

## Relationship Between White Pan pH and Saponin Content of the Sugar

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In previous work on saponin content of refined sugar and its relation to floc production in beverages, (1)<sup>2</sup>, (2), it was found that sugar containing less than 2 p.p.m. saponin would very likely not produce floc in susceptible carbonated beverages. It was also determined that at some factories all the sugar produced contained less than 2 p.p.m. saponin. At others all the production analyzed 6 p.p.m. or more of saponin. At still other factories the saponin content in the granulated sugar varied rather widely.

While attempting to correlate saponin concentrations in various factory syrups with concentrations observed in granulated sugar, the campaign composite white massecuites from the seven operating factories were autoclaved to effect solution of the grain, diluted to 66.5 Brix and stored in the refrigerator. The next day it was noted that a scum had formed on the solution from Factory B (high saponin sugar producer). This scum was found to be 65 percent saponin. This massecuite composite had an unusually low pH value of 6.5, which led to the hypothesis that perhaps the soluble saponin originally present in the standard liquor had been precipitated by pH drop in the white pan. As the first step in checking this hypothesis, saponin was determined on all seven white massecuites without previous filtration. As may be noted in Table 1, the range was from 52 to 107 p.p.m. on solids with a pH range from 6.5 to 8.8. There was no correlation whatever between saponin content of these campaign composite

Table 1.—pH and Saponin Content of Unfiltered White Massecuite Composites.

Factory	Massecuite pH	Saponin p.p.m. on D.S.
R	8.5	52
S	7.7	70
C	8.0	71
B	6.5	75
U	7.2	79
T	8.5	83
A	8.8	107

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<sup>2</sup> Numbers in parentheses refer to literature cited.

white massecuites and the saponin content of the corresponding sugar. As a matter of fact, the 107 p.p.m. massecuite was from Factory A, a very low saponin sugar producer.

If the foregoing hypothesis is correct, the saponin concentration in the filtrate of filtered low pH massecuite should be much lower than on the same massecuite unfiltered. This was exactly the case as shown in Table 2.

Table 2.—pH and Saponin Content of Unfiltered and Filtered Massecuite Composites.

Factory	Massecuite	pH	Saponin p.p.m. on D.S.	
			Unfiltered	Filtered
A	White	8.8	107	101
B	White	6.5	75	10
A	Low Raw	9.1	560	455
B	Low Raw	6.0	470	75

Note that in high pH massecuites, approximately the same concentration of saponin was obtained from both filtered and unfiltered solutions. On the other hand, about 85 percent of the saponin contained in low pH massecuite was retained on the filter.

Aliquots of the low pH massecuite from Factory B were treated with NaOH solution to raise the pH to 7.6 and 8.3 respectively. The treated aliquots were stirred for 10 minutes, filtered and saponin determined on the filtrate. Similarly, portions of the high pH massecuite from Factory A were brought to pH 6.6 and 5.9 with HCl, filtered, and the filtrate examined for saponin. Results are shown in Table 3. Progressive increase in pH puts progressively more saponin in solution. A progressive decrease in pH progressively precipitates the saponin in the massecuite.

Table 3.—pH and Saponin on Massecuites Treated With NaOH and HCl.

Factory	pH	Saponin in Filtrate p.p.m. on D.S.
B	6.5	10
	7.6	21
	8.3	33
A	8.8	107
	7.2	94
	5.9	73

It had been demonstrated many times (2), both in the factory on the white centrifugals and in the laboratory on the laboratory centrifugal that no amount of wash water would effect a decrease of more than 1 or 2 p.p.m. saponin in the sugar. It had also been established that recrystallizing a refined granulated sugar of high saponin content by re-boiling in the laboratory vacuum pan did not significantly lower the saponin content of the resultant sugar. However, at the time this work was done, no adjustment in pH was made on the syrup feeding the vacuum pan. To determine the effect of higher pH during recrystallizing, the following experiment was set up. Refined granulated sugar containing 16.2 p.p.m. saponin was dissolved in distilled water to a 65 Brix syrup. A pan was boiled without any pH adjustment. A second pan was boiled from an aliquot of the syrup adjusted to pH 7.9 with sodium hydroxide and a third from an aliquot of the syrup adjusted to pH 9.0. Each pan was drained, boiled on an intermittent feed to a normal grain size, Brixed to about 92.0, spun in the laboratory centrifugal, and washed with boiling distilled water in an amount equivalent to 20 quarts per factory sized machine. The resultant sugar was then analyzed for saponin. Results are shown in Table 4.

Table 4.—Saponin on Sugar Re-boiled at Various pHs.

Pan No.	pH	Saponin p.p.m.
1	7.1	14
2	7.9	5
3	9.0	2

As a result of these laboratory findings a careful study was made of the pH history throughout the process of each of the seven factories and some rather significant facts were noted.

1. Factories (A) having sugar production consistently low in saponin were those in which the pH remained constant at a value of not less than pH 8.5 from thin juice to molasses, or were factories in which there was a rise in pH through the process from thin juice to molasses, provided the pH of the white pan was not below 8.5.

2. Factories (B) producing sugar consistently high in saponin were those which had a drop in pH from thin juice to molasses resulting in a white pan pH below 8.5; usually considerably below.

3. Factories (C) in which saponin in the sugar was variable were those in which the pH carried through the process at a constant figure but this pH was lower than 8.5.

Typical examples of each of these are shown in Table 5.

Table 5.—Average pH Values on Process Juices at Three Factories.

Station	pH		
	Factory A	Factory B	Factory C
Second Carbonation	9.0	9.2	9.5
Thin Juice	8.2	8.6	8.1
Thick Juice	8.8	7.4	8.0
Standard Liquor	8.7	7.4	8.0
White Pan	8.8	7.0	8.0
High Raw Pan	9.0	6.6	7.8
Low Raw Pan	9.1	6.4	7.8

The next step obviously was to apply this information to full scale production. In the case of type "C" factories, this turned out to be relatively easy. The pH of thin juice was maintained high enough so that the white pan was boiled at pH 8.6 or higher. To do this and still get some sulfur dioxide in the juice it was necessary to raise second carbonation pH considerably higher than had been carried in the past, higher in some cases than the optimal second carbonation alkalinity. Table 6 shows typical pH values under this system.

Table 6.—Typical pH Values Operating in the Higher pH Range.

Station	pH
Second Carb.	9.7
Thin Juice	9.1
Thick Juice	8.8
Standard Liquor	8.7
White Pan	8.7
High Raw Pan	8.6
Low Raw Pan	8.6

Operating in this manner resulted in practically one-hundred percent of the sugar analyzing less than 2 p.p.m. saponin.

The problem of producing low saponin sugar at Factory B was not so simple. Here the juice contains a high concentration of nitrogen compounds. These continuously break down through

the five evaporators and through all three pans with the evolution of ammonia liberating the corresponding organic acids into the juice, resulting in a progressive drop in pH through the evaporators and through the sugar end. No combination of lime addition nor of alkalinities in first and second carbonation was found to have much effect on this decomposition. Even with second carbonation pH raised to 9.8 and thin juice to 9.4 the white massecuite was still no higher than pH 8.0 and saponin concentration in the sugar was as high as 10 p.p.m.

The only way to get the white pan pH up to the required 8.6 was to add caustic soda. It was added into the charge line of the pan as the 25 percent solution. Sufficient was added with the graining charge to bring it to pH 8.8 to 9.0. To maintain this pH it was necessary to add caustic at intervals until the pan was ready for Brixing. A pH meter was set up on the pan floor and the massecuite checked about every 15 minutes. This was necessary because if the pH at any time during the boiling cycle dropped more than about 0.3 pH the sugar would be above 2 p.p.m. saponin. Conversely, if the pH became too high (9.4) considerable color developed. A typical boiling cycle is shown in Table 7. By using this procedure it was possible to consistently produce sugar in the range 0.5 to 1.5 p.p.m. saponin.

Table 7.—Typical Boiling Cycle on White Pan With Caustic Soda Addition.

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Graining charge 700 cu. ft. pH 8.1
41.2 lbs. caustic added—pH 9.0
At 800 ft., pH 8.7—15.5 lbs. caustic added, pH 9.0
1000 ft., pH 8.7—10.3 lbs. caustic added, pH 8.9
1200 ft., pH 8.7—10.3 lbs. caustic added, pH 8.8
1400 ft., pH 8.7—10.3 lbs. caustic added, pH 8.8
1500 ft., pH 8.6—10.3 lbs. caustic added, pH 8.8
1600 ft., pH 8.6—10.3 lbs. caustic added, pH 8.9
Total caustic added 108.2 pounds
1650 cu. ft. massecuite pH 8.8

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It is obvious that this is not an ideal solution to the problem of floc-producing substances in refined beet sugar. The technique is difficult to control, is expensive and was used only for the sugar to be allocated to the beverage industry. Even with good control there is a continued color build-up in the syrups and purities tend to get somewhat out of hand. For this reason the system is operated intermittently, allowing time for the sugar end to clear out between cycles. However, in spite of the mani-

fest disadvantages of high operating pH values and caustic addition to white pans where necessary, such technique has solved a very perplexing problem for us. Certainly there must be some better method of eliminating the few parts per million of these offending non-sugars. Further developments by the industry's technologists are awaited with interest.

### Summary

Evidence is presented that the concentration of saponin in refined granulated beet sugar may be influenced by pH conditions in the white pan during the boiling cycle. Methods of controlling this pH under two sets of conditions are presented.

### Literature Cited

- (1) WEST, ROBERT R. and GADDIE, ROBERT S. Determination of saponin in granulated sugar. method of H. G. Walker adapted to routine control. Jour. Am. Soc. Sugar Beet Tech. IX (2):146-152.
  - (2) GADDIE, ROBERT S. Studies on the control of saponin concentration in refined beet sugar. Jour. Am. Soc. Sugar Beet Tech. IX (2):189-192.
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