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PROGRESS REPORT I

PROCESSING OF SUGAR BEETS

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ABSTRACT

This report presents the progress on this project since April 1, 1952. The research problem under investigation is described. A survey of pertinent literature is included. Some preliminary experimental work has been accomplished.

I. INTRODUCTION

The conventional batch diffusion process has been most extensively used for the extraction of sugar from sugar beets; however, it has certain limitations and disadvantages. The exploration of new methods may lead to more efficient and economical production of beet sugar to the benefit of sugar beet growers and processors, especially to those located in the general beet-growing area of Michigan and Ohio, where a considerable portion of beet sugar processing industry is faced with an extensive renovation program of plants.

A joint meeting of representatives of the Michigan beet-sugar industry and selected scientific personnel of the University of Michigan was held March 1950, to discuss problems facing the Michigan industry. At this meeting Professor J. C. Brier, Professor of Chemical Engineering, suggested some methods of improving the diffusion process, the present key to the recovery of sugar from sugar beets. Also, Professor L. E. Brownell, Associate Professor of Chemical Engineering, suggested the possibility of developing a new process by which sugar could be recovered from sugar beets without diffusion. The proposed process involved rupturing the wall of the cells of the sugar beet so that the juice could be removed by pressing, washing, filtering, or centrifuging without diffusion. The proposed method of rupturing the cells was the use of steam

followed by a sudden reduction in pressure. Representatives of the beet-sugar industry considered the ideas to have merit.

These ideas were discussed with representatives of the Bureau of Agricultural and Industrial Chemistry, U.S.D.A. It was agreed that the proposed method had sufficient potentiality to warrant laboratory scale investigation of the proposed approach. This resulted in Contract No. A-1s-33464 between the U. S. Department of Agriculture and the Engineering Research Institute of the University of Michigan. This contract specifies that sugar beets shall be processed to remove the cell contents in the following ways:

a. "Explode the cells by preheating beets under pressure, then suddenly releasing the pressure so that steam will be instantaneously developed. Results will be obtained for at least three different pressure differentials. The maximum cossette or particle size will be established at the optimum pressure differential. The cell contents will be removed from cell debris by water washes followed by centrifuging or filtration.

b. "Pretreat beet cossettes with each of the compounds calcium oxide and aluminum sulfate at three pH values between six and nine to increase the brittleness of the cell walls. Examine the effect of these pretreatments by subjecting the chemically treated cossettes to the explosion process described under part (a) of this section. The pressing characteristics of such pretreated cossettes are to be measured using a laboratory-type press such as the Carver press. [The minimum pressure required to rupture the cells as well as the recovery of juice from cossettes exploded at this pressure are to be determined. If the recovery of juice is comparable to that obtained by explosion of untreated cossettes for the same amount of energy, then water-washing followed by centrifugation or filtration will be used to determine the ease of removal of cell contents from cell debris.]

c. "Diffuse the cell contents from beet cossettes in a continuous countercurrent manner. Experiments will be conducted at two temperatures in a range from 65 to 80°C. The effect of pretreatment as described in part (b) will be measured at the more effective temperature...."

This research is estimated to require a minimum of two years. A review of the budget, personnel, and equipment required indicated that it would be inadvisable to attempt to perform research studies a, b, and c simultaneously. Therefore, the following plan as been adopted: Professor L. E. Brownell will supervise the study of the process involving cell rupture (items a and b) during the first year. It is estimated that this study will require about 60 per cent of the budget. Professor J. C. Brier will supervise the studies on continuous countercurrent diffusion during the second year.

II. REVIEW OF LITERATURE

A. Continuous Diffusion Process

The continuous diffusion process is based upon the use of a machine called the continuous diffuser, which conveys the beet cossettes and juice continuously (usually countercurrently from cell to cell and concurrently within each cell) during diffusion. The difference between one continuous diffusion process and another is primarily a difference in design of the diffuser. At present, the continuous process is the only process used commercially other than the batch diffusion process. Some of the continuous diffusion processes are described below:

1. Bergé Continuous Diffuser (1)

This machine was designed by Julien Bergé and has been used at the Raffinerie Tirlemontoise, Belgium. It consists of a large revolving drum, separated into "cells" by a helix attached to the interior surface. As the drum and its helix revolve, it moves the juice and cossettes in such a way that they travel countercurrently through the drum but concurrently within each cell. The drum revolves at a speed of 20-22 revolutions per hour.

2. Smet Diffuser (2)

This machine is a modification of Bergé diffuser, in which a double helix replaces the single helix. One complete revolution of the drum causes the juice to travel two compartments forward and cossettes one compartment backward, thus, the speed of the juices is doubled. The drum revolves at 23-25 revolutions per hour. The capacity of the diffuser is about 500-600 tons of beets per day.

3. Silver Continuous Diffusers (3)

a. Chain-type Silver Diffusers

The chain-type diffuser consists of a series of about 21 U-shaped cells approximately 2 feet wide and 12 feet deep. Each cell is set several inches higher than its predecessor so that the juice may flow by gravity through the cells. The cossettes are fed into the diffuser at one end of the lower tier and are carried by drag chains with perforated steel-plate flights through the entire length of the diffuser and finally discharged at another end.

b. Scroll-type Silver Diffuser

In the scroll-type diffuser, the cossettes are pushed through tank-type cells by large scrolls, concurrently with the juice, while the operation in countercurrent from cell to cell. The cossettes are transferred between cells by a wheel type of lift.

4. Oliver-Morton Continuous Diffuser (4,5)

This design was developed by W. V. Morton, patented by Spreckels Sugar Company and made by Oliver United Filters, Inc. It is similar to the Silver Scroll-type continuous diffuser but employs a different method of transferring cossettes from cell to cell. In each cell there is a series of six sets of fingers at the end of and attached to the scroll. As the fingers revolve they lift the cossettes from the cell and then pass through a stationary comb of grid bars, which takes the cossettes from the fingers, whence they are pushed into the next cell. The flow of cossettes is countercurrent to the juice from cell to cell but concurrent within each cell.

A 12-cell test unit was installed and operated at Spreckel's Woodland, California refinery. Each cell was 4 feet wide, 8.5 feet long and 5 feet deep, and had a round bottom. The capacity was about 500 tons of beets per day. The test results were so satisfactory that a full-scale 24-cell unit was built at Spreckel's Woodland plant and a 28-cell unit at the company's refinery at Spreckel, California.

5. Hildebrandt Continuous Diffuser (6)

This diffuser consists of two vertical cylinders of different height, separated about 4 feet from each other and usually about 50 feet high and 7 to 10 feet in diameter. The cylinders are connected at the bottom by a short third cylinder for transfer of cossettes and juice from one vertical cylinder to the other. Each of the cylinders is provided with a perforated scroll to move the cossettes at the desired rate. The cossettes and juice move countercurrently to each other.

6. Other types

There are many other types of continuous diffusers described in the literature, some of which might be worthy of further investigation, but these designs will not be discussed in this review.

The continuous diffusers possess the advantages of saving labor and providing higher sugar extraction; however, they pose a number of problems that are yet to be solved. Some of these problems are microbiological actions, corrosion, foam, and reduction of the size of the diffusers.

B. Pressing Process

1. Pressing-Washing Process

Pressing sugar beets to obtain the juice is a very old process. Frank K. Archard made his first beet sugar in 1799 by pressing grated sugar beets. The modern version of this process is, however, much more refined and efficient.

In the process described in German Patent No. 553,375 (January 9, 1930, to E. Seldis, C. A., 26, 4728, 1932), the beets are cut in the presence of warm dilute juice, then heated directly with steam, freed from the bulk of the added juice, and pressed. The pressed beets are repulped with water and pressed again. The juice from the second pressing is added to the next batch of beets in the cutting machine.

In the process described in U. S. Patent No. 2,332,062 (October 9, 1943, to T. R. Cutler, Br. C. A. B., 111, 152, 1946) the washed beets are sliced, pulped by grinding or crushing, then pressed for removal of juice. Pulping and pressing are carried out in the cold and without the addition of water. The pulpy residue is mixed with hot liquid from the third pressing at a temperature not less than 85°C, in an amount of about 40-60 per cent of the saturated mash produced and again pressed. The liquid is combined with the first-press liquid at 85°C for sugar recovery. A third pressing is given after treatment of the pulp with water at 98-100°C, and the liquid is returned to the pulp for the second pressing.

2. Pressing-Diffusion Process (7)

A combined pressing-diffusion process was practiced by certain British beet sugar factories. In this process the beet cossettes are immersed in 4-5 times their weight of juice heated to around 100°C, their temperature thus being raised to around 85°C, the mixture is passed over a screened scraper conveyor, which takes the slices to a press where about 50 per cent of the mass is expressed as juice, to be mixed later with factory diffusion juice. Then the slices are conveyed to the diffusion

battery. The juice which has passed through the screen of the scraper conveyor is returned together with a portion of diffusion juice, after heating to around 100°C, to the scalding tanks for re-use there. It is claimed that a high extraction of sugar and a small draft of about 104 are made possible by this process.

3. Pressing-Chemical Treatment Process (8)

Bonelli patented a process in 1943 to extract sugar from sugar beets by chemical treatment and pressing.

The beets are cleaned, reduced to a pulp with lime not exceeding about 0.8 per cent, and subjected to a preliminary pressing, followed by successive washings and a second pressing. Juice from the pressings is heated and treated with 0.1-0.3 per cent lime, then saturated with carbon dioxide to an alkalinity of 0.01-0.02 per cent, filtered, boiled, and centrifuged in the usual way. It is claimed that purer juice is obtained as a result of the action of the lime on the cellular tissue as well as on the juice, rendering the tissue more readily removable. In the usual process, the pectic matter, especially protopectin, during hot diffusion is hydrolyzed and is only partially removed with hot defecation or refining. Much of it passes through all stages of purification and goes into the molasses. It is claimed that the pectic material is wholly precipitated by cold lime treatment. Other impurities, such as albuminous and nitrogenous matters, remained intact but coagulated in the successive phases of saturation and defecation in cold.

Bonelli made further studies on his process in 1945 and suggested the following modifications:

(1) The amount of lime used for the preliminary defecation should be increased to give an alkalinity of 0.2 per cent in the juice.

(2) In the secondary defecation the amount of lime should not be less than 0.5 per cent, and double saturation should be used to give a more clear, colorless juice, which is easily filtered.

He claimed that this process was a fundamental advance in beet-sugar manufacture but admitted that further industrial-scale testing was required.

C. Desiccation Process

This process was suggested almost a century ago by Schuzenbach but it was never successfully carried out because of the lack of efficient drying equipment. Numerous modifications of this process have been patented since then.

1. De Vecchis Desiccation Process (9,10)

Beet slices are dried under such conditions that no appreciable invert sugar formation and caramelization occur. The temperature should be held not under 80-90°C nor over 100-105°C, and sufficient time should be allowed to coagulate the albuminous and other similar substances. It is claimed that the cell membrane of the beet cell is ruptured by heat and that the subsequent extraction is no longer an osmotic but a simple leaching process. A concentrated juice of about 50° Brix can be obtained by extracting the dried beets. The juice obtained has entirely different characteristics from those of fresh beet juice. Filtration can be accomplished without difficulties, because the solid scum formed with the addition of superphosphate gives the filter cakes a grainy structure having a high permeability.

2. Oxford Process

This process is an outgrowth of the De Vecchis process. The beet slices are dried continuously by means of hot air (93°C) to a final moisture content of about 5 per cent by weight. It is claimed that the cell membrane of the beet cell is not ruptured by heat, therefore the prolonged drying advocated by De Vecchis is unnecessary. The drying can be done in about one hour. The dry cossettes are extracted in a continuous diffuser to produce a juice of 50° Brix, which is first treated with a quantity of calcium superphosphate, warmed to 80°, and treated with excess of lime to render the juice adequately alkaline. Mechanical separation may be used to advantage prior to chemical treatment.

The following are a few of the patents relating to various modifications of the desiccation process:

(1) Brit. 274,131, July 10, 1926, to K. Cuker (C.A., 22, 2075, 1928). Materials are subjected to a gentle pressing or suction which does not injure the cell membrane and are subjected to an oxidizing current of gas which dries the cells and concentrates their juice, thus raising the osmotic pressure and accelerating the diffusion process.

(2) Brit. 293,066, Feb. 23, 1927, to R. G. Farnell (C.A., 23, 1523, 1929). Dried beet cossettes are extracted with water at a temperature below 100°C, preferably, 50-60°C. Pressure may be varied during the extraction.

(3) Brit. 294,520, Feb. 23, 1928, to R. G. Farnell (Br. C.A.B., 832, 1928). Beet cossettes are dried at 100°C or less at atmospheric pressure and the

pressure is suddenly reduced to facilitate the breaking down of the structure of beets.

(4) Brit. 329,112, Apr. 11, 1929, to I. Tishchenko (C.A., 24, 553, 1930). Raw beet shavings are treated with alkali to neutralize the acids contained in the gases used for subsequent drying. Juice obtained is more readily purified.

The desiccation process has been investigated by several factories, including the Sanguinetto plant in Italy, Eynsham plant in England, and the experimental factory of the Russian Sugar Trust at Derjugin, Russia. These factories used the process for years, and the process was critically examined by independent experts for a number of sugar-beet-growing countries.

The conclusions reached by various experts after examining the desiccation process were (13, 14, 15):

1. Drying was successfully accomplished without appreciable inversion and discoloration.

2. High fuel consumption prevented the process from being commercially feasible.

3. The process of making sugar from dried beets had not been improved to a point where a first-class product could be produced economically.

III. DISCUSSIONS OF SOME PROCESS LIMITATIONS

Microscopically, the sugar beet is made of elongated cells approximately 0.07 mm long and 0.02 mm in diameter, running lengthwise. The cell wall is covered with a semipermeable protoplasmic membrane which in the living cell has the function of regulating the fluid flow into and out of the cell in order to maintain the life of the protoplasm. The juice cannot pass through the cell wall of the living cell, since the protoplasmic membrane is almost completely impermeable to the juice. Therefore, the first step in the recovery of the juice is to destroy the membrane. The method commonly used for this purpose is heating the cossettes to a temperature about 60°C. At this temperature the protoplasmic membrane shrinks and comes off the cell wall. The cell wall is very thin and permeable, and juice contained within the cell can pass through it to other cells and to intercellular spaces. In this way the cell wall loses its semipermeable characteristics and becomes a permeable

membrane through which the exchange of matter takes place directly by diffusion. From the technological point of view it is important to know the change in the composition of the diffusion juice as compared with the composition of cossettes. According to the experiments carried out in six sugar factories by the Sugar Research Institute in Prague (16), the following amounts of some more important nonsugars passed from the cossettes to the diffusion juice in the usual diffusion process:

K_2O, Na_2O, SO_3, Cl----------76.9%

harmful nitrogen----------90.1%

Similar data can be obtained from the table of the composition of sugar beets and intermediate products given by Dr. H. Classen in "Die Zuckerfabrikation mit besonder Berucksichtigung des Betribes". It should be borne in mind that the composition of sugar beets is far from constant and that they contain a great variety of nonsugars for which there are often no reliable analytical methods. Nevertheless, the results obtained by the Sugar Research Institute in Prague (16) and given above concern components which are of great importance in beet-sugar production, since they cannot be removed by the usual methods of purification. They show that even in the usual diffusion process a considerable amount of nonsugars pass to the diffusion juice. If, therefore, instead of using the diffusion process, the cell were destroyed and the juice recovered by simple methods as pressing, spinning, or leaching, the quality of the juice should not be seriously affected. On the other hand, substituting this simple method of recovery for the complicated method of diffusion would probably shorten the time of recovery and enable the process to be carried out in a small, compact, continuous plant.

IV. PRELIMINARY EXPERIMENT ON EXTRACTION OF BEET SUGAR BY RUPTURING BEET CELLS

Preliminary experiments were made to determine whether or not it is possible to rupture the cell membrane of the sugar beet by a sudden reduction in pressure. An autoclave equipped with a quick-opening valve was fabricated from pipe fittings, as shown in Figure 1. A cage made of perforated stainless steel was placed in a stainless steel beaker inside the autoclave. The cage which confined the beet cossettes during explosion and the beaker are shown in Figure 2.

The sugar beets for use in these experiments had been stored at 4°C since the end of 1951 campaign. The beets were cut manually with a V-corrugated knife with vertical splitters, 46 divisions per 165 mm. The cross section of the cossettes

was rather irregular, and the length ranged from 1/4 to 3 inches, but were in the general range of 1 to 3 inches, as shown in Figure 3.

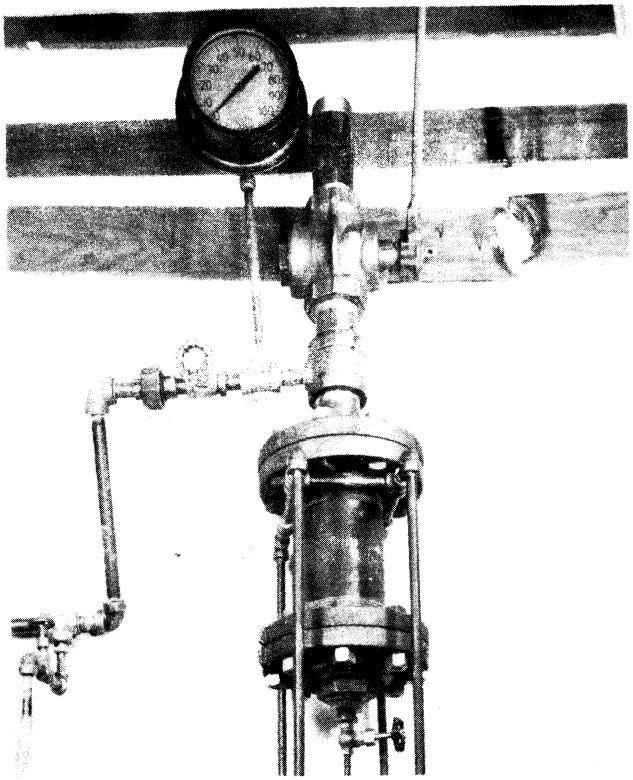

Figure 1. View of Preliminary Explosion Chamber

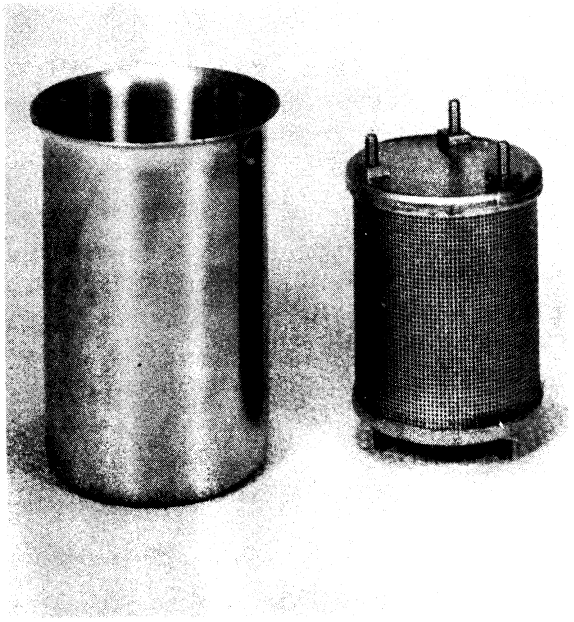

Figure 2. Stainless Steel Cossette Retainer

Figure 3. Untreated Raw Cossettes

Three series of tests were conducted.

1. Explosion of cossettes by treating under various steam pressures and suddenly releasing the pressure into atmosphere. For each run 100 gm of cossettes was used. The experimental conditions of each run are given in Table I.

TABLE I

EXPLOSION TESTS INTO ATMOSPHERIC PRESSURE

	Test Number			
Conditions of Tests	1	4	5-A	5-B
Steam pressure, psig	20	50	110	110
Time at pressure (including the time required to reach the pressure), minutes	10	10	8	4
Time loading and unloading, minutes	5	5	5	5
Time releasing the pressure to 0 psig	Less than two seconds			

Observations:

Run No. 1. The cossettes after treating were soft and retained the original shape. The juice could be easily squeezed out and had a light color. A frozen section of 30μ thickness was cut with a freezing microtome and examined (unstained) under a microscope at a magnification of 100 times. The microscopic examination showed that the cells were intact but swollen.

Run No. 4. The cossettes were similar in appearance to those from Run No. 1 except darker in color and softer. The microscopic examination indicated that very few cells were ruptured.

Run No. 5-A The cossettes were dark brown in color and pulpy and had a strong caramel odor. It is evident that this treatment is too severe.

Run No. 5-B The cossettes retained their shape, were light brown in color and had no caramel odor.

The microscopic examination showed that the cells were greatly swollen and more cells were ruptured.

2. Explosion of beet cossettes by cooking under steam pressure and suddenly releasing the steam into an evacuated vessel.

For this experiment the autoclave was connected to a vessel of about 300 times its volume. A vacuum of 22 inches of mercury was maintained in the vessel by a single-stage steam ejector. The experimental conditions of those series of experiments are given in Table II.

TABLE II

EXPLOSION TESTS INTO VACUUM

	Test Number			
Conditions of Tests	10	11-A	11-B	12
Steam pressure, psig	30	60	60	110
Time at pressure (including the time required to reach the pressure), minutes	15	10	20	7
Time, loading and unloading, minutes	5	5	5	5
Time, releasing the pressure to vacuum	Less	than	one	second
Vacuum, inches Hg	22	22	22	22

For the preparation of samples for microphotographs cossettes were fixed in Nawaschin's fluid for 24 hours, and paraffin sections of 10μ thickness were prepared employing the usual technique. For each sample two sections were stained, one with Harris Hematoxylin and the other with Light Green SF (1 per cent in 95 per cent ethanol). The former indicate cell membrane and the latter indicates substances of cellulose origin. Figures 4 and 5 are photomicrographs of untreated raw cossettes.

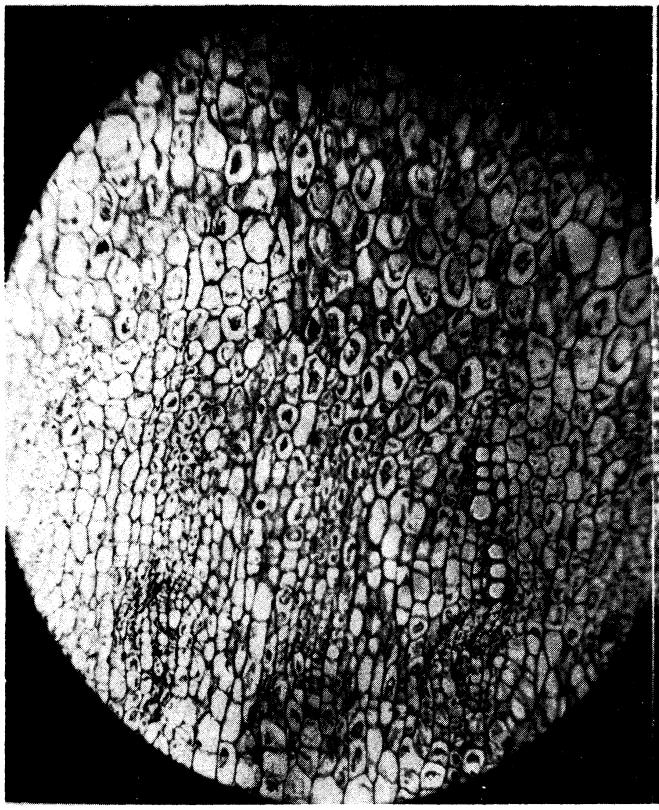

Figure 4. Cell Structure of Raw Cossettes

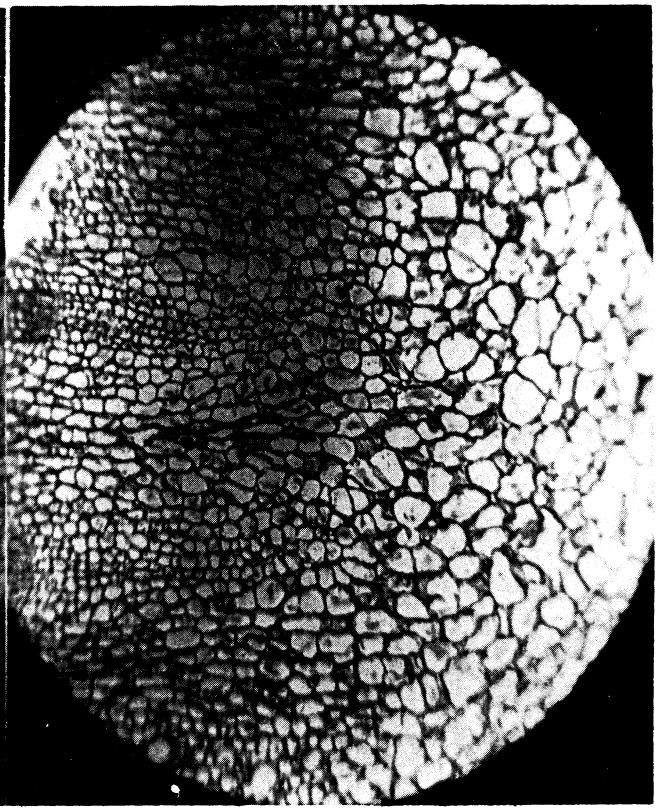

Figure 5. Cell Structure of Raw Cossettes

Observations:

Run No. 10 The treated cossettes were very soft and had a light color. See photomicrographs of Figures 6 and 7.

Run No.11-A The cossettes were similar to those of Run No. 10, but had a brown color. See photomicrographs of Figures 8 and 9.

Run No. 11-B The cossettes were mashed and had a brown color. See photomicrographs of Figures 10 and 11.

Run No. 12 The cossettes were mashed and had a dark brown color. See photomicrographs of Figures 12 and 13.

The photomicrographs show that most of the cell membrane is ruptured by this treatment; however, the explosion is not severe enough to cause the cossettes to become liquified and thereby permit the sugar content to be extracted from the debris by centrifuging. This is especially true for the runs employing low steam pressure.

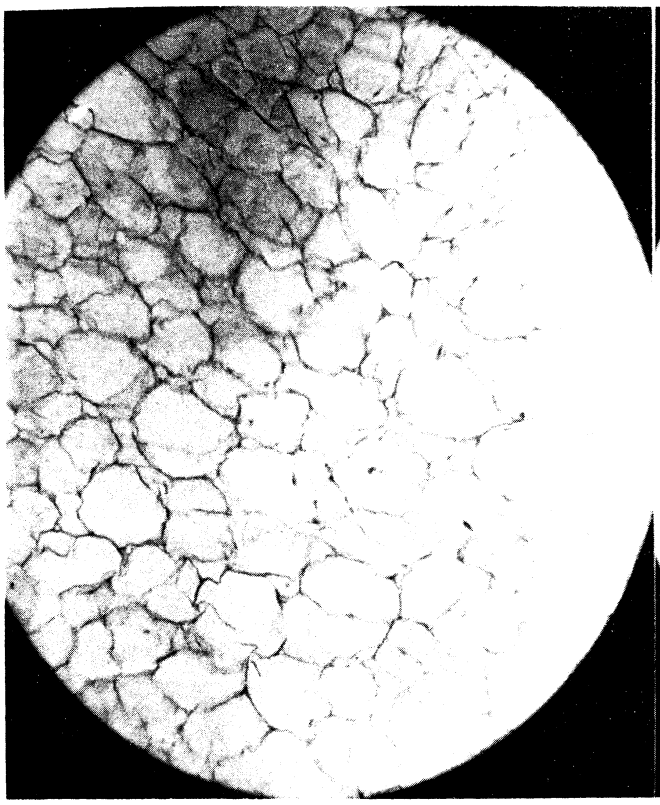

Figure 6. Cossettes Exploded from 30 psig to 22-inch Hg Vacuum (15 minutes)

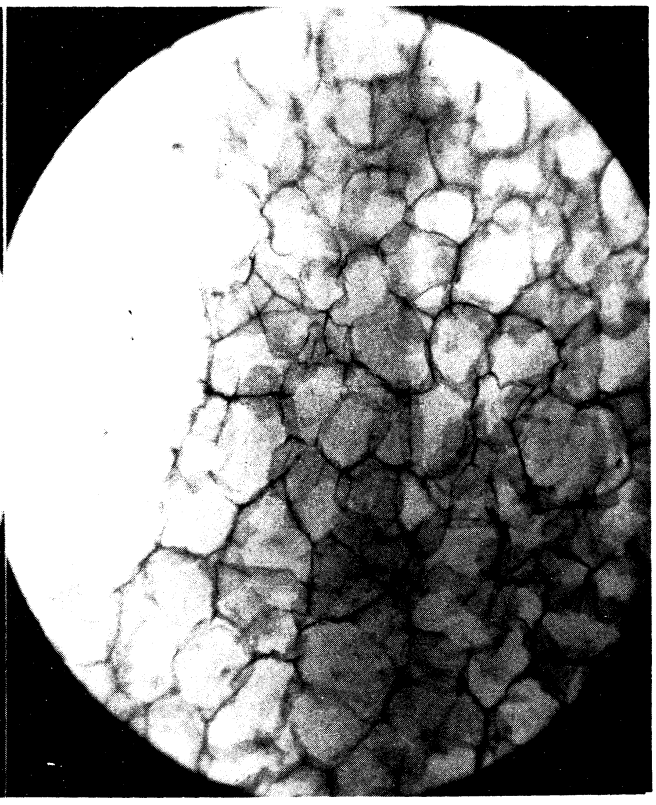

Figure 7. Cossettes Exploded from 30 psig to 22-inch Hg Vacuum (15 minutes)

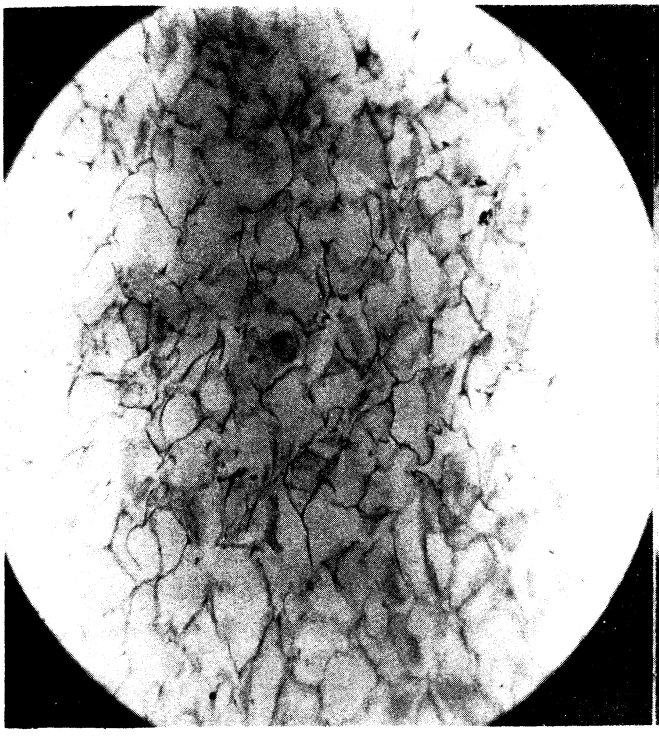

Figure 8. Cossettes Exploded from 60 psig to 22-inch Hg Vacuum (10 minutes)

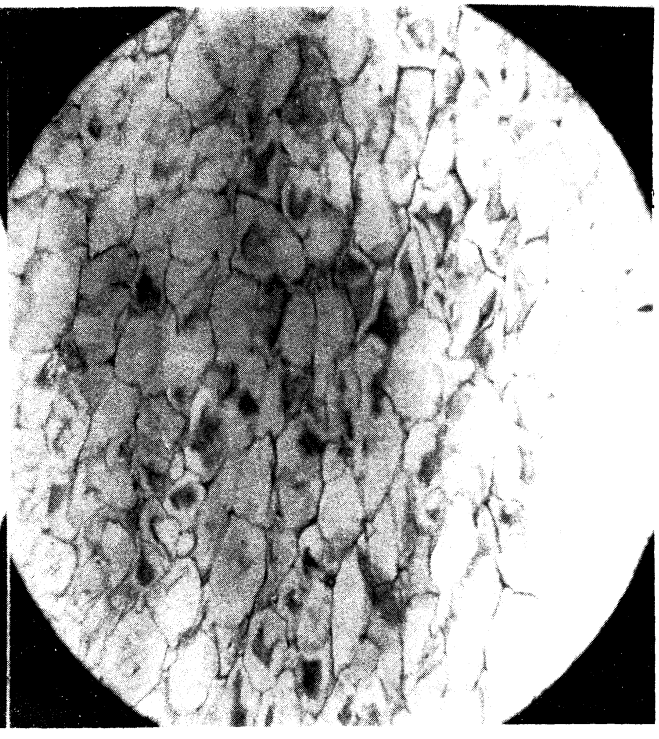

Figure 9. Cossettes Exploded from 60 psig to 22-inch Hg Vacuum (10 minutes)

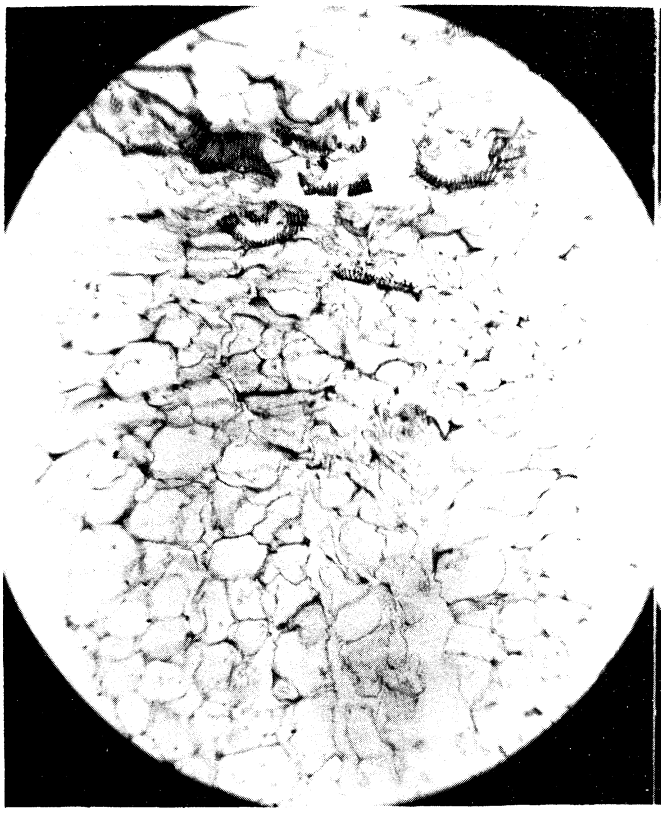

Figure 10. Cossettes Exploded from 60 psig to 22-inch Hg Vacuum (20 minutes)

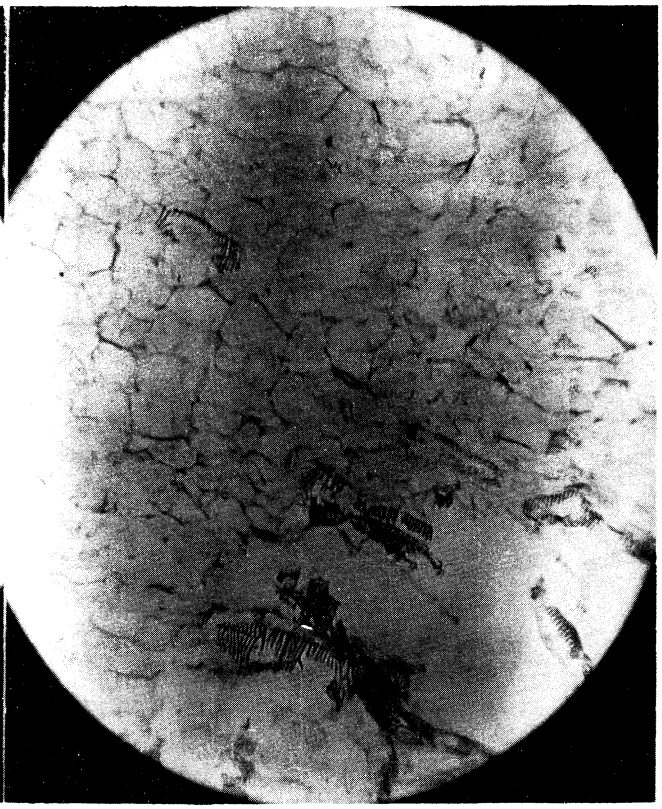

Figure 11. Cossettes Exploded from 60 psig to 22-inch Hg Vacuum (20 minutes)

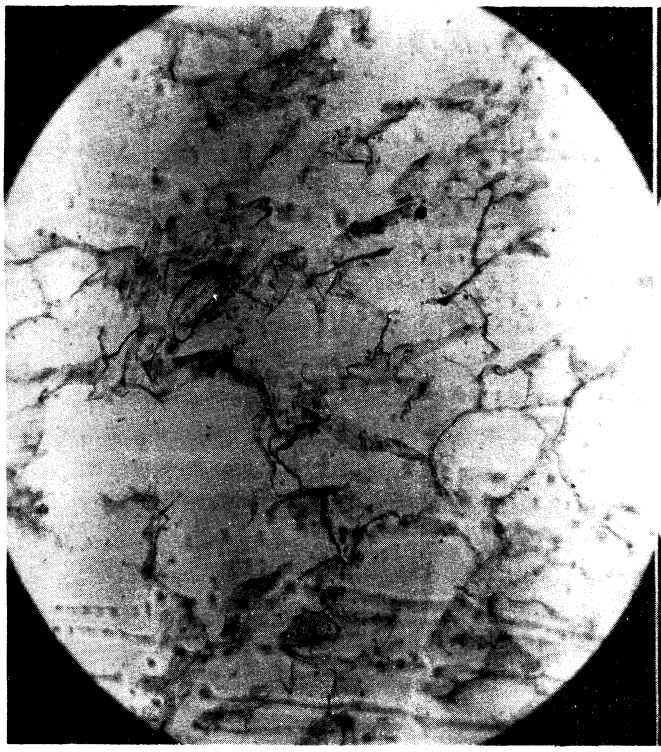

Figure 12. Cossettes Exploded from 110 psig to 22-inch Hg Vacuum (7 minutes)

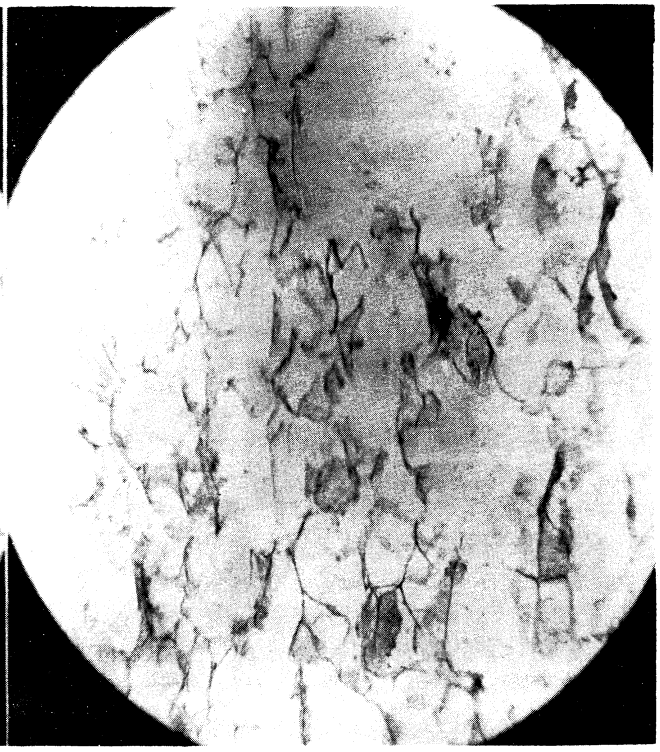

Figure 13. Cossettes Exploded from 110 psig to 22-inch Hg Vacuum (7 minutes)

3. Pressing Test

A laboratory-model Carver hydraulic press with cage filter equipment was employed.

For each run 300 gm of beet cossettes was used. In order to obtain a rough idea of per cent recovery of sugar, optimum treating pressure, and optimum extraction pressure for further investigation, the density of the juice expressed was determined by a Westphal balance, and the total soluble solid content in juice was calculated by means of sugar tables (Browne and Zerban, Physical and Chemical Methods of Sugar Analysis). There is no end point on volume of juice obtainable at each pressure. Therefore, a definite duration of pressing time was employed for each pressure increment, namely, 20 minutes for the first increment and 10 minutes for each subsequent pressure increments. Experimental data are given in Tables III, IV and V. A plot of total soluble solids in juice extracted at various pressures is shown in Figure 14.

TABLE III

VOLUME OF JUICE (cc) AT VARIOUS EXTRACTION PRESSURES

Force in lbs., $3\frac{1}{2}$" diameter plunger	Raw Beets	Treated Beets		
		10 psig, 10min. 22" vacuum	30 psig, 10min. 22" vacuum	60 psig, 10min. 22" vacuum
1,200	30	140	130	----------
2,000	--	162	147	145 @ 2500#
4,000	72	185	170	----------
8,000	90	202	183	165 @ 7200#
12,000	105	212	190	169

TABLE IV

PROPERTIES OF JUICE

Properties	Raw Beets	Treated Beets		
		10 psig, 10min. 22" vacuum	30 psig, 10min. 22" vacuum	60 psig, 10min 22" vacuum
Color	Greenish grey	Brownish grey	Brown, but more transparent	Blackish brown, caramel odor
Density	1.0560	1.0680	1.0760	1.0942
Wt. of raw beet gm	300	300	300	300
Wt. of pressed residue	180	46	35	30

TABLE V

TOTAL SOLUBLE SOLIDS IN JUICE EXTRACTED AT VARIOUS EXTRACTION PRESSURES

Force (#) on $3\frac{1}{2}$" diam plunger	Raw Beets		Treated Beets					
			10psig, 10min 22" vac		30psig, 10min 22" vac		60 psig, 10 min. 22" vac	
	gm/300gm	% on beet	gm/300 gm beet	% on beet	gm/300 gm beet	%on beet	gm/300 gm beet	% on beet
1,200	4.6	1.5	25.8	8.9	26.6	8.9	---	---
2,000	---	---	29.9	10.0	30.1	10.1	36.7 @ 2500#	12.2
4,000	11.1	3.7	34.2	11.4	34.8	11.6	---	---
8,000	12.9	4.6	37.3	12.4	37.4	12.5	41.7 @ 7200#	13.9
12,000	16.2	5.4	39.2	13.1	39.3	13.1	42.6	14.2

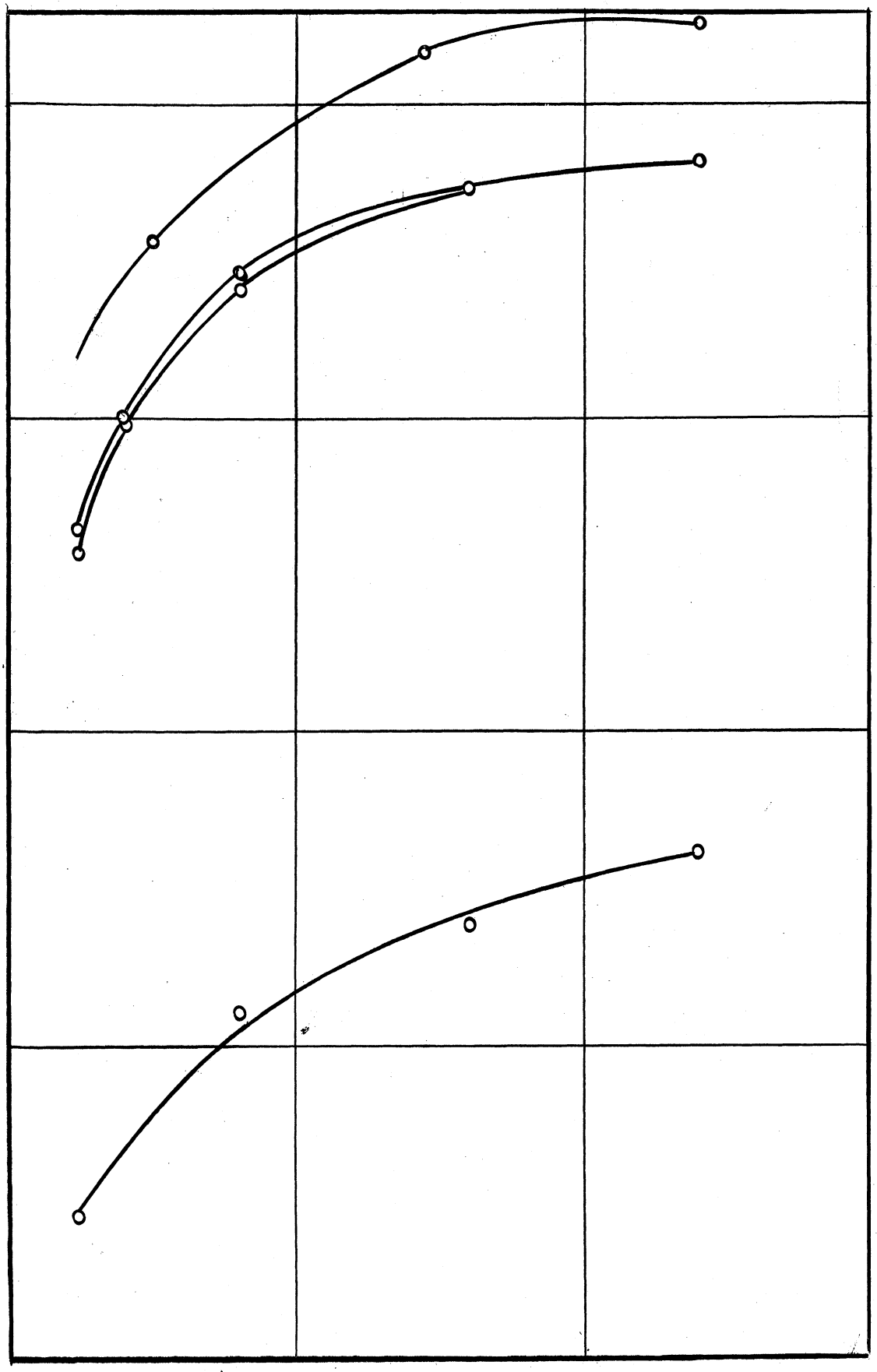

Figure 14. Total Soluble Solids in Juice Extracted At Various Pressures

V. NEXT EXPERIMENTS PLANNED

A. Preliminary Experiments

In the next few months it is planned to conduct some preliminary experiments to determine the influence of variable factors such as temperature, time of retention and pressure difference, on the destruction of the cell wall and on the apparent purity and invert sugar content of the resulting juice. The experiments will be carried out with and without an intermediate disintegration of the treated cossettes.

In these preliminary experiments it is proposed that the following experimental technique be used:

A portion of the sample of raw cossettes prepared for an explosion test will be subjected to hot digestion at a temperature of 70-80°C for about 60 minutes and the resulting juice used as a reference. Sugar, Brix an invert sugar will be determined.

One part of the cossettes after explosion will be digested in a similar way but at about 20°C and for a short time (2-5 minutes) and sugar and Brix of the juice determined. Another part of the exploded cossettes will be hot-digested in a manner analogous to that used for raw cossettes, the resulting juice polarized and Brix and invert sugar determined.

A comparison of the apparent purity and invert sugar content of the juice prepared by hot digestion of the exploded cossettes with that prepared by hot digestion from raw cossettes will give a rough idea of the change of the quality of the juice as a result of process conditions. The extent to which the cells have been opened can be determined approximately by the difference in the polarization of the juice prepared by hot and cold digestion of the exploded cossettes.

B. Experiments with Pretreatment of Cossettes

Using a similar experimental technique, the influence of pretreatment of cossettes with calcium oxide and aluminum sulfate will be investigated.

C. Experiments with Factory Diffusion Juice as a Reference

An average sample of cossettes will be taken from the factory beet slicer over a two-hour period. A portion of these

cossettes will be exploded at the optimum conditions determined in section A. The composition of the resulting juice will be compared with that of the factory diffusion juice obtained from the cossettes sampled. Determination of apparent purity, Ph, and the colloids is proposed.

The juice prepared by the explosion process and the factory diffusion juice will then be subjected to the usual purification process and the purity, color (spectrophotometric), ash (by electrical conductivity), and lime salts content of the resulting thin juices compared.

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APPENDIX

ANALYTICAL METHODS

Sugar in the Beet

If a mechanical rasp is not available, grind half of the beet length-wise with a hand grater 40 x 25 cm with openings 2 mm (approximately 150 openings per 1 dm^2). Collect the pulp in a round-bottom enameled bowl, mix well, removing at the same time coarse particles. The mixing should be done as rapidly as possible to avoid evaporation. Weigh out 26 gm of the well-mixed sample on a tared aluminum scoop or on a counterpoised piece of parchment or glazed onion-skin paper about 4 inches by 4 inches. Transfer the scoop with the sample to a capsule of Monel metal or nickel provided with a cap and rubber gasket, add 177 ml* of lead water (1 volume of lead subacetate solution and 40 volumes of water**), put on the cover shake the capsule vigorously and place it for 30 minutes in a water bath at 75° to 80°C. The capsule should be shaken at least every 10 minutes during heating. After heating cool to 20°C, shake well, filter, and polarize in a 400-mm tube. The reading gives the percentage of sugar directly.

Literature:

1) Browne & Zerban, op. cit., p. 365-8
2) Sazavsky & Sandera, Cukrovarnicka Analytika, p. 331
3) Polish Sugar Institute: Chemical Control in Sugar Factories, p. 130.

Fresh Cossettes

Mix well in an enameled bowl the total amount of prepared cossettes and take an average sample from different places of the bowl (approximately 300-500 gm). Grind the sample with a

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* For beets cultivated in Southern California an average juice content is lower; therefore, add 178.4 ml. See Bachler (Browne and Zerban, p. 368). For local conditions inquire in local sugar factories.

** Prepared according to German Pharmacopoeia: see Browne & Zerban, p. 311, IV.

meat chopper. The "Enterprise" chopper No. 41 or similar types are recommended in the United States. In other countries "Keystone" choppers with ten holes in the plate are favored. Weigh out 26 gm of the well-mixed sample as rapidly as possible and determine sugar by the method given for beets.

Literature:

1) Sazavsky & Sandera, op. cit., p. 336-337.
2) Polish Sugar Institute, op. cit., p. 15.
3) Browne & Zerban, op. cit., p. 341.

Sugar Content, Bx, and Purity of the Diffusion Juice

Remove air by keeping the sample of the juice for 5-10 minutes under vacuum (at least 20 inches Hg). Determine Bx in the deaerated sample. Weigh out as rapidly as possible 52 gm of juice and wash it into a 100-ml volumetric flask; add 5 ml of lead subacetate solution, make up to the mark mix, filtrate, and polarize in a 200-mm tube. The reading divided by two gives the percentage of sugar. If the filtrate for polarization is turbid, add one drop of acetic acid. It is a better practice to determine Bx by means of a pycnometer than by a refractometer.

Literature:

1) Sazavsky & Sandera, op. cit., p. 340.
2) Czechoslovak Society for Research and Testing of Materials, Standard Methods for Chemical Control in Sugar Factories, (1946) p. 13-14.
3) Browne & Zerban, op. cit., p. 59-63.

Ofner's Iodometric Method for Determining Invert Sugar in the Beet or in the Diffusion Juice.

Fifty grams of juice expressed from the pulp (or 50 gm of diffusion juice) is transferred to a 250-ml volumetric flask, clarified with neutral lead acetate solution; the volume is completed to the mark and the solution filtered. 100 ml of the filtrate is transferred to a 200-ml volumetric flask and deleaded with a solution of $Na_2HPO_4 \cdot 12 \cdot H_2O$; the volume is completed to the mark and the solution filtered. 50 ml of the filtrate (equals 5 gm of juice; in case of greater amount of invert, a smaller amount is taken and completed to 50 ml) is transferred to a 300-ml Erlenmeyer flask and mixed with 50 ml of the copper reagent. A small quantity of powdered pumice stone or talcum is added to prevent overheating during the boiling period. The flask is placed on a wire gauze resting on an asbestos plate with a round hole about 6 cm in diameter. The liquid is heated

to boiling in four to five minutes and then gently boiled with a smaller flame for exactly five minutes. The flask is then rapidly cooled in running water, and 15 ml of N hydrochloric acid is carefully run in along the inner wall of the tilted flask. Immediately after acidifying, the standard iodine solution is added, the flask being slowly revolved after the first few milliliters have been run in. The iodine must be added in excess; a total of 5 to 20 ml is used, depending on the amount of invert sugar present. The flask is stoppered immediately after the addition of the iodine, which is allowed to act for two minutes, with occasional agitation. Then 5 ml of a 0.5 per cent starch solution is added, and the excess of iodine is titrated back with the standard thiosulfate. The difference between the milliliters of iodine solution added and the milliliters of thiosulfate used in back titration gives the amount of the iodine solution used. Now a correction must be made, which is determined as follows: 50 ml of the clarified and deleaded solution is transferred to an Erlenmeyer flask and mixed with 50 ml of the copper reagent; then 15 ml of N hydrochloric acid is added. Immediately after acidifying, the standard iodine solution is added in excess. The flask is stopppered and after two minutes the excess iodine is titrated back with the standard thiosulfate in the presence of 5 ml of starch solution. The amount of milliliters of iodine solution used and increased by 0.1 ml for each gram of sucrose in the 50 ml is deducted from the milliliters found in the actual analysis. Each milliliter of the iodine solution thus corrected corresponds to 1 mg of invert sugar or 0.02 per cent.

Instead of using the expressed beet juice, a juice obtained by cold digestion of a fine beet pulp may be used for the same purpose.

Literature:

1) Czechoslovak Soc., op. cit., p. 45.
2) Browne & Zerban, op. cit., p. 837-839.