# Liming of Sugar Beet Cossettes

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The prospect of improving sugar beet processing by pretreating beets with lime has intrigued beet technologists for many years. Proposed systems have included pressing limed brei (2,3,4,12)[2], pressing limed and neutralized brei (13,14), diffusion of limed and neutralized beet chips (14), diffusion with dilute lime solution (7), and diffusion of cossettes after treatment with a solution of lime and sugar (10). To our knowledge, none of these processes has found commercial acceptance. This paper reports our investigation of the effects of lime on beet tissue and some results of processing limed cossettes.

The effects of lime on beet tissue are due primarily to the reactions of pectin under alkaline conditions since other pulp components are relatively inert. About 30% of the water-insoluble portion of the beet is pectin. Pectin is a linear polymer of galacturonic acid units which are partially methylated and acetylated. In sugar beet pectin, about 50% of the carboxyl groups are methylated, and about 30% of the hydroxyl groups, which are in positions 2 and 3, are acetylated.

Previous knowledge of the effects of alkali on pectin (9,11) may be summarized as follows: Two reactions may occur when pectin is treated with alkali. One reaction is ester hydrolysis, which produces methanol and acetate as byproducts. The other reaction is polymer degradation by glycosidic bond cleavage. Figure 1 shows a portion of the pectin molecule and the reactions with hot or cold alkali. At low temperature the predominant reaction is ester hydrolysis. The resulting pectic acid is highly resistant to alkaline degradation. The calcium salt of pectic acid is insoluble, and, if lime is the alkali used to deesterify the pectin, the divalent calcium cross links acid groups to form a rigid calcium pectate structure.

At high temperature, not only are the esters hydrolyzed, but glycosidic bonds adjacent to esterified carboxylic groups are broken (11). Since about half of the carboxyl groups are originally esterified, the pectin chain is almost completely destroyed. This pectin degradation makes the associated hemicellulose soluble in water, and thus greatly reduces the mechanical strength of the beet tissue. Although the degradation product shown in

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[2] Numbers in parentheses refer to literature cited.

Figure 1 has a galacturonic acid end group, it may actually be unsaturated, as is the case with enzymatic degradation (1). In either case the glycosidic bond is broken adjacent to the esterified carboxyl group. The degradation reaction occurs only at esterified loci, and thus previous cold de-esterification protects the pectin from subsequent hot alkaline degradation.

Below 25° C the principal alkaline reaction is de-esterification, but degradation increases rapidly at higher temperatures. At 75° C there is so much degradation that sugar beet pulp loses its structure and becomes mushy. The temperature dependence could be explained by lower activation energy for de-esterification than for degradation, as is the case under acid conditions (9).

In order to take advantage of these properties of beet pectin, fresh cossettes should be treated with lime at low temperature. This treatment would stabilize the pectin by de-esterification, and at the same time form a calcium pectate precipitate *in situ*. This insoluble matrix would hinder the extraction of colloids during diffusion, but allow free diffusion of sugar.

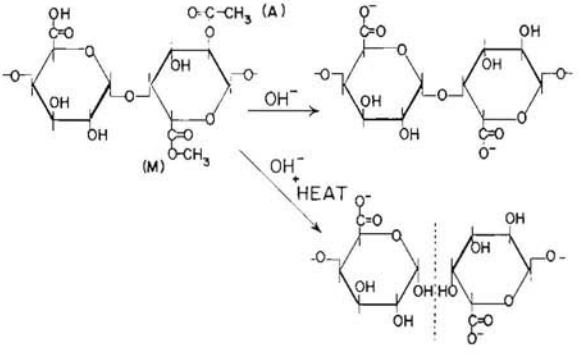


**Figure 1.—Alkaline reaction of pectin. De-esterification (top) and degradation (bottom). M indicates a methylated carboxyl, A. indicates an acetylated hydroxyl group. Dashed line indicates a broken glycosidic bond.**

## Lime Penetration

Esterified pectin can be detected in plant tissue *in situ* by reaction with alkaline hydroxylamine and ferric chloride (6). This reaction was used to follow the de-esterification of sugar beet pectin with lime. Five centimeter rectangular plugs were cut from a sugar beet with a microtome, and soaked in 1% lime slurry. Sections examined after ten minutes showed de-esterification to a depth of 0.1 mm at 25° C and 0.2 mm at 45° C. This corresponds to 3 and 7 cells, respectively. No gradation of partially de-esterified pectin was apparent. The sharp demarcation between de-esterified and unreacted pectin indicates that the rate controlling step is diffusion of lime.

Similar tests with cossettes showed that after ten minutes liming at 25° C only a narrow band of esterified pectin remained in the center of the cossette. The greater penetration into cossettes can be explained by considering the cossette structure shown in Figure 2. The numerous transverse cracks allow the lime to penetrate rapidly which results in more extensive de-esterification than would be predicted from the results on microtome-cut pieces.

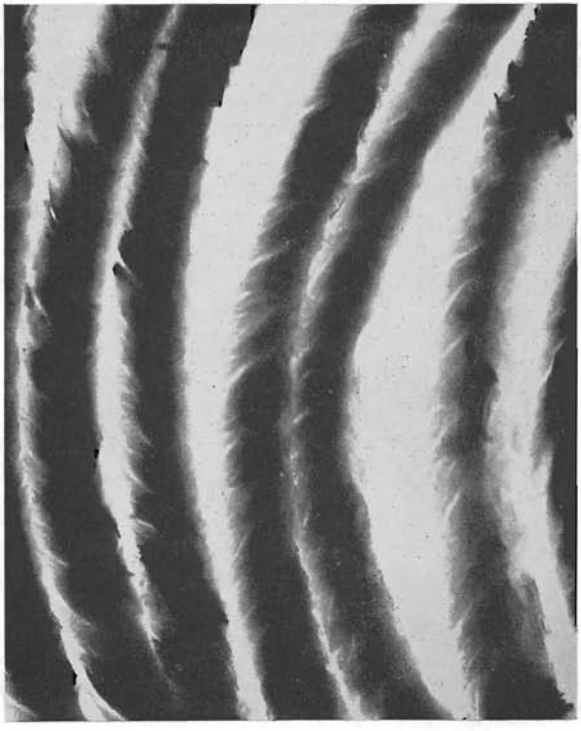

**Figure 2.—Sugar beet cossettes showing transverse cracks.**

## Saponification Rates

De-esterification and deacetylation rates were obtained by measuring the residual ester and acetyl content of pulp after reaction with lime. Cossettes were mixed with dry $Ca(OH)_2$ and held at 25° C. Samples were withdrawn at timed intervals and the acetyl and methoxyl content of the marc measured (5). The results were calculated as percent saponification, and are shown in Figure 3. The rate of demethylation is greater than the rate of deacetylation at this temperature. Demethylation stabilizes pectin, but deacetylation does not, and furthermore, it produces

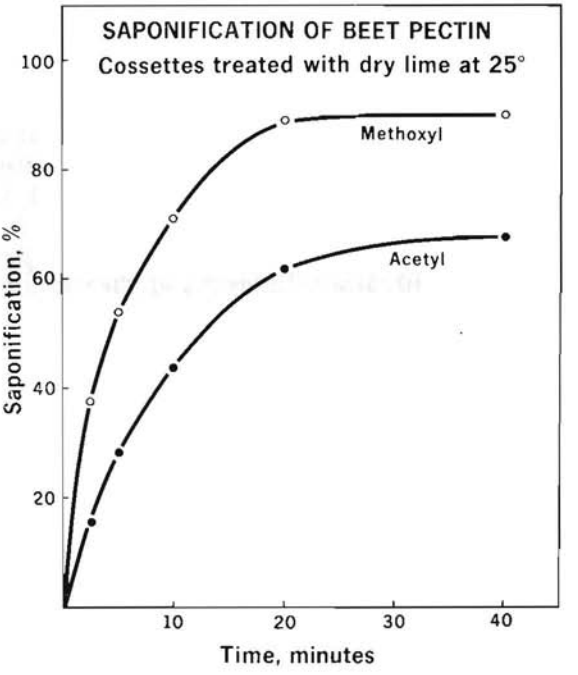


Figure 3.—Saponification of beet pectin. Rate of de-esterification and deacetylation. Cossettes treated at 25° C with dry $Ca(OH)_2$, 1.3% on beets.

acetic acid as a byproduct. Since demethylation is more rapid than deacetylation, it may be possible to restrict deacetylation and still stabilize pectin by sufficient demethylation.

## Processing Tests

Several different processing procedures were evaluated by examining diffusion juice and thin juice from treated beets. Diffusion was carried out in a Brüniche-Olsen laboratory diffuser with a feed rate of 9 kg of beets per hour. First carbonation was continuous, in a Dorr-type laboratory carbonator (8). Thin juice was made by batch second carbonation of filtered first-carbonation juice. Cossettes were limed in the following four ways: 1) dry $Ca(OH)_2$ preliming, 2) lime slurry preliming, 3) direct liming in the diffuser, and 4) countercurrent washing with limed diffusion juice. Cossette liming was either at room temperature (less than 25° C) or at 40° C, using 0.4 to 1.0% CaO on beets. Diffusion temperature was 70° or 25° C with alkaline beets, and was 70° C with limed beets which had been neutralized with

$CO_2$. Liming at 40° C was unsatisfactory because of pulp deterioration. Lime slurry treatments were difficult to manage because of the rapid extraction of sugar, even at low temperature. For continuous operation, a system of adding treatment liquor to the diffusion juice at a constant rate would be needed. Dry liming was easier, and still gave satisfactory treatment to the beets. Treatment levels above 0.4% CaO on beets were unnecessary. Diffusion juice from alkaline beets was clear and light colored. Juice from neutralized beets was darker, with more colloids, but still much lighter and clearer than normal diffusion juice. All of the diffusion juices were subjected to first carbonation, with additional lime. Sedimentation and filtration rates were determined on the first carbonation juices, and the color, lime salts, and purity of the second carbonation juices were measured. Table 1 is a summary of the results from the best three treatments. The major differences are in the total lime consumption and in the lime salts and total anions. The final mud volumes are a reflection of the amount of lime used.

**Table 1.—Processing comparisons for prelimed beets**

| | Cossettes limed at room temperature with dry Ca $(HO)_2$, (0.4% CaO on beets) | | | |
|---|---|---|---|---|
| | Control | Alkaline | | Neutral |
| Preliming time, min. | none | 30 | 30 | 10 |
| Diffusion temp., °C. | 70 | 25 | 70 | 70 |
| First carbonation: | | | | |
| Lime added (% CaO on beets) | 2.0 | 0.5 | 0.5 | 1.0 |
| Sedimentation resistance | 25 | 32 | 31 | 34 |
| Mud volume (%) | 9.0 | 5.0 | 5.0 | 7.0 |
| Second carbonation: | | | | |
| Lime salts (CaO/100 Bx) | .05 | .52 | .39 | .18 |
| Anions (meq./100 Bx) | 31 | 50 | 55 | 51 |
| Purity (%) | 91.6 | 89.5 | 90.2 | 90.0 |
| Color (OD/100 Bx) | 2.0 | 2.0 | 2.8 | 2.8 |

The total lime consumption for the alkaline-diffused beets was 0.9%, for the neutralized beets 1.4%, and for the control beets 2.0% CaO on beets. Lime salts and anions are high in the second carbonation juice from the limed beets because of acetate liberated from the pectin. The increase in total acids was matched by the increase in volatile acids, which we assume to be acetic acid. These excess anions prevent the removal of lime by carbonation without the addition of corresponding cations. The sodium carbonate needed to reduce the lime salts to the level of the control is calculated to be 0.05 to 0.15% on beets. The increase in anions, calculated as acetate, plus the increase in lime salts, calculated as calcium ion, is 1.3 to 1.7 grams per 100

grams of solids in second carbonation juice. Within experimental error, this is the same as the observed drop in purity for juices from limed beets. Prelimed cossettes do retain more colloids and insoluble calcium salts than do untreated cossettes, but these are materials which would be removed during carbonation. There is no net change in the amount of soluble nonsugars in second carbonation juice, except for acetate produced by deacetylation of pulp pectin.

Pulp from prelimed beets is much tougher than normal pulp. Laboratory tests showed that up to 40% solids could be obtained using a static press but that the pulp was too tough to be pressed in a high-speed screw press. We were unable to test pulp from prelimed beets in a factory press without introducing the system to an entire factory, but we have made some factory tests on pulp from a continuous slope diffuser by adding lime after diffusion. Pulp treated with 0.15% CaO becomes hard and tough in the same way as do the cossettes. Dry hydrated lime or a slurry of lime was added to the pulp in the conveyor system feeding the presses. The temperature was 45° to 50° C. Although the trials were not extensive, it does seem that lime-hardened pulp can be pressed satisfactorily. In one trial, about 100 tons of pulp were treated at the rate of 0.15% CaO on pulp over a period of two hours, and the pulp was pressed on two or three Stearns-Rogers presses to about 83% moisture, which was the same as for untreated pulp. On two occasions, lower moistures were obtained for brief periods, but the presses plugged, either because the bottom plates were too tight or because not enough pressure could be developed to force the pulp through the discharge slot. The power consumption on the presses was not excessive, even when they were plugged.

## Summary

Cossette preliming at low temperature stabilizes the beet structure to permit alkaline diffusion without disintegration of the pulp. Considerable retention of otherwise extractable colloids is observed, leading to a reduction in the lime needed for clarification. The production of acetate by alkaline deacetylation reduces the purity of clarified juice and increases the amount of lime salts.

The preliming of cossettes must be carried out below 40° C and sufficient time must be allowed for the pectin to be stabilized before it is subjected to heat. Alkaline diffusion may presumably be conducted over a wide range of temperature. The high pH will preclude fermentation at low temperatures, and the stabilized pectin will permit unusually high temperatures

during diffusion. Another advantage of preliming cossettes is the production of partially clarified juice, which might be suitable for direct ion exchange treatment.

**Acknowledgment**

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