Effects of Some Variables on First Carbonation

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A sugar beet processing laboratory (5) has been built at the Western Regional Research Laboratory, Albany, California, as a result of an agreement between the Beet Sugar Development Foundation, Fort Collins, Colorado, and the U. S. Department of Agriculture (2). A key unit of this processing laboratory is a continuous, Dorr-type, model carbonator.

The purpose in building a model carbonator was to further the study of the effects of beet composition on processing behavior. In order to do such experiments, the interdependence of operating factors and the reproducibility of observation, separated from feed variability, must be known. The techniques of operation and analysis needed development before elaborate experiments were begun. For these reasons, a set of experiments on storage of a large sample of diffusion juice was planned.

Diffusion juice was collected from a nearby factory with no attempt at immediate cooling. This juice was stored in a 1° C. room with toluene and chloroform preservatives. A marked deterioration after 2 weeks was observed. A second lot of juice was frozen and held at −15° C. The purity of freshly thawed samples was constant during as much as 3 months of frozen storage. Carbonation quality of thawed juice was very similar to that of the fresh juice. Unfortunately, it was later found that thin juice from the frozen stock had lost most of the protein fraction of its colloids, as compared to thin juice from the fresh diffusion juice.

A study of various means of holding unfrozen juice suggested the procedure finally used. In this procedure 300 gallons of diffusion juice from the Roberts battery at Holly Sugar Co., Alvarado, California, was collected and cooled within minutes to 5° C. in a heat exchanger. Twelve p.p.m. of Terramycin and 40 p.p.m. of phenyl mercuric nitrate were added immediately. The juice was held in drums in a 1° C. room until used. The result was juice which showed no change during the 5-day periods required for each series of experiments. A slight change was observed between 2 replicates which were begun at 55 days and 75 days of juice age, respectively. This juice had been extracted from beets harvested at the end of the Imperial Valley's spring campaign and was consequently low in purity.
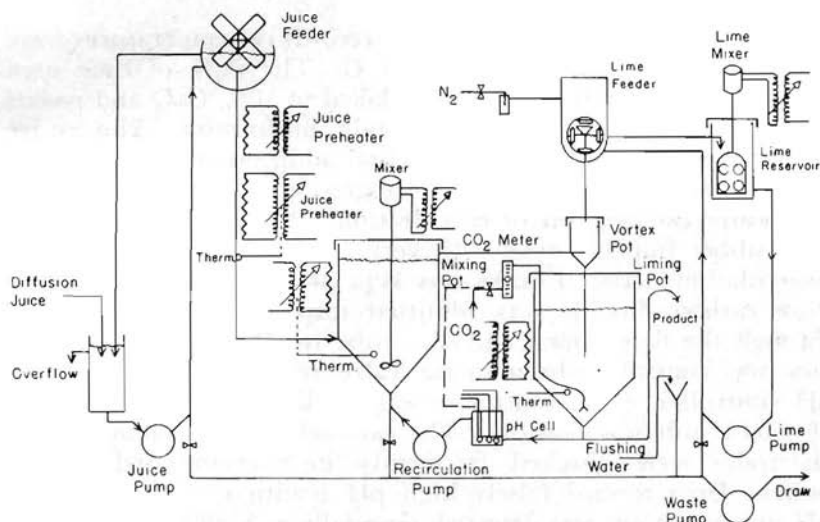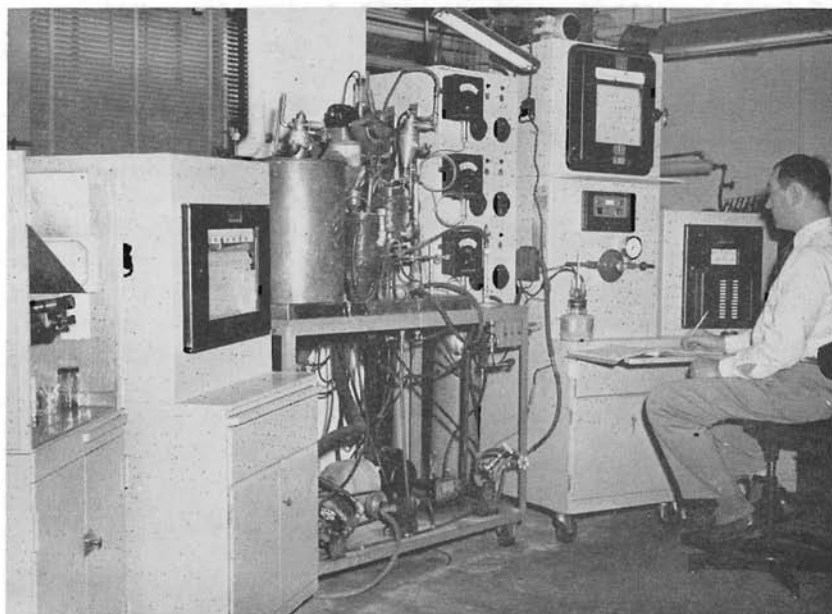
Figure 1.—Schematic of dorr carbonator.

Figure 2.—Photo of carbonator.

The 3-gallon-per-hour model Dorr-type carbonator used is shown schematically in Figure 1 and in a photograph, Figure 2. This device is based on a development by R. A. McGinnis of Spreckles Sugar Co. (8). The carbonation tanks and juice feed are electrically heated. The temperature used throughout this

experiment was 80° C. Continuous records of temperatures were kept, showing control to be ±0.5° C. The milk of lime used was prepared from builder's lime, slaked to 10% CaO and passed through a wet colloid mill to obtain uniformity. The entire lime system up to the point of actual addition to the juice was kept under a water-saturated nitrogen atmosphere to prevent premature carbonation or dehydration. Pumps were slow moving, rubber impeller type with very gentle action. The rate of recirculation between tanks was kept at 7 times the feed rate. Pure carbon dioxide was admitted into the juice-lime slurry through the flared open end of a tube in the liming tank. Gas flow was controlled by a motor valve responding to a 3-mode pH controller. The controller was modified so that sensitivity of ±0.01 unit was possible. The calomel-glass high-temperature electrodes were checked frequently in thermo-stated, stirred buffer. Drift toward falsely high pH readings, as rapid as 0.1 pH unit per hour, was detected, especially at high pH. To avoid this drift, both electrodes had to be dipped in strong acid as often as every 15 minutes during critical parts of the experiment. Control of temperature to ±0.3° C. at the pH cell was required for meaningful pH values. Alkalinity titrations were made at 5-minute intervals, using pH 8 as an end point.

The experiment was designed to include 4 levels of alkalinity: 0.07, 0.08, 0.11 and 0.14% CaO; 2 levels of lime addition: 1.5 and 2.5% CaO based on beet weight; and 2 levels of retention time in carbonator: 20 and 40 minutes. A factorial experiment involving the 16 conditions, repeated once, was used, with orders of performance within each replicate randomized separately.

Results are compared so as to show all main effects and simple interactions with alkalinity. Other effects are considered error. Additional estimates of error were obtained where possible by considering the variation in retention time to be without effect.
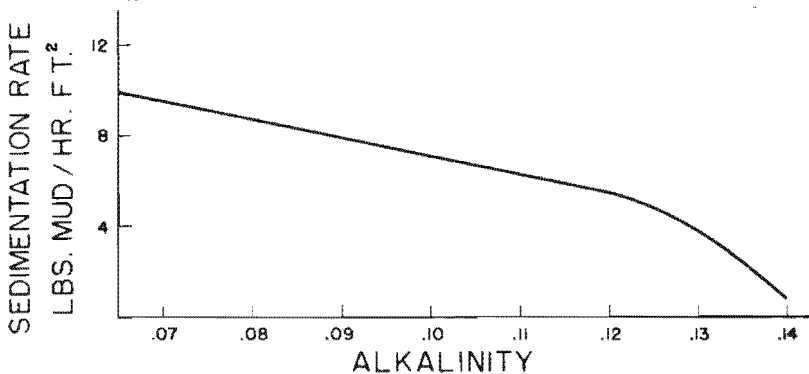

Figure 3.—Sedimentation vs. alkalinity.

Sedimentation is shown in Figure 3 as a function of alkalinity for the average of both levels of each of the other factors. Sedimentation rates were calculated from the Dorr-Kynch test (9), using 80° C. and an underflow ratio of 5.5 pounds of fluid per pound solids. The curve shows the usual relation, but values are low, due to the poor beet quality. The coefficient of variation for this test is 10 percent for equivalent conditions.
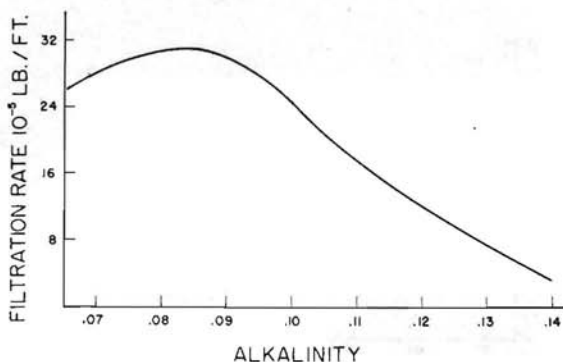

Figure 4.—Filtration vs. alkalinity.

Filtration rate versus alkalinity is shown in Figure 4. Filtration rate expresses the conductivity of unit thickness of filter cake to flow of unit viscosity filtrate when measured at 70° C. with a constant-pressure difference of 15 cm. Hg. Filtration rate is defined by (6)

$$\frac{1}{a} = \frac{\mu ws}{Pg}$$

where $\frac{1}{a}$ is filtration rate, μ is viscosity, w is cake solids per unit volume filtrate, P is pressure difference used, g is gravitation constant, s is slope of curve of time divided by true filtrate volume versus true filtrate volume. The apparatus used for filtration measurement is similar to that described by Bernhardt (3). The cake is sheltered from agitation of the slurry. The true filtrate volume is the collected volume at any time plus the volume between the septum and the collector. Coefficient of variation of filterability was 40% but some later data show improvement to 20% when the septum is turned from the vertical to the horizontal position with the chamber entrance up. The test appears satisfactory with this modification.

Thin juice was prepared batchwise from samples of each carbonation slurry. This was done by hot filtration followed by heating to boiling temperature in a jacketed kettle; carbon dioxide

was bubbled into the juice for 3 minutes followed by further boiling for 5 minutes. After second filtration the resulting juice was analyzed for purity, color, lime salts, total nitrogen, amino nitrogen, ammonia nitrogen, and total anions.

Thin juice purity versus alkalinity is shown in Figure 5. The effect is statistically significant, since the coefficient of variation was 0.6%, but the effect may apply only to this one juice.
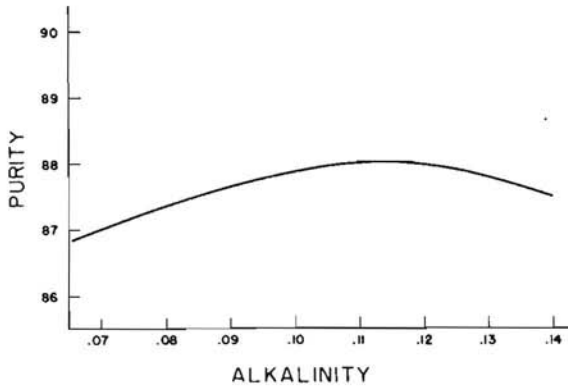

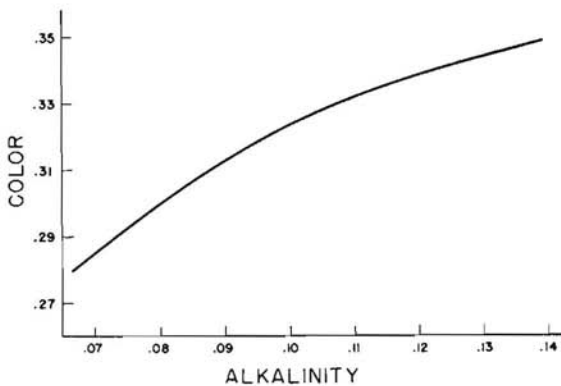Figure 5—Purity vs. alkalinity.

Figure 6.—Color vs. alkalinity.

Color versus alkalinity is shown in Figure 6. Color is given as optical density of a 1 cm. sample at pH 7 for this juice at 420 mu less optical density at 720 mu, corrected to 10% RDS. The surprising result shown may arise from the low pH of the carbonation slurries, which at .07 alkalinity was only 9.5. The coefficient of variation for color was 8%.

Lime salts versus alkalinity are shown in Figure 7. Determination was by versene titration. The result was not unusual (4).
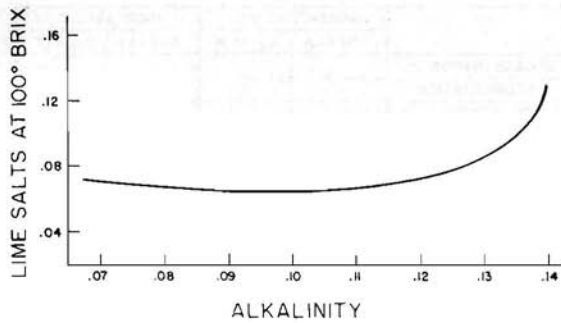
Figure 7.—Lime salts vs. alkalinity.

The coefficient of variation was 20%, which suggests that some important factor, such as time, was not under adequate control in second carbonation or in subsequent analysis.

Total nitrogen by Kjeldahl versus alkalinity is shown in Figure 8. This does not reflect the purity maximum found at alkalinity 0.11. The coefficient of variation for total nitrogen was 1%.
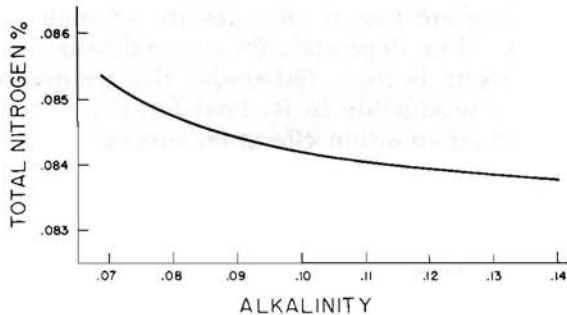

Figure 8.—Total N vs. alkalinity.

Amino nitrogen found by Van Slyke manometric analysis (7) on the thin juice follows the same trend as total nitrogen and constitutes over 40% of the total. Ammonia nitrogen found by MgO distillation (1) of the thin juice was independent of treatment and represented 8% of the total nitrogen.

The effects of 1.5% and 2.5% lime rates and 20- and 40-minute retention times are shown in Figure 9 together with all significant interactions with alkalinity. Values are not shown, since they are peculiar to the diffusion juice used. The effect of increased lime rate on sedimentation and filtration rates is deemed favorable, since it more than compensates for the additional mud quantity. Increased lime, as expected, was generally

	INCREASED LIME		INCREASED TIME	
	0.07 ALK.	0.11 ALK.	0.07 ALK.	0.11 ALK.
1ˢᵀ CARBONATION				
SEDIMENTATION	+	+	O	O
FILTRATION	+ +	+ +	O	O
THIN JUICE				
PURITY	O	O	O	O
LIME SALTS	+	+	−	O
COLOR	+	+	−	− −
TOTAL ANIONS	+	+ +	O	O
TOTAL NITROGEN	+ +	+	+	+
AMINO NITROGEN	+	+	+	+
AMMONIA NITROGEN	O	O	O	O

+   FAVORABLE EFFECT
−   ADVERSE EFFECT
O   NO EFFECT

Figure 9.—Lime and time effects.

favorable throughout. The effects of retention time were gen-erally nil, except for the adverse color increase with longer re-tentions, especially at higher alkalinities. Purity was essentially unaffected by either variable.

In summation, the trends with changes in alkalinity, lime rate, and retention time are shown. Results are anomalous for color versus alkalinity. The dependability of lime salts from experi-ment to experiment is low. Otherwise the technique used is satisfactory and reproducible in its final form. It is suitable for our study of beet composition effects on processing.

Literature Cited

(1) Association of Official Agricultural Chemists. 1955. Official Methods of Analysis. 8th ed. p. 371.

(2) Beet Sugar Development Foundation. Cooperative Agreement No. 12-14-100-111 (74) between Beet Sugar Development Foundation and U. S. Department of Agriculture.

(3) BERNHARDT, W. O. 1954. An experimental micro-filter. Proc. Amer. Soc. Sugar Beet Tech. VIII (2) :284.

(4) McGINNIS, R. A. 1951. Beet Sugar Technology. Reinhold Pub. Co., Chapter 8.

(5) MORGAN, A. I., BARTA, E. J., and KOHLER, G. O. Design of a sugar beet processing laboratory. In preparation for J. Amer. Soc. Sugar Beet Tech.

(6) PERRY, J. H. 1950. Chemical Engineers Handbook. McGraw-Hill Pub. Co., p. 967.

(7) PETERS, J. P., and VAN SLYKE, D. D. 1946. Quantitative Clinical Chem-istry, Williams & Wilkins.

(8) SKAAR, K. S., and McGINNIS, R. A. 1944. Purification of sugar beet juice. Ind. Eng. Chem., 36, 574.

(9) TALMAGE, W. P., and FITCH, E. B. 1955. Determining thickness unit areas. Ind. Eng. Chem., 47, 38.