Studies on the Control of Saponin Concentration in Refined Beet Sugar

ROBERT S. GADDIE[1]

All commercial granulated sugars contain traces of acid-insoluble substances which precipitate in acidified aqueous solution and which, at sufficient concentration, show up as a white flocculent precipitate. The floc present in beet sugar is composed of a mixture of saponin and its derivatives, of fats, and of various other adsorbed minerals and colloids. (1)[2] Because these substances gradually precipitate in certain carbonated beverages and acidified pharmaceutical syrups, it is necessary that sugar be produced as free as possible from these floc-formers.

Certain information of a very general nature on the concentration of floc in the granulated sugars produced by the seven factories of the Utah-Idaho Sugar Company was obtained during the 1952 and 1953 campaigns. The general nature of the information was due in large part to the extreme qualitative value of the then known tests. In 1953, Walker and Owens, working on this problem, found a fairly large percentage of the acid-insoluble constituents in beet sugar to be saponin and Dr. Walker devised a method of analysis for this component. Even though the method had not been published and had, in fact, not been tested thoroughly, Walker very kindly made available all the information he had on the problem. With our adaptation of this test it was determined that a measure of saponin concentration in a sugar gave an excellent measure of that sugar's tendency to produce floc in susceptible beverages. The maximum safe concentration of four p.p.m. for sugar going into these susceptible beverages (2) was further determined. With these facts established, it was decided to make exhaustive tests on sugar from all seven of the Utah-Idaho factories during the 1954 campaign and attempt, within the practical limits of full scale operation and on present knowledge, to control the saponin concentration in the final sugar.

The number of analyses which could be made was limited by the two spectrophotometers available. However, a minimum of three samples, representing three to six strikes of sugar from each factory each day, was analyzed. A study of these analytical results showed that each factory's sugar production fell into one, and only one, of three classifications. Typical examples of these three are shown in Table 1.

Table 1.—Saponin Concentration in Sugar Samples from Three Different Factories.

Factory	No. of Samples	Saponin Concentration ppm		
		Maximum	Minimum	Average
A	232	24	6	14
B	298	3	0	1
C	631	17	1	6

[1] General Chemist, Utah-Idaho Sugar Co., Salt Lake City, Utah.
[2] Numbers in parentheses refer to literature cited.

The results gave quantitative confirmation to deductions arrived at from the general information obtained the two previous years.

a. Sugar from some factories always contains relatively high concentrations of saponin, (A in Table 1).

b. Sugar from some factories always contains very low concentrations of saponin, (B in Table 1).

c. Saponin concentration in sugar from some factories varies within wide limits (C in Table 1).

Because the saponins are only one of many non-sucrose constituents in purified beet juice and because very efficient sugar-end operation nearly always results in improved sugar quality, it is logical to suppose that saponin concentration in granulated sugar would be lowered by high pan purities, precision sugar boiling technique to avoid crystal agglomerates and as near perfect separation of mother liquor from crystals as possible. On this supposition it was decided to try this procedure at Factory C (Table 1). Standard liquor purities were held up to near 93.0, high raw sugar purity was maintained at 98.5 to 99.0 and sufficient wash water was applied at the centrifugals to bring the color index of the sugar to the lowest practical value. Typical results are shown in Table 2. Colors are given in terms of reference basis units (3).

Table 2.—Saponin Concentration in Selected Sugar Samples from Factory C.

Sample No.	White Pan Purity	Color Index	Saponin p.p.m.
1	92.5	34	12
2	92.8	29	8
3	92.1	34	11
4	92.6	31	10
5	92.9	27	6
6	91.9	22	3
7	93.1	17	3
8	93.1	13	1
9	92.5	17	1
10	92.2	19	2
11	93.2	19	1

Variations in pan purity within the limits at which operations were conducted did not have any measurable effect on floc, but floc concentration at this factory was found to be almost directly proportional to color index. However, in order to obtain a sugar with a color index below 22, it was sometimes necessary to wash with as much as 26 quarts of water per machine. This would fill up the white side in about 24 hours and so would have to be discontinued temporarily. Throughout most of the campaign, however, it was possible to obtain sugar containing less than four p.p.m. saponin by washing to such a color index.

As soon as results from the work at factory C were available, identical conditions were established at factory A where saponin concentration had been running consistantly high. All operating conditions were directed toward the production of an extremely even, relatively coarse grain in the

vacuum pans, and as near perfect washing in the white centrifugals as could be obtained. In spite of these efforts, results obtained at Factory C could not be duplicated in Factory A, even for short periods. There seemed to be no correlation whatever between saponin concentration and any other physical or chemical characteristic of the sugar. This is illustrated in Table 3.

Table 3.—Saponin Concentration in Selected Sugar Samples from Factory A.

Sample No.	White Pan Purity	Percent Ash	Color Index	Saponin p.p.m.
1	93.1	.0050	17	12
2	93.8	.0054	20	8
3	93.6	.0058	23	13
4	93.4	.0038	24	15
5	93.3	.0040	35	8
6	93.5	.0044	35	10
7	93.5	.0046	36	6

Periodic samples of sugar were made into a massecuite, purged, and washed in the laboratory centrifugal with what would be equivalent in factory practice to 40 quarts of hot distilled water per machine without effecting a decrease of more than 1 or 2 p.p.m. of saponin.

Since the saponins are highly surface-active, adsorbing agents such as activated carbon should remove at least a portion of those left in the purified juice (4). Therefore, the addition of activated carbon (Darco S51) to the standard liquor was started. The first 48 hours, .3 pounds per ton of beets was added. For the next 48 hours this was increased to .5 pounds and 48 hours later to .6 pounds per ton of beets. At this point, the standard liquor filters had about reached the limit of their capacity and higher carbon additions were not practical. During these periods, sugar boiling was carefully watched and 25 quarts of centrifugal wash water per machine were used. No decrease in average saponin concentration in the sugar was effected by any of these carbon additions. Adsorbing agents, such as activated carbon, have selective adsorption properties which do not confine themselves to the saponins. Because of this, Sabine predicted, and our work has shown, that in many cases the quantity of carbon which would probably be required would raise production costs higher than could be justified.

Saponins all form emulsions with oil (6). In testing the solubility of the sugar beet saponins in the various common defoamers used in sugar manufacturing, McGinnis (7), found that they were least soluble in Balab. Therefore, for a period of 24 hours, all foam oils (except Balab) were taken out of the process and Balab used as sparingly as possible. Saponin concentration in the sugar produced during this period ranged from 6 to 13 p.p.m.

Triterpenes form insoluble compounds with heavy metal ions (4). This explains the high (about 95%) elimination from diffusion juice, as the calcium complex, when lime is added at the carbonators. Hoping to take further advantage of this property, two things were tried. First, milk of lime equivalent to about .15% CaO on beets was added to the filtered 1st carbonator juice going to 2nd carbonation. This was continued for 3 days

and when no decrease in floc was obtained the second experiment was started. This consisted of adding fresh milk of lime equivalent to approximately .07% CaO on beets to the standard liquor at the high melter. This was then sulfured to pH 7-8 to 8.0 using liquid SO_2 and filtered on the standard liquor presses using .6 pounds filteraid per ton of beets. After a 72 hour run in which no significant decrease was effected in the average saponin concentration in the sugar, this procedure was also discontinued.

It is highly probable that methods of processing influence, to some extent, the concentration of saponins in the final sugar. Eis, et al (4) have shown, that even at the same factory, the concentration of triterpenes in diffusion juice is widely variable. Although 95-97% is eliminated in carbonation, in cases of high triterpene incidence, the removal is still inadequate.

References

(1) WALKER, HOWARD G. JR., and OWENS, HARRY S. 1953. Beet sugars; acid insoluble constituents in selected samples. Agr. and Food Chem. 1:450.

(2) WEST, ROBERT R. and GADDIE, ROBERT S. 1956. Determination of saponin in granulated sugar: method of H. G. Walker adapted to routine control. Journ. Amer. Soc. Sugar Beet Tech. 9:146-152.

(3) Bottler's Sugar Survey (1952)

(4) EIS, F. G., CLARK, L. W., MCGINNIS, R. A., and ALSTON, P. W. 1952. Floc in carbonated beverages. Ind. & Eng. Ch. 44:2844.

(5) SABINE, F. M. 1952. The floc problem in beet sugar. Proc. Amer. Soc. Sugar Beet Tech. pp. 760-763.

(6) MCILROY, R. J. 1951. The plant glycosides. Edward Arnold & Company, London. p. 64.

(7) MCGINNIS, R. A. Private communication.