grade of fat and is free from unsaponifiables introduced by the Twitchell reagent. With high grade feed stocks, it is possible to make a good grade of fatty acid without distillation, which can be used for soap and many products. By a suitable bleach the product can be made generally lighter in color than the original fat.

5. With higher grade fats, the glycerine concentration in the sweet water can be 13 to 18%, or in general more concentrated than obtained by the Twitchell method. A light lime treatment is all that is required to coagulate impurities and the concentrated glycerine is practically ash free.
6. The process has exceptional heat economy, splitting more than five pounds of fat for every pound of high pressure steam used.
7. All of the inherent advantages of a continuous and countercurrent process are realized, including ease of control and operation, uniformity of product, low labor cost, high through-put, low uniform steam and power consumption, small space requirement, and low interprocess inventory.
8. The overall cost of splitting is less than by usual methods.

The process carries out in a modern continuous way an operation which has for almost a century been handled by a batch method. Since it is a continuous process it fits well with any other continuous process, for example those processes involving solvent extraction prior to splitting and distillation, solvent separation [Emersol process (7)], and neutralization (for the manufacture of soap) following splitting.

Fatty acids are basic chemical building blocks and can be used as intermediates for a multitude of products. By fractional distillation and solvent crystallization individual fatty acids can be prepared which can be used as starting materials to make specific chemical compounds.

The standard process can be modified to meet specific requirements. In the case of certain special products, such as drying oils, it may be desirable to operate at a somewhat lower temperature and pressure, even though the column size must of necessity be larger. For plants having capacities less than about 3,000 lbs. per hour, it may be good engineering to use a lower pressure, thereby having a column size which can be more easily fabricated, and will afford access for inspection of the interior. By the use of a small amount of catalyst the through-put can be increased greatly or the operating temperature may be lowered.

In addition to its use in fat splitting, the process may be of general interest for carrying out certain types of chemical reactions. Any two immiscible (or partially miscible) liquids which react reversibly at elevated temperature and pressure might be handled in the same type of plant. As an example, the hydrolysis of chlorobenzenes with aqueous caustic to form phenols might be carried out in this type of apparatus.

The problems involved in the commercialization of this method of operation have been successfully solved. Commercial plant results have demonstrated that most of the commercial fats can be split to a high degree and at a low cost. The process is finding wide application for purposes such as fatty acid manufacture and soap making. The general processing method and plant equipment may be adapted to other chemical reactions between two immiscible liquids.

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Preparation and Properties of Cottonseed Protein Dispersions[1]

JETT C. ARTHUR, JR., and MELVIN K. KARON, Southern Regional Research Laboratory,[2] New Orleans 19, Louisiana

THE utilization of vegetable proteins in the manufacture of fibers, films, adhesives, paper and textile sizes, cold water paints, and related products is dependent on various physical properties of the proteins *per se* and of their aqueous dispersions. A number of these properties, *e.g.*, dispersibility in various media and the viscosity characteristics of concentrated dispersions have been investigated in the case of soybean and peanut proteins. Similar but less extensive investigations have been reported for cottonseed proteins. These investigations include the factors which influence the peptization of protein in solvent-extracted and hydraulic-pressed cottonseed meal (1), cottonseed proteins prepared under different processing conditions (2), the peptization of cottonseed proteins by various acids and bases (3, 4), the pigments of cottonseed (5, 6, 7, 8, 9), methods for separating the pigment glands from cottonseed (8, 10), and the viscosity characteristics of relatively concentrated dispersions of cottonseed protein (11).

The last mentioned article is also concerned with the difficulties of avoiding gel formation during the preparation of cottonseed protein dispersions in high protein concentration and in preparing dispersions having tacky and viscous characteristics.

The industrial utilization of cottonseed meal and proteins has heretofore been retarded because the extracted proteins are not appreciably dispersible in aqueous alkali solutions below pH 11.7. The present report is concerned with an investigation of methods for preparing nongelling dispersions of cottonseed

[1] Presented at the 21st Fall Meeting of the American Oil Chemists' Society, October 20-22, 1947, at Chicago, Illinois.

[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

proteins in high protein concentration which have good tackiness and desirable viscosities for industrial use.

Experimental

Cottonseed Meal. A quantity of cottonseed was cracked and flaked, and the pigment glands, meal, and most of the oil were separated by means of the mixed solvent flotation process (8, 10). The meal was re-extracted with *n*-hexane to remove the residual oil, after which the bulk of the solvent was removed from the meal by aeration at room temperature, and the meal was further dried in a vacuum drier at a temperature below 99°C. The dried meal contained 0.60% oil, 9.33% nitrogen, and 0.02% gossypol, calculated on a moisture-free basis.

Cottonseed Protein. In order to provide a basis for selecting the conditions for extracting and precipitating the cottonseed protein the optimum pH of the extracting alkali and the precipitating acid solutions were determined as a function of percentage of nitrogen dispersed. In Figure 1 the percentage of total

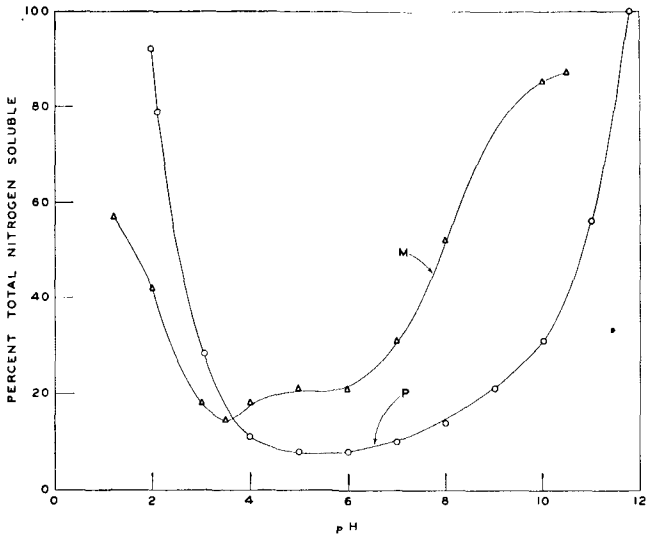

FIG. 1. Effect of pH on the nitrogen solubility of cottonseed meal (M), isolated protein (P).

nitrogen dispersed by alkali and acid from cottonseed meal or isolated protein is shown as a function of the pH of the extracting solution. The optimum concentration of the extracting salt solution was determined in a similar manner. When sodium sulfite concentrations of 0.05, 0.10, 0.20, and 0.40 N at pH 7.5 were used for extraction, the percentages of nitrogen dispersed were 55, 55, 73, and 80, respectively. A 0.20 N solution of sodium sulfite was selected as the extraction medium because the use of

TABLE 1

Effect of Trichloroacetate Ion on 20% Dispersions of Cottonseed Protein (Na_2SO_3-SO_2) and 14% NaOH[a] at 25°C. and pH 12.5.

Trichloroacetate ion, per cent[a]	Microscopic appearance	Relative tackiness
0.00	Coacervates	.....[b]
1.25	Clear	200
2.50	Clear	300
3.75	Clear	125
7.50	Coacervates	100
15.00	Coacervates	.....[b]
20.00	Two phases	.....[b]
30.00	Two phases	.....[b]

[a] Expressed as weight per cent of protein.
[b] No elongation of extruded protein filament before breaking.

TABLE 2

Effect of Method of Preparation of Cottonseed Protein on Its Properties

Extractant pH	Precipitant pH	Analytical data			Dispersion	
		N,%	P,%	Ash,%	Microscopic appearance	Relative tackiness
NaOH 9.1	HCl 4.2	16.6	0.86	0.89	Coacervates	.....[a]
NaOH 9.1	H_2SO_4 4.0	15.4	1.12	3.4	Coacervates	.....[b]
NaOH 9.1	SO_2 4.4	15.2	0.92	3.2	Coacervates	.....[a]
Na_2SO_3 0.2N 7.4	SO_2 4.0	16.4	0.35	1.9	Clear	300
Na_2SO_3 0.2N 7.5	H_2SO_4 4.0	16.4	0.30	1.9	Clear	100
Na_2SO_4 0.2N NaOH 7.0	H_2SO_4 4.0	16.1	0.70	2.8	Clear	.....[b]
Na_2SO_4 0.2N NaOH 7.3	SO_2 4.0	16.4	0.52	2.2	Clear	.....[b]

[a] Slight elongation of extruded protein filament before breaking.
[b] No elongation before breaking.

higher concentrations of this salt gave only slight increase in the amount of nitrogen dispersed and resulted in a small increase in the final yield of isolated protein.

The cottonseed meal prepared as described above was suspended in a dilute solution of alkali or salt in the ratio of 10 liters of solution to 1 kilogram of meal, and the suspension was stirred for 2 hours at room temperature, after which the extract was separated from the insoluble residue by centrifugation. The protein was precipitated from the alkaline extract by the addition of acid, and the supernatant liquor was decanted. The precipitated protein was washed several times with water and twice with acetone, after which it was air-dried at room temperature (12). This procedure was followed for the preparation of a number of protein products in the course of which various alkalies or acids were used. The details of the preparation and composition of these products are summarized in Table 2.

Apparatus. The absolute viscosities of the concentrated protein dispersions were measured with a Höppler rolling ball viscometer. The theoretical aspects of this viscometer are discussed by Höppler (13) and its application by Hubbard and Brown (14). Burnett, Roberts, and Parker (11) discussed the advantages of the use of this instrument in determining the rheological properties of concentrated protein solutions. The viscometer balls were calibrated by means of standard viscosity oils provided by the National Bureau of Standards. Prior to use, the viscometer and accessories were washed with water and ethanol and dried with air.

A constant temperature viscosity bath fitted with a motor-driven stirrer, cooling coil, mercury thermoregulator, and electronically controlled heating coil was used to maintain the viscometer and its contents at temperatures constant within ±0.02°C. during the period required for making the determinations.

To evaluate the relative stringiness or tackiness of the concentrated cottonseed protein dispersions an instrument was constructed which consisted essentially of a motor-driven 100-ml. syringe, a 40-hole

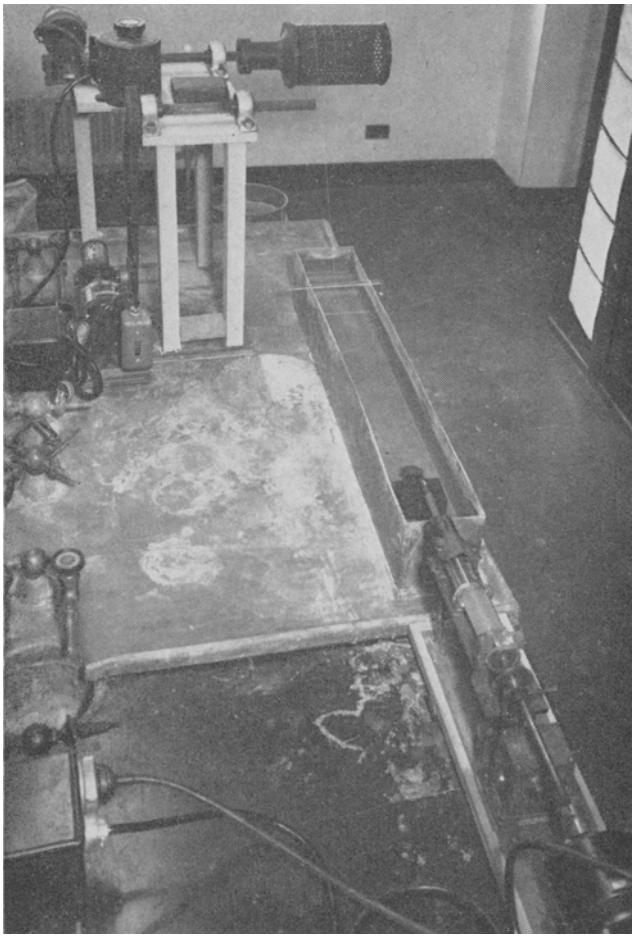

FIG. 2. Apparatus for determining tackiness of protein dispersions.

platinum jet similar to that used for extruding protein fibers, and a precipitating bath, 4x5x36 inches.

The design and method of operation of this instrument is illustrated in Figure 2. This apparatus was applied in determining tackiness as follows. Protein dispersion was ejected from the syringe through the 40-hole jet at a fixed rate of flow into a bath containing an aqueous solution of 2% hydrochloric acid and 10% sodium chloride. The protein coagulate was guided to the end of the bath and onto a revolving godet. The speed of the godet was increased until the 40-filament tow of coagulated cottonseed protein dispersion broke. From the rate at which the dispersion was ejected from the syringe and the peripheral speed of the godet at the time the filament broke, it was possible to calculate the maximum percentage of elongation that the filament underwent in the coagulating bath prior to breaking. The percentage of elongation was taken as the relative measure of the stringiness or tackiness of the protein dispersion.

Viscous Dispersions. Ordinarily when efforts are made to prepare viscous alkaline dispersions of cottonseed protein in concentrations of 5 to 25%, the resulting products are gels, and microscopic examination of these gels show that they are not clear but contain particles and coacervates of protein. These products also exhibit poor tackiness. The effect of specific ions upon the physical properties of colloidal solutions in preventing or delaying gel formation has been reported many times. As early as 1887 Hofmeister (15) and Lewith (16) reported the effect of specific ions upon the physical properties of protein dispersions. Höber (17) refers to similar observations of the effect of specific ions on the viscosity of protein dispersions.

In the present investigation cottonseed protein dispersions were prepared with the aid of anions selected from the Hofmeister series which had been shown to delay or prevent gel formation in colloidal solutions and particularly in protein dispersions. Among the anions investigated were trichloroacetate, borate, citrate, and nitrate. Data covering the effect of the trichloroacetate ion on aqueous cottonseed protein dispersion are summarized in Tables 1 and 2 and Figures 3 and 4.

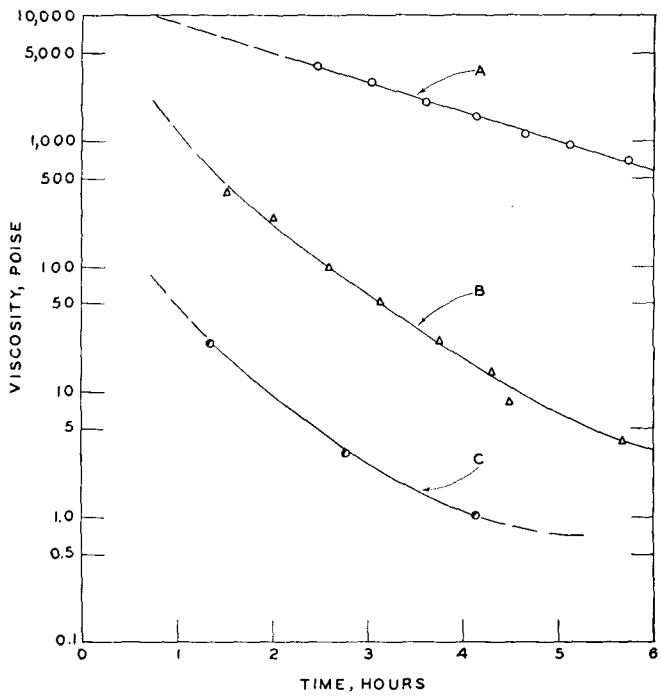

FIG. 3. Effect of sodium hydroxide on the viscosity of a dispersion of 20% protein and 2.5% trichloroacetate at 25°C. and pH 12.5; (A) 12% NaOH, (B) 14% NaOH, (C) 16% NaOH.

Data relative to the effect of the trichloroacetate ion on alkaline dispersions of cottonseed protein (Na_2SO_3-SO_2 preparation, Table 2) in 20% concentration at 25°C., pH 12.5 and 14% of sodium hydroxide expressed as weight-per cent of total protein are shown in Table 1. From these data it may be seen that with increasing concentration of the trichloroacetate ion the relative tackiness of the protein dispersion increases to a maximum at 2.50 weight-per cent of protein, after which the relative tackiness decreased sharply.

Data relative to the effect of the method of preparing cottonseed protein on the relative tackiness of its dispersion in 20% concentration in the presence of 2.50 weight-per cent trichloroacetate ion and 14 weight-per cent sodium hydroxide at pH 12.5 and 25°C. are shown in Table 2. These conditions correspond to those at which the Na_2SO_3-SO_2 preparation (Table 1) exhibited maximum tackiness.

The data in Table 2 show that the method of preparation of the protein effects the tackiness of alka-

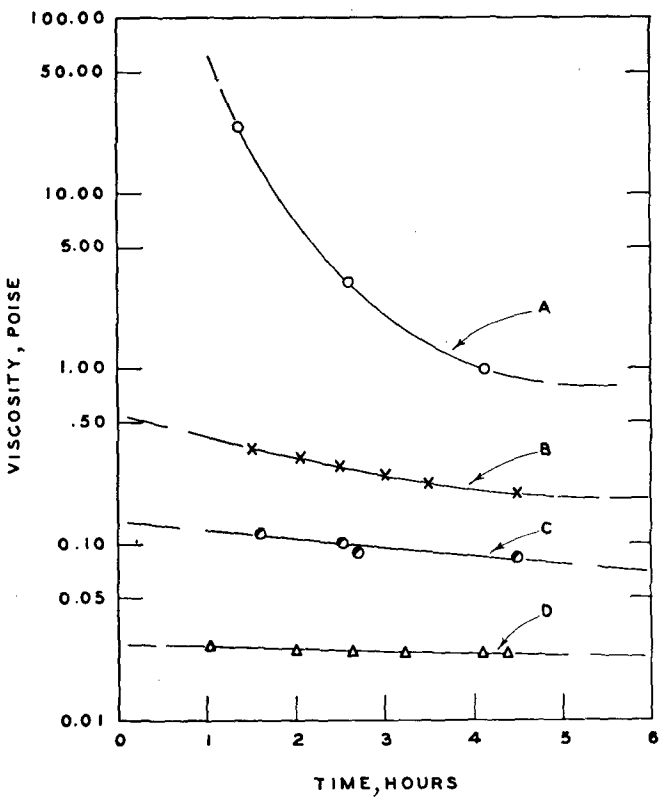

FIG. 4. Effect of protein concentration on the viscosity of its dispersions in 16% NaOH and 2.5% trichloroacetate at 25°C. and pH 12.5; (A) 20% protein, (B) 15% protein, (C) 10% protein, (D) 5% protein.

line dispersions. For example, the protein extracted by sodium sulfite gave alkaline dispersions having greater relative tackiness than proteins extracted with sodium hydroxide or with sodium hydroxide plus sodium sulfate.

The dependence of the viscosity of alkaline protein dispersions on the sodium hydroxide concentration is shown in Figure 3. With increasing concentrations of sodium hydroxide the viscosities of the dispersions decrease sharply.

The dependence of the viscosity of dispersions on the concentration of protein is shown in Figure 4. With increasing concentrations of protein the viscosities of the dispersions increase sharply.

Acknowledgment

The authors wish to thank the Engineering and Development Division for supplying the solvent-extracted cottonseed meal used in this investigation and the Analytical, Physical Chemical and Physical Division for determining the nitrogen and residual oil contents of the meal.

Summary

1. A method has been described for preparing non-gelling, tacky dispersions of cottonseed protein in which trichloroacetate ion has been used to prevent gelation.

2. It has been shown that the properties of isolated cottonseed protein depend on (1) the method of isolating the protein (2) the concentration of protein in the dispersion, (3) the concentration of sodium hydroxide used to disperse it, and (4) the addition of trichloroacetate ion to prevent gelation.

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