

A New Continuous Steffen Process

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Introduction

Recent drives towards increased efficiency in extracting sugar from sugar beets has also involved the Steffen process. Main efforts have been directed towards the development of a continuous process which meets the basic requirements for the Steffen reaction. These are as follows:

1. Suitable lime quality.
2. Intimate contact between the CaO and the sucrose while avoiding slaking of the active CaO
3. Provisions for cooling.
4. Avoidance of excessive agitation and beating which leads to obnoxious foaming, particle reduction, and filtration difficulties.

In addition to these points, there are a host of desirable refinements such as:

- a. Highest possible nonsugar concentration. This is to minimize energy cost when concentrating final waste.
- b. Improvements in saccharate cake purity to minimize nonsugar recycle to carbonation.

Objective

The importance of minimizing Steffen nonsugar recycle to carbonation cannot be overemphasized. Nonsugars recycled with the so-called saccharate cake are the most noxious types and add considerably to the color and limesalt burden in this juice. Their removal in carbonation is at best only very limited.

The impact of nonsugar cycle via saccharate cake is actually much more serious than reflected by the conventional methods for cake purity determination. The high available CaO/cake nonsugar ratio in the cake allows for the absorption of about 1/3 of the nonsugars on the highly active calcium carbonate formed during the gassing procedure of the CO₂ gassing method. In actual factory operation, however, the total nonsugar load introduced with the raw juice reduces the available CaO/nonsugar ratio to only about 10% of that prevailing for the saccharate cake (1)².

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²Numbers in parentheses refer to literature cited.

A material balance over a five-year period between two adjacent factories, one a Steffen house, the other a straight house, may illustrate the impact of nonsugar carry-over from the Steffen house. It is realized that such a comparison is a risky business at best, even for factories located in close proximity.

Table 1.—Comparison of nonsugar balance. (All values % on Beets.)

		Steffen House			
Av. CaO	Non-Sugars				
	Raw Juice	Steffen Cake	Total In	Mol.	Δ
3.22	2.14	0.46	2.6	2.27	.33

Straight House			
Av. CaO	Non-sugars		
	Raw Juice	Mol.	Δ
2.27	2.28	1.85	.43

The evidence shows a larger non-sugar elimination through carbonation for the straight house even though it operated at only 70% of the available CaO that was used in the Steffen house. In a number of extensive laboratory studies originating in 1964 with detailed material balance around each system and operating under controlled conditions with identical raw materials, it became evident that the saccharate cake nonsugars are a liability in carbonation. At equal low available CaO on raw juice nonsugars (< 1.0), the straight house always exhibited a higher non-sugar elimination. Only at the higher available CaO on raw juice nonsugars (> 1.0), was there any indication of some elimination of saccharate cake nonsugars. Tables 2, 3, and 4 show the results for some of these tests.

Limesalts and color in thin juice were always higher with the Steffen house regardless of the average CaO/raw juice nonsugar ratio. Likewise, thin juice purities were always substantially lower for the Steffen house.

Improved non-sugar elimination in the Steffen process is therefore of paramount importance for any process modification.

Some understanding about the reaction mechanism is required to achieve this end. Unfortunately, very little is actually known about the true reaction in the Steffen process.

There is, however, substantial evidence to indicate a two-stage reaction. For the first phase, any type of lime will do as long as it is

Table 2.—Laboratory comparison for nonsugar elimination using milk of lime and saccharate milk. Average values after 18 comparisons with variegated CaO/raw juice nonsugar.

A. Starting Purity:

	Raw Juice	Saccharate Cake	
		CO ₂ Gassing Method	(NH ₄) ₂ CO ₃ Method
Apparent Purity	87.82	92.61	85.77
True Purity	85.36	86.82	80.58

B. Final Purities of Thin Juice:

	Straight House	Steffen House
Apparent Purity	91.23	90.9
True Purity	90.32	89.46
Color Index at 420	2041	2541
Limesalts	0.13	0.27
Available CaO/Raw Juice		
Nonsugars (Via TP) (0.59-1.07)	0.74	0.81

C. Nonsugars Introduced (% on Raw Juice Nonsugars):

	Raw Juice Straight House	Steffen House Raw Juice	Steffen House Saccharate Cake As Determined By:	
			CO ₂ Gassing Method	(NH ₄) ₂ CO ₃ Method
Apparent Purity	100	100	5.57	12.15
True Purity	100	100	8.71	14.04

D. Nonsugars Eliminated (% of Total Introduced):

	Straight House	Steffen House	
		CO ₂ Gassing Method	(NH ₄) ₂ CO ₃ Method
Apparent Purity	30.6	24.8	29.3
True Purity	37.6	29.9	33.3

E. Total Eliminated Nonsugars as % of Nonsugars Removed in Straight House:

Apparent Purity	100	85.6	107.4
True Purity	100	86.4	101.0

F. Percent of Removed Nonsugars Credited to Saccharate Cake:

	CO ₂ Gassing Method	(NH ₄) ₂ CO ₃ Method
Apparent Purity	-79.3	18.6
True Purity	-58.5	2.7

active. Even hydrated lime will suffice. This reaction phase will not produce an insoluble calcium/oxide/saccharate complex in the cold.

A prerequisite for the second phase of the Steffen reaction to precipitate an insoluble CaO complex in the cold is a properly burned lime.

The next condition requires that the properly burned lime be finely ground. Other conditions pertain to the limerock itself.

Hence, it is this second phase reaction which is the critical determinate for the so-called Steffen process.

Material balances carried out around the system and using various means of cake decomposition established that the CaO/saccharate complex carries the bound nonsugars primarily in the form of their calcium salts. This CaO/saccharate complex has apparently a high affinity for these calcium salts at the prevailing high alkalinities of the Steffen reaction.

The data indicate that the carbonation of raw juice in the presence of saccharate cake nonsugars does remove slightly more nonsugars than the straight house if cake nonsugars are accounted for by the $(\text{NH}_4)_2\text{CO}_3$ method. If saccharate cake nonsugars are accounted for by the CO_2 gassing method, however, then the total nonsugar removal is substantially less than for the equivalent straight house. Slightly more available CaO was used on the average for the Steffen house in this series.

A second series of tests consisting of 50 sets of comparisons was carried out with equal amounts of available CaO/raw juice nonsugars for each comparison, although variations in available CaO/raw juice nonsugars were used with different comparisons (range 0.42 - 1.92).

The average for these tests and their respective evaluation is shown in Table 3.

It will be noticed again that nonsugar removal is higher for the Steffen operation when the accounting of saccharate cake nonsugars is done by the ammonium-carbonate method but lower with a material balance via the CO_2 gassing method than the respective straight house. An increased rise in nonsugar removal for the Steffen system over the straight house is also noticeable for the higher ratio of available CaO/raw juice nonsugars in Table 3 when compared with the data in Table 2.

In a separate laboratory test, raw juice was treated as for Straight house and Steffen house purification to obtain thin juice

Table 3.—Laboratory comparison for nonsugar elimination using milk of lime and saccharate milk. Average of 50 sets of comparison equal CaO/raw juice nonsugars within each comparison but variations between sets.

A. Starting Purity:

	Range	Raw Juice	Saccharate Cake	
			CO ₂ Gassing Method	(NH ₄) ₂ CO ₃ Method
Apparent Purity		87.81		
True Purity		85.00		
% Cake Sug./R.J. Sugar (AP)				12.5
% Cake Sug./R.J. Sugar (TP)				11.9
% Cake N.S./R.J. N.S. (AP)			6.9	14.2
% Cake N.S./R.J. N.S. (TP)			10.1	17.3
Avail. CaO/R.J. N.S. (TP)	(0.42-1.52)	0.91		0.91
Avail. CaO/R.J. N.S. (AP)	(0.61-1.92)	1.11		1.11

B. Thin Juice:

Apparent Purity		91.63		91.4
True Purity		90.26		89.5

C. Nonsugars Introduced as % on Raw Juice N.S.:

	Raw Juice Straight House	Raw Juice Steffen House	Steffen House Saccharate Cake CO ₂ Gassing Method	(NH ₄) ₂ CO ₃ Method
Apparent Purity	100	100	6.9	14.2
True Purity	100	100	10.1	17.3

D. Nonsugars Eliminated % of Total Introduced:

	Straight House	Steffen House CO ₂ Gassing Method	(NH ₄) ₂ CO ₃ Method
Apparent Purity	34.2	28.3	32.2
True Purity	38.9	31.3	35.9

E. Total Eliminated Nonsugars as % Nonsugars Removed in Straight House:

Apparent Purity	100	88.4	107.7
True Purity	100	88.7	108.4

F. Percent of Removed Nonsugars Credited to Saccharate Cake:

	CO ₂ Gassing Method	(NH ₄) ₂ CO ₃ Method
Apparent Purity	-57.5	18.5
True Purity	-43.5	18.9

under equal available CaO/raw juice nonsugars of 0.75. An aliquot portion from the saccharate cake sample used for carbonation with raw juice was also decomposed via ammonium carbonate. The final filtrate from the ammonium carbonate decomposition after steam distillation was added to straight house thin juice to represent the same ratio of available CaO/raw juice nonsugars.

Table 4 details the resulting purity comparison.

Table 4.—Comparison of thin juice purity from Straight House, Steffen House, and synthetic Steffen House techniques at equal available CaO/raw juice nonsugars (via true purity).

	Straight House		Steffen House	
	I	II	Normal	Synthetic
Apparent Purity	91.03	91.15	90.84	90.91
True Purity	90.49	90.72	89.89	89.63
Limesalts	0.131	0.123	0.278	
Color at 420	1867	1799	2689	3882

In this test there seems to be no difference in the purities of Steffen thin juice whether prepared by carbonation of the cake in the presence of raw juice or whether by decomposing the cake via ammonium carbonate independently and adding the final filtrate to thin juice prepared in the normal straight house fashion. However, carbonation of saccharate cake in the presence of raw juice does show an improved color over the synthetic Steffen thin juice. But color and limesalts of normal Steffen thin juice are vastly inferior when compared against normal straight house thin juice.

It was reasoned that this type of affinity would express itself in surface adsorption of these calcium salts on the CaO/saccharate complex where this complex functions as a substratum. Hence, surface area could be a controlling factor.

Logically, surface area, hence, surface adsorption, would decrease with increasing particle size. It was further reasoned that reduced shear and agitation would favor the formation of large particles.

Large particles would, of course, also improve filtration and washing. Insufficient displacement of mother liquor is the most obvious source of nonsugar carry-over.

Results and Discussion

These deductions led to the first objective for process modification which is as follows: Reduce or eliminate agitation and shear. The recently developed RT process is also a move in that direction(2). This objective demanded an answer to the problem of obtaining an intimate contact between the powdered lime and the diluted molasses without agitation. Furthermore, the contact must

be instantaneous to avoid hydration of the lime. This was an impossible task with powdered lime.

A wet lime slurry or paste in an inert liquid carrier may, however, be a suitable alternative. Wet grinding of burned lime would likely be far more efficient than dry grinding.

Furthermore, if a suspension or paste of finely ground lime in an inert liquid carrier could be produced, it is a simple matter of metering continuously both components.

Instantaneous in-lime mixing with a motionless static mixer is then assured.

Such a technique would eliminate vigorous agitation which is harmful to efficient filtration and cake washing. It also improves lime utilization because of the improved grinding efficiency and reduces potential slaking, hence, generation of exothermic heat during the second reaction phase, the latter because the active CaO is encapsulated in an inert carrier until it is combined with the soluble dicalcium saccharate.

A series of experiments were carried out to test the applicability of wet grinding lime and its use in the Steffen process. It was found that a large variety of organic liquids were suitable carriers. Simple mixing of the resultant lime suspension with a molasses solution gave efficient precipitation of the CaO/sucrose complexes.

Filtration coefficients were on the average substantially lower when using the lime suspension than with powdered lime.

Cake purities averaged nine points higher for a series of nine tests using mineral oil as the lime carrier when compared against powdered lime coolers. The respective correlation coefficient was 0.94. Hence, the results were not by chance.

Isopropanol was used in subsequent tests for grinding crushed lime in a one gallon laboratory ball mill. Figure 1 correlates grinding time to residual sugar in the cold waste. One hour grinding time appears to be sufficient.

Figure 2 relates the effect of liquid carrier to lime ratio on residual sugar in cold waste. This data applies to the conditions of the laboratory tests only. Larger commercial-sized ball mills may be optimized at a lower ratio. The minimum ratio is dependent upon desired particle size, milling equipment, and the pasting qualities of the carrier. The pasting point is that ratio at which each CaO particle is encased in the liquid carrier. This point is probably below a ratio of 0.2. As revealed earlier, only part of the lime needs to be ground.

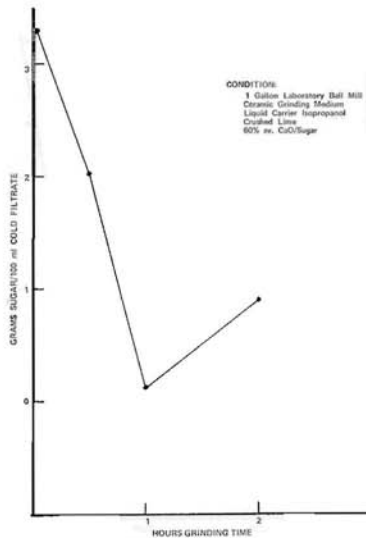


Figure 1.—Effect of grinding time on sugar losses.

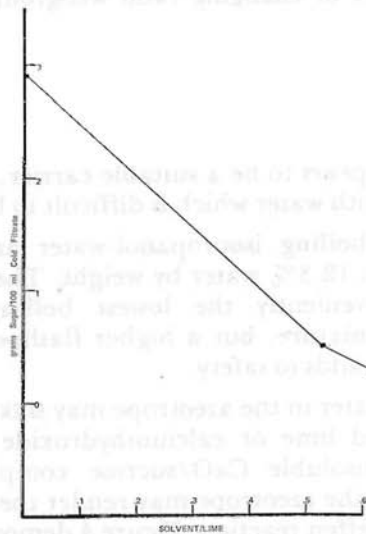


Figure 2.—Effect of solvent/lime on completeness of CaO/sucrose complex precipitation 60% CaO/sugar.

Figure 3 demonstrates the effect of changing ratios between MOL and wet ground CaO on the residual sugar in cold waste. The breakpoint is about 1:1 using 80% CaO on sugar.

The wet ground lime process is only of practical interest if the liquid carrier can be economically recovered for reuse. Separation

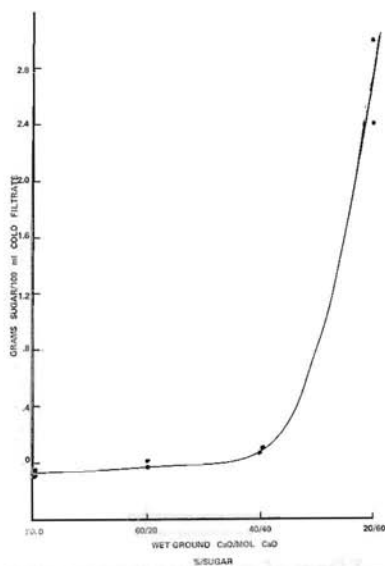


Figure 3.—Effect of changing ratio wet-ground CaO/MOL on sugar in cold filtrate.

is possible either by distillation and condensation or decantation for water immiscible liquids. Because of the hot process, distillation is compatible for solvents having a boiling point below 90°C.

Isopropanol appears to be a suitable carrier. However, it does form an azeotrope with water which is difficult to break.

The constant boiling isopropanol-water azeotrope boils at 80.4°C and contains 12.3% water by weight. The constant boiling azeotrope has conveniently the lowest boiling point of any water/isopropanol mixture, but a higher flashpoint than the absolute alcohol which adds to safety.

However, the water in the azeotrope may slake the lime during the grinding. Slaked lime or calciumhydroxide is ineffective in precipitating the insoluble CaO/sucrose complex. Hence, the presence of water in the azeotrope may render the wet ground lime unsuitable for the Steffen reaction. Figure 4 demonstrates the effect of water in isopropanol on residual sugar in cold waste. It confirms that Steffen lime has insufficient affinity for water to break the azeotrope.

The preliminary tests lead to the assembly of a continuous process outlined in Figure 5. It consisted of:

- a. Molasses dilution tank
- b. Precooler

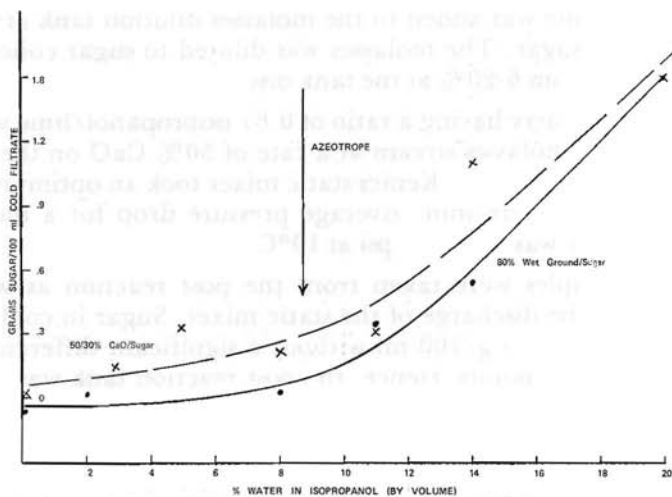


Figure 4.—Effect of water in IPA on sugar in cold filtrate.

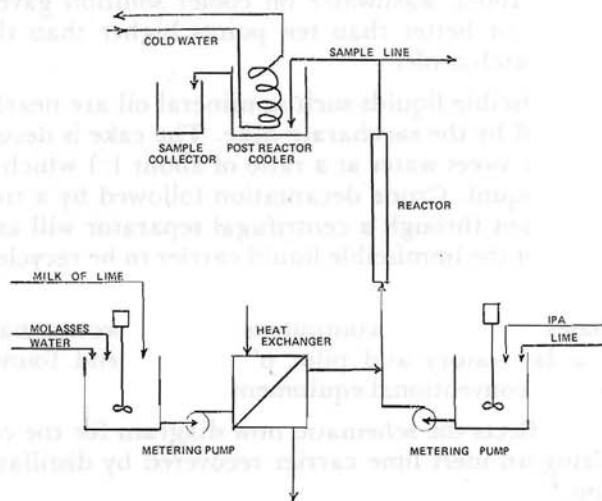


Figure 5.—Schematic flow diagram for continuous Steffen process bench model.

- c. Lime slurry tank
- d. Reactor
- e. Post cooler

Milk of lime was added to the molasses dilution tank at a rate of 30% CaO/sugar. The molasses was diluted to sugar concentrations ranging from 6-20% at the tank discharge.

The lime slurry having a ratio of 0.67 isopropanol/lime was injected into the molasses stream at a rate of 50% CaO on the sugar in the molasses. The 1/2" Kenics static mixer took an optimum flow rate of about 4 liters/min. Average pressure drop for a molasses with 10% sugar was about 25 psi at 10°C.

Grab samples were taken from the post reaction as well as directly from the discharge of the static mixer. Sugar in cold waste averaged about 0.25 g/100 ml without a significant difference between the sample points. Hence, the post reaction tank was not required. However, substantial heat was generated in the post reaction tank reflecting a delayed slaking after the CaO/sucrose complex had formed.

Cold filtrate RDS averaged 7.45°C. With 50% washwater on cooler solution, cake purities by the CO₂ gassing method averaged about 92.5% with a washwater RDS of 6.5.

The use of 100% washwater on cooler solution gave a cake purity of 96.6% or better than ten points higher than the equivalent dry lime batch cooler.

Water-immiscible liquids such as mineral oil are nearly quantitatively retained by the saccharate cake. The cake is decomposed with thin juice or sweet water at a ratio of about 1:1 which releases the immiscible liquid. Crude decantation followed by a treatment of the super-natant through a centrifugal separator will assure required refining of the immiscible liquid carrier to be recycled to the lime wet grinding process.

All phases of this new continuous Steffen process have been studied on a laboratory and pilot plant scale and found to be compatible with conventional equipment.

Figure 6 reflects the schematic flow diagram for the complete system utilizing an inert lime carrier recovered by distillation and condensation.*

Summary

It appears that the new process combines all the advantages demanded for a continuous Steffen process.

Its advantages over the conventional system may be summarized as follows:

1. Wet grinding of the lime
2. Uniform particle size

*Patents applied for.

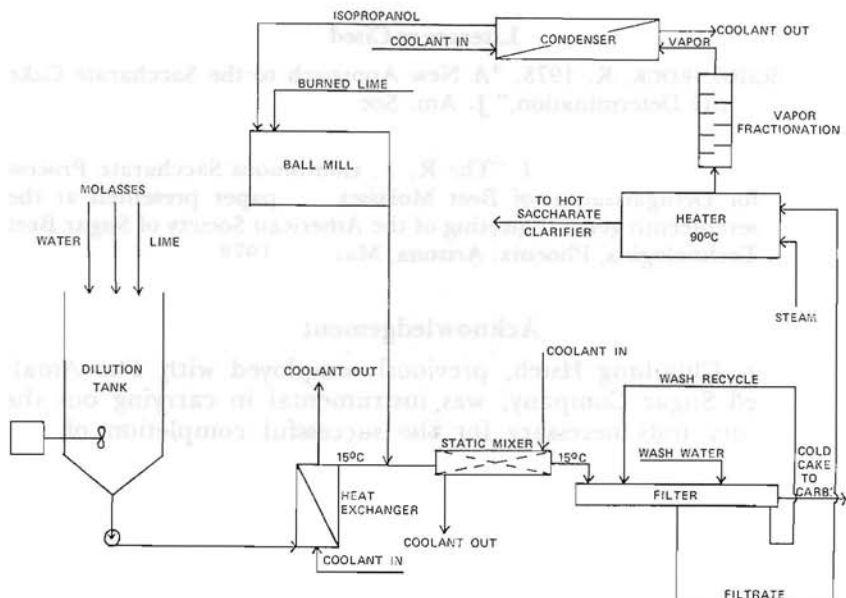


Figure 6.—Continuous calcium-saccharate process.

3. Smallest possible CaO particle
4. Elimination of lime dust
5. Elimination of partial air slaking
6. Possibility of storing the lime suspension
7. Efficient recovery of the inert liquid carrier
8. Grinding < 50% av. CaO/sugar
9. Reduced mechanical wear and tear
10. Convenient metering of the lime slurry
11. Truly continuous operation
12. Instantaneous in-line reaction
13. Elimination of violent agitation
14. No foam
15. Reduced cooling requirements
16. High Cake purity
17. Reduced nonsugar load
18. Improved filtration and washing
19. Increased nonsugar concentration of the Steffen waste

Literature Cited

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