

Salt Elimination During Diffusion of Sugar Beets

A. E. GOODBAN AND J. B. STARK¹

Received for publication June 25, 1962

For the production of white sugar from sugar beets, sugar must be separated from other soluble impurities in the factory juices. The ease with which this can be done, the ultimate yield of sugar, and the cost of production are largely determined by the amount and character of these impurities. Most of the non-sugars come from the beets, but some are introduced with the water used for diffusion. All of the soluble impurities in the battery supply water do not leave the diffuser in the juice, because a portion diffuses into the pulp and is discarded at the tail of the diffuser. The extent of juice contamination is dependent upon the quality of the diffusion supply water, the draft (9)², and the equilibrium distribution of impurities between juice and pulp (9). The contamination of juice by ash constituents of the supply water has been estimated by various authors to amount to 33 to 50% of that present in the water (1,5,6), and water containing 250 ppm of chloride is considered to be unsuitable for diffusion (1).

The present study was undertaken to assess the magnitude of the problem, and to devise a system to reduce the contamination of the juice by ash constituents of the diffusion supply water. Consideration of the theoretical distribution of a soluble additive in a diffuser leads us to the belief that, since the water used for diffusion usually is made up of two kinds, one of which is free of ash constituents, it should be possible to alter the fraction of supply-water solids that are eliminated with the pulp.

Experimental

The diffuser used in these experiments is a laboratory Bruniche-Olsen continuous countercurrent diffuser (4). Beets were obtained from the Woodland factory of the Spreckels Sugar Company. They were washed and then stored in moist pine shavings at 1° C prior to use (4). Sodium was determined by use of a flame photometer attachment on a Beckman DU spectrophotometer, and chloride by means of an Aminco automatic chloride titrator.

For each test in the diffuser, about 200 pounds of beets were removed from storage and sliced into standard cossettes, then mixed in a plastic-lined cement mixer, in order to have a homogeneous supply during the day. Two runs were made in the

¹ Western Regional Research Laboratory, Western Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, Albany, California.

² Numbers in parentheses refer to literature cited.

diffuser, the first to establish the distribution of salt from the supply water between pulp and juice, and the second to determine the effect of altered supply water management on this distribution. Feed rates to the diffuser were 9.0 kg. of cossettes and 12.0 liters of water per hour. Product rates were 6.6 kg. of pulp and 12.6 liters of juice per hour. The draft was 146 and diffusion temperature 70° C, with a retention time of 57 minutes for beets and 27 minutes for juice. Measurements were made on grab samples of pulp and juice for sodium chloride, refractometric solids, and polarization sugar.

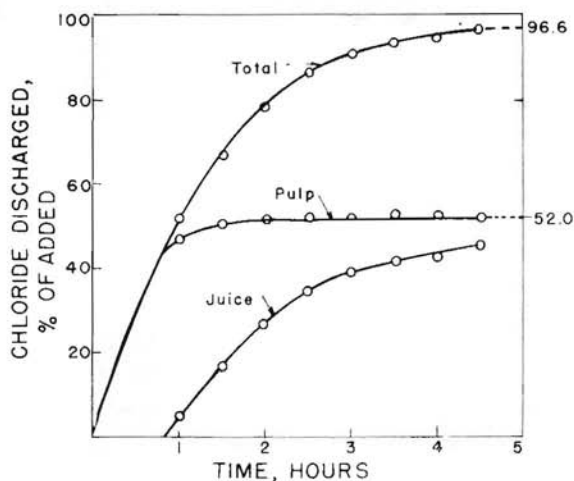


Figure 1.—Distribution of added chloride during normal operation.

For the first run, L21, the diffuser was operated with distilled water as the battery supply until the pulp and juice solids became constant, in order to establish a base concentration of sodium and chloride in the pulp and juice. The battery supply was then changed to 5% NaCl and diffusion continued without other changes. Figure 1 shows the resulting distribution of chloride in the pulp and juice, expressed as a percentage of the chloride added, after subtraction of the base concentration of chloride introduced by the beets. Equilibrium concentration of chloride was reached in the pulp much more quickly than in the juice. Of more interest is the observation that although the chloride in the pulp water reached essentially the same concentration as that in the battery supply, about 48% of the added chloride was carried over into the juice, because the battery supply volume was almost double the pulp water volume. Countercurrent diffusion works very well in reverse, and pulp was shown to be an efficient extractor of chloride ions, but the large excess of

water in the juice over water in the pulp permitted a great deal of salt to leave the process with the juice.

One way to increase the removal of salt by the pulp is to decrease the draft, but this is not desirable because it would severely decrease the extent of sugar extraction. An alternative is to split the battery supply into two streams, introducing one containing salt at the tail end, and the other containing no salt nearer the head end. This would give the desired low draft at the tail end, to favor diffusion of salt into the pulp, and the desired high draft at the head end to favor diffusion of sugar out of the cossettes. Accordingly, a second run (L22) was made in the same way as Run L21, except that the battery supply water was split into two streams. One half of the battery supply was 5% sodium chloride, introduced at the tail end, and the other half of the battery supply was distilled water, introduced through a hole in the trough cover, 12 inches forward of the tail. The effective diffusion length of the apparatus is about 38 inches, therefore, the distilled water was introduced about one third of the length of the diffuser from the tail. The results of Run L22 are shown in Figure 2. The flow rate of the salt supply dropped at 2.5 hours, but equilibrium was reached by 5 hours, as shown by the total recovery figure of 99.8%. Splitting the battery supply into two streams resulted in a much more favorable distribution of salt between the pulp and the juice.

A comparison of Runs L21 and L22 is given in Table 1, in order to evaluate the effect of the split stream. The amount of chloride in the juice at equilibrium is given as the difference

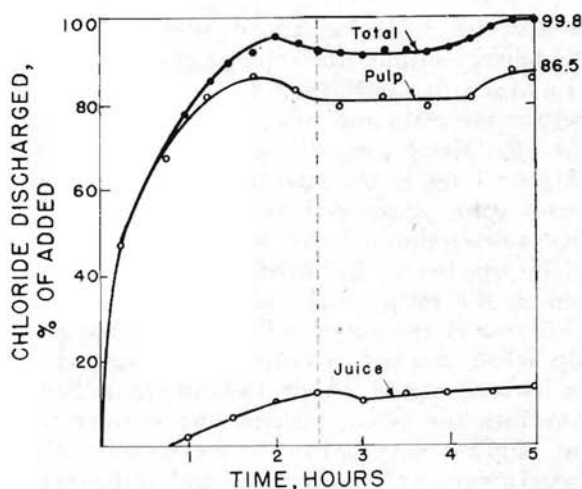


Figure 2.—Distribution of added chloride using dual battery supply.

between 100 and the pulp chloride. Figure 1 shows that the pulp chloride has reached a constant value, but that the juice concentration is still increasing slowly, and should be 48% at equilibrium. The material balance is as good on L22 as on L21, so that the change in flow rate of salt during L22 apparently did not prevent equilibration. There was an increase in pulp sugar with the split stream under the conditions of this experiment. We would expect that loss in a commercial battery would be less, because it would be possible to introduce the pure water at a point proportionally closer to the tail end of the battery, so that the desired high draft would hold for a greater fraction of the total diffuser length.

Table 1.—Equilibrium distribution of NaCl introduced in battery supply water.

	Single battery supply Expt. L21	Dual battery supply Expt. L22
Pulp chloride, % of added	52	86.5
Juice chloride, % of added	48 ¹	13.51
Pulp sucrose, pol	0.21	0.32
Cl:Na ratio pulp	1.49	1.53
juice	1.63	2.34
Excess Na in pulp, g/kg	0.95	0.85
Excess Na in pulp, Meq/kg	41	37

¹ Estimated equilibrium value. Measured values were 44.6% for L21 and 13.3 for L22.

The results for sodium are not quite the same as those for chloride. It is apparent that a disproportionate amount of sodium is carried out with the pulp in each case. The sodium deficit in the juice is more apparent in Run L22 where the Cl:Na ratio is 2.34 instead of 1.54 as in NaCl. The total amount of sodium in excess of the chloride in the pulp is about the same in each run. The explanation for this exchange capacity of the pulp is the presence of uronic acid polymers in the pulp (2,3,8). We have found previously that the pulp solids are about 20% anhydrouronic acid (a measure of uronic acid polymers), and approximately two thirds of the acid groups are free carboxyls (7). Since wet pulp is about 5 to 6% solids, this means there are about 37 to 45 milliequivalents (meq) of free acid per kilo of wet pulp. This would mean a total capacity for cation exchange amounting to 0.85 to 1.03 g Na/kg wet pulp. This calculated value agrees with the observed exchange value, even though the excess sodium figure is calculated from a small difference of rather large numbers.

To verify this exchange capacity, a composite sample of pulp from the distilled water portion of Runs L21 and L22 was heated at 70° C for 30 minutes with an equal weight of water containing CaCl₂ or NaCl. The calcium ion concentration was then

measured in the supernatant. The results in Table 2 show that there was a cation exchange in the pulp and, furthermore, that sodium could displace the calcium that was already bound by the pulp. The total exchange capacity of this pulp is estimated to be 47 meq/kg of wet pulp (28.8 plus 17.9). This is not a precise estimate for two reasons. First, it is quite possible that a single batch equilibration with sodium ion is not sufficient to displace all of the calcium bound by the polyuronide carboxyl groups. Second, the free carboxyl groups of 28.8 meq/kg shown by the more concentrated calcium solution could be high, because some of the calcium may be bound as $(CaCl)^+$ instead of Ca^{++} . These two effects are in opposite directions and tend to cancel each other. The indicated capacity is sufficient to explain the observed effect of sodium exchange by the pulp in the diffuser.

Table 2.—Exchange capacity of pulp.

Salt added ¹ meq/kg pulp	Ca ⁺⁺ exchange observed meq/kg pulp
CaCl ₂ 70.6	28.8 adsorbed
CaCl ₂ 21.1	14.2 adsorbed
NaCl 850.0	17.9 desorbed

¹ Exhausted pulp heated 30 minutes at 70° C with equal weight of salt solution.

The theoretical distribution of a soluble additive in a diffuser has been studied by Stitt (9). Equations were developed predicting the concentration of additive at any point in the diffuser in terms of the number of cells, the relative volume of liquid in the juice and in the cosettes, the rate of movement of juice and beets, and the point in the diffuser where the additive is introduced. The present data have been compared with the results predicted from these equations. One of the assumptions made in developing the equations was that d , the ratio of juice volume to beet liquor volume, remained constant throughout the diffuser. This was not true for these runs, for d was 1.92 at the tail of the diffuser, and 1.55 at the head end. The explanation for this may be that when the beets are heated, their capacity for retention of juice is reduced. This reduction of volume occurs simultaneously with diffusion. The reduction in volume is calculated to be about 23% beginning-to-end of the diffusion. The water associated with the pulp was reduced from 18.2 to 15.5 g of water per gram of marc, a reduction of 15%. Thus, it may be seen that part of the reduction in volume is due to the loss of soluble solids from the water inside the beets. If it is assumed that the volume ratio of 1.92 holds for the portion of the diffuser where active diffusion is occurring, the number of theoretical

cells calculated from the pulp sugar loss of 1.2% is 5.6 cells. This leads to the prediction for Run L21 that the pulp will remove 51.5% of the added chloride while the observed value was 52%.

In the split stream experiment, the battery supply at the tail end was 6 liters per hour of salt water, and the pulp water volume was 6.24 liters per hour, so that d equals 0.96. Using this ratio, and 5.6 theoretical cells, the calculated fraction of chloride in the pulp is 81.1%. The observed value is 86.5%. The advantage of the dual water supply scheme is that in order to achieve this elimination of chloride in the pulp with a single supply of water it would be necessary to reduce d to 0.96 for the entire length of the diffuser, in which case the pulp sugar loss would be 16.2% instead of 1.9%, as found for Run L22.

In order to apply this system of water management to a factory diffuser, it is necessary to have two sources of water, one containing salts and the other free of salts. Fortunately, this is the case in factories where there is no return of pulp press water, for some of the battery supply is make-up water from outside the factory, and the rest is condensate from the evaporators. In this case, the make-up water would be added at the tail of the battery, and the condensate some distance forward. The idea of using more than one stream of water into the diffuser can also be applied to the case of pulp press water return, but not in the same way. In this case, the press water contains some sugar which can be saved, and the object then is to *reduce* the amount of soluble solids from the water which will be lost in the pulp. To accomplish this, the pulp water is introduced ahead of the condensate water, at a point where the sugar is slightly higher in the cosettes than in the pulp water. In either case, the application would consist of supplying two different sources of water at two points in the diffuser instead of mixing them outside the diffuser and supplying the mixture at the tail end.

Summary

It has been shown that diffusion water salts can be eliminated in the sugar beet pulp by altering the method of introducing water to the diffuser. In a small continuous diffuser of 5.6 theoretical cells, the salt elimination in the pulp is increased from 52% to 86% by supplying the water containing salt at the tail end, distilled water about one third of the way forward in the diffuser. The increased sugar loss in the pulp is very small by this procedure. The results were found to be in good agreement with the theory developed by Stitt (9). Application of a similar system to the return of pulp press water in order to increase the amount of sugar in the juice is also discussed.

Acknowledgment

The authors wish to thank Mr. Harold Lukens for the sodium analyses, and E. J. Barta, K. Smith, and R. L. Patterson for assistance with the diffuser.

Reference to a company or product name does not imply approval or recommendation of the product by the U. S. Department of Agriculture to the exclusion of others that may be suitable.

Literature Cited

- (1) BÖTTGER, S. 1951. Betrachtungen über den einfluss von salzhaltigem Betriebswasser bei der Zuckerfabrikation. *Zeit, Zuckerind.* 1: 61-63.
 - (2) CARRUTHERS, A., and J. F. T. OLDFIELD. 1957. Absorption and desorption of calcium ions by cosettes. *Int. Sugar J.* 59: 277-281.
 - (3) DEUEL, H., K. HUTSCHNEKER, E. STUTZ and J. C. FREDERIKS. 1957. Anionwirkung auf Ca-Na-Gleichgewichte an Kationenaustauschern. Ionenaustauscher. 9. *Helv. Chim. Acta.* 40: 2009-2014.
 - (4) MORGAN, A. I., JR., E. J. BARTA and G. O. KOHLER. 1959. Development of a sugar beet processing laboratory. *J. Am. Soc. Sugar Beet Technol.* 10: 563-570.
 - (5) MÜHLPFORTE, H. 1959. Über den einfluss der gewässerversalzung auf die Zuckerfabrikation. *Wasserwirtschaft-Wassertechnik.* 9: 348-349.
 - (6) MÜHLPFORTE, H. 1960. Über salzhaltiges Diffusionswasser in den Zuckerfabriken. *Die Zuckererzeugung* 4: 276-278.
 - (7) OWENS, H. S., E. A. McCOMB and G. W. DEMING. 1954. Composition and percentage of marc in some varieties of inbred sugar beets. *J. Am. Soc. Sugar Beet Technol.* 8: 267-271.
 - (8) SPEISER, R., C. H. HILLS and C. R. EDDY. 1945. The acid behavior of pectinic acids. *J. Phys. Chem.* 49: 328-343.
 - (9) STITT, F. 1957. Theoretical steady state distribution of an additive in sugar beet diffusers. *J. Am. Soc. Sugar Beet Tech.* 9: 611-631.
-