Effect of Cossette Liming on Diffusion Juice

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Received for Publication October 7, 1982

INTRODUCTION

The sugar beet processing industry has a goal of minimizing energy usage for economic survival. Therefore, different alternatives to attain this goal are being tried and tested. The sugar beet processing industry is considered one of the top energy users in the food industry (16). A survey by Karren (11) showed that some factories use over 10,000 BTU per lb of sugar produced. With the high price of energy, the impact of any savings in energy consumption will be significant on the profitability of operation. It could even mean survival or demise of some operations. Randall, et al. (14) mentioned that 50% energy savings in drying of pulp could be obtained by preliming. This would amount to approximately 12% reduction in total energy consumption for beet-sugar production.

Reducing cost by treating fresh sugar beet cossettes with lime has been considered for some time, but so far the process has not been applied commercially. There has been no systematic study on the characteristics or quality of juice obtained from fresh sugar beet tissues treated with lime. As far back as 1905, Weinrich obtained patents for processing brei or chips which had been limed then neutralized (19,20,21). Loof and Pohl (12) reported using dilute lime water in the diffusion of cossettes. Other reserchers (2,3,4,17) have studied liming sugar-beets in one form or another, including Susic (15), Goodban and McCready (9), Bobrovnik, et al. (1) and Degtyar (8).

However, the studies mentioned above were carried out

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for reasons not primarily concerned with changing processing to consume less energy. With escalating energy costs, pretreatment of sugar beets with lime may offer the industry a potential advantage that has not been focussed on before.

The major advantage of using lime results from the crosslinking of calcium hydroxide with the pectic substances that hold the cells together, forming insoluble calcium pectate to prevent the usual degradation and peptization of the pectin. Camirand, et al. (6), described the reaction of pectin with lime in detail. With the present commercial system, juice extraction is carried out at 75-80°C to prevent microbial fermentation and to denature the beet cells. However, in the presence of lime, beet cells denature at lower temperature. In addition, high pH prevents fermentation. Therefore, extraction of juice could be accomplished at lower temperatures.

This study compares the characteristics of raw and purified juice from both untreated and lime treated beet tissue. Apparent purity was determined as the most important characteristic of juice quality. Also determined were lime salts, methanol and acetate, impurities which may be affected by beet liming.

MATERIAL AND METHODS

<u>Cossette</u> <u>Liming</u>. Lime was applied as either dry reagent grade $Ca(OH)2$ powder or as a thin juice slurry. Procedures used were reported by Camirand, et al (6).

<u>Diffusion</u> <u>Juice</u>. Experimental diffusion juice was produced by a bench-scale countercurrent, semi-continuous extraction process, developed in beet-sugar laboratories to simulate factory diffusion. Coarse cotton bags, filled with 200g of cossettes, were transferred at 15 minute intervals among a series of 12 beakers, each normally containing 200ml hot water.

Each cossette sample was contacted with 4 or 5 aqueous extraction liquors at about 75°C for sugar extraction. Final juice refractory dissolved solids (RDS) was usually about 12. Final sugar residue in the pulp was normally about 0.5-1.5%.

Juice Pufification. Thin juice was obtained by the oxalate procedure of Brown and Serro (5). Normally, 0.25-0.5% additional lime, based on juice (reported as CaO, but added as $Ca(OH)_2$), was added to the limed juice to aid in purification, although reasonably good thin juice could be prepared without it.

Apparent Purity and Color. The pale yellow thin juices or limed diffusion juices were analyzed directly in a 200-mm tube Rudolph Autopol III saccharimeter. RDS was obtained with a Bausch & Lomb Precision Model Refractometer. Apparent purity was calculated from saccharimeter and refractometer readings (as Polz and Brix) and the corresponding apparent density of the solution. Cossette purities were obtained by the same method after slurrying the cossettes with twice their weight of water. Color of raw and thin juices, given as ICUMSA (International Commission for Uniform Methods of Sugar Analysis) color units, were determined in a Varian Spectrophotometer at 420nm, after adjusting the pH to 7.0 and the RDS to approximately 10.0.

Total Alkalinity and Lime Salts. Total alkalinity of diffusion juice from prelimed cossettes (expressed as % CaO) was determined by titrating the juice with 1/28N HCl, using phenolphthalein as indicator.

Lime salts (as % on solids) were obtained by titrating thin juice samples with 1/28N EDTA, using hydroxyl napthol blue (HNB) as indicator. The RDS of the sample was first adjusted to a range of 11-17; then HNB buffer diluted with water was added to the sample.

Methanol and Acetate Ion. These impurities were obtained by Cochrane's gas chromatographic technique for free fatty acid (FFA) determination (7). Raw juices were initially treated with 4.0M HCl and centrifuged. The supernatant (1.5ul) was injected into a nickel column, using Chromosorb 101 packing at 180°C. Methanol and acetate (obtained as acetic acid) were expressed in millimolar concentrations.

RESULTS AND DISCUSSION

Color. Diffusion juice obtained from control samples was the normal grayish-black, turbid raw juice. In contrast, diffusion juice from limed cossettes was clear and light straw colored, after the small amount of particulate matter quickly settled. Table 1 shows the effect of lime on color of raw diffusion juice. Control diffusion juice had colors of 220 or higher. Diffusion juice obtained from cossettes treated with 0.5% dry lime had color of 36.4, while raw juice obtained from cossettes treated with 2 and 4% slurry lime had color of 12.2-20.2. After purification by the oxalate method, the color values of control and all limed thin juices were similar, ranging between 8.3 and 13.9 (see Table 1).

Table 1. Effect of lime on color of raw and thin juices (incl. S.D.).

Method of Application	Temperature (°C)	Lime added (%)	Color (ICUMSA units) Raw	Thin
Dry lime	22	0	220*	10.0 (1.4)
		0.5	36.4 (3.0)	9.9 (2.8)
		1.5	20.2 (0.9)	8.9 (1.1)
Slurry	4	0	220	8.3 (0.7)
		2	20.2 (3.9)	13.9 (4.3)
		4	25.8 (5.2)	10.6 (2.1)
	22	0	220	12.0 (2.3)
		2	19.8 (2.4)	12.0 (2.8)
		4	12.2 (3.2)	10.0 (3.3)
		10	27.8 (0.5)	11.6 (0.2)
	33	0	220	11.0 (0.9)
		2	15.7 (0.6)	10.5 (3.0)
		4	14.6 (3.6)	10.2 (2.6)

*Color levels higher than 220 could not be determined because of the opacity of these solutions.

Brix, pH, Alkalinity and Lime Salts. It has been reported that sucrose solutions treated with metal hydroxides could result in colloidal turbidities, sirupy gels, or flaky precipitates under appropriate conditions. If calcium hydroxide is used, the compounds formed are commonly know as calcium saccharates (10, 13). If insoluble saccharates are formed, with sucrose bound in these compounds, the Brix readings will consequently be lower. The results presented in Tables 2 (dry liming) and 3 (Slurry liming) suggest that little or no sucrose was lost by treating the cossettes with lime. Control juices had Brix readings of 11.1 to 15.6, whereas limed juices had Brix reading of 12.4 to 17.1. Apparently, the conditions used in these experiments were not favorable to the formation of insoluble saccharates.

Table 2. Effect of dry lime at room temperature on the Brix, pH, total alkalinity of raw juice and lime salts of thin juice (incl S. D.).

Lime Level (% as CaO)	Contact Time (min)	Sucrose Conc. (°Brix)	pH	Total Alkalinity (%CaO)	Lime Salts (% on solids)
0.0	0	15.6	6.6	---	0.266 (0.04)
0.5	10	14.6	10.7	0.10 (0.01)	0.738 (0.04)
0.5	20	17.1	11.0	0.07 (0.01)	0.571 (0.08)
1.5	10	15.8	11.6	0.34 (0.04)	0.644 (0.05)

As expected, pH of the juice increased as the amount of lime used increased. Control juices tended to be neutral or very slightly acid, while lime treated samples were alkaline, with pH's of 11.0-11.5. The alkalinity of the juice was also expected to increase as increasing amounts of lime were used in treating cossettes before diffussion. The data presented in Tables 2 and 3 confirm this expectation.

In slurry liming experiments, control thin juices had lime salts of 0.110-0.240%, compared to 0.331-0.516% for cossette-limed juices. It seems from Table 3 that lime salts increased with an increase in lime level or treatment time, as lime salts at 4% lime level were higher in each group than those at the 2% lime level. However, statistically (t test) there was no significant difference. This problem may be related to differences in beet batches, for an increase in lime salts with increasing lime application could be predicted based on the reaction of Ca(OH)2 with beet pectin. Increasing lime level did increase total alkalinity of the juice (significant at the 99% confidence level).

Table 3. Effect of slurry lime on the Brix, pH, total alkalinity of raw juice and lime salts of thin juice (incl S. D.).

Lime Level (% as CaO)	Temp (°C)	Contact Time (min)	Sucrose Conc. (°Brix)	pH	Total Alk. (as % CaO)	Lime Salts (% on solids)
0	4	0	11.1	6.4	---	0.110 (0.03)
2	4	15	13.0	11.1	0.18 (0.0)	0.413 (0.08)
4	4	15	13.7	11.5	0.45 (0.02)	0.535 (0.06)
0	22	0	13.2	6.2	----	0.120 (0.03)
2	22	15	13.8	11.1	0.17 (0.02)	0.331 (0.08)
4	22	15	15.0	11.5	0.40 (0.06)	0.367 (0.02)
0	22	20	13.1	6.4	----	0.110 (0.02)
1	22	20	12.4	9.9	0.03 (0.01)	0.439 (0.10)
2	22	20	12.4	11.4	0.23 (0.02)	0.441 (0.01)
10	22	20	12.4	11.6	0.92 (0.0)	0.516 (0.01)
0	33	0	14.7	6.6	----	0.240 (0.02)
2	33	15	13.8	11.1	0.17 (0.02)	0.385 (0.09)
4	33	15	14.5	11.5	0.35 (0.05)	0.396 (0.07)

No significant effect of treatment temperature, contact time or form of lime used was observed on Brix or pH, except that juices from beets treated with dry lime were significantly (99% level) higher in Brix than juices from beets treated with slurried lime. No correlation could be found between total alkalinity and either treatment temperature or contact time.

Apparent Purity. Results of apparent purity determinations for both dry and slurry liming experiments, as summarized in Table 4, show that in general the aparent purities of juices from limed cossettes are lower than those of control juices. There was considerable variability from sample to sample, and reported results are averages from several determinations. The only juice from limed cossettes with markedly higher apparent purity than its corresponding control was that from cossettes treated with

4% lime slurry at 22°C.

Since methanol and acetate produced as byproducts of the reaction between lime and pectin during the liming process would probably remain in the juice during purification, this would contribute to the lowered apparent purities of cossette-limed juice compared to control juice. Table 5 shows that cossette-limed diffusion juices do have higher methanol and acetate ion contents than do the control samples. Diffusion juice from cossettes treated with 0.5% lime (as CaO) applied dry had 22.09 or 23.56 millimoles of methanol compared with the control which had 2.38 millimoles. Also, limed samples had 42.09 or 46.09 millimoles acetate ion (expressed as acetic acid) compared with the control which had 7.94 millimoles.

Table 4. Apparent purity of cossettes and limed juices (incl S.D.).

Treatment Conditions			Apparent Purity		
	Lime Level (% as CaO)	Temperature (°C)	Cossettes (%)	Raw Juice (%)	Thin Juice (%)
Dry Lime	0	22	81.6 (1.8)	88.9 (1.2)	94.3 (0.9)
	0.5	22	91.5 (2.2)	89.4 (1.0)	91.5 (0.4)
	1.5	22	89.8 (1.9)	77.0 (1.0)	88.6 (1.6)
Slurry Lime in Thin Juice	0	4	85.7 (5.5)	90.1 (1.0)	94.9 (1.6)
	2	4	87.7 (2.5)	88.5 (1.7)	92.9 (1.8)
	4	4	87.5 (3.0)	75.5 (0.7)	89.3 (1.8)
	0	22	85.7 (1.8)	84.0 (1.3)	92.3 (1.5)
	2	22	92.6 (0.1)	80.0 (0.7)	89.4 (2.0)
	4	22	90.5 (2.2)	83.6 (1.0)	96.5 (2.0)

1 Cossette Purities were determined before liming.
2 Additional 0.5% Ca(OH)2 added to limed juices in purification procedure.

On the basis of only one juice sample for cossette treated with slurried lime (average of five determinations), it appears that this treatment might produce juices with lower methanol and acetate impurities than juices from cossettes treated with dry lime. Since slurry liming was carried out by dipping cossettes into a slurry of $Ca(OH)_2$ in thin juice, it would be reasonable to assume that there would be some leaching of acetate, or other constituents, from the cossettes into the slurry. Analysis of the slurry dip solutions indicated this to be correct (results not shown). Valeric acid was also detected, but no correlation with specific treatment was observed.

Camirand et al. (6) mentioned that slurry liming of cossettes would be favored over dry liming because of relative ease of operation, minimized cossette damage, and the good potential for using available equipment such as Oliver Morton or Robert batteries, after slight modification.

Table 5. Methanol and acetic acid impurities of control and limed diffusion juices.* (incl S.D.).

	Lime Level (% as CaO)	Methanol (Mmoles) Control	Methanol (Mmoles) Limed	Acetic Acid (Mmoles) Control	Acetic Acid (Mmoles) Limed
Dry Lime	0.5	2.38(1.8)	22.1(6.1)	7.94(1.7)	42.1(14.7)
	0.5	2.38(1.8)	23.6(4.0)	7.94(1.7)	46.1(10.9)
	1.0	1.87(0.5)	19.3(0.1)	6.72(0.1)	54.0(0.1)
Slurry Lime	4.0	6.13(3.3)	20.7(5.6)	11.0(2.2)	37.6(5.9)

*All results are averages of five or more determinations.

Energy Reduction in Juice Production and Processing. There are several energy consuming areas of the beet-sugar production scheme where cossette liming has the potential to reduce energy consumption. One of these areas is juice heating. In current sugar beet processing, all sugar-containing streams must be heated to 70°C or higher to prevent microbial attack on the sucrose. However, increasing the pH by liming of beet tissue plasmolyzes the beet cells (18) and kills most or all microorganisms. The high pH could effectively prevent microbiological activity and allow extraction of sugar and processing of juice at lower than current temperatures. Of course, sugar extraction at lower temperatures would necessitate greater residence time of cossettes in the extractor and thus larger extractors.

Since liming plasmolyzes beet cells, there is another possible approach to sugar removal which would result in reduction of energy. Early experiments with ground beet tissue showed that beet juice flowed freely after liming. This might be expected, since liming plasmolyzes the cell, inactivating the semipermeable membrane of the cell wall which retains the liquid in the cells. It is much easier to press juice from limed ground beets than from unlimed ground beets, for the same reason (14). Up to 90% of the juice can be expressed from limed tissue, compared to no more than 60% from unlimed ground tissue. Theoretically, energy could be saved by grindng and liming beets, pressing at ambient temperature to remove most of the juice, then perhaps extracting the remaining 10% of the sugar with either cold or hot water.

Since raw juice from limed cossettes is very light in color, with little colloidal material, it may be possible to feed raw juice, after settling of particulates, directly to ion exchange or membrane separation systems for purification and concentration to thick juice. This would completely eliminate the first and second carbonation steps, resulting in significant energy reduction. Also, membrane concentration is less energy intensive than multieffect evaporation. If this approach were successful, it would fit in perfectly with cold processing of beet juice or with expression of beet juice, as well as with hot diffusion.

CONCLUSION AND SUMMARY

The addition of lime to sugar beet cossettes results in energy saving in several processing steps, including reduction of overall lime consumption and decreased thermal energy requirements in pulp drying (14). Beet liming also increases total pulp solids because pectin is retained in the pulp instead of decomposing and leaching into the juice (6).

The diffusion juice from limed beets is clear, light and straw-colored compared to the grayish-black and turbid diffusion juice from control beets. The color of limed juice was 12.2 to 20.2 ICUMSA units instead of 220 or higher for control diffusion juice. In addition to the superior color, limed diffusion juice contained less colloidal material. Therefore, it may be possible to feed raw juice, after settling of particulates, directly to ion exchange or reverse osmosis units, this would completely eliminate the first and second carbonation steps, resulting in further process energy reduction.

Limed diffusion juice could be purified without additional lime. However, slightly increased purities could be obtained with the addition of 0.25-0.5% lime on juice. Purities of limed and unlimed thin juices were comparable, although normally the purities of thin juice from limed beets were slightly lower, presumably because of the methanol and acetate impurities generated by the liming reactions. These impurities remain in the juice even after the normal purification steps. However, methanol should volatilize during concentration, and the deleterious effect of acetate may be small because of its negative melassegenic factor.

Other experimental variables, like method of lime application (dry or slurry), treatment temperature, or contact time, had little effect on Brix, pH, or lime salts in the final juice.

ACKNOWLEDGMENTS

This project was partially funded by the Department of Energy under Interagency Contract Number EM-78-I-01-5182.

The authors wish to thank Gary McDonald for analytical work, Alex Hill for laboratory assistance, Spreckels Sugar Co., Woodland, California, for supplying sugar beets and other assistance, and the Beet Sugar Development Foundation and Jim Fischer for loan of equipment and for general support.

Reference to a company and/or product named by the De-

partment is only for purpose of information and does not imply approval or recommendation of the product to the exclusion of others which may also be suitable.

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