The Occurrence and Elimination of Saponin in Process Juice

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There are several processing means available to the beet sugar industry which affect the saponin content of refined sugar. They can be classified in two main groups: 1. Those which constitute major additions to, or departure from, the conventional process such as ion exchange or granular carbon; 2. Those which amount to minor changes in processing or which can be brought about through a change in process variables such as temperature, pH, flow, etc.

Ion exchange and granular carbon both can be very effective in the removal of saponin but represent a large capital investment.

The results presented in this paper point up the relative merits of a few of the less expensive possibilities the industry can use for the control of saponin. Until the use of ion exchange or other more positive methods of control are economically feasible the development of a cheap, reliable control method must be found. To accomplish this objective, three possibilities are shown, based upon the chemical and physical properties of saponin.

a. By taking advantage of the reduced solubility of saponin at low pH levels.

b. By formation of relatively insoluble calcium salts of saponin compounds.

c. The prevention of coagulation or crystallization of saponin by maintaining a high pH thus causing the saponin to pass through the process and be eliminated in the molasses.

The last method proved to be the most effective and is discussed in detail. The less effective methods are only briefly described in the following section.

Experimental

Full scale factory trials were carried out over periods of several days after the various means of saponin control exhibited some promise of success in laboratory experiments. The results of these tests are discussed as follow:

Trial No. 1 Reduction of Battery Supply pH

This trial consisted of reducing the pH of battery supply water to about 6.0 to 6.5 in an attempt to prevent or minimize extraction of saponin in the diffusion process. It was established in the laboratory that the extraction of saponin from beets increased when the battery supply water was in the alkaline range

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and especially when it contained ammonia. This treatment did not significantly reduce saponin in process liquors and in granulated sugar.

Trial No. 2 Reduction of Standard Liquor pH

The next trial consisted of reducing the pH of standard liquor to 5.0 to 5.5 pH with sulfur dioxide ahead of first filtration. The reduced pH caused some saponin to precipitate and be removed by filtration. The pH of the filtrate was next adjusted to 7.0 with NaOH, filtered and sent to the pan floor. This procedure also failed to reduce the saponin content of standard liquor sufficiently to prevent co-precipitation or crystallization of saponin in the white pans.

Trial No. 3 Effect of Additional Lime on the Solubility of Saponin

It is possible to form insoluble calcium salts of saponin compounds. This is strongly pointed out by the fact that over 97 percent of the saponin entering the system in diffusion juice is eliminated during carbonation. The addition of milk of lime up to approximately 0.25% on beets at second carbonation resulted in only a minor reduction of saponin in Thin Juice. Pilot plant experiments in pre-defecation have shown a 50 percent reduction of saponin in thin juice when compared with conventional carbonation. Further work along this line may prove to be rewarding.

Trial No. 4 Effect of High pH on Solubility of Saponin

R. S. Gaddie (1)[2] demonstrated the potential of high pH boiling as a means of saponin control in white sugar. Gaddie added caustic soda to the white pan in rather large amounts in an effort to maintain the pan pH at 8.5. It was believed that by careful control of carbonation and sulfitration that the pH of the white pan could be maintained at satisfactory levels without the addition of caustic soda.

A study of the saponin content of factory juices correlated with the white pan pH over an entire campaign pointed to the fact that when the saponin content of standard liquor was less than 60 ppm on dry substance that the resulting sugar met the specifications of the soft drink industry providing the white pan pH was 8.0 or over.

Table 1 illustrates the ranges of saponin in factory liquors. Factories 3 and 4 produced low "floc" or low saponin sugar almost at will by maintaining the white pan pH at 8.0 or over. Factories 1 and 2 experienced difficulty in being able to control the saponin in sugar except during the periods when the saponin in standard liquor was less than 60 ppm.

[2] Numbers in parentheses refer to literature cited

Table 1.—Distribution of Saponin in Liquors (1958-59 Campaign).

Factory	Saponin ppm on D.S.			
	1	2	3	4
Diffusion Juice				
High	9,193	5,926	4,756	3,860
Low	4,208	3,117	2,620	2,599
Average	6,429	4,709	3,516	3,282
Thick Juice				
High	173	206	159	191
Low	60	61	65	42
Average	103	110	105	83
Standard Liquor				
High	104	101	78	80
Low	51	48	44	26
Average	74	67	55	40
Number of Samples	90	95	150	150

It has been possible to maintain white pan pH over 8.0 by careful attention to carbonation and sulfitation. First carbonation alkalinity must be held as high as possible. With beets processed by The Amalgamated Sugar Company the optimum alkalinity is around 0.10. It is necessary to carry second carbonation alkalinity at .025 to .028 in order to use sufficient sulfur to stabilize the thin juice and to prevent a drastic pH drop through the evaporators. This has been accomplished by sulfuring to a thin juice pH of 9.0 to 9.2. Thick juice pH will then range from 8.0 to 8.4.

Table 2.—Effect of White Pan pH on Saponin in Sugar (Twin Falls Factory 1959-60 Campaign).

Date	pH White Fillmass	Saponin in Sugar ppm
12-26-59	8.5	1.3
12-27-59	8.3	1.7
12-28-59	8.5	2.0
12-29-59	8.6	1.7
12-30-59	8.4	1.3
12-31-59	8.7	1.3
1-1-60	7.1	7.9
1-2-60	6.8	4.9
1-3-60	7.1	7.0
1-4-60	6.6	6.0
1-5-60	7.2	7.0
1-6-60	7.1	7.9

Table 2 demonstrates the effect of white pan pH on the saponin content of white sugar. From December 26 to 31 all sugar produced met bottlers' standards. In the following period, the pan pH was deliberately reduced.

It is imperative that when a control such as this is in force certain conditions must be satisfied. Carbonation and sulfitation levels must be steady and uniform. Uneven control at this point can and will cause wide fluctuations in the thick juice pH. If the pH of thick juice drops below 8.0 for only a short time it is possible that the resulting sugar will be too high in saponin content for bottling purposes even though the pan pH indicates a satisfactory value. The failure of an entire day's sugar production to meet specifications has been traced to faulty operation for periods as short as 30 minutes at the carbonation of sulfitation stations. Continuous records of thick juice pH are more apt to pinpoint faulty operations than hourly laboratory data in a process such as this.

It is also necessary to establish upper limits for the concentration of saponin in sugar based on a quantitative analytical method and on a visual estimation. Assuming that most of the bottlers employ the Spreckels visual estimation method using acidified sugar solutions, a 24-hour development period and a strong light beam as their standard, the following study was made.

Four hundred and eighteen daily production sugar samples were analyzed for saponin by the visual Spreckels method and by a reliable quantitative method. The samples were selected in a random manner attempting to cover a wide range of saponin content in order to establish a reliable upper limit.

Table 3.—Frequency Distribution of Saponin in Sugar to Establish Limits of Acceptance.

Intervals Saponin ppm Quantitative	Spreckels Test Value (Visual)			
	Less than 1	1	Over 2	Total
0 - 2.1	157	6	1	164
2.2 - 3.2	111	22	5	138
3.3 - 5.9	18	41	22	81
Over 5.9	0	2	33	35
Total	286	71	61	418

If upper range is set at 2.1 ppm odds are 19 to 1 against putting sub-standard sugar on market.

At 2.6 ppm odds are 10 to 1 against putting sub-standard sugar on the market.

Table 3 is the frequency distribution of this study. A Spreckels test value of less than one showing only a slight haze at the most after 24 hours was chosen as the basis for acceptable bottlers sugar.

By setting the quantitative upper limit for acceptable sugar at 2.1 ppm the risk of placing a sub-standard sugar on the market is about 5 percent. By raising the limit to 2.6 ppm the risk increases to about 10 percent. However, if the sugar at 2.6 ppm is examined by the Spreckels test after 48 hours, a pinpoint development of floc will show up about 75 percent of the time.

At the start of the 1959 campaign the sugar rating standards were revised to include a penalty factor on sugar produced in certain factories if the saponin content exceeded 2.1 ppm.

The results have been gratifying in that at three factories, the to date average saponin content in sugar produced is 3.1, 2.3 and 2.4 parts per million. The percent acceptable bottlers sugar for the same factories is 57, 66 and 56% respectively for this year against an over-all average of about 38 percent a year ago and 15 to 20 percent in previous years.

The method of controlling saponin in sugar by high pH boiling is not without some inconsistancies. For example, one factory has been able to maintain satisfactory low levels of saponin in standard liquor which enables them to boil sugar at 7.5 pH. Opinions vary as to whether or not extraction of sugar is influenced adversely by high pan pH. At times, when the level of saponin soars to over 175 ppm in thick juice due to poor carbonation, the saponin level in sugar goes up to 5.0 ppm or more even when boiled from 8.5 pH liquors.

By using the techniques described in this paper it has been possible to supply bulk and liquid sugar outlets with an adequate amount of low floc sugar to allow multiple end-use of the product during peak seasons for bottling and canning deliveries.

Literature Cited

(1) Gaddie, R. S. and West, R. R. 1958. Relationship between white pan pH and saponin content of sugar. Jour. Am. Soc. Sugar Beet Tech. X (2) : 171-176.