

# The "Floc" Problem in Beet Sugar

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In view of the fact that Ed Maudru, general chemist for Holly Sugar Corporation, and I have been assigned as moderators for the floc problem in white sugar, I believe it would be well to trace briefly the history of this new subject in our operations before we begin a general discussion on this subject. Since Mr. Maudru, as general chemist, would be primarily concerned with the chemical phases of this problem, and I with operating problems, I will to the extent possible limit my remarks in connection with this subject to the operating phases.

## History

During the course of the past several years several complaints with respect to the use of beet sugar have been brought to our attention by the Seven-Up Company of St. Louis, Missouri, through Dr. B. C. Cole, who is in charge of the product department. This department is actually the quality control laboratory for the bottlers who hold franchises to put up this product. Periodic samples are furnished this laboratory where they are tested for taste, uniformity and quality. In making their tests they have at times found off-quality in their product, which was ascribed to the use of beet sugar. The following factors they claim have given the Seven-Up Company trouble with beet sugar were described as:

1. "Delayed action" floc.
2. "Fertilizer" odor.
3. "Metallic" taste.
4. Foam at the filters and in the syrup.

These descriptions are based on the physical tests of odor and flavor made by personnel trained and qualified for this type of work. The "delayed action floc" occurs most consistently in Seven-Up through the use of beet sugar at the pH of the formula used, and from their standpoint is the most important objection to the use of beet sugar.

Since the so-called floc test is the one under discussion my remarks will be limited to this subject.

In 1950 this was called to our attention before we had any knowledge of their problem and Dr. G. A. Vacha and I visited the St. Louis office of the Seven-Up Company. Efforts in the factory laboratory to reproduce this "floc" with citric acid and sugar solutions were not too successful. In fact, little "floc" formation resulted and yeast and mold growth occurred before a "floc." Apparently the Western Beet Sugar Producers' Committee through R. S. Gaddie furnished the various companies with the so-called "Coca-Cola Floc Test" which is used by that company. The test will be described later merely as a matter of information.

In July, 1951, Dr. Cole contacted representatives of the sugar corn- regarding a new "floc" test which they developed and which requires less than 24 hours for a "floc" to form, although the 24-hour period is recommended for evaluating purposes. The test briefly is to heat a 10 percent sugar solution at 100° C.-115° C. for one hour after having been adjusted to an approximate pH of 2.70 with hydrochloric acid. Tests on heating at the boiling temperature for one hour apparently work satisfactorily, but

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not much experience has been had with tests at lower temperatures than 100° C, which normally requires an autoclave. The details of this test were tabulated and Mr. Gaddie of Utah-Idaho Sugar Company was appointed to work out the specifications for the racks (2 1/2 inch diameter holes) and the globes to be used for such tests *in order* that these tests would be made on a uniform basis.

The other "Roc" tests which were previously used are:

**Coca-Cola Floc Test:** A 50-55.0 BX sugar solution is adjusted to 1.50 pH to 1.60 pH with phosphoric acid and allowed to stand for 10 days to check "floc" development. The sample is one gallon and placed in a one-gallon jug for the 10-day period. The jugs should be sterile.

**Holly Sugar Corporation:** 40 gms. sugar, 30 ml. H<sub>2</sub>O, 1/2 gm. citric acid allowed to stand for five days (no heating). Containers should be sterile.

**Great Western (Also used by Utah-Idaho):** 10 gms. sugar, 25 ml. H<sub>2</sub>O, 2 1/2 gms. citric acid/100 ml. using 1 ml. acid solution for the test and heating to 80° C.-90° C. for one hour. Remove and take reading after one hour.

**Seven-Up Test:**

*Beet Sugar Floc Test*

(On One Gallon Basis and One Quart Basis)

1. Use 400 grams sugar (approx. 10 percent solution) to one gallon. Use 100 grams sugar for 1,000 cc. or one quart. (1,000 ml. Florence flask).

2. Add distilled water to volume (neck of bottle or flask).

3. Add 1/2 cc. concentrated HCL (pH approximately 2.70) to one gallon solution. Add 4 cc. N HCL for 1,000 cc. or one quart.

4

4. Heat to 100° C. for one hour.

5. Observe after 24 hours over light beam.

6. Report results as below:

- 0 — No definite general floc.
- + — Barely visible, very fine, general floc.
- ++ — Very definite but fine pin-point type floc.
- + + + — Larger (1/64 inch) but pin-point (not loose) floc.
- + + + + — Large, loose snowflake-type floc with clearing of solution.

Notes: Insufficient experience has been had with respect to these tradings as compared to floc formation in bottled Seven-Up. Did not anticipate difficulty with "0" or "+." "++" and over doubtful for Seven-Up.

This test apparently exaggerates the trace compound, or trace compounds which "floc" out under the conditions of the test. However, it is an impurity which definitely influences the use of our sugar for specific purposes since cane sugar while containing such tract impurities does not form a floc under these conditions. The Seven-Up Company is next to Coca-Cola in national distribution, and a large number of plants are located in areas where the bottlers are receptive to beet sugar and want to use it. When trouble occurs, however, with a "floc" formation in their product they receive complaints, and since the product quality is carefully checked by the St. Louis laboratory, they recommend switching to cane sugar, which they claim clears up this trouble. The Seven-Up Company through Dr. Cole, however, has been cooperative in trying to work out these problems with the industry.

The companies working on this problem have the following observations to make from their experience:

1. The floc forms in all sugar end products starting with thick juice.

2. High pan purities result in low floc formation, on the basis of tests on all pans at Carlton during the 1950 campaign.

3. The floc tendency increases as deteriorated storage beets are worked.

4. High carbon addition 0.5 pound to 1.5 pound per ton of beets apparently reduces or eliminates the floc as determined by the Coca-Cola floc test. There is not enough experience with the Seven-Up test to note the carbon influence, but apparently the carbon effect is not so noticeable.

In studying this problem from an operating standpoint, numerous floc tests were made prior to and during the 1951 campaign period, and to our surprise and satisfaction very few if any of the samples tested exhibited any tendency to form a floc based on the tests used. Except in infrequent cases, the floc tests did not indicate more than a "+." While many of the floc tests made on the 1950 production did indicate at some plants a condition of higher floc properties in the sugar, there was a possible explanation that any low amounts found in 1951 were due to the higher quality of beets processed. In southern California the purity of the beets averaged approximately 0.5 percent higher than in 1950. At Clarksburg, California, the purities were 1.2 percent higher, and for the 1951 campaign as a whole, the average purity of the beets sliced was .61 percent higher than in 1950. In the Red River Valley the average purity was slightly over 2.5 percent higher than in 1950.

If the substance or substances producing floc in beet sugar depended on the concentration in the beet, then there is some logical explanation for the lower floc encountered in the 1951 production. Some preliminary work indicated that there was a possibility of absorbing this floc-producing substance by treating solutions of granulated sugar with de-colorizing carbon or compounds such as Adsorbol or a mixture of the two. The cost of such treatments on granulated sugar after it is produced is prohibitive and if sufficient quantities of materials outlined were applied on a factory scale any amounts which would probably be required would raise the production costs higher than justified for the known potential market for Hoc-free sugar. If used in the bottler's plant there would be the absorption of desirable flavor characteristics.

There is a serious question in our minds also that such treatment would be entirely effective on sugar end liquors, since, for example, there are non-sugars for which these materials would have selective absorption properties which would not confine themselves to whatever substances causes the floc. A number of years ago J. C. Keane, while working with the United States Department of Agriculture, did some work which indicated that at least certain mineral constituents were crystallized out at the end of the boiling operations or that their solubility was such at that concentration that they adhered to or were absorbed on the outer surfaces of the sugar crystal and that increased washing resulted in a lower ash content of sugar produced.

During our 1951 operations we, therefore, concentrated as much as possible on good centrifugal work and, while all of you are familiar with

centrifugal operations, experience has indicated that in plant supervision the following points should be checked into carefully and followed up by frequent and careful supervision:

1. All centrifugals should be checked to be certain that mechanical operation is good and that the machines operate at full speed.
2. Individual machine cycles should be carefully adjusted for the grain size of the sugar boiled.
3. In prewashing the sugar, the water should go on as soon as possible before the machine gets up speed and the second washing should also go on at the proper time and preferably before all the syrup leaves the sugar wall.
4. Wash water temperature and pressure should be carefully regulated in order to provide sufficient water for proper washing. The quantity of such water delivered per machine also should be carefully checked.
5. Nozzle adjustments should be carefully checked and maintained in order that uniform washing is always obtained.

In view of the fact that in 1951 most of the sugar end products, including intermediate sugar, exhibited floc-forming tendencies when subjected to the so-called Seven-Up test, the indications were that with the substances being present in intermediate products prior to standard granulated it might still be found in the final sugar if centrifugal operations on the white, intermediate and low raw sides were not carried on as efficiently as possible. There still remains the possibility, however, that the concentration of floc-forming substance in the sugar end products in 1951, due to the higher purity of the beets, was lower than would normally crystallize or become insoluble at the density of pans boiled.

In any event, the result of 1951 operations, in general terms from our company's experience, has been that the floc problem has not existed to the extent that it did in 1950. As previously stated, there is evidence from past experience that good centrifugal operations and washing operations always result in a better final product and, while there is a limit to the quantity of wash water which can be used in centrifugal operations, it is felt that proper attention to such operating details under all conditions will result in improvement in sugar quality, which we are all striving for, and should in turn result in a sugar relatively "floc" free.

In view of the fact that the bottling industry is the second largest industrial user of sugar, we should do everything possible, within reasonable economic limits, to furnish a sugar which will not result in complaints from the trade, especially having in mind that when complaints with respect to the "floc" problem result it is claimed to have been eliminated by using high quality cane sugar. This does not necessarily mean, however, that all cane sugars are free from this "floc" problem.

In view of the fact that a discussion of this problem has not given everyone an opportunity to observe the tests such as we had the opportunity to do in St. Louis, we have prepared floc tests representing the "+," "++" and "+ + ++" floc test in sugar which were furnished by the Seven-Up people in St. Louis, and which were kindly prepared by Mr. Gaddie of the Utah-Idaho Sugar Company. He has also furnished us with the facilities for viewing the occurrence of this material in the sugars furnished.