was added at the melter in a continuous stream from the slurry circulating line. In the operations at the Green Bay factory, using the calcium chloride treatment of the juice and employing a procedure of pre-coating the press with filter aid, it was found practical to dispense with the subsequent continuous addition of filter aid (2). At Missoula, however, the filter operations indicated that the continuous addition of filter aid was desirable.

The flow of juice at Missoula is from the melter located in the sub-basement to surge tank located on the second floor and thence through the filter feed pump to the presses. The juice is double filtered. No difficulties from pipe scaling were encountered despite the relatively long flow line.

Filter Operation Effects

The immediate obvious effect on operations following on the start of the treatment of the thick juice with calcium chloride was a marked slowing up of flow through the filters and a very substantial reduction in the length of the press cycles. This condition was not entirely unexpected as some previous warning had been given that such might occur as a result of the mass precipitation of accumulated colloidal gummy materials circulating in process in the remelt stocks.

Under normal operating conditions prior to the test run with calcium chloride addition press cycles on the first liquor were about 40 to 48 hours, with three to four presses on the line. For the first few days after starting the calcium chloride addition the filter service of the presses dropped to cycles of less than 8 hours. A dense smeary type of precipitate was collected on the cloths rendering them almost completely impervious to further filtration after a few hours run. This sludge was indicative of the removal from the juice of a substantial amount of gum-like organic non-sugars still remaining despite the normal lime defecation and carbonation and the filtration of the thin juice.

To assist in the filtration under these difficult conditions first encountered a supplemental dosage of filter aid was added directly into the outlet well in the first liquor surge tank each time a clean press was put on the line. By forcing a rapid flow through wide open valve into the new press while pinching back at the same time on the old presses, a moderate pre-coat was formed. In later operations a separate pre-coat mixing tank was provided, which then afforded improved facilities for more efficient pre-coating of the filters. This moderate pre-coating effected by the first makeshift arrangement significantly improved the flow through of juice, but despite this aid the filter cycles were still discouragingly short.

With determined effort, in the face of the obvious filtration difficulties, to carry the test through at least one round of crystallizers, presses were cleaned as often as necessary, frequently two or three a shift. After about the fourth day of the test run gradual improvements in flow rates and press cycles were noted.

The rate of calcium chloride addition was then stepped up slightly for the next few days to a dosage of about 0.4 pound per ton of beets sliced. Since no relapsing detrimental effect with respect to filterability of the juice was noted due to the increased dosage of calcium chloride, but that rather filtration improvement was observed, the dosage was subsequently raised by increments until towards the latter part of campaign a dosage of slightly over 1 pound per ton of beets was being used.

After the first 2 weeks or so of struggling with filtration difficulties under the calcium chloride treatment the filter flow rates and the press cycles were restored again to satisfactory operating performance. Due to the greater amount of solids removed a regular schedule was set up to pull each press on a 24-hour cycle, staggering the start of the three presses on the line at 8-hour intervals.

For the Missoula conditions, judged on the basis of the increase in lime salts in the filtered standard liquor, 0.9 pound calcium chloride per ton of beets sliced was considered about optimum. In the later stages of the test period, when the calcium chloride addition was in excess of 1 pound per ton of beets, the lime salts, normally at a very low figure at that factory, had increased to nearly double of the 0.008 weekly average shown in the period just prior to the start of the treatment. Whether a moderate increase in lime salts resulting from the calcium chloride addition would necessarily be harmful in causing scaling is doubtful. As chlorides, the lime salts would not be scale forming. In appreciable excess the added lime salt might hinder boiling and retard crystallization.

Sulfite Reduction

As concerns the results achieved in the line of the primary purpose for which the calcium chloride treatment of the juice was advanced, that of lowering the sulfites in the first liquor for final products of sugar and molasses with lowered SO_2 content, it was apparent that the treatment had been effective. In the first liquor, with weekly averages of 800 to 900 parts per million SO_2 prior to starting treatment with calcium chloride the SO_2 content in the latter stages of the test run had dropped to about 400 parts per million.

Sulfites in the sugar produced at Missoula have generally been quite high. During the period of the test run a marked decrease in the sulfites in sugar produced was noted, with an average for a weekly period as low as 6 parts per million.

Likewise in the molasses a reduction in sulfites was obtained. The average of daily tests on molasses produced for the weekly period prior to starting with the calcium chloride treatment of the juice was in the range of 8900 parts per million SO_2. During the final weeks of the test run the sulfites in molasses were down to about 2400 parts per million.

Sugar ash followed somewhat the same pattern as the sulfites in the sugar during the test run. Starting with an ash of 0.016 and 0.013 percent, respectively, as the weekly averages on sugar produced in the 2 weeks before starting the treatment of first liquor with calcium chloride, the ash decreased to 0.010 in the latter weeks of the run. Comparative operating and test data are given in table 1.

Table I.—Comparative operating and test data relative to calcium chloride treatment of thick juice at Missoula.

Week ending 1945 campaign	Weekly averages: Beets sliced per day (tons)	Calcium chloride per ton beets (pounds)	Analysis: Sugar ash (percent)	SO_2 First liquor	PPM: Sugar	PPM: Molasses	Lime salts first liquor	Molasses percent on beets
Oct. 28	1429	None	.016	828	25.5	8900	.008	4.13
Nov. 4	1286	None	.013	888	14.5		.008	3.62
Nov. 11*	1510	.15	.012	966	16.1		.013	4.00
Nov. 18	1534	.51	.013	733	16.8		.010	4.21
Nov. 25	1512	.89	.010	453	9.4	3668	.012	3.72
Dec. 2	1502	.93	.010	340	6.3	2378	.014	4.20
Dec. 9	1521	1.05	.011	442	14.2	2429	.014	4.20

*Calcium chloride addition started November 8.

Effect of Raw Fillmass

Quite aside from the beneficial effects in reducing sulfites in the end products, a supplementary, but equally, and probably more important result achieved, considered from the operating standpoint, which was very obviously associated with the calcium chloride treatment of the first liquor, was the pronounced freeing up of the raw fillmass. The Missoula raw fillmasses were notably sticky and difficult to purge. The improvements in the pan boiling characteristics of the low purity green syrups and the spinning qualities of the crystallizer fillmass in the centrifugals following the use of calcium chloride in the first liquor were unmistakable. The usual former stickiness and stringy, gummy characteristics were largely eliminated. Color also had improved. The effect was so apparent and so favorable that the operators accustomed to the former sticky masses spoke of it as remarkable.

This feature in itself as concerns the improvement in the workability of the low grade products is considered now the more important advantage and has merited the continuance of the calcium chloride treatment of the first liquor at the Missoula factory.

Experience at East Grand Forks Factory

Without detracting in any way from the obvious merits of the calcium chloride treatment of juices observed in operations at Missoula, the citation of those experiences would not be complete without reference to the less favorable experience in attempts made at introducing the like processing treatment at the East Grand Forks factory. In several different starts made at that factory in successive campaigns, filtration difficulties encountered were such that it was practically impossible to keep up the juice flow through the filters and the trials had to be discontinued.

To insure most favorable conditions after the first unsuccessful start at using calcium chloride in the juice at East Grand Forks, the special mixing tank recommended was built and installed but this did not relieve the filtration obstacle. The smeary, gummy residues deposited in the filters

were evidence that the treatment with calcium chloride was coagulating organic non-sugar matter in the juice, the removal of which undoubtedly would have been beneficial. The practical operating difficulties, however, in those attempts at East Grand Forks, required the interruption of the treatment before the possible benefits to be derived at that factory in subsequent processing could be ascertained or realized in the manner as profitably experienced at the Missoula factory.

Summary

The treatment of first liquor with calcium chloride ahead of filtration at the Missoula factory proved effective in reducing sulfites in granulated sugar and in molasses. Coagulation of organic non-sugars of a gummy nature by the calcium chloride treatment initially caused filtration difficulties. The elimination of the organic non-sugar material improved the purging qualities of the raw fillmass. A dosage of about 0.9 pound of calcium chloride per ton of beets sliced was found to be optimum for Missoula conditions without unduly increasing lime salts in the filtered first liquor. Filtration difficulties in trial runs at East Grand Forks prevented the adoption of the process of calcium chloride addition in operations at that factory.