contents leveled off to values of 58 and 10%, respectively, at an absorption of about 2 pounds of ammonia per unit of phosphorus pentoxide.

In general, the physical conditions found to be most conducive to efficient ammonia absorption by superphosphates were low ammoniation rates, high temperature (for triple superphosphate), prolonged reaction periods, and such attributes of the superphosphate as fineness, softness, and high moisture content.

Acknowledgment

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Economical Waste Disposal for Pear Canneries; Color of Alkaline Sugar Solutions Studied

SUGAR RECOVERY

Recovery of Sugars from Pear-Canning Waste

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A process for the recovery of sugars and other soluble solids from peelings, cores, and trimmings wasted in canning pears involves conversion of the milled waste into a calcium pectate gel from which a clear, pectin-free juice is easily pressed, removal of excess calcium by means of an ion-exchange resin, and removal of color by precipitation and by adsorption with activated carbon. The purified juice, when used as a sirup base for canning pears, replaces about one third of the refined sugar now used in canning this fruit. The pomace, which amounts to about 25% of the waste, can be dried in a continuous rotary dryer for use as a feed. Pilot-plant operation indicates that the process offers a profitable solution to the pear-canning industry's serious waste-disposal problem.

A PROCESS FOR THE RECOVERY OF SUGARS and other soluble solids from pear peelings, cores, and trimmings for use as a sirup base for canned pears has been described in a preliminary report (*9*). The steps in this process were adapted from conventional methods for treating fruit wastes, preparing fruit juices, and refining sugar solutions. The steps were specifically selected and designed to provide as simple and economical a process as possible, consistent with the quality essential to a sirup base for canned pears. The present report describes a pilot plant application of this process and presents pilot plant and laboratory data on several of the unit operations involved.

The recovery of sugars from fruit cannery wastes in a form suitable for use as a sirup base has been proposed by a number of workers. Such processes appear logical, as sugars are the main constituent of economic value in fruit

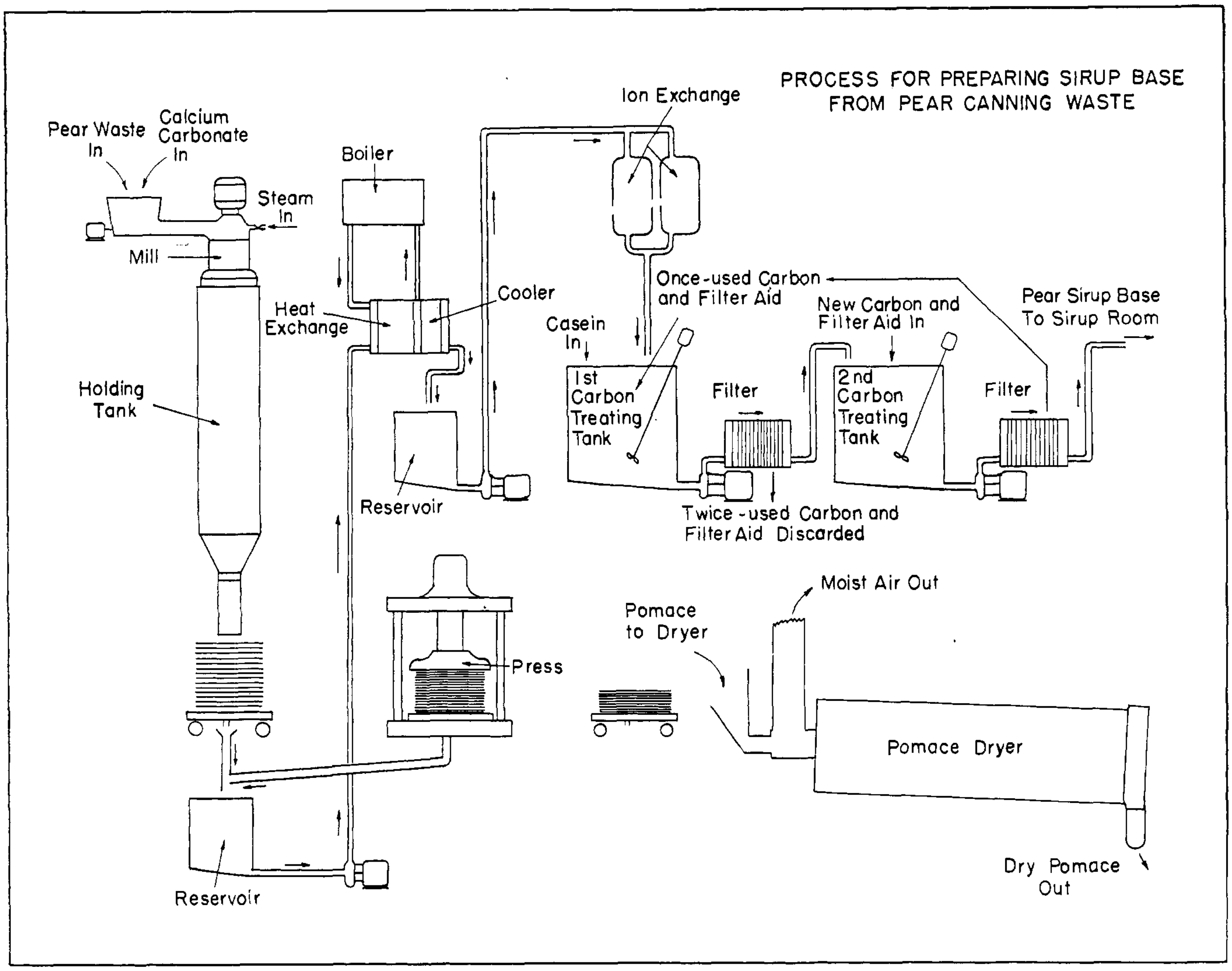


Figure 1. Flow sheet for preparing sirup base from pear-canning waste

wastes, and canners producing the waste require large quantities of refined sugar in processing the fruit. Probably the best known application of such a process is that described for pineapple (*7*). Bartlett pears, with their relatively high sugar content and heavy waste loss during preparation for canning, also offer a good opportunity for the application of a sugar-recovery process.

Several workers have proposed methods designed specifically for the recovery of sugars from pears and pear-canning wastes. Erickson and Ryan have proposed several processes, one of which consists of removing the soluble solids from diluted, finely ground pears by filtration after treatment with phosphoric acid and lime under suitable conditions (*3*). They proposed that juice recovered in this manner be decolorized to produce a clear, substantially water-white sirup. A second method involved treatment of diluted, finely ground pears with lime to a definitely alkaline pH to promote extraction of sugars, and removal of the juice by pressing (*4*). In a modification of the second method, the expressed juice was treated with additional lime and then acidified with phosphoric acid to eliminate certain difficultly removable impurities (*5*). Chong and Cruess (*2*) proposed treating the ground pear waste with a commercial pectic enzyme preparation to facilitate separation of juice from insoluble pulp in a conventional rack-and-cloth press. They considered filtering and decolorizing juice obtained in this manner advisable before use as a sirup base for canned pears.

Ash (*1*) proposed a process similar to that of Chong and Cruess, but followed the enzyme digestion with a heat treatment and addition of water, and removed the insoluble solids by means of a press or filter. Juice recovered by this method was decolorized with activated carbon.

Pilot Plant Studies

A pilot plant, designed to operate continuously at a rate of about 4 pounds of waste per minute, was installed and operated at the Apple Growers Association cannery, Hood River, Ore., in 1950. A flow diagram of the process, showing essential parts of the pilot plant, is given in Figure 1. The pomace-drying equipment shown in this figure was not part of the pilot plant, and steam injection into the mill has been substituted for a tubular heat exchanger used in the pilot plant. Photographic illustrations of the pilot plant are shown in Figures 2 through 4.

Nearly 30 days of successful operation were realized during the season. A total of 2311 gallons of pear juice was refined, which had an average soluble solids content of slightly over 10%. Liquid sugar was added to bring the solids up to 30%, the ingoing Brix for Choice pears, and this juice sirup was used in preparing an experimental pack of 1431 cases (24 No. 2-$^1/_2$'s) of pears. The pack was marketed under a special permit issued by the U. S. Food and Drug Administration. The product was considered by consumers to be as acceptable as pears canned in a conventional sugar-water sirup and in many

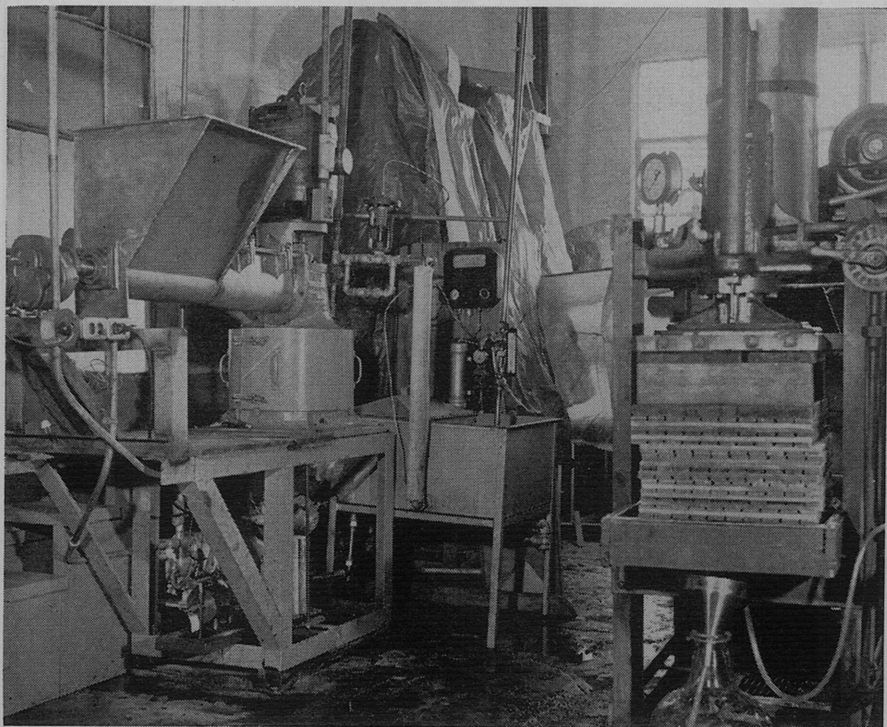


Figure 2. Waste treating and pressing section of pilot plant

Waste and calcium carbonate were fed into hopper (upper left) and pulped by attached mill. Pulp was then pumped through tube immersed in constant temperature water bath (center) and delivered to vertical tank (upper right, above press) where gel was allowed to form. Gelled waste was pressed in hydraulic press (lower left)

instances the flavor of the juice-sirup pack was preferred.

Cost estimates (*9*), revised on the basis of this study, indicate that the process would be profitable under conditions of adequate waste volume. Capital investment, including equipment, installation, and building, but exclusive of a pomace dryer or steam plant, would be about $90,000 for a plant capable of processing waste at a rate of 4 to 6 tons per hour. Capital depreciation at 10% annually would amount to $2.25 per ton when 4000 tons are processed annually. Labor is estimated at $3.00 per ton when waste is available at a rate of 4 tons per hour. Of this, about $1.50 would be for operation of the juice press. The cost of materials, which are relatively independent of volume, would be somewhat less than $3.00 per ton and half of this would be for decolorizing carbon. The total cost of $8.25 per ton would be substantially less as the volume increased to 6 tons per hour and 6000 tons per year.

Based on a recovery of 165 pounds of soluble solids per ton of waste at $8.25, the cost would be 5 cents per pound of soluble solids. Assuming that these are acceptable, pound for pound, in place of refined sugar, the savings can be estimated from the cost of refined sugar. A refined sugar cost of 8 cents per pound would result in a saving of about $20,000 annually when 4000 tons of waste are processed at a rate of 4 tons per hour. When 6000 tons of waste are processed at a rate of 6 tons per hour, the saving would be about $45,000.

Unit Operations

Treatment Of Waste

Probably the most difficult step in the handling of fruit wastes is the economical separation of soluble from insoluble solids. To facilitate this separation, lime treatment has frequently been employed and numerous patents have been issued covering liming procedures, particularly as applied to citrus wastes. These methods, as applied to citrus, have been reviewed by von Loesecke (*12*). A more recently issued patent by Neal (*8*) also describes a lime treatment for citrus and other fruit wastes. The treatment used in the present study depends on naturally occurring pectic enzymes in pear waste to convert the pectin, in the presence of added calcium, into a solid calcium pectate gel (Figure 5), from which clear, pectin-free juice can be easily pressed in a rack-and-cloth press.

To produce this gel, calcium carbonate was added manually at a rate of about 0.35% to the waste as it was fed into the screw feed hopper of a mill provided with a 0.25-inch screen. The pulp was then heated to between 90° and 120° F. and held for 1 hour. The amount of calcium carbonate added was not critical and the mill provided adequate mixing. Pulp treated in this manner was converted into a suitable gel throughout the season.

A temperature between 90° and 120° F. was attained by pumping the pulp through about 14 feet of 1-inch stainless-steel pipe immersed in a water bath held at 140° to 165° F. In commercial operation the required temperature could be attained by direct injection of steam into the mill. Dilution by condensate would amount to about 2%. The gel was formed in a cylindrical aluminum tank 1 foot in diameter and 5 feet high, which was supplied with a slotted sliding gate at the lower end. This tank was mounted on a hydraulic press in such a manner that opening the gate would deliver gelled pulp by gravity to the press pan where cheeses were made. The tank, which held 2 hours' production of pulp when full, was provided with an electronic level controller that turned on a signal light when the tank was about half full and controlled the pulp delivery pump to maintain the level above this point. Pulp drawn from the tank when the signal light was on was therefore held at least 1 hour.

An alternative procedure consists of raising the pH of the pulp to about 8.2 by addition of a calcium hydroxide slurry. At this pH a suitable gel is formed without heating and the pH drops below 6.5 during gel formation. The method has the disadvantage that it requires accurate proportioning of slurry and pulp, followed by thorough mixing to produce a uniform gel structure. Successful operation of this alternative process was not achieved in the pilot plant operation, because adequate equipment for thoroughly mixing the calcium hydroxide slurry with the pulp was not available. Although gels produced by calcium hydroxide treatment could be pressed without difficulty, pectin remaining in the juice when the treatment was not uniform caused fouling of the heat exchanger, clogging of the ion exchange column, and difficulty in carbon removal and filtration.

Pressing

Juice was pressed from the gelled pulp with a hydraulic press equipped with nylon cloths and 17-inch racks. A practically clear, amber juice was obtained and no difficulty was experienced in stripping the cloths from the pressed pulp. In a trial run on a commercial 48-inch rack press, about one third of the total yield of juice was obtained as free-run while building the cheeses another third in the press during the initial pressing, and the final third under pressures of about 100 pounds per square inch. Experimental data showed that a juice yield of about 75% of the pulp could be expected. Pomace from the full scale pressing was dried without difficulty in a single pass through a commercial rotary steam tube dryer.

Boiling To prevent excessive darkening, the juice must be heated to boiling as soon as possible. Heating up to 190° F. did not prevent later darkening, while boiling effectively halted darkening and precipitated 10 to 20% of the pigment already present. Heating was accomplished in the pilot plant by pumping the juice through a countercurrent heat exchanger constructed of about 30 feet of stainless steel tubing $^3/_8$ inch in outside diameter, jacketed with tubing $^5/_8$ inch in outside diameter. Raw juice passed through the inner tube into a $^1/_4 \times 1 \times 4$ foot tank, where it was heated to boiling by means of a steam coil. The boiling juice flowed by gravity through the $^5/_8$-inch jacket and was cooled by incoming raw juice. The juice was then passed through a water-cooled coiled block tin tube to assure complete cooling.

The volume of juice obtained from waste is about equal to the volume of sirup required to can the pears from which the waste originated. The finished juice can be regulated to the exact volume desired by control of evaporation in the boiling step. In this study about 10% of the volume was evaporated at this point to compensate for dilution encountered in later operations.

Adjustment of pH During the course of the pilot plant study the pH of boiled, cooled juice was found to vary from 5.3 to 6.7; the average value from over 100 readings was 5.7. This lower than normal acidity is caused by residual calcium from the treating operation. In order to use the juice as a sirup base for canned pears, it is necessary to increase the acidity. Sirup prepared from juice adjusted to pH 4.4 to 4.5 resulted in a canned product with a pH of about 4.2, which was considered pleasingly tart (*9*). The flavor was noticeably more tart than that of pears of the same pH which had been canned in a water-sugar sirup.

Adjustment of the pH was accomplished in the pilot plant by passing the juice through a cation exchange resin in a hydrogen cycle, using experimental exchange units with 4-inch-diameter columns. Application of ion exchange resins in the food industry has been reviewed by Felton (*6*). In the authors' process the ion exchange treatment was limited to a partial removal of cations, principally calcium, to produce the desired pH. The operation consisted simply of passing juice through the exchange column until the pH of the mixed effluent reached the desired point.

Juice from properly gelled pulp presented no difficulty in clogging the columns, provided it had been strained. The pH of the initial effluent was about 2.3 and that of the final effluent was the same as the ingoing juice. Figure 6 shows typical pH curves obtained on pear waste juice with a number of resins, using a laboratory column 2 cm. in diameter. It is apparent from this figure that the cut-off point for a mixed effluent at a pH between 4.4 and 4.5 is not critical. Capacities shown in this figure were not realized with the pilot plant columns. Resin 1, for example, had a capacity of about 27 volumes when operated to a mixed effluent pH of 4.5 in the column 4 inches in diameter, whereas in the column 2 cm. in diameter the capacity was about 35 volumes. This resin showed no appreciable loss of capacity during 50 consecutive cycles.

Decolorizing. The color of the juice was measured with a photoelectric colorimeter, using a 420 mμ filter and a water blank. Color was stabilized in the sample taken for measurement by adding 5 ml. of 2*N* hydrochloric acid to 50 ml. of juice. This stabilizing treatment, which reduced the pH to between 1 and 2, prevented darkening during delay before measurement. A standard curve was prepared by blending a colored with a completely decolorized juice. To obtain sufficient accuracy in the high color range, a second curve was prepared by diluting 10-ml. blends of these same juices to 100 ml. after addition of 5 ml. of 2*N* hydrochloric acid. All samples were filtered before measurement.

To be suitable for use as a sirup base for canned pears, the juice must be almost colorless. Canning experiments have shown that pear juice decolorized to the point where its transmittancy is 85% of water at 420 mμ (absorbance equals 0.0655), or higher at a 1 to 1.1 dilution, gives a canned product comparable in color to a conventional sugar-water pack (*9*). For convenience in conducting decolorizing studies and in operating the pilot plant, this amount of

Figure 3. Boiling and ion exchange section of pilot plant

Juice from press was pumped through small tubular countercurrent heat exchanger to rectangular tank (top), where it was heated to boiling. Final cooling of boiled juice occurred in coil immersed in water tank (center) before delivery to reservoir (lower center). Boiled and cooled juice was then pumped through ion exchange columns (left)

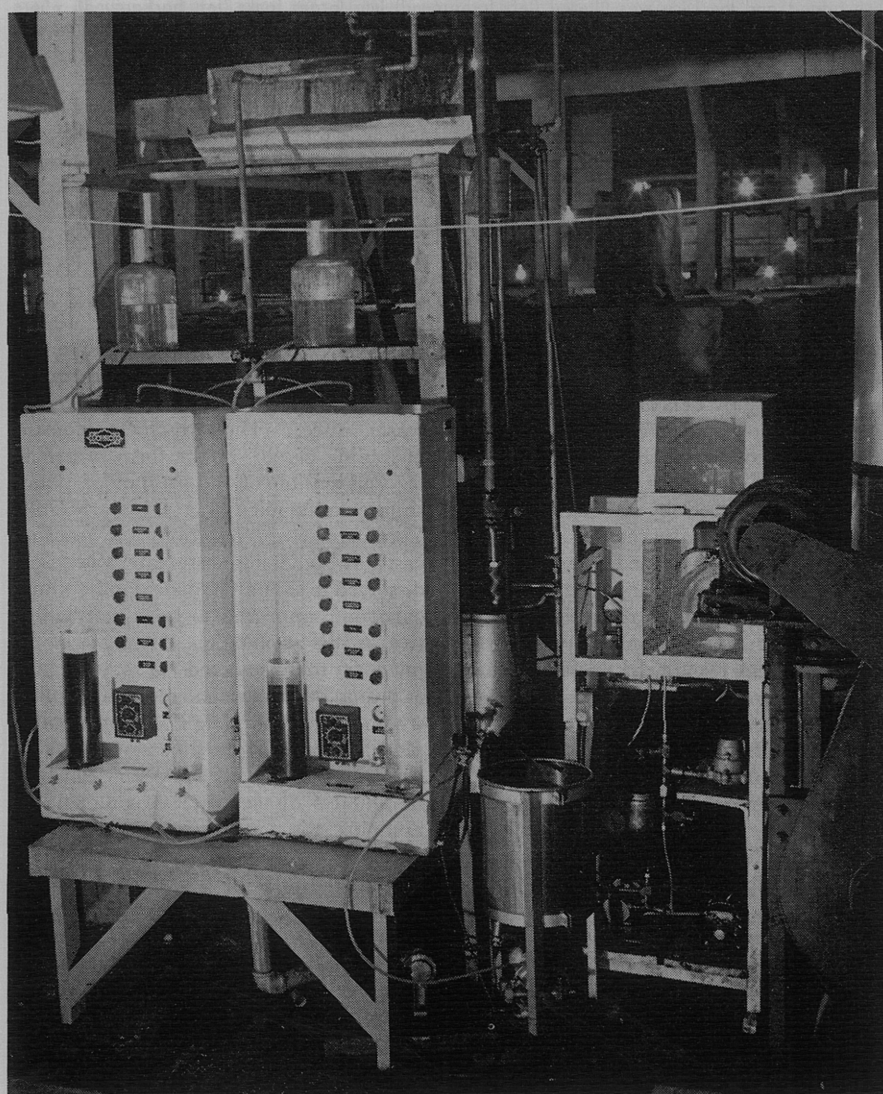


Figure 4. Decolorizing section of pilot plant

pH of juice from ion exchanger was adjusted in small tanks (right). Gelatin and first cycle carbon were then added and the juice was filtered (left foreground) into larger tanks (left background), where second carbon treatment was made. Finished juice from second carbon treatment was filtered into largest tank (center background) for storage until it was pumped to cannery sirup room

color was assigned a value of one color unit. Standard curves showing the transmittancy of pear juice at the two dilutions in terms of this color unit are shown in Figure 7.

The color of boiled and cooled juice was observed to vary from 18.1 to 45.8 color units during the course of the pilot plant study, with an average value of 28.2 for 125 measurements. The amount of color present at this point is determined largely by delays in handling the waste and in bringing the juice to boiling. This color was removed by two operations. About one third of the color was removed by the addition of gelatin, which also served to flocculate suspended solids formed during the boiling and ion exchange treatments. The color was further reduced by use of activated carbon in a two-stage, countercurrent cycle. In this operation carbon recovered from the second treatment was used for the first treatment of a subsequent batch. The gelatin treatment was carried on concurrently with the first carbon treatment and both carbon and precipitate were removed in a single filtration.

In the pilot plant study gelatin, dissolved in a small amount of juice, was added to ion exchange–treated juice at the rate of 2 to 3 ounces per 100 gallons. While the gelatin treatment was consistently effective in removing color, the effect on suspended solids varied from complete and rapid flocculation to the formation of a hydrophilic sol. Filtration of juices that failed to flocculate was more difficult than when good flocculation occurred, but practical filtration rates were obtained in all cases.

A casein treatment similar to that described by O'Neal, Meis, and Cruess (*10*) has since been developed, which was equally as effective as gelatin in removing color and gave positive coagulation of colloidal materials under all conditions encountered in pear waste juice. The treatment consists of adding casein, in the form of a 5 to 6% suspension of the sodium or potassium salt, to juice at a pH above 5.25. Commercial preparations of sodium caseinate, potassium caseinate, and casein dissolved in sodium hydroxide or sodium carbonate appeared to be equally effective in this application. The pH of the juice is then lowered to 4.4 by the addition of ion exchange–treated juice, where rapid, complete flocculation takes place. The amount of casein added is such that the final concentration will be about 4 ounces per 100 gallons. In applying the casein treatment the ion exchange cycle is modified so that the final two thirds of the effluent from a spent column, which would be at a pH above 5.25, is placed in the first carbon treating tank. The casein suspension is then added and initial effluent from a fresh column is added until the pH drops to 4.4.

The effectiveness of this treatment apparently depends on the acid coagulation of casein, and the pH limits of dispersion and coagulation are critical. The effect of the pH at which the casein is dispersed on color removal from pear juice is shown in Figure 8 and the effect of coagulation pH is shown in Figure 9. Figure 10 shows the effect of casein concentration on color removal. In each case the point on the ordinate gives the number of color units in the original juice.

When carbon is applied in a two-stage process, the desired final color can be reached with a considerably lower dosage than in a single treatment. The low final color requirements for juice to be used as a sirup base make a two-stage process essential to economic operation. Equations for determination of countercurrent dosage have been derived from the Freundlich equations (*11*), where M_{cc} is the countercurrent dosage and M_s is the single-treatment dosage. These equations,

$$\frac{M_{cc}}{M_s} = \frac{C_i - C_f}{C_o - C_f}$$

$$\frac{M_{cc}}{M_s} = \frac{(C_f)^{1/n}}{(C_i)}$$

which are independent of K from Freundlich's equation, involve six variables. M_s and $1/n$ are determined from the isotherm, while C_o, the initial color, and C_f, the final color, are given. The factors C_i, the intermediate color, and M_{cc} remain unknown. C_i and M_{cc} were determined from the above simultaneous equations after M_s and $1/n$ had been evaluated from experimental isotherms. These factors were determined for casein-treated pear-waste juice with two types of commercial vegetable carbon manufactured for use in decolorizing sugar solutions. The best average values from several tests were: $M_{cc} = 0.35\%$ and $C_i = 6.0$ color units for carbon A, and $M_{cc} = 0.60\%$ and $C_i = 6.0$ color units for carbon *B*. Table I shows C_i and C_f values obtained in the application of a two-stage countercurrent treatment on a juice having an initial color, C_o, of 35 color units.

Figure 5. Pear waste after treatment

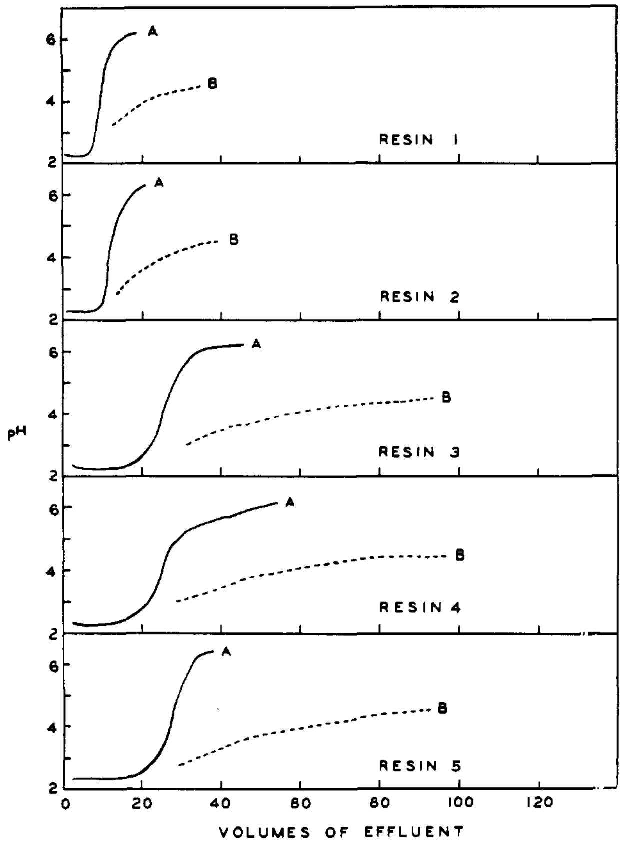


Figure 6. Typical pH curves obtained by cation exchange treatment of pear waste juice in hydrogen cycle

—. pH of effluent from exchanger
----. pH of mixed effluent
Resins 1 and 2. Sulfonated phenolic base resins
Resins 3, 4, and 5. Styrene nuclear sulfonic–type resins

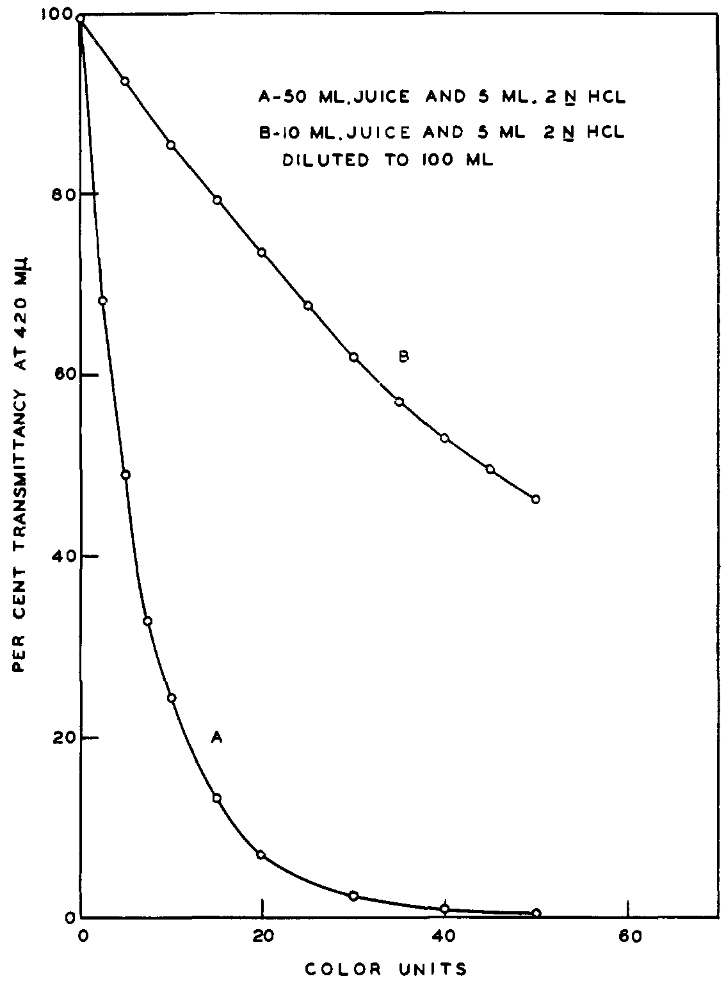


Figure 7. Standard curves for converting transmittancy to color units

The exact values required for M_{cc} and C_i to obtain a C_f of 1.0 color unit will vary with initial color. Use of a carbon dosage adequate to reduce color to or below the desired level in all juices encountered is believed desirable for

Table I. C_i and C_f Values Obtained in Countercurrent Treatment of Pear Waste Juice with Initial Color, C_o, of 35 Color Units

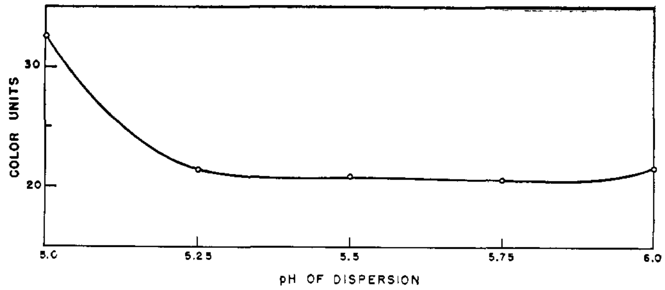
Carbon	M_{cc}, %	C_i, Color Units	C_f, Color Units
A	0.35	6.4	1.0
B	0.60	6.7	1.1

commercial operation, since pear canners would find it impractical to apply a minimum countercurrent treatment efficiently. The above results confirm countercurrent dosages previously established in the pilot plant studies for carbon A. In these studies a countercurrent dosage of 0.35% for carbon A was found adequate for practically all juices encountered. A safe countercurrent dosage of 0.75% was established for a third commercial vegetable carbon, C. In selection of a carbon for commercial use, cost would be important. Although carbon B was found to be less effective than carbon A in removing color from pear juice, the relative cost of these carbons would be about the same, because carbon B is considerably less costly than carbon A.

Filtration Filtration is the most critical operation in this process. Only by the complete removal of pectin in the lime gelatin step is the removal of carbon by filtration made practical. Filtration was accomplished in the pilot plant study by means of a horizontal plate-and-frame filter using either a cotton cloth or filter paper media. Two types of carbon, A and C, were used in juices that had been treated with gelatin. Although accurate determination of optimum filter aid dosages was not possible under pilot plant operating conditions, the information obtained is useful in estimating filter requirements.

In the first filtration step use of a fairly retentive diatomaceous filter aid in a 2 to 1 ratio with carbon A usually gave adequate filtration rates with a filter area of 6 square feet per 100 gallons of filtrate, even when flocculation was poor. Best results with carbon C,

Figure 8. Effect of dispersing pH on color removal by casein flocculation



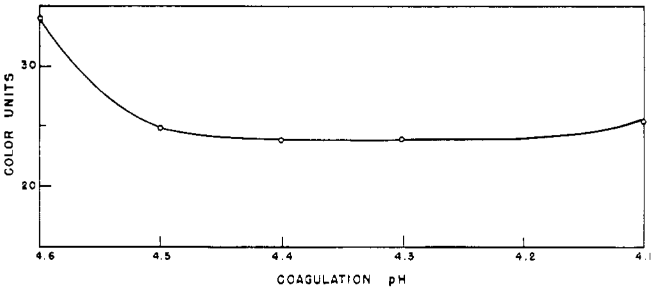


Figure 9. Effect of coagulation pH on color removal by casein flocculation

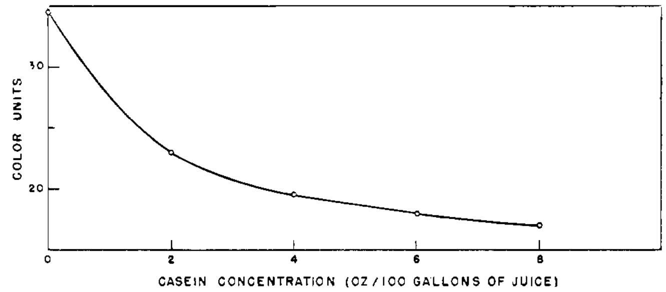


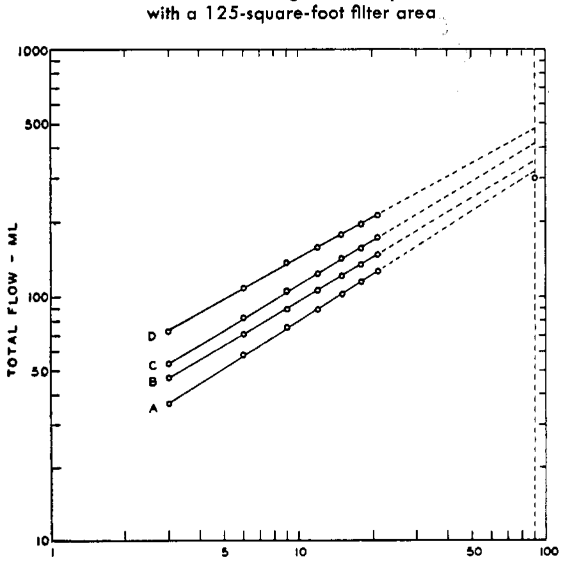
Figure 10. Effect of casein concentration on color removal by casein flocculation

which was more finely divided than A, were obtained in the first filtration by using a very porous filter aid in amounts up to 1% of the juice or slightly above a 1 to 1 ratio with the carbon. Filtration rates were slower with this carbon, however, in part because of the greater filter-cake thickness encountered. The second filtration, which removed the carbon and filter aid for re-use in the first treatment, presented no problem with either carbon, since suspended solids precipitated by the gelatin were no longer present.

Laboratory studies were made with a constant-pressure bomb filter to determine the most suitable grade and dosage of filter aid for casein-treated juice. Six grades of diatomaceous filter aid, ranging in relative flow ratings from 1 to 10, were studied at pressures of 10, 20, and 30 pounds per square inch. Carbon A and carbon B, in dosages of 0.35 and 0.60%, respectively, were used in these studies. The filter consisted of a 0.005-square-foot area of cotton duck. Filtrations were made at 78° F.

Figure 11. Typical filtration curves obtained with a bomb filter on casein-treated pear waste juice containing 0.60% carbon B and using a filter aid with a flow rating of 1

A and B. 0.3% filter aid at pressures of 10 and 20 pounds per square inch, respectively
C and D. 0.4% of the same filter aid at pressures of 10 and 20 pounds per square inch, respectively
The point on the broken vertical line at 90 minutes represents the minimum rate required to filter 2000 gallons of juice in 90 minutes with a 125-square-foot filter area



Considerable variation was observed between flow rates of different juices. Data from a great many tests, however, indicate the range of filtration rates that might be encountered in commercial operation. In these studies flow rates tended to decrease slightly as successively coarser grades of filter aid were used. This is contrary to usual filtration observations, but may be due to the nature of the casein floc. Filter aids having relative flow ratings of 1, 2, and 3 appeared to be about equal in flow rate and filtrate clarity.

In considering optimum dosages of filter aid, the over-all conditions of a commercial operation must be considered. A commercial pear-canning operation in which 10 to 15 tons of pears are canned hourly would produce up to 1000 gallons of juice per hour. The ion exchange treatment would divide this flow into 2-hour cycles of 2000 gallons each. Allowing a 90-minute filtration cycle and 125 square feet of filter area, an average filtration rate of 10.7 gallons per square foot per hour would be necessary. Under these conditions filter aid dosages of 0.35% for carbon A and 0.40% for carbon B gave adequate filtration rates at pressures of 20 to 30 pounds per square inch in all cases for the first filtration. Figure 11 presents some typical filtration curves plotted on log-log paper.

General Considerations

The effect of iron contamination on the efficiency of the refining process was studied to determine the metal requirements of processing equipment. The pilot plant was operated several days, during which the boiler was filled with iron turnings and other iron scrap, and the waste was in contact with iron in the pulp-holding tank. This modification showed that iron contamination did not interfere with the refining process and did not affect the quality of pears canned in such juice. This will make possible a considerable saving in equipment costs of a commercial installation. Corrosion resistance would appear to be the main consideration in selecting suitable metals. In this process the natural corrosiveness of pear juice is reduced, up to ion exchange operation, by the neutralizing effect of the calcium.

During operation of the pilot plant, continual difficulty was encountered from microorganism build-up in the equipment, which tended to cause souring of the juice. In designing a commercial plant special care should be given to permit easy and thorough cleaning.

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