Recovering Potato Proteins Coagulated by Steam Injection Heating

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Recovering proteins from potato juice of different concentrations was studied, including those concentrations which simulated typical waste effluents discharged by U. S. potato starch factories. Studies included: the effect of pH and temperature on the percent recovered using steam injection heating; a comparison of gravity settling; filtration and centrifugation; and a comparison of drum, air, and freeze drying of the precipitated proteins. These studies led to the development of a process which quantitatively recovers the proteins by the steam injection heating of acidified protein water to a temperature of at least 99° followed by filtering in a plate and frame filter press, and then drying the precipitate on a double drum dryer.

Current public concern over environmental quality has caused the USDA to study some of the waste disposal problems facing the potato processor. Thus far, the Eastern Center has directed most of its attention to the starch factory effluent because it has the highest BOD and is the least amenable to conventional treatment. It also is expected that the basic approach and findings can be applied to other pollution problem areas involving heat coagulable proteins.

Essentially, this problem has strong economic implications. The already marginal potato starch industry, which uses culls and scraps, faces stiff competition from other domestic starches as well as imported starches. Thus, the cost of impending mandatory waste treatment threatens to price potato starch out of the market. The manufacturer has the choice of closing down or trying to reclaim values to offset all or part of the effluent treatment costs. The Eastern Center has based its research on the latter alternative, *i.e.*, rendering the effluents suitable for discharging by first recovering such valuable components as protein, amino acids, organic acids, and potassium. An ion-exchange process for recovering the potassium, amino acids, and organic acids has been developed at this Center (Heisler *et al.*, 1970, 1972; Schwartz *et al.*, 1972). When conventional downflow ion-exchange columns are used, for efficient operation the protein concentrations must be lowered to less than 180 ppm or else they will precipitate in the ion-exchange columns. Since the protein level in the protein water obtained from U. S. plants typically contains 1500–4000 ppm, protein removal must be virtually complete.

Reports of a Dutch process (Peters, 1960) indicated that steam injection precipitation might achieve the required goal by overcoming problems encountered in other attempts to recover potato proteins, *i.e.*, scaling, low yields, and slurries difficult to filter. Apparently the only disadvantage of this method is the dilution incidental to steam condensation. Many other processes for recovering proteins by coagulation have been reported in Europe and the U. S. For example, Xander and Hoover (1959) recovered the proteins from potato juice by heat coagulation and then further treated the deproteinized juice to produce a protein hydrolyzate. However, none of these processes have used steam injection heating and the conditions necessary to produce an easily filterable precipitate have not been defined. Since no published engineering data or pertinent processing information were available, the experimental studies which this paper discusses were made to determine suitability of this approach.

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These studies were: effect of steam injection coagulating temperature, solids concentration, and pH on the recovery and filterability of the heat-coagulated proteins; gravity settling of the precipitated proteins; finding a dewatering system meeting processing requirements; and finding an appropriate drying method.

These studies led to the development of a process which virtually quantitatively recovers the proteins. It comprises steam injection heating of acidified protein water (pH 4.2–5.5) to 104.4°, filtering in a plate and frame filter to remove the protein, and then drum drying to yield a soft, grayish, bland product.

MATERIALS AND METHODS

Preparation of Protein Water. The flow diagram (Figure 1) shows the preparation of the protein water used in our pilot plant experiments. Potatoes (70 kg/batch) first are washed thoroughly in a rod-reel type washer by high pressure water sprays (7.8 atm). They are then ground in a Rietz Disintegrating Mill, during which time the sodium bisulfite is added. The resulting slurry is pumped to a solid bowl continuous centrifuge. From the pulp, about 60–62% of the undiluted juice is spun off; the cake leaving the centrifuge contains 38–40% solids. An equal weight of water is added to the cake, which is reground and centrifuged, the resulting liquid is blended with the juice, giving a liquid containing 3.4–4.4% solids. With this method *ca.* 93% of the soluble solids is recovered.

Steam Injection Heating. The experimental unit used is shown in Figure 2 and consists of a metering pump, a steam pressure regulator, the injector unit, a surge chamber, and a vapor–liquid separator and condenser. The surge chamber ensures steady (nonpulsating) operation. The vapor–liquid separator and condenser were necessary to make a complete material balance when operating above 100°. The valve in the injector outlet was used to throttle the liquid–steam mixtures to reach temperatures above 100°. The valve in the outlet from the surge chamber maintained a slight pressure in the chamber to prevent foaming. The liquid, if superheated, was flash cooled to 100° in the vapor–liquid separator.

Sedimentation Studies. All experiments were carried out using 4.4 atm of steam to superheat the protein water to 104.4°, flash-cooling to 100°, and then collecting it in a series of 18 jars (475 ml). The jars were quickly filled in consecutive order, the whole procedure taking about 1.5 min. The jars were sampled in consecutive order at 10-min intervals by pipetting 30 ml of the liquid from the center; the samples were analyzed for protein using the procedure described under "Determination of Coagulable Protein." Then these values (in ppm) were plotted against the reciprocal of the corresponding elapsed time, and the curve was extrapolated to zero, *i.e.*, $t \rightarrow$ infinite time as in Figure 3. The value C_∞ on the ordinate scale is the

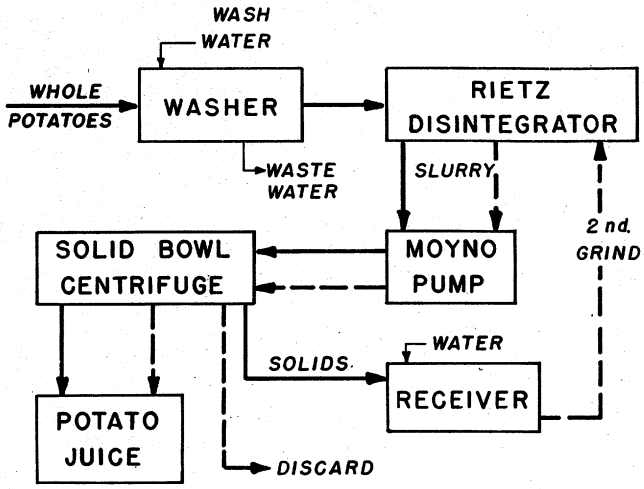

Figure 1. Preparation of potato protein water.

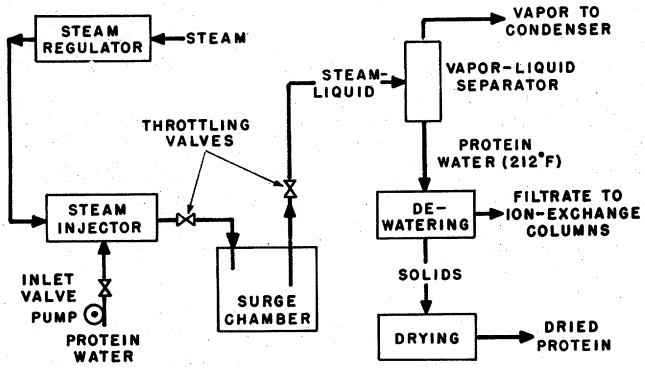

Figure 2. Flow sheet of the protein recovery process.

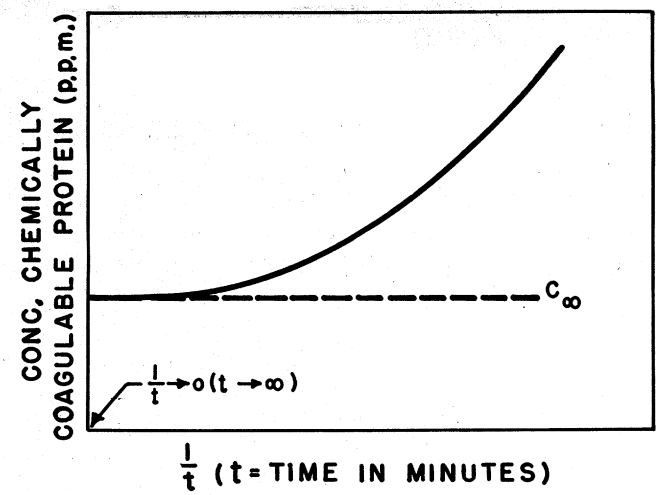

Figure 3. Determination of C_∞.

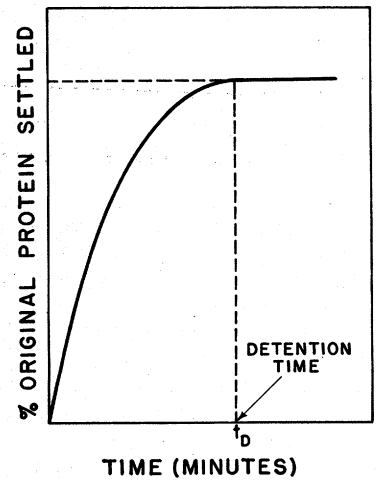

Figure 4. Determination of detention time.

amount of protein that never settles; subtracting this value from the protein originally present (C_0) and dividing this concentration by C_0 and multiplying by 100 gives the percentage of proteins originally present, which theoretically is the maximum amount that can settle out even in an infinite period of time. Practically, however, this value usually can be approached closely in a finite time. This finite time is the so-called detention time, an engineering parameter necessary to size gravity settling equipment. It is determined as shown in Figure 4 by plotting the percent of the original proteins settled against the corresponding time. The value t_D is the time after which $dC/dt = 0$, *i.e.*, the concentration no longer changes and is the residence time required to settle out the maximum amount at that pH and solids content. Since we are dealing with protein-containing liquids, pH and concentration significantly affect precipitation and hence their effect must be determined. Two concentrations of protein water, 1.5 and 2.8%, were used to duplicate the protein water of most American starch plants, the soluble solids of which lie in this range. The pH of fresh protein water is normally 5.8 to 6.2; lower pH's down to 2.00 were obtained by acidifying with HCl.

Filtration and Centrifugation Studies. Two types of filters were used in the pilot plant studies. One was a plate and frame type filter press with 30.5 cm × 30.5 cm plates (total filter area = 21,860 cm^2). The other was a 930 cm^2 continuous rotary vacuum horizontal drum filter with 30% of the filtering area submerged and a recirculating pump to maintain proper level in the feed trough. In the centrifugation studies, a bench type air-driven Sharples super centrifuge with a vertical rotating solid bowl was used, bowl speed was 50,000 rpm. In these studies the effect of pH and coagulating temperature and the range of solids were extended to include undiluted protein water (*ca.* 5% solids).

Drying Studies. Three types of drying were evaluated: freeze drying, air drying, and drum drying.

Determination of Coagulable Protein. The proteins were precipitated from a 5–10-ml sample of protein water by adding approximately 40 ml of 15% trichloracetic acid and 0.1 ml of 0.1% sodium tungstate. The centrifuged precipitate was washed once with 2% trichloracetic acid before transferring the pellet to a Kjeldahl flask for analysis by the official AOAC microKjeldahl method (AOAC, 1970).

Moisture and Solids Determination. Moisture contents of potato slurries and the cakes from the centrifugation and filtration studies were determined by the official AOAC vacuum oven method (AOAC, 1970). The vacuum oven was run for 11 hr at 70° under a partial vacuum ≥ 25 mm. The solids content of protein water was also determined by the vacuum oven method after the bulk of the water was removed by evaporation on a steam bath.

RESULTS AND DISCUSSION

Gravity Settling. In selecting a solid–liquid separation method, gravity settling is usually considered first. Even if one contemplates using filtration or centrifugation, the most economic operation usually is attained by first concentrating the slurry by clarifying, thickening, or flotation-thickening (Dahlstrom, 1968). In gravity settling, the two important parameters to be determined are residual solids in the colloidal size range which are too small to settle and the detention (residence) time. The former tells us how sharp the separation is, while the latter is necessary for sizing the clarifier based on the volumetric flow rates. Heretofore, the nonsettling solids have been of

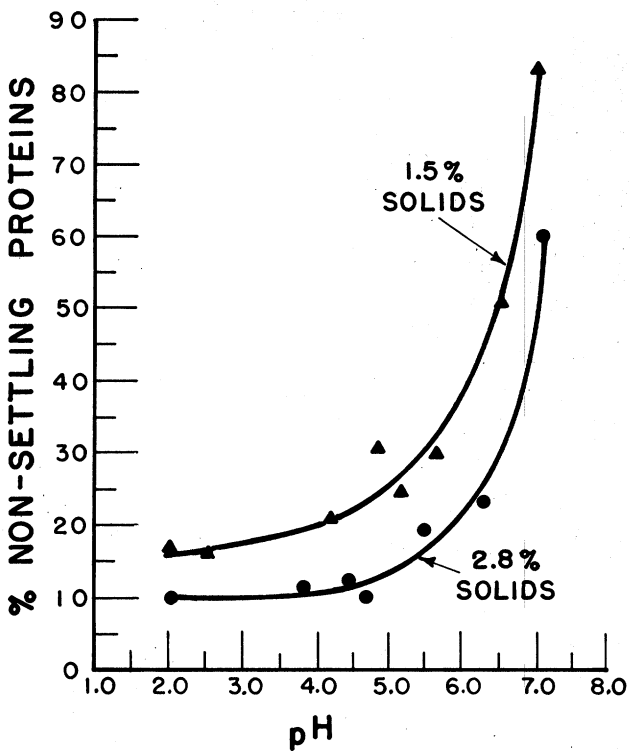

Figure 5. Effect of pH and concentration on protein recovery.

minor importance since the settling operation was generally confined to treating metallurgical slurries or those containing similarly agglomerated solids. However, with the increased interest in waste treatment, the colloidal range has become increasingly more important and can no longer be ignored (Fitch, 1962). The methodology described in this paper to study the settling of coagulated proteins is similar to that described by Fitch for metallurgical slurries and, as far as the authors are aware, is the first extension of this method to coagulated protein systems. Based on the authors' experience, the method could be a useful tool in studying coagulate protein systems, as the following discussion will show.

The results of this study are summarized in Table I, which shows C_∞, the concentration (ppm) of proteins which do not settle and remain dissolved or suspended indefinitely, the percent of nonsettling proteins, % NSP, obtained by dividing the initial concentration C_0 into C_∞ and multiplying by 100, and the corresponding detention times.

Figure 5 in which % NSP is plotted against pH shows the effect of pH and concentration more vividly. With slight acidification the amount of nonsettling proteins decreases significantly and then continues to decrease slightly over the pH range 5.0 to 2.0. The curves show that increasing the concentration also decreases the amount of nonsettling proteins. However, even though good yields can be obtained by acidification, settling is inadequate for our purpose. Table I reveals that not one value of C_∞ is below 180 ppm, the maximum protein content that can be tolerated for removal of the other compounds by ion exchange.

The detention times for the 2.8% protein water ranged from 60 to 78 min, while for the 1.5% protein water, they ranged from 40 to 72 min. The values are quite erratic and there is no correlation between these values and the other data reported, *i.e.*, pH. The average of all the values for the 1.5% protein water was 50 min and for the 2.8% protein water, the average was 67 min. In a more dilute solution, the precipitate settles faster and has more nonprotein material, *e.g.*, starch associated with it. In a more concentrated solution, the precipitate settles more slowly

Table I. Effect of Solids Content and pH on % Nonsettling Protein and Corresponding Detention Times

1.5% TS (C_0 = 1450 ppm of protein)[a]				2.8% TS (C_0 = 3090 ppm of protein)			
pH	C_∞, ppm	% NSP	Detention time, min	pH	C_∞, ppm	% NSP	Detention time, min
2.00	240	16.6	64	2.00	310	10.0	72
2.50	230	15.9	62	3.80	347	11.2	60
4.22	300	20.8	40	4.45	369	11.9	60
4.85	440	30.5	56	4.65	313	10.1	70
5.19	350	24.3	50	5.50	592	19.1	60
5.70	430	29.8	50	6.30	710	22.9	78
6.60	730	50.6	45	7.10	1860	60.2	70
7.10	1200	83.2	72				

[a] C_0 = initial protein concentration. C_∞ = protein concentration at infinite time. NSP = nonsettling proteins. TS = total solids.

but the protein content of the precipitate is higher. From an engineering point of view, the detention times are quite reasonable and if it were not for the rigid requirements imposed by conventional downflow ion-exchange columns, gravity settling could be used.

Filtration and Centrifugation Studies. The sedimentation studies led directly to three separate studies: feasibility of meeting the stringent residual protein requirements of less than 180 ppm; the effects of concentration and coagulation temperature on filterability; and the feasibility of using a continuous filter.

To meet the requirements of less than 180 ppm of residual protein, it was found necessary to acidify the protein water to a pH of 5.5 or less before heat coagulating by steam injection. The acidified slurries could be easily filtered or centrifuged, whether hot or cold, producing filtrates containing less than 100 ppm of protein.

The beneficial effect of concentration was apparent in the filtration studies using nonacidified protein water. Increasing the concentration increased the yield of protein removed. In fact pure potato juice (5.0–5.5% solids) produced filtrates containing less than 100 ppm of residual proteins. However, the precipitates obtained were gelatinous, making filtration difficult; more frames were needed for these slurries than were needed to handle the same volume of acidified slurries.

Since the coagulation temperature in all the foregoing experiments was 104.4°, it was decided to investigate lower coagulation temperatures to see how critical this parameter is, especially when using acidified (pH of 3.0–5.5) protein water. While temperatures as low as 82.2° produced filtrates containing less than 180 ppm of protein, the precipitates thus obtained were gelatinous and filterability was poor. In fact, a minimum coagulation temperature of 100° was found necessary to produce easily filterable slurries. Using nonacidified undiluted potato juice and a coagulation temperature of 82.2–99° removed only 80–85% of the proteins. These slurries were also difficult to filter. The results of the filtration studies are summarized in Table II.

Attempts to filter the slurries with a continuous rotary vacuum filter proved fruitless regardless of concentration, pH, coagulation temperature, or type of filter cloth used. While the slurry was being recirculated, the floc was broken down and blinded the cloth.

Drying Studies. Air drying in a conventional tray drier gave a dried product which was black, hard, and horn-like and which was very difficult to grind. However, double drum drying (3 rpm, 2.6–3.4 atm steam and 0.20 mm drum clearance) gave a fairly soft, grayish product. Freeze drying gave a white soft product; however, this method is probably too expensive. Use of the drum dryer required

Table II. Effect of Concentration, pH, and Coagulation Temperature on the Filterability of Protein Water Slurries at 70–180°F

% solids	pH	Coagulation[a] temp, °C	Initial protein, ppm	Residual protein, ppm	% protein recovered	Filtrate	Characteristics of precipitate[b]
5.0–5.5	5.8–6.2	104.4	8250–8785	<100	99	Pale amber Sparkling clear	(Grey) Gelatinous
4.0–4.5	5.8–6.2	104.4	4895–5958	724–905	85	Sparkling clear	Gelatinous
1.5–2.8	5.8–6.2	104.4	1500–4000	375–980	75	Sparkinlg clear	Gelatinous
5.0–5.5	5.8–6.2	82.2–100	8250–8785	1659–1320	80–85	Sparkling clear	Gelatinous
1.5–5.5	3.0–5.5	100–104.4	1500–8785	<100	99	Pale yellow Sparkling clear	Bulky flocculent Precipitate (white)
1.5–5.5	3.0–5.5	82.2–93.3	1500–8785	<100	99	Sparkling clear	Gelatinous (white)

[a] Temperature leaving injector before flash cooling. [b] Cake after filtration contained 12–15% moisture.

Table III. Comparison of Three Separation Methods

Method	% solids in slurry	% solids after concn	Character of the concentrated phase	Best drying method[a]
Gravity settling	0.27–0.70	1.3–3.5	Slurry	Further dewatering necessary
Filtration (plate and frame)	0.27–0.70 ——— 1.3–3.5[b]	12–15	Pasty or gelatinous[c]	Drum drying Freeze drying
Centrifugation (solid bowl)	0.27–0.70 ——— 1.3–3.5[b]	25–35	Dry, crumbly, similar to dried putty	Air drying Freeze drying Other (Baran, 1960)

[a] Refers to feasibility of methods studied. [b] After gravity settling. [c] Acidified cake lighter in color and bulkier; *i.e.*, not gelatinous.

feeds containing 12–15% solids to get a good sheet. The cake produced by centrifugation contained 25 to 35% solids and had to be diluted before drum drying. On the other hand, filtration produced precipitates containing 12–15% solids and could be readily drum dried. Hence, pressure filtration is more suitable for drum drying. Use of centrifugation would require the use of other means of drying, *e.g.*, dispersion spray nozzles (Baran, 1964). Comparison of the three solid–liquid separation methods with appropriate drying methods is summarized in Table III.

CONCLUSION

From the foregoing studies we can conclude that to recover proteins quantitatively from potato starch factory effluent, the effluent should be acidified to a pH of 5.5 or less, heated to at least 99°, pressure filtered in a plate frame filter press, and then dried on a double drum dryer.

While the process described is simple, efficient, and technically feasible, an economic analysis of the overall process, *i.e.*, protein recovery followed by removal of the other constituents using ion-exchange, indicated that this process is not economically feasible (Stabile *et al.*, 1971). Apparently, the most economically viable procedure is to concentrate the entire effluent, dry it, and then use it as a feed for monogastric animals, as a fermentation medium, or even as a component of pet foods. Nevertheless, publication of these studies is justified in view of the current interest in recovering proteins, since the techniques discussed may be applicable to other protein recovery problems where less stringent technical and economic requirements must be met. Perhaps even processes for recovering potato proteins already reported, *e.g.*, Xander and Hoover (1959), might find steam injection heating advantageous.

ACKNOWLEDGMENT

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