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Edited by D. R. HELDMAN



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#### JOURNAL OF FOOD PROCESS ENGINEERING

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### CONTENTS

Meetings vii
Ultrafiltration of Whole Milk with Hollow Fiber Membranes
C. A. GAROUTTE, C. H. AMUNDSON and C. G. HILL, JR., University of Wisconsin, Madison, Wisconsin
Effect of Time and Fish Temperature on the Blunting Rate of Filleting Knives
<b>TADEUSZ MATUSZEK</b> , The Technological University, Gdańsk,Poland203
Effects of Recovery Methods on the Functionality of Protein Concentrates from Food Processing Wastes
D. KNORR, University of Delaware, Newark, Delaware 215
The Effect of Heat and Shear on the Viscoelastic Properties of Soy Flour Dough
<b>D. G. BAIRD</b> , Virginia Polytechnic Institute and State University, Blacksburg, Virginia
Literature Abstracts
Author Index
Subject Index

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#### MEETINGS

#### **APRIL 1983**

April 5-8: Better Process Control School. University of Wisconsin, Madison, Wisconsin. Contact: C. E. Johnson, Dept. of Food Science, Babcock Hall, University of Wisconsin, Madison, WI 53706.

April 10-14: TEMA 83–4 International Trade Fairs: Food Fair, Hotel and Restaurant, Food Tech and Pak Tek, Bella Center. Copenhagen, Denmark. Contact: Bella Center A/S, Center Blvd., DK-2300 Copenhagen S, Denmark.

April 11-13: 64th Annual Meeting of the Dairy and Food Industry Supply Association. Boca Raton Hotel and Club, Boca Raton, Florida. Contact: F. J. Greiner, Dairy and Food Industry Supply Association, Inc., 6245 Executive Blvd., Rockville, MD 20852.

April 11-14: Better Process Control School. University of California, Davis, California. Contact: R. C. Pearl, Dept. of Food Science and Technology, University of California, 250 Cruess Hall, Davis, CA 95616.

April 18-21: Better Process Control School. Purdue University. Contact: L. F. Chen, Food Sciences Institute, Purdue University, West Lafayette, IN 47907.

April 25-28: Better Process Control School. Penn State University. Contact: G. D. Kuhn, Dept. of Food Science, 116 Borland Bldg., Penn State University, University Park, PA 16802.

#### **MAY 1983**

May 9-12: Better Process Control School. Cornell University, Rochester, NY. Contact: D. L. Downing, Dept. of Food Science and Technology, Cornell University, Geneva, NY 14456.

#### **JUNE 1983**

June 19-22: 43rd Annual Meeting of the Insitute of Food Technologists and Food Expo. New Orleans, LA. Contact: C. L. Willey, Institute of Food Technologists, Suite 2120, 221 N. LaSalle St., Chicago, IL 60601. June 26-29: Summer Meeting of the American Society of Agricultural Engineers. Montana State University, Bosman, Montana. Contact: Mark A. Purschwitz, American Society of Agricultural Engineers, PO Box 410, St. Joseph, MI 49085.

#### **SEPTEMBER 1983**

September 1-9: The 16th International Congress of Refrigeration. Paris, France. Contact: Joseph W. Slavin, American Society of Heating, Refrigerating and Air-Conditioning Engineers, 1791 Tullie Circle, NE, Atlanta, GA 30329.

September 18-23: 6th World Congress of Food Science and Technology. Dublin, Ireland. Contact: Dr. R. L. Joseph, An Foras Taluntais, Dunsinea, Castle Knock Co., Dublin, Ireland.

September 26-28: 3rd International Congress on Engineering and Food. Dublin, Ireland. Contact: ICEF Secretariat, Institute of Engineers of Ireland, 22 Clid Rd., Dublin 4 Ireland.

#### ULTRAFILTRATION OF WHOLE MILK WITH HOLLOW FIBER MEMBRANES

#### C. A. GAROUTTE and C. H. AMUNDSON

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Whole milk was concentrated using Romicon PM50 membranes. Rententate and permeate were analyzed for total solids, protein (TKN) and fat at five concentration levels. The effects of pressure, concentration, fiber length and several milk pretreatments on flux were examined. A 5X concentrate was prepared containing 37.2% solids, 13.3% protein and 17.8% fat. The highest initial and 4X fluxes were obtained using a pressure differential of 1.4 kg/cm<sup>2</sup> - 2.11/0.7 (20 psig - 30/10). Flux declined slowly with concentration to 3X (20% loss) and decreased more rapidly thereafter. Higher fluid velocities were deemed responsible for 27% higher initial flux and 47% higher 4X flux when a shorter module was employed (63.5 versus 109.2 cm). Clarification, pasteurization and/ or homogenization had little effect on flux at either level.

#### **INTRODUCTION**

There is interest in the ultrafiltration of whole milk because of its potential importance in cheese manufacture. The ultrafiltration of skim milk has already proven to be a desirable pretreatment step in the commercial manufacture of various soft cheeses (Chapman *et al.* 1974; Matthews *et al.* 1976; Maubois and Mocquot 1976). The advantages of using ultrafiltration include the incorporation of whey proteins in the cheese and increased production capacity (Matthews *et al.* 1976; Maubois and Mocquot 1976).

In cheese operations currently using ultrafiltration, the milkfat is removed before the milk is ultrafiltered. If hard cheeses are to be manufactured by ultrafiltration, it may be desirable to concentrate whole milk directly and, therefore, eliminate separation. There have been few studies on the ultrafiltration of whole milk because of the fouling problems that occurred when lipid-containing materials were ultrafiltered on cellulosic membranes. These problems are minimal on the new anisotropic-polymeric membranes. The hollow fiber features a high membrane area-to-volume ratio which makes it promising for this application (Baum *et al.* 1976). Of the studies reported on whole milk (Chapman *et al.* 1974; Glover 1971; Thompson and de Man 1975; Yan *et al.* 1979), few have gone beyond a three-fold concentration and only Thompson and de Man (1975) used hollow fibers.

This work was conducted to evaluate the usefulness of ultrafiltration in cheese making. The following variables were studied: (1) flux and its dependence on operating pressure, concentration and fiber length, (2) the composition of both process streams during concentration and (3) the effect of several milk pretreatments on flux.

#### MATERIALS AND METHODS

#### **Materials**

Apparatus. A modified, model HF 2SSS ultrafiltration unit (Romicon, Inc., Woburn, Massachusetts) was used. A larger pump was installed for higher operating pressures and velocities. Sanitary disc valves were placed at each end of both modules so that the modules could be used independently or simultaneously. One long module (109.2 cm) and one short module (63.5 cm) were used in parallel with respective membrane areas of 2.47 and 1.39 m<sup>2</sup>. The PM50 polysulfone membrane with a molecular weight cutoff of 50,000 daltons and an inside diameter of 1.1 mm was used. The recommended maximum operating pressure and temperature were 1.76 kg/cm<sup>2</sup> (25 psig) and 75°C, respectively.

Figure 1 is a flow diagram of the ultrafiltration system. The system included a 378.5 liter (100 gal) steam-jacketed, feed tank, a 7.5 horsepower centrifugal pump, a stainless steel prefilter, two modules, sanitary disc valves, and temperature and pressure gauges.

**Milk Supply**. Raw whole milk was obtained from the University of Wisconsin dairy plant. The milk was received at  $4^{\circ}$ C and stored at that temperature until used. Storage never exceeded four hours. Milk pretreatments included pasteurization (63°C for 30 min) and homogenization using a two-stage Manton-Gaulin Type 125 "E" homogenizer at 140/35 kg/cm<sup>2</sup> (2000/500 psig).

#### Methods

Chemical.(1)Fat-Mojonnier ether-extraction method of Atherton and Newlander (1977).

(2)**Protein**–Semi-micro method for total Kjeldahl nitrogen (X 6.38) as described by Bradstreet (1965).

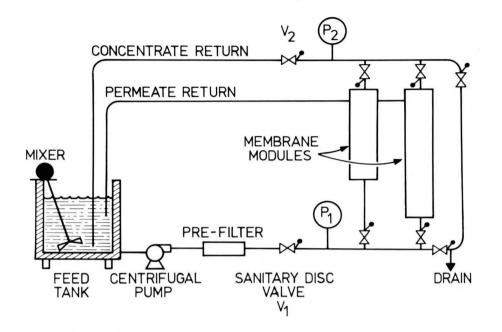


FIG. 1. FLOW DIAGRAM OF THE ULTRAFILTRATION SYSTEM

(3)**Total Solids**-Mojonnier vacuum-oven method of Atherton and Newlander (1977).

#### Procedure

Startup. The system was drained and sanitized with 200 parts per million (ppm) available chlorine (Wyandotte Antibac B) at 55°C for 10 min using inlet and outlet pressures of 2.11 and 0.35 kg/cm<sup>2</sup> (30 and 5 psig), respectively. Following the sanitation cycle, the system was flushed with 189.3 liters (50 gal) of softened water and the soft water permeate flux measured at 55°C using 1.76 and 1.05 kg/cm<sup>2</sup> (25 and 15 psig) as the inlet and outlet pressures. A clean membrane was characterized by a soft water flux of  $\geq$  305 gallons per square foot of membrane per day (GSFD) on 3.86 m<sup>2</sup> of membrane area. If flux was satisfactory, the tank and modules were drained and the tank filled with milk.

**Operation**. The milk was pumped out of the feed tank and through the membranes by the high speed centrifugal pump. Operating pressures were adjusted to the desired value. Feed temperatures were monitored by thermometer and held at  $55^{\circ}C\pm 1^{\circ}C$  using periodic injections of steam or cold water. Uniformity of feed temperature and feed composition was maintained by a rotary mixer attached to the feed tank.

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The following data were recorded: (1) nature and pretreatment of feed, (2) inlet and outlet pressures, (3) feed temperature, (4) time and (5) flux. Samples of permeate and retentate were taken after one min of operation at zero recovery. Permeate samples were stored at -27.8 °C until analyzed. Retentate samples were stored at 4 °C and analyzed within 24 h.

Flow rates and flow velocities were determined in triplicate by measuring the volume of concentrate and permeate collected in a precalibrated portable vat over a unit time span. Fluxes were monitored by measuring the volume of permeate separated with time or by measuring pounds removed and converting to volumetric units using densimetric measurements. These measurements were performed in quintuplicate.

**Cleaning**. All cleaning operations were conducted at zero recovery with permeate ports open. The feed tank and modules were drained and rinsed with 378.5 liters (100 gal) of softened water (55°C) immediately following operation. A 1% (w/w) solution of Tergazyme (proteolytic detergent from Alconox, Inc. of New York) was then circulated through the system for about one minute at 55°C with inlet and outlet pressures of 2.11 and 0.35 kg/cm<sup>2</sup> (30 and 5 psig), respectively. This cleaning solution was left overnight in the module to remove proteinaceous deposits on the membrane.

The cleaning solution was drained the next morning and flushed from the system with 378.5 liters (100 gal) of softened water at the same operating temperature and pressure. After rinsing, a 1% (w/w) solution of sodium hydroxide was circulated through the system for 20 min. This solution was then drained and the tank and modules rinsed with 378.5 liters (100 gal) of softened water. A solution of 10 ppm available chlorine was circulated for 10 min and allowed to stand in the system until next use.

A 1.2% solution of formaldehyde was used to store ultrafiltration modules not in frequent use. A 1% (w/w) solution of technical grade hydrochloric acid was used in a twenty minute cleaning cycle after every third or fourth run to maintain a flux above 305 GSFD.

#### **RESULTS AND DISCUSSION**

#### Dependence of flux on pressure, concentration and fiber length.

Since flux affects productivity, high fluxes are desirable and process parameters are adjusted accordingly. Although flux increases with temperature, temperatures between 50 and 55°C are usually the upper limit to avoid denaturation of milk proteins. Pressure, concentration level and fiber length are three additional parameters that can affect the rate of permeate removal.

**Pressure**. A study was conducted to determine the inlet and outlet pressure settings that would result in high flux during operation. Under the limits imposed by the system, four sets of inlet and outlet pressures were evaluated: 1.4 and 0.7, 1.76 and 0.35, 1.76 and 1.05, and 2.11 and 0.7 (inlet and outlet pressures, respectively, in kg/cm<sup>2</sup>). These pressures were chosen in a manner such that the effects of pressure differential ( $\triangle P$ , inlet pressure – outlet pressure) and average trans-

membrane pressure  $(\triangle P_T, \frac{\text{inlet pressure + outlet pressure}}{2})$  could be examined simultaneously. The effects of these pressure variables were investigated at two concentration levels: 1X (0% reduction in initial feed weight) and 4X (75% reduction in initial feed weight). 1X and 4X fluxes were termed initial and final flux, respectively. Reported values represent the average of five measurements after one minute of operation at zero recovery.

Figures 2 and 3 show the effects of pressure differential and average transmembrane pressure on initial and final flux, respectively, using  $3.86 \text{ m}^2$  of total membrane area at  $55^{\circ}$ C. Each bar represents the

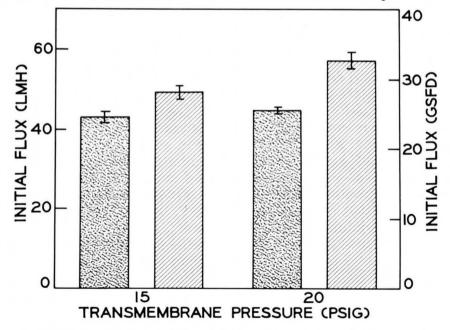


FIG. 2. EFFECTS OF PRESSURE DIFFERENTIAL ( $\triangle P$ ) AND AVERAGE TRANSMEMBRANE PRESSURE ( $\triangle P_T$ ) ON INITIAL FLUX  $B \triangle P = 10 \text{ psig}, (0.7 \text{ kg/cm}^2)$   $\Box \triangle P = 20 \text{ psig}, (1.4 \text{ kg/cm}^2)$ 

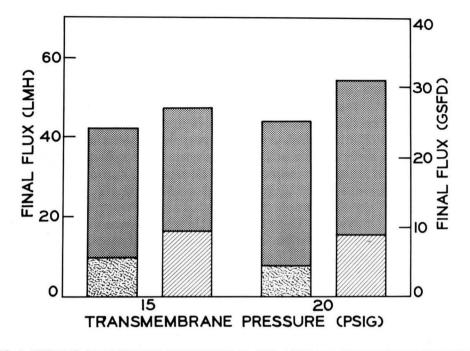


FIG. 3. EFFECTS OF PRESSURE DIFFERENTIAL AND AVERAGE TRANSMEMBRANE PRESSURE ON FINAL FLUX
initial flux per Fig. 2 initial flux △P = 10 psig (0.7 kg/cm<sup>2</sup>)
Initial flux △P = 20 psig (1.4 kg/cm<sup>2</sup>)

average of duplicate runs under one set of operating pressures in kg/ cm<sup>2</sup> (left to right: 1.4/0.7, 1.76/0.35, 1.76/1.05, and 2.11/0.7). The highest initial flux, 54 liters per square meter per hour (LMH), was obtained when  $\triangle P$  and  $\triangle P_T$  were equal at 1.4 kg/cm<sup>2</sup> (2.11/0.7). At both transmembrane pressures, fluxes were highest when the larger  $\triangle P$  (1.4 kg/cm<sup>2</sup>) was used. Although an increase in  $\triangle P_T$  from 1.05 to 1.4 kg/  $cm^2$  at a  $\triangle P$  of 0.7 kg/cm<sup>2</sup> did not substantially increase flux, a similar increase in  $\triangle P_T$  at a  $\triangle P$  of 1.4 kg/cm<sup>2</sup> did cause a significant increase. The results of this study show that initial flux is more dependent on  $\triangle P$ than  $\triangle P_T$  and that operation at large  $\triangle P$ , in this case 1.4 kg/cm<sup>2</sup>, is desirable for high initial flux. The effects of  $\triangle P$  and  $\triangle P_T$  on final flux (4X) were similar to those observed on initial flux. Pressure differential exhibited a greater effect on final flux than transmembrane pressure, and as  $\wedge P$  increased, flux also increased. However, unlike the effects at 1X, the effect of  $\triangle P$  was less pronounced and changes in  $\triangle P_T$  had little or no effect on flux.

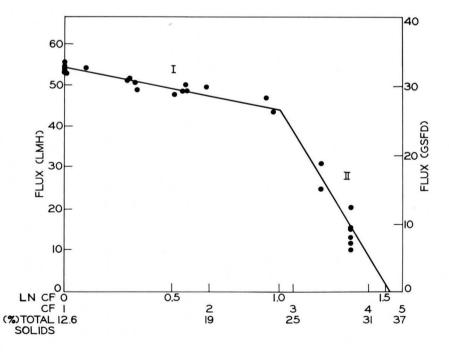
The results of these experiments showed that the highest flux at both concentration levels was obtained when  $\triangle P$  and  $\triangle P_T$  were equal at 1.4 kg/cm<sup>2</sup> (2.11/0.7). This combination of inlet and outlet pressures was used in the remainder of the study.

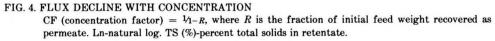
**Concentration**. The relationship between flux and concentration will indicate what concentration levels can be reached by ultrafiltration and, to some degree, how efficiently they can be obtained. Figure 4 shows how flux declined with the natural log of the concentration factor (ln CF) when operating on  $3.86 \text{ m}^2$  of membrane area at  $55^{\circ}$ C using inlet and outlet pressures of 2.11 and 0.7 kg/cm<sup>2</sup> (30 and 10 psig), respectively. The total solids content of the retentate and the concentration factor (CF) have been provided for reference and discussion.

There was a curvilinear loss of flux with concentration, shown here as two lines of best fit through over 40 flux measurements taken in seven identical runs to a 5X concentration level. Two regions of flux decline are evident, labeled I and II. Equations 1 and 2 were developed by regression analyses to describe the flux loss of each region in terms of concentration.

$$J_{\rm I} = 54.1 - 8.82 \ln {\rm CF} \qquad (r = 0.94) \tag{1}$$

$$J_{\rm II} = 110 - 67.8 \ln {\rm CF}$$
 (r = 0.93) (2)



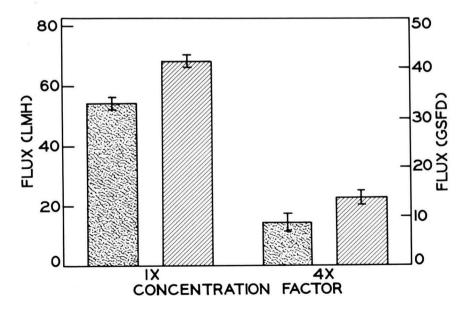


where  $J_I$  and  $J_{II}$  represent fluxes for regions I and II, respectively.

Fluxes began at 54 LMH and decreased to a value of 45 LMH near a three-fold concentration of milk. It is speculated that flux in region I is controlled by the hydrodynamics of the boundary layer as well as the membrane. The loss of flux would then be attributed to the increasing concentration of macro-molecules in the boundary layer. Region II flux began near a three-fold concentration of milk (45 LMH) and appeared to reach 0 LMH near a five-fold concentration. Blatt *et al.* (1970) suggested that transition to region II occurs when certain species present in the boundary layer exceed their solubility limit. A gel or precipitate would then be formed with flux governed primarily by the permeability of the gel. This transition occurred at about 25% total solids in the retentate. A five-fold concentration appears to be the maximum level attainable under these conditions.

Fiber Length. If higher concentrations are desired, the efficiency of permeate removal in region II must be increased. The results in Fig. 5 show that a method is available and that it is especially suited for use at high concentration levels.

The bar graph shows the effects of membrane area and fiber length on initial and final flux using inlet and outlet pressures of 2.11 and 0.7 kg/cm<sup>2</sup>, respectively. The waved bars correspond to the combined



membrane area of the long and short modules  $(3.86 \text{ m}^2)$  and the hatched bars to the membrane area of the short module alone  $(1.39 \text{ m}^2)$ . Each bar is the average of four measurements taken in four identical runs.

When the long module was closed at the 1X level and the pressures readjusted to their original values (2.11 kg/cm<sup>2</sup> inlet, 0.7 kg/cm<sup>2</sup> outlet) for the short module alone, there was a 40% decrease in flow rate through the system. However, the fluid velocity increased 15% resulting in a 23% increase in membrane flux. This was a rise in flux from 55 to 68 LMH.

When the long module was closed at the 4X level, there was a 33% reduction in flow rate through the system, a 35% increase in fluid velocity and a resultant 47% increase in flux. It increased from 15 to 22 LMH.

It is assumed that the higher flux is the result of increased fluid velocity through the shorter module. Although the short module exhibited higher flux at both levels, it is more beneficial at the 4X level. This suggests that shorter modules should be used at high concentration levels to increase the efficiency of permeate removal.

#### **Retentate and Permeate Composition**

**Retentate**. Since the level of concentration needed for cheese manufacture is unknown, the composition of the retentate was determined at five concentration levels (Table 1). The values represent the average of triplicate runs. A five-fold concentration of milk was the highest level tested. At this level, the retentate contained 37.2% total solids, 13.3% protein (TKN) and 17.8% fat. Since this is near the maximum concentration level obtainable under the conditions used and the solids content of a finished low moisture cheese is roughly 60%, it is evident that the solids content of such a cheese can not be reached by ultrafiltration

Concentration	Total	Protein	Fat
Factor	Solids	(TKN)	q
	$\overline{100g}$	$\overline{100g}$	$\overline{100g}$
1X	$12.56 \pm 0.25$	$3.22 \pm 0.17$	$3.75 \pm 0.09$
2X	$18.99 \pm 0.21$	$5.71 \pm 0.10$	$7.50 \pm 0.01$
3X	$25.01 \pm 0.20$	$8.46 \pm 0.04$	$10.91 \pm 0.08$
4X	$31.10 \pm 1.22$	$10.91 \pm 0.41$	$14.62 \pm 0.28$
5X	$37.18 \pm 0.33$	$13.33 \pm 0.09$	$17.80 \pm 0.04$

Table 1. Average retentate composition at five concentration levels

Homogenized, pasteurized whole milk

3.86 m<sup>2</sup> of HF-43PM50 membranes

 $\triangle P$  and  $\triangle P_T = 20$  psig (1.4 kg/cm<sup>2</sup>)

alone. If such levels are to be attained, the concentrate will require fortification with additional solids and/or an extra concentration step, such as thermal evaporation.

**Permeate**. Table 2 shows the average composition of the permeate stream at the same concentrations. These, also, are the average of triplicate runs. The solids content of the permeate stream was 5.5% at the 1X level and increased to 6.5% at 5X. The protein (TKN) content increased from 0.1 g/ml to 1X at 0.7 g/ml at 5X. The rejection of nitrogenous matter remained essentially constant at 95%. Some whey proteins were detected in the permeate during the initial stages of concentration. This was determined by addition of 12% (w/v) trichloroacetic acid. Fat was completely retained by the membrane even though globule size had been reduced by homogenization.

#### Effects of Several Milk Pretreatments on Initial and Final Flux

Since various pretreatments such as clarification, pasteurization and, possibly, homogenization may be desirable prior to cheese making, their effect on flux was determined.

Clarified, pasteurized and combined pasteurized and homogenized milk samples were prepared and fluxes determined at 1X and 4X. Unclarified raw milk was used as the control. Except for the pasteurized sample, the initial (1X) flux of the treated milks was not statistically different at the 95% confidence level. The average initial flux of pasteurized-homogenized milk (51 LMH) was about 3 LMH lower that the control. However, at 4X, the difference in flux between the control and the treated milk was not statistically significant at the 95% confidence level.

Concentration	Total	Protein	Fat
Factor	Solids	(TKN)	a
	$\overline{100g}$	$\overline{100g}$	$\overline{100g}$
1X	$5.51 \pm 0.07$	$0.13 \pm *$	0.0
2X	$5.78 \pm 0.03$	$0.25 \pm 0.02$	0.0
3X	$5.98 \pm 0.06$	$0.31 \pm 0.02$	0.0
4X	$6.22 \pm 0.06$	$0.49 \pm 0.03$	0.0
5X	$6.54 \pm 0.07$	$0.70 \pm 0.05$	0.0

 Table 2. Average permeate composition at five concentration levels

 $\triangle P \text{ and } \triangle P_T = 20 \text{ psig (1.4 kg/cm^2)}$ 

\*<0.01

Homogenized, pasteurized whole milk

<sup>3.86</sup> m<sup>2</sup> of HF-43-PM50 membranes

<sup>55°</sup>C

#### CONCLUSIONS

This work demonstrated that ultrafiltration can be used to pretreat whole milk to at least a 5X concentration. Flux was more dependent on pressure differential than transmembrane pressure with the highest initial and 4X flux obtained using a pressure differential of 1.4 kg/cm<sup>2</sup> (2.11/0.7). Flux declined slowly with concentration to the 3X level (20% loss) and decreased more rapidly thereafter, appearing to reach a value of 0 LMH near a five-fold concentration level. The use of shorter modules at high concentration levels provided a means of increasing flux at these levels. Clarification, pasteurization and/or homogenization did not seriously affect flux initially or at 4X.

#### ACKNOWLEDGMENTS

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#### EFFECT OF TIME AND FISH TEMPERATURE ON THE BLUNTING RATE OF FILLETING KNIVES<sup>1</sup>

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#### ABSTRACT

The blunting rate of the disc knives in the filleting process is influenced by the anatomical location of the cut, cutting time and temperature of the fish body. The head cutting, abdominal-bone cutting and spinal bone separating knives blunt faster due to the structural differentiation in the fish body. The longer cut at the spinal bone separation also increases the blunting rate. Lowering the fish temperature from  $285^{\circ}K$  to  $258^{\circ}K$  (+12 to  $-15^{\circ}C$ ) accelerated the blunting rate of the knives about 2.4 times.

#### INTRODUCTION

The commercial filleting of fish consists of separating fish meat (fillets) from the bones by means of disc knives. The efficiency of the cutting process is affected by the type of deformation of the fish tissue under and around the cutting edge (Matuszek 1972). This deformation depends on the shape of the cutting edges, condition of the blade, cutting speed, and cutting resistances of scales, skin, fish meat and bones. The cutting resistance may vary and is influenced by many factors. It determines the rate of the cutting edge blunting.

In the cutting process, it is necessary to apply a sufficiently large force to overcome the resistance of the fish due to its elastic and plastic deformation, as well as frictional and cohesive forces. Cuting forces on the disc knife are composed of: (a) force initiating the cutting process (i.e., causing contact stresses at the knife/fish interface), (b) forces of frontal resistance on knife edges, (c) frictional forces on the surface of knife wedge, and (d) frictional forces on side surface of the knife (including cohesion and adhesion). By influencing the speed of blunting, these forces affect the effectiveness and the life of the disc knife.

<sup>&</sup>lt;sup>1</sup> Based on the dissertation submitted in partial fulfillment of the Ph.D. degree to the Faculty of Mechanical Engineering at the Technological University, Gdańsk, Poland.

#### T. MATUSZEK

The maximum resistance during fish cutting occurs on the cutting edge, on the so-called intermediary surface created by the external rounding with the arc radius  $\rho_i$  (Fig. 1). The main effect of change in the knife sharpness is noticeable on this surface. Blunting of the knife blade is caused primarily by the backbone, skeleton, crane and the bones of the torso with fins, and secondarily by the general firming up of the flesh influenced by the length and temperature of fish storage.

Blunting is a process of continuous change in the knife microgeometry. Previous studies on the fish cutting process have not generated any specific information on the blunting speed of the knives. Proselkov and Pielejev (1967) and Kawka (1971) made no mention of the allowable knife wear and of the characteristics of the cutting process. Similarly, none of the patent descriptions (Barrete and Carignan 1974; Baader 1969; Rydberg and Petterson 1969; Kloster and Kloster 1969; Michal 1969; Yamanashi 1974; Yoshida 1969) contain any essential details concerning the design of the different machine parts and cutting tools.

Lack of numerical values is particularly conspicuous when it is necessary to determine the real cutting forces and conditions of cutting according to the technological partitioning of the fish. Difficulties with the design of fish fixing clamps occur when the magnitude of the blade

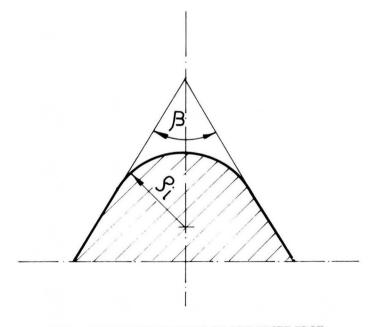


FIG. 1. SCHEMATIC DRAWING OF THE KNIFE EDGE  $\beta = \text{knife angle}, \rho_i = \text{blunting arc radius.}$ 

wear and progressive blunting are neglected. Also, an evaluation of the machine performance depends fundamentally on the knife blunting rate (Druseik and Aboltinsh 1971). Yakubow (1971a, b), who evaluated forces occurring during tuna and big-cod cutting, emphasized the effect of changes in the cutting-edge geometry on the cutting process when interpreting the results. However, he did not explain the issue. Kawka (1972) totally neglected the aspects of knife blunting and assumed incorrectly that knife sharpness is constant during the cutting process. Hence, his interpretations of optimum feed velocity  $(V_p)$  and knife speed  $(V_o)$  must be criticized on methodological grounds. This is further supported by the fact that his experimental results did not correspond to the actual industrial experience.

The purpose of the research described in this paper was to study and quantify the process of knife blunting during redfish filleting.

#### **RESEARCH SCOPE AND METHODOLOGY**

The experimental work was carried out from October to December, 1976, on board of a fishing ship in the North West Atlantic Ocean.

The normal filleting unit Baader 150 equipped with standard disc knives was used for processing fresh redfish within 2 h of catch (Fig. 2). The nine knives used in filleting of redfish were made of steel 4H13 and had the following edge angles: 22° for the decapitating knives ( $O_q$ ,  $O_d$  in Fig. 2), 20° for the abdomen slitting knives (B), 25° for the rib cutting knives ( $W_1$ ,  $W_p$ ), 22° for the lower filleting knives ( $D_1$ ,  $D_p$ ), and 30° for the upper filleting knives ( $G_1$ ,  $G_p$ ). The speed coefficient  $\lambda = V_0/V_p$  was 38 for  $O_g$ ,  $O_d$  knives and ranged from 10.0 to 11.6 for the other knives. Knives which cut belly slices and are normally part of the Baader 150 unit were not used in filleting of redfish.

The hardness of the knife surfaces was about  $55^{\circ}$  HRC. The roughness of those surfaces was identical for the sharp and blunt knives and had values from 1.27 to 1.40  $\mu$ m. Surface hardness was measured with the PMT-3 micro-hardness tester against scale 3 according to Rockwell. The surface analyzer C. Zeiss Jena ME-10 was used to evaluate the surface and measure its roughness.

For the evaluation of blunting, the radius  $\rho_i$  of the circle inscribed in the edge angle of the disc knife was measured (Fig. 2) by taking imprints in plastic silicone (Dentaflex manufactured in Czechoslovakia and Lastic 3c manufactured in West Germany) and quantifying the radius with a metallographic microscope Ergaval with occular attachment (C. Zeiss, Jena). After establishing the enlargement, the picture

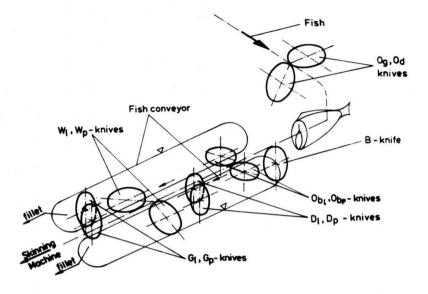


FIG. 2. SCHEMATIC OF THE FILLETING MACHINE BAADER 150 WITH STANDARD DISC KNIVES

 $O_g$ ,  $O_d$ -head cutting, B-abdomen slitting,  $Ob_1$ ,  $Ob_p$ -belly cutting,  $D_1$ ,  $D_p$ -lower filleting,  $G_1$ ,  $G_p$ -upper filleting,  $W_1$ ,  $W_p$ -abdominal bone cutting.

on the focus screen and the master radius were compared and measured. Each of the reported values is an average of four imprints made of areas located equidistantly on the knife cutting edge.

A sharp knife had a radius of  $5\mu$ m. The blunting limit of  $45 \mu$ m was considered as the maximum still acceptable from the standpoint of high-quality filleting (Matuszek 1981). Blunting greater than  $45\mu$ m lowers the quality and efficiency of the filleting process by about 30% since the strips can be used only for fish meal. In the course of this research the author concluded that for knives with  $\rho_i > 45\mu$ m, the cutting line did not coincide with the determined cutting planes for certain sections of the fish. The fillets obtained by cutting with such blunt knives contained pieces of fins, bones and traces of black peritoneum.

Measurements of knife wear were made during filleting of fresh fish  $(275-278^{\circ}K)$  and during decapitation of fish previously quick frozen to  $258^{\circ}K$  and tested frozen  $(258^{\circ}K)$  and thawed  $(288^{\circ}K)$ . Measurements for the different elementary operations on fresh fish were made up to 50 h of effective work of an originally sharp knife. Measurements for the decapitation of frozen and thawed fish were made up to 20 min of operating time.

#### BLUNTING RATE OF FILLETING KNIVES

#### RESULTS

#### **Effect of Time**

Progressive blunting of disc knife edges during the filleting process of fresh fish is shown in Table 1, and a summary of the statistical evaluation of the data is given in Table 2. It will be seen that knife  $O_d$ reached the limiting radius value after 40 h. Knives  $O_g$ ,  $W_p$  and  $G_1$ showed the limiting radius value at the time of the 50 h measurement. Thus, four out of the nine knives in the set were too blunt after 50 h of continuous use to produce good quality fillets. The severest wearing out took place during the decapitation process (knives  $O_d$ ,  $O_g$ ) due to the thickest skin, greatest quantities of bone and gristle, and greatest cohesiveness of the fish body structure.

#### **Effect of Temperature**

Table 3 shows the effect of fish temperature on the changes in knife edge radius over a 20 min working perod for beheading knives operating at two throughput rates: 35 and 40 fish per min. Using the method of least squares, the data were fitted to straight lines (Fig. 3 and 4) which indicated that the rate of blunting (slope value) was very similar for the two knives and was highly temperature dependent (r =0.97-0.99). The relationship may be expressed by the following equations:

for 
$$-15^{\circ}$$
 C  $\rho_{Q_1} = 0.58 \ t + 5.6$   
 $\rho_{Q_2} = 0.52 \ t + 6.8$   
for  $+5 \ to +12^{\circ}$  C  $\rho_{Q_1} = 0.10 \ t + 5.8$   
 $\rho_{Q_2} = 0.04 \ t + 5.8$ 
(1)

where t is time in minutes and Q is the throughput rate ( $Q_1 = 35$  fish/min and  $Q_2 = 40$  fish/min).

time (h)	$O_d$	$O_g$	В	$D_1$	$D_p$	<i>W</i> <sub>1</sub>	$W_{p}$	$G_1$	$G_p$
0	8	6	6	8	8	8	8	8	10
5	11	9	9	11	12	10	12	11	14
10	14	12	14	14	16	15	17	14	17
15	21	19	15	19	21	19	24	19	24
20	25	26	25	16	23	26	25	24	21
25	29	31	24	26	26	31	28	26	31
30	35	36	31	30	31	30	34	31	28
35	36	42	31	28	30	36	40	36	34
40	45	44	35	32	36	38	41	42	39
50	48	48	38	36	39	43	45	45	41

Table 1. Changes in knife radius,  $\rho_i$  (µm), with filleting time of fresh fish tested at 275-278°K

Knife	r	C.V. Max.	$S_{\hat{y}}^{z}$	$\pm \overline{y}$	$\pm \triangle p_1$	$\pm  riangle y_i$	$\pm \Delta \overline{y_i}$
0,	0.994	22.8	2.9	26.0-28.5	0.087	1.2-2.3	4.1-4.5
0"	0.995	17.0	2.1	26.1 - 28.2	0.073	1.0 - 1.9	3.5-3.8
B	0.985	16.1	4.1	21.3-24.2	0.01	1.5 - 2.7	4.9 - 5.4
$D_1$	0.973	27.8	5.4	20.2-23.7	0.12	1.7-3.1	5.6 - 6.2
$D_n$	0.995	14.1	6.0	23.3-24.8	0.049	0.7 - 1.3	2.3-2.6
W,	066.0	17.4	3.3	24.2 - 26.9	0.092	1.3 - 2.4	4.3-4.8
W	0.993	11.7	2.3	26.2-28.5	0.007	1.1-2.1	3.6 - 4.1
G,	9660	18.0	1.2	24.7-26.4	0.056	0.8 - 1.5	2.7-2.9
<i>b</i> <sup><i>b</i></sup>	0.978	15.2	5.4	24.2-27.5	0.11	1.7 - 3.1	5.6 - 6.2

Table 2. Statistical evaluation of the change in knife radius with time

 $\pm \overline{y}$  confidence interval for the grand means

 $\pm \bigtriangleup p_1$  – % confidence interval for the regression coefficient

 $\pm \Delta y_i - \text{confidence interval of individual observations}$  $\pm \Delta \overline{y}_i - \text{confidence interval of average values}$ 

	Frozen Fish (258°K)	Thawed Fish (278-285°K)
Time (min)	$Q_1 =$	35 Fish/Min
0	6	6
5	8	6
10	11	6
15	15	6
20	17	7
	$Q_2 =$	40 Fish/Min
0	6	6
5	10	6
10	12	7
15	16	7
20	16	8

Table 3. Effect of time, temperature and filleting rate on decapitating knife edge,  $\delta_1$  (µm)

#### **Mathematical Treatment of the Blunting Process**

The process of knife blunting is linear with time and may be expressed by the equation:

$$\rho_i(t) = \delta(t) + b,$$

where b is a probability variable corresponding to the initial knife edge radius,  $\rho_0$ , and  $\delta$  is the velocity coefficient of knife wear.  $\delta(t)$  is the sum of a large number of components reflecting different physical processes leading to knife blunting which may vary with the type of knife, type of fish and processing conditions such as temperature and throughput.

If one assumes that all the components are of the same order, then the distribution  $\delta(t)$  may be described by means of normal distribution. If b is also assumed to have a normal distribution, it follows that  $\rho(t)$ is a Gaussian process having the parameters:

 $E\{\rho(t)\}$  for distributor, and  $D^2\{\rho(t)\}$  for variance

Tables of Laplace's function were used to find the confidence limits at the confidence level  $\alpha = 0.05$ . For some variables corresponding to the individual observations  $(x_i = t_i, y_i = \rho_i)$  an equation of a straight line passing through these points was determined. This straight line was given in the form of a regression equation  $\hat{y} = a_1 \cdot x + a_2$ . The values of  $a_1$  and  $a_2$  were selected according to Gauss's postulate and a condition of a "minimum squares line" was satisfied.

#### DISCUSSION

It is evident that the most severe blunting takes place on the head cutting knives  $(O_d, O_g)$  during the decapitation process. This is because of greater skin thickness, larger quantities of gristle and bone elements

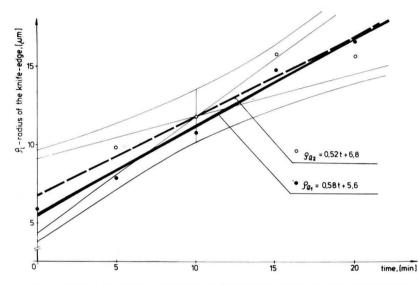


FIG. 3. BLUNTING SPEED OF HEAD CUTTING KNIVES FILLETING FROZEN REDFISH AT -15°C (258°K)

Each point in the diagram is the average of four measurements from imprints on the cutting knife edge. Dark lines represent regression lines for the two throughput rates  $(Q_1 \text{ and } Q_2)$ .

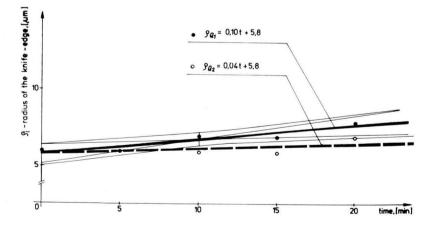


FIG. 4. BLUNTING SPEED OF HEAD CUTTING KNIVES FILLETING REDFISH AT +15 °C TO  $-12^{\circ}$ C (278-285 °K)

Each point in the diagram is the average of four measurements from imprints on the cutting knife edge. Dark lines represent regression lines for the two throughput rates  $(Q_1 \text{ and } Q_2)$ .

and greater cohesion of the internal fish body structure (Dunajski 1980) encountered by these knives. The temperature of the fish has a large effect on the blunting speed since at lower temperatures the fish body acquires a monostructure in the sense of continuous media mechanics (Matuszek 1972). This is an oversimplification since different amounts of water will freeze out at different temperatures. This also affects the anisotropy of the fish body.

The function equation for edge blunting in the filleting unit Baader 150 according to changing factors and based on the analysis of variance (Kulmanowa 1969) (Matuszek 1972) is:

$$\delta(t) = 1.046 \cdot T_s^{0.38} \cdot t^{0.34} \cdot k_f^{2.20 \cdot 10^{-3}} \cdot \lambda^{2.67 \cdot 10^{-4}}$$
(2)

where  $\delta(t)$  is the sum of factors leading to knife blunting,  $T_s$  is fish body temperature (°K), t is time,  $K_f$  is capacity coefficient and  $\lambda$  is velocity coefficient. The value 1.046 partially includes the effect of variable surface and length of fish being cut and the different percentages of structural components in these places.

Comparison of variances of selected elements characteristic of the blunting process shows that the fish temperature has the biggest impact on the knife blunting rate. This confirms the author's hypothesis that blunting occurs faster when the substances being cut have a more cohesive structure. From the above, a correct statement can be drawn on the higher elasticity and brittleness of such biological materials (Kulmanowa 1969), the occurring strains and the high resistance of the medium (Proselkov and Pielejev 1967).

The values of  $\rho_i$  for knives in continuous filleting operations indirectly confirm the values of the elementary resistance during fish cutting reported by Proselkov and Pielejev (1967).

According to the results reported here, these temperature-dependent characteristics of the raw material determine the dynamics of knife wear at different temperatures of the biological medium at equal use times.

Values of the peripheral speed of knife edge ( $V_o$ ), of the fish feed rate ( $V_p$ ) and of the operating rate (Q) could not be varied at will since the knife wear studies were carried out under actual processing conditions. This is the reason for the succession of coefficients  $k_f$  and  $\lambda$  in Equation 2 and their minimum influence on  $\delta / t/$ .

#### CONCLUSIONS

1. In the period 0-50 h of effective work the wear of knife edges in the filleting process follows a linear course of constant blunting velocity.

#### T. MATUSZEK

- 2. Based on the analysis of variance, the temperature of the fish was found to have the greatest effect on the rate of blunting. Lowering the fish temperature from  $+12^{\circ}$ C to  $-15^{\circ}$ C increased the rate of knives blunting 2.4 times during a 20 min work period.
- 3. There is a working time limit for the entire set of 9 knives in the filleting machine of about 40 h of continuous cutting, after which blunting in some knives reaches about  $45\mu$ m and does not permit high quality cutting and filleting of fresh redfish.

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#### EFFECTS OF RECOVERY METHODS ON THE FUNCTIONALITY OF PROTEIN CONCENTRATES FROM FOOD PROCESSING WASTES<sup>1</sup>

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#### ABSTRACT

The multiple effects of the method of protein recovery on food protein functionality are presented. The results indicate the influence of method of protein coagulation (heat coagulation versus coagulation at room temperature) and type of coagulant (HCl, FeCl<sub>3</sub>, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, citric acid) on protein solubility, water binding capacity and baking properties. Dehydration conditions such as air inlet/outlet temperature (120-125/42-42°C to 190-195/70-72°C) or pH of the protein coagulate (pH 4 to 8) as well as methods of dehydration (freeze-, spray-, drum-drying) affected the water binding capacity and protein solubility of plant protein concentrates. Insignificant correlations (P < 0.01) existed between solubility values and water binding capacity data. Marked effects of the interactions between method of dehydration, protein source or dehydration temperature and pH of the coagulate on protein functionality were found.

Based on the data presented it is proposed to develop processes for the production of protein concentrates with "tailored" functionality by selecting specific protein recovery processes and by mixing differently processed protein concentrates.

#### INTRODUCTION

Protein sources for protein concentrates are commonly divided into two groups: conventional or traditional and unconventional or alternative ones (Altschul 1976; Anon 1975). While traditional proteins have become accepted both from the standpoint of desirable organoleptic

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properties and safety for human consumption over the centuries(Circle and Smith 1975; Orr 1978; Pirie 1975), unconventional proteins are relatively new, have been introduced within the past 30 years and do not enjoy wide acceptance (Forsythe and Briskey 1977; Worgan 1973).

Unconventional protein sources are either wastes or byproducts of food production and/or food processing operations or they are presently under-utilized products (Bender *et al.* 1970; Green and Kramer 1979; NAS 1975; Richardson 1975). Examples of unconventional protein sources for the processing of protein concentrates have been given by many authors (Edwards *et al.* 1980; Knorr 1977; Kramer and Kwee 1977; Lusas 1979; Ohlson 1973; Pirie 1971, 1979; Saunders and Kohler 1975; Sosulski 1979; Tannenbaum 1977).

The utilization of food processing wastes for protein concentrate production has several advantages, such as the availability and concentration of large amounts of material and the already available food processing operations. The amounts of these wastes have been reported to range from 8 to 65% of the raw material (Ben Gera and Kramer 1969; Gilde 1972). Pimentel and Pimentel (1979) estimated that 18 million metric tons (dry matter) of waste accumulate annually from U.S. food production.

There is also a need for the recovery of nutrients, especially proteins from food processing wastes to reduce pollution effects of these effluents (Green and Kramer 1979). It is important to stress that the long range goal in food processing operations sould aim towards minimizing food wastes during food processing rather than recycling after processing.

Processing of plant protein concentrates usually consists of three main steps: (1) pretreatment of the raw material to give a protein solution, (2) protein coagulation and (3) dewatering the protein coagulates. Many different methods and processes are used for pretreatment of the raw material. The most common are mechanical operations such as size reduction, expression centrifugation or concentration (Pirie 1975; Kohler and Knuckles 1977; Bramsnaes and Olsen 1979; Lucas 1979), thermal operations such as steam injection or heating (Betschart *et al.* 1975a; Olsen and Anjou 1979) and/or chemical operations such as enzyme inactivation, pH adjustment or enzyme treatment (Knorr *et al.* 1977; Betschart *et al.* 1975a; Grant 1974). Protein coagulation is carried out at various temperatures with or without pH adjustment of the protein solution (Birch *et al.* 1976; Knorr 1980; Pirie 1975), using acids such as hydrochloric acid, sulfuric acid or phosphoric acid. More recently ferric chloride, aluminum sulfate or citric acid has been used for pH adjustment (Dosanjh 1977; Knorr 1980; Meister and Thompson 1976). Heat coagulation is frequently applied and heat exchange is commonly carried out by steam injection (Kohler and Knuckles 1977; Stabile *et al.* 1971; Knorr 1977). This results in protein concentrates of low solubility with limited application in foods (Kinsella 1976). Protein coagulation at room temperature has been used more recently for energy reasons and to reduce thermal denaturation of the proteins (Doe 1977; Finley and Hautala 1976; Meister and Thompson 1976; Knorr 1980).

Filtration (Strolle et al. 1973; Stabile et al. 1971), centrifugation (Meister and Thompson 1976; Rosenau et al. 1978; Kohler and Knuckles 1977), decanting (Peters 1972; Mester and Thompson 1976), reverse osmosis and ultrafiltration (Lawhon et al. 1978, 1979) have been used to concentrate protein coagulates. Hot air drying (Sarkki 1979; Strolle et al. 1973), spray drying (Bramsnaes and Olsen 1979; Kohler and Knuckles 1977), freeze drying (Betschart et al. 1975a; Strolle et al. 1973) and drum drying (Betschart et al. 1975b; Bramsnaes and Olsen 1979) have commonly been applied for the further dewatering.

The term protein functionality is of recent origin and its application has been widened, not always with adequate precision and consistency, over the last 20 years (Bakel 1976; Briskey 1970; Pour-El 1976, 1979). Recently protein functionality has been redefined (Pour-El 1979) as follows: Functionality is any property of a substance besides its nutritional ones, that affects its utilization.

Proteins are added to formulated foods to impart one or more functional properties and to improve nutritional value. Functional characteristics considered in determining which protein to add include foaming, whipping, gelling, fat and water absorption, retention of moisture during processing, dispersion characteristics, solubility, texture, heat stability, freeze-thaw stability, etc. (Kinsella 1976; Schoen 1977). Sensory properties (e.g., color, flavor, taste), "hydrophilic" properties (e.g., water binding, foaming, protein solubility), "hydrophilichydrophobic" properties (e.g., fat binding, emulsification) and textural properties (e.g., softness, elasticity, hardness) are the most important functional properties in food application (Briskey 1970; Kinsella 1976; Knorr 1980).

The objective of this paper was to identify effects of protein coagulation and dehydration of food protein concentrates on the functionality of these products. This is part of an ongoing study to identify functional proteins from food processing wastes by selecting specific protein recovery processes.

#### **EXPERIMENTAL**

#### **Protein Coagulation**

Potato protein concentrates were processed on a pilot plant scale from Russet Burbank potatoes containing 2.05% crude protein and 23% total solids (Knorr *et al.* 1977). Protein coagulation was carried out after grinding the potatoes, diluting the resulting slurry with water to simulate commercial potato processing wastes and after removal of the solids in a horizontal flow, decanter-type centrifuge (Type P-3000 S, Sharples Co., Philadelphia, PA). The resulting protein solution (protein water) contained 1.1% crude protein. Protein was coagulated by steam injection (Edwards *et al.* 1975) at 98°C after adjusting the pH of the protein water to pH 4.8 or at 20 to 22°C by using HCl, or FeCl<sub>3</sub> · 6H<sub>2</sub>O  $Al_2(SO_4)_3$ , or citric acid as coagulants. For optimum protein yield the pH values were adjusted to pH 3 using FeCl<sub>3</sub> · 6H<sub>2</sub>O or HCl, to pH 4 with citric acid, or to pH 5 with  $Al_2(SO_4)_3$  (Knorr 1980).

Rice bran protein concentrate was prepared from PROTEX\* (Riviana Foods, Inc., Houston, TX) a high protein, defatted rice bran produced during the solvent extraction milling of rice (crude protein @ 19%) by heat coagulation at 80°C and pH 5.0 (Connor *et al.* 1976). Soy protein concentrate was prepared from HIZYME 280\* (Central Soya, Chicago, IL), an enzyme-active defatted soy flour (crude protein content @ 56%) by diluting with  $10 \times$  volume of water, adjusting to pH 4.5 with HCl and separating the coagulate.

#### Dewatering

Protein precipitates were recovered using a high speed disk type solid discharging centrifuge (Model BRPX-207S, De Laval Separator Co., Poughkeepsie, NY). Dry protein concentrate powders were obtained after adjusting the pH of each precipitate to 7.0.

Freeze drying was conducted at 25°C in a modified Stokes vacuum oven. Spray drying was carried out using a Bowen laboratory model conical type dryer with inlet and outlet temperature of 210 to 220°C and 105 to 110°C, respectively. A steam heated Buflovak double drum dryer with stainless steel drums of 30 cm diameter by 45.5 cm length was used for drum drying. All protein concentrates were ground by using a hammermill to pass through a 0.28 mm sieve.

Spray drying experiments with commercial heat coagulated potato protein coagulates (Agrar Industrie, Vienna, Austria) were also conducted on a pilot plant scale (Model No. 1, Anhydro A/S Copenhagen, Denmark). The dry matter content of the protein slurry was kept constant at 15% (w./w.) and three different pH levels of the slurry (pH 4, 6 and 8) were studied (Giokas 1977). Air inlet/outlet temperatures at 120-125/40-42, 155-160/54-56, or 190-195/70-72°C were used.

#### Analysis

Total nitrogen, total solids, crude fat and ash were determined according to standard AOAC methods (AOAC 1975). Trichloracetic acid (TCA)/heat treatment as reported by Finley and Hautala (1976) was used to determine TCA coagulable protein in the protein water. Protein solubility was evaluated following a method described by Betschart (1974), except that an ammonia selective electrode was used after Kjeldahl digestion. Water binding capacity and foaming capacity were determined with minor modifications of the methods of Sosulski (1962) and Lawhon *et al.* (1972). Baking experiments were carried out according to the procedures reported earlier (Knorr and Betschart 1978).

#### **RESULTS AND DISCUSSION**

#### **Composition and Protein Functionality**

The data from Table 1 indicate that equally high protein recoveries could be obtained from heat coagulation and coagulation at room temperature with citric acid or ferric chloride. Hydrochloric acid and

Method of Protein Coagulation	Protein Yield (% of Crude Protein in	Total Nitrogen	Crude Fat	Ash
	Protein Water)		(% Dry Matte	r)
		Mean ± Standa	rd Deviation	
Hydrochloric acid at				
98-99°C (pH 4.8)	$35 \pm 2 \ (95)^{a}$	$12.3 \pm 0.3$	$2.4 \pm 0.1$	$7.0 \pm 0.2$
Citric acid at				
22-24°C (pH 4.0)	$39 \pm 2 (105)^{a}$	$6.0 \pm 0.1$	$1.6 \pm 0.2$	$36.3 \pm 0.5^*$
Aluminum sulfate at				
22-24°C (pH 4.0)	$28 \pm 1 \ (76)^{a}$	$7.7 \pm 0.1$	$1.6 \pm 0.1$	$38.2 \pm 0.3^{**}$
Hydrochloric acid at				
22-24°C (pH 3.0)	$23 \pm 1 \ (62)^{a}$	$10.5 \pm 0.1$	$2.3 \pm 0.1$	$20.9 \pm 5.1$
Ferric chloride at				
22-24°C (pH 3.0)	$40 \pm 1 \ (108)^{a}$	$8.8 \pm 0.8$	$1.4 \pm 0.1$	$24.9 \pm 0.5$

Table 1. Protein yield and proximate composition of spray dried potato protein concentrates coagulated under different conditions (after Knorr 1980)

\* ash content after additional washing step:  $1.8 \pm 0.1$ 

" ash content after additional washing step:  $3.4 \pm 0.4$ 

\*\*\* ash content after additional washing step:  $14.7 \pm 2.1$ 

<sup>a</sup> data in parenthesis indicate percent yield precipitated of all TCA/heat coagulable protein  $(37 \pm 2\%)$  of the crude protein)

#### D. KNORR

Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> were less effective coagulants. Coagulation with HCl at 98-99°C resulted in significantly (P < 0.05) higher yields than coagulation with HCl at 22-24°C. Total nitrogen content of potato protein concentrates, coagulated at room temperature, was lower and ash content was significantly higher in comparison to the heat coagulated sample. One possible explanation for the lower ash content in the heat coagulated samples is that modification of the tertiary structure of a protein molecule resulting in separation of binding sites would be expected to be accompanied by loss in binding. An additional washing step during processing of the protein concentrates significantly reduced ash content of protein samples coagulated at 22 to 24°C (Table 1).

A comparison of the effects of different methods of protein coagulation and of additional heat treatment of the protein coagulates on protein functionality is given in Table 2. These data indicate the effect of heat coagulation or heat treatment of the coagulates on protein functionality. Thermal denaturation of protein can significantly reduce protein solubility. Differently heat treated (boiled) protein coagulates resulted in freeze dried protein concentrates of low and insignificantly different protein solubility. In connection with the effects of protein composition and denaturation or solubility, the recent findings by Shen (1981), indicating that the exposed hydrophobic surface area of native (sov) proteins was 50% of that of the denatured proteins, are of major interest. Earlier studies regarding the effect of different dehydration methods of protein coagulates on loaf volume showed increasing loaf volume with increasing heat application during drying (Betschart et al. 1975b; Knorr and Betschart 1978, 1981). Thus loaf volume would increase where raw materials were prepared by freeze-drving, spraydrying, and drum-drying, respectively.

Treatment of Coagulation	Solubility (%)	Specific Loaf Volume* (ml/100g)	Deformation (% Initial Thickness)
	Mea	n ± Standard Devia	ation
Protein coagulation with			
ferric chloride at 22-24°C			
Untreated	$81.0 \pm 2.8^{a}$	$173 \pm 3^{a}$	$10.9 \pm 2^{a}$
Boiled for 20 min.	$14.5 \pm 2.1^{b}$	$271 \pm 5^{b}$	$50.0 \pm 5^{b}$
Protein coagulation with			
hydrochloric acid at 98°C			
Untreated	$12.5 \pm 0.7^{b}$	$162 \pm 5^{c}$	$15.4 \pm 5^{a,c}$
Boiled for 20 min.	$13.5 \pm 2.1^{b}$	$167 \pm 5^{\mathrm{a,c}}$	$10.0 \pm 1^{c}$

Table 2. Effect of heat treatment of potato protein coagulate on the protein solubility and baking properties of freeze dried protein concentrates

white breads fortified with potato protein concentrate (10% replacement of wheat flour)

a,b,c means with different letters indicate significant differences (P < 0.01)

The effects of different methods of protein coagulation at 22 to  $24^{\circ}$ C in comparison with protein coagulation at  $98-99^{\circ}$ C on protein solubility of spray dried potato protein concentrates are shown in Table 3. These data also confirm the effects of heat treatment during protein coagulation as indicated earlier. The use of citric acid or ferric chloride at 22-24°C as coagulant resulted in the highest protein solubility, reaching 85 to 92% at pH 7 in comparison with approximately 12% for the heat coagulated samples (Knorr 1980).

Table 3. Effect of method of protein coagulation on protein solubility of spray dried potato protein concentrates

Method of Coagulation	Protein Solubility at pH 7.0(%) Mean $\pm$ Standard Deviation (n=4)	
Hydrochloric acid at 98-99°C	$11.5 \pm 0.7^{\mathrm{a}}$	
Citric acid at 22-24°C	$90.7 \pm 7.3^{b}$	
Aluminum sulfate at 22-24°C	$75.5 \pm 5.7^{\circ}$	
Hydrochloric acid at 22-24°C	$56.0 \pm 1.0^{\rm d}$	
Ferric chloride at 22-24°C	$87.5 \pm 2.1^{b,e}$	

a,b,c,d,e means with different letters are significantly different (P < 0.05)

The combined effects of coagulation methods and variable pH of the protein concentrate on water binding capacity of potato protein concentrates are illustrated in Table 4. The results of a two way analysis of variance indicate a significant effect of pH (of the protein concentrate) and method of coagulation as well as significance of their interaction (pH  $\times$  method of coagulation) on water binding capacity of potato protein concentrates. These data stress the complexity of the effects of processing on protein functionality and provide guidance about how to develop "tailored" protein concentrates with requisite functionality.

Table 4. Effect of method of coagulation<sup>1</sup> and  $pH^2$  of the protein coagulate on water binding capacity of spray dried potato protein concentrates

Source of Variation	Sum of Squares	Degrees of Freedom	F-value
pH-value	80,190.4	2	175.5*
Method of coagulation	106,339.7	4	116.4*
pH value $\times$ method of coagulation	432,300.3	8	236.5*
Residuals	10,281.0	45	
Total	629,111.4	59	_

• significant (P < 0.01)

 $^1$  coagulation with hydrochloric acid, citric acid, aluminum sulfate and ferric chloride at room temperature and with hydrochloric acid at  $98^\circ \rm C$ 

<sup>2</sup> pH values 5, 6 and 7

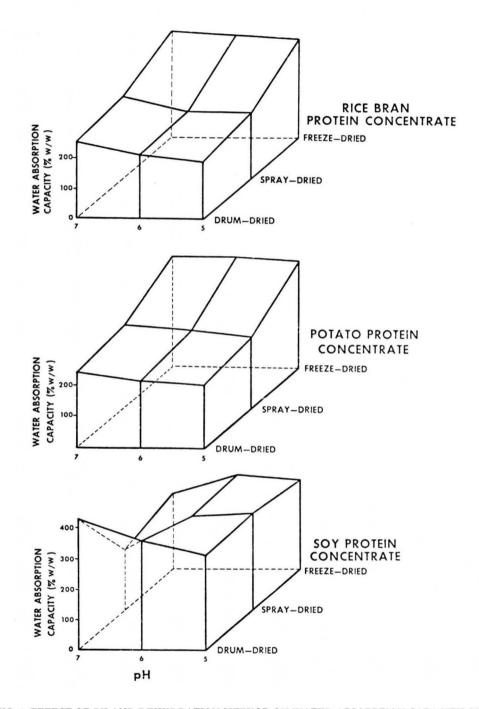


FIG. 1. EFFECT OF PH AND DEHYDRATION METHOD ON WATER ABSORPTION CAPACITY OF RICE BRAN PROTEIN CONCENTRATE, POTATO PROTEIN CONCENTRATE AND SOY PROTEIN PROTEIN CONCENTRATE

#### Effect of Dehydration of Protein Coagulates on Protein Functionality

The effect of freeze-, spray- or drum-drying as well as the pH of the protein concentrate on the water binding capacity of soy protein concentrates, heat coagulated rice bran protein concentrates and potato protein concentrates is shown in Fig. 1. The data indicated (with one exception) an increase in the water binding capacity with increasing pH. The drying process also markedly affected the water binding capacity of the proteins and followed the order freeze-dried > spray-dried > drum-dried samples. The lowest value ( $192 \pm 5\%$ ) was found for drum-dried rice bran protein concentrate at the lowest pH and the highest value ( $348 \pm 33\%$ ) for the freeze-dried sample at the highest pH investigated (pH 7). It may be assumed that heat during drying results in additional structural changes of the heat coagulated samples.

The multiple effects of drying temperature and pH of the coagulate (heat coagulated) on protein solubility were also studied (Table 5). The data in Table 5 show a significant influence (P < 0.01) of drying temperature and pH value of the protein coagulate as well as their interaction on solubility of potato protein concentrates.

Source of Variation	Sum of Squares	Degrees of Freedom	F-value
Drying temperature	0.57	2	57.0 <sup>•</sup>
pH value	0.62	2	62.1 <sup>*</sup>
Drying temperature $\times$ pH value	0.22	4	11.1*
Residuals	0.18	36	
Total	1.59	44	

Table 5. Effect of drying temperature<sup>1</sup> and pH of the protein coagulate<sup>2</sup> on protein solubility of spray dried potato protein concentrate

<sup>1</sup> inlet/outlet temperature 120-125/40-42, 155-160/54-56, 190-195/70-72°C

<sup>2</sup> pH 4, 6, 8

' significant for P < 0.01

The variations in solubility as shown in Tables 3 and 5 are due to both the degree of protein denaturation and changes in the interactions between the molecules, (causing association and dissociation), which can also be caused by other contributions such as nonprotein portion and solution (Hutton and Campbell 1977; Hermansson 1977).

The multiple effect of different protein source, variable drying method and variable pH of the protein concentrates on water binding capacity of protein concentrates is given in Table 6. The results of a 3way analysis of variance showed significant influence of all variables and all the interactions on water binding capacity thus indicating the complex correlation between processing of protein concentrates and

Source of Variation	Sum of Squares	Degrees of Freedom	F-value
pH value	43,115.0	2	284.2°
Drying method	120,489.2	2	794.2°
Protein source	107,477.6	2	708.5
$pH \times drying method$	6,530.4	4	21.5
$pH \times protein source$	5,151.2	4	16.9°
Drying method $\times$ protein source	106,535.7	4	351.1°
$pH \times drying method \times protein source$	13,234.1	8	21.8
Residuals	6,144.0	81	
Total	408,677.2	107	_

Table 6. Effect of variable  $pH^1$ , variable drying method<sup>2</sup> and variable protein source<sup>3</sup> on water binding capacity of protein concentrates (three-way classification)

• significant (P < 0.01)

<sup>1</sup> pH values pH 5, 6, 7

<sup>2</sup> freeze-, spray-, drum-drying

<sup>3</sup> potato protein, soy protein, rice bran protein

protein functionality. There was also a significant effect of the different protein sources on water binding capacity indicating the different water binding capacities existing for the various protein sources.

#### **Relationship between Protein Solubility and Water Binding Capacity**

The relationships between protein solubility and water binding capacity were studied at pH 5, 6 and 7 for differently processed rice bran protein concentrates, soy protein concentrates and potato protein concentrates. Mean values for solubility and water binding capacity as well as some details about processing of the protein concentrates are given in Fig. 2. As expected, low solubility existed for the heat coagulated samples and generally increased with increasing pH. The effects of method of coagulation, drying method and pH on water binding capacity of some of the protein concentrates has been discussed previously (see Fig. 1, Tables 4 and 6). Correlation coefficients between solubility and water binding capacity of protein concentrates are given in Table 7 indicating insignificant correlations (P < 0.01) for all protein concentrates examined. As stated by Hutton and Campbell (1981), the reported relationship between water absorption and solubility of proteins has not been consistent. Possible explanations with regards to the reported differences have been given by Fennema (1977), who also suggested to express water uptake on the (more appropriate) basis of grams of water per unit of protein interfacial area instead of mass of water per unit mass of protein. Data by Hutton and Campbell (1977) on the effect of pH and temperature suggested that water binding and solubility may be related up to a certain point, perhaps maximum hydration, at which solubility continues to increase and hydration does

Protein Concentrates	Sample Size (N)	Correlation Coefficient (R)
Freeze dried	9	-0.738**
Spray dried	18	-0.564**
Drum dried	9	+0.613*
Values at pH 5	13	-0.350*
Values at pH 6	13	-0.481*
Values at pH 7	13	-0.624
All rice bran protein samples	9	$+0.302^{*}$
All soy protein samples	9	-0.713**
All potato protein samples	21	-0.430**
All samples	39	-0.378**

Table 7. Correlation between mean values<sup>a</sup> of solubility and water binding capacity of various protein concentrates

<sup>a</sup> two replications for solubility

four replications for water binding capacity

<sup>b</sup> See Fig. 2 for protein concentrates used

' insignificant correlation for P < 0.05

" insignificant correlation for P < 0.01

not. In our case, the correlation between solubility and water binding can also be affected by the presence of nonprotein components in various concentrations and by possible qualitative differences in the nature of the protein surface of the protein concentrates.

In summary, the data presented outlined the significant effect of processing of protein concentrates on functionality and also indicated the multiple interactions between various factors influencing protein functionality. This can lead to the "tailoring" of protein concentrates with desired functionality during their processing instead of the commonly applied chemical, mechanical, physical or enzymatic modifications of the protein concentrates *after* the protein recovery process.

Research should be focused for establishing the correlation between processing of protein concentrates and protein functionality. The potential for protein mixtures consisting of differently processed (e.g., mixture of native proteins with partially denatured proteins) to give high nutritional quality and desirable functionality should be investigated.

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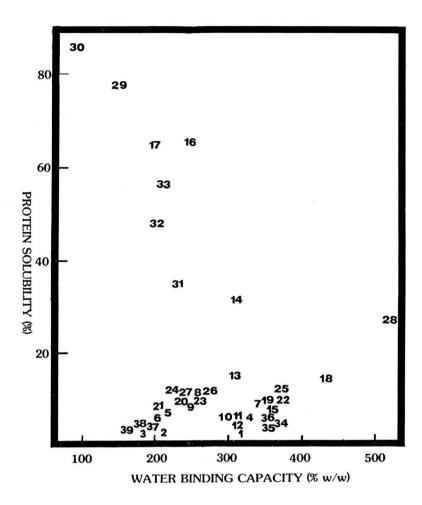


FIG. 2. RELATIONSHIP BETWEEN PROTEIN SOLUBILITY AND WATER BINDING CAPACITY OF VARIOUS PROTEIN CONCENTRATES Rice bran protein concentrate 1-9. Sou protein concentrate 10-18

Soy protein concentrate 10-18. Potato protein concentrate 19-39. Data for pH 5: 1-3, 10-12, 19-21, 28, 31, 34, 37. Data for pH 6: 4-6, 13-15, 22-24, 29, 32, 35, 38. Data for pH 7: 7-9, 16-18, 25-27, 30, 33, 36, 39. Freeze dried samples: 1, 4, 7; 10, 13, 16; 19, 22, 25. Spray dried samples: 2, 5, 8; 11, 14, 17; 20, 23, 26, 28-36. Drum dried samples: 3, 6, 9; 12, 15, 18; 21, 24, 27. Hot air dried samples: 37-39 (commercial product). Heat coagulated: 1-27, 34-39. Coagulated at room temperature: 28-33.

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#### THE EFFECT OF HEAT AND SHEAR ON THE VISCOELASTIC PROPERTIES OF SOY FLOUR DOUGH

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#### ABSTRACT

This paper is concerned with the effect of heat and shear on the rheological properties of defatted soy dispersions and doughs of 30 to 60% flour by weight. Capillary and rotary rheometers (Rheometrics Mechanical Spectrometer) were used to heat doughs up to 75°C and shear them simultaneously. In one series of experiments using a modified Instron capillary rheometer the doughs were heated to several different temperatures and then sheared. The linear viscoelastic properties of the dough which were determined by means of a plate-plate rheometer were then compared to the fresh dough and those of a heated but nonsheared dough. In another experiment a cone-and-plate rheometer was used to heat and shear the dough. The linear viscoelastic response of the dough was used to monitor any changes as it was heated and sheared. It was observed that only in the higher moisture dispersions (i.e., moisture contents greater than 50%) were there any signs of an increase in rheological properties which might be associated with "cooking". It was concluded that cooking of soy flour doughs most likely does not involve the formation of a permanent network formed by covalent chemical bonds. Hence, in extrusion cooking processes involving soy flour doughs, it may not be necessary to treat the rheological properties of the dough as a thermosetting system.

#### **INTRODUCTION**

Soy flour doughs are frequently processed using high-shear cooking extruders (Harper 1978). During extrusion-cooking processes, a network structure is formed which gives the extruded product texture and water binding characteristics. The exact nature of the formation of the network structure in an extruder is poorly understood but it is associated with protein denaturation and gelation (Hermansson 1978). Some researchers have proposed that the network structure formed during cooking is held together by covalent bonds (Circle *et al.* 1964; Remsen and Clark 1978; Harper 1979) while others have viewed the bonds to be less permanent (Castimpoolas *et al.* 1970; Ewart 1977; Bloksma 1973; Baird 1981a). When it comes to developing models for extrusion-cooking processes, it is necessary to know the nature of the bonds that hold the network together. Since one of the key elements in extrusion modeling is the rheological properties of the dough, then covalent crosslinks will certainly require a different approach than that for physical entanglements. Even if the bonds are only physical entanglements or hydrogen bonds, it is necessary to know how heat and shear change the rheological properties of the dough.

There are few studies reported in literature pertaining to the effect of temperature on soy dough rheology. Most studies have been concerned with high moisture dispersions (greater than 85% moisture) in which gelation occurs at some critical temperature. For example, Circle and coworkers (1964) studied aqueous dispersions of soy isolate from 8% to 14% flour. The viscosity of the dispersions went up sharply at 65°C. Because small amounts of sodium sulfite and cysteine prevented gel formation on heating they concluded that crosslinks promulgated by disulfide bonds must be responsible for gel formation.

However, even in the case of dispersions, it is not clear whether gelation, which may also occur in extrusion cooking, involves a crosslinked network. Castimpoolas and Meyer (1970) found that on heating soy bean globulins a progel formed as indicated by a rise in viscosity. On cooling the sample a very viscous gel formed. However, the progelgel step was reversible which led them to conclude that the bonds in the gel state were noncovalent in nature. Hence, it may be that gelation is a phenomenon associated only with high moisture dispersions. Castimpoolas and Meyer proposed that soy protein molecules unfold on heating exposing groups capable of hydrophobic bonding. Furthermore, Hermansson (1975) found that for concentrations of soy flour greater than 16%, gels formed without heating. Studies involved with doughs seem to indicate that gelation does not occur in these systems.

Baird (1981) measured the linear viscoelastic properties of soy isolate doughs and dispersions at temperatures up to  $85^{\circ}$ C. He observed that only for dispersions of 15% flour and less did the rheological properties increase with increasing temperature. Otherwise the rheological properties decreased with increasing temperature. He concluded that gelation was associated with the amount of water available for swelling the globular proteins. Jao and coworkers (1978) measured the rheological properties of defatted soy doughs using capillary dies attached to the end of the extruder at temperatures from 100°C to 160°C and reported that the viscosity decreased with increasing temperature. Hence it seems that gelation and the corresponding rise in viscosity of soy protein dispersions occurs only for high moisture dispersions.

However, there seems to be a significant difference in opinion in the literature as to what cooking entails in terms of changes in rheological properties. Even Hermansson (1978) who reports doubts about the formation of a crosslinked network structure, proposes that the kinetics of the network formation must be studied. Remsen and Clark (1979) tried to account for the changes in soy dough rheology during cooking using a model which has been used to describe the viscosity increase in a curing polymer system (i.e., one that undergoes crosslinking at a critical temperature). In other words they viewed cooking as a crosslinking reaction. However, their model for cooking kinetics was never confirmed by independent sets of experiments. Furthermore, they used viscosity data obtained from high moisture dispersions to evaluate the parameters in their model. Hence, the few reported studies on soy dough rheological properties at elevated temperatures seem to indicate that gelation does not occur or at least the dough viscosity does not rise at some critical temperature. However, it seems that most views on cooking of soy doughs are based on our knowledge of high moisture dispersions.

The purpose of the present paper is to investigate the effects of heat and shear on the rheological properties of defatted soy flour doughs and dispersions. Because of our interest in food extrusion processes, it is necessary to know if the rheological properties of defatted soy doughs increase at some critical temperature and whether the changes occur as a function of time. Although we can not simulate the exact thermal and shear history of dough in an extruder, we can at least carry out experiments resembling some of the conditions in an extruder. In this research we use a cone-and-plate, plate-plate, and capillary rheometers to shear, heat, and follow changes in dough rheological properties. In particular, we have measured changes in the linear viscoelastic properties of defatted soy doughs in order to determine changes in dough structure as a result of heating and shearing.

#### **EXPERIMENTAL**

#### Materials

Defatted soy flour (Soyafluff 20 manufactured by Central Soya; Fort Wayne, Ind.) was used in this study. Since it contains no starch it was believed that any changes in the dough rheological properties upon heating should be due to protein denaturation. Mixtures of 30, 40, 50 and 60% by weight in distilled water were prepared by mixing in a Sorvall Omnimixer for 5 min at  $25^{\circ}$ C. The 30 and 40% samples are referred to as dispersions because of their significantly lower viscosity as compared to the 50 and 60% materials which we refer to as doughs.

#### Equipment

A modified Instron capillary rheometer (Model 3211) was used to heat and shear the dispersions and doughs. The modification is described in more detail elsewhere (Baird 1981b). Basically, the existing barrel and plunger system were replaced by a barrel which fit inside the existing barrel and which was attached to a larger reservior. The new plunger and reservior fit more tightly together ensuring that the plunger always scraped the walls of the reservior removing any cooked and hardened materials. Three capillaries of 0.052 in. dia. and L/D of 10, 15 and 20 were used. The dynamic mechanical mode of a Rheometrics Mechanical Spectrometer [RMS] (Model 602) was used to determine the linear viscoelastic properties of the soy flour mixtures. Furthermore, the instrument was operated in the steady shear mode as a means of providing a shear field during the heating of a sample. Both the parallel plate (25 mm dia.) and cone-and-plate (0.04 radian cone angle and 25 mm dia.) attachments were used. The cone-and-plate has the advantage of providing uniform shear throughout the specimen. The temperature could be maintained to  $\pm 1.0^{\circ}$ C.

At elevated temperatures the doughs tended to dry out at the specimen edge which made it difficult to obtain information about the cooking kinetics. A ring was machined to fit around the bottom plate which contained fabric saturated with water. In this way the moisture evaporated from the fabric rather than the dough during heating.

#### Procedure

Soy flour mixtures were heated from 3 to 5 min until the center of the reservior came up to the test temperature. As soon as the center reached the test temperature, a temporary cover was removed from the top of the reservior and the plunger installed. The mixtures were then extruded while being held at 25°C, 50°C and 75°C at apparent shear rates (where  $\dot{\gamma}_a = 4Q/\pi R^3$  and Q is the volumetric flow rate and R is the capillary radius) of 34, 224 and 1120 s<sup>-1</sup>. The extrudates were then collected in glass bottles. As soon as the doughs reached 25°C the linear viscoelastic properties of the dough were determined with the RMS. The extruded doughs were also checked for moisture evaporation but no significant moisture loss was detected. The cone-and-plate geometry of the RMS was also used to study the nature of the cooking process and the changes in the rheological properties. The loss, G'', and storage moduli, G', and the complex viscosity  $|\eta^*|$  (these functions are defined in detail elsewhere, J. D. Ferry 1970) where determined before heating and shearing the dough sample. The doughs were then heated to temperatures of 50°C and 75°C while being sheared at various shear rates. The time required to obtain a stable temperature was of the order of 2 to 3 min. The sample was sheared and heated for 5 min after a stable temperature was reached. At the end of this period G' and G'' were again measured. Finally the dough was cooled to 25°C and G'' and G'' redetermined.

Another experiment which was readily carried out in the RMS was to execute a time sweep during the heating cycle. In this way G' and G'' were automatically determined every 30 s at a predetermined angular frequency ( $\omega$ ) of 1 rad/s. The effects of shear on changes in dough properties could then be separated from those of heat alone. The linear viscoelastic properties were determined because it was felt that in this region of deformation the strains are small enough that there would be negligible effect on the kinetics of any chemical reactions.

#### **RESULTS AND DISCUSSION**

#### **Capillary Rheometer Studies**

The first experiment was designed to study the effect of heat on the linear viscoelastic response of the doughs. The soy flour mixtures were heated to 50°C and 75°C and held for 5 min. Then G and G' and  $|\eta'|$ were determined as a function of angular frequency ( $\omega$ ). The results for the 40% dispersion and 50% dough are presented in Fig. 1 and 2, respectively. For the 50% dough G' and  $|\eta'|$  are observed to decrease with increasing temperature. On the other hand,  $|\eta^*|$  and G' increase with increasing temperature for the 40% dispersion. This behavior of only the high moisture dispersions exhibiting an increase in rheological properties at 75°C has been previously observed for soy isolate doughs (Baird 1981a). Because the shape of the G' curves remains nearly unchanged as a function of temperature, it is believed that the increase in G' for the 40% dough is not due to the formation of a permanent network structure. If a permanent network structure had been generated, the G' versus  $\omega$  curve would have exhibited a plateau as is the case for crosslinked polymers (Ferry 1970). However, because G' depends to some degree on  $\omega$ , we can speculate that the proteins are highly confined but not permanently crosslinked.

#### D. G. BAIRD

However, because G' and  $|\eta^*|$  increase with temperature for the 40% dispersion and not the 50% dough, a mechanism is still needed to explain this behavior. We believe that the increase in rheological properties which has been associated with protein gelatin (Hermansson 1978; Circle *et al.* 1964) may be due to the expansion of the globular

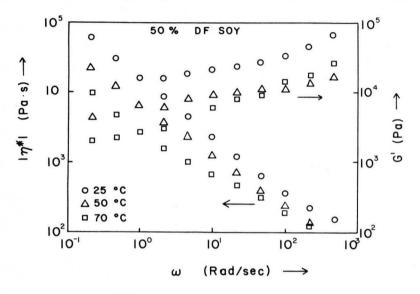


FIG. 1. VALUES OF G' AND  $|\eta^{*}|$  VERSUS  $\omega$  FOR A 50% DEFATTED SOY FLOUR DOUGH HEATED TO 25°, 50° AND 75°C

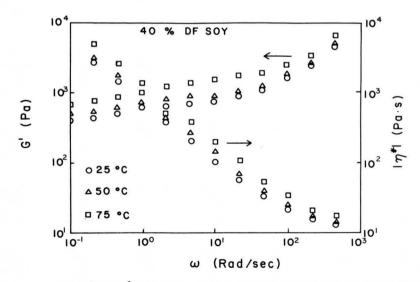


FIG. 2. VALUES OF G' AND  $|\eta^{\bullet}|$  VERSUS  $\omega$  FOR A 40% DEFATTED SOY FLOUR DISPERSION HEATED TO 25°, 50° AND 75°C

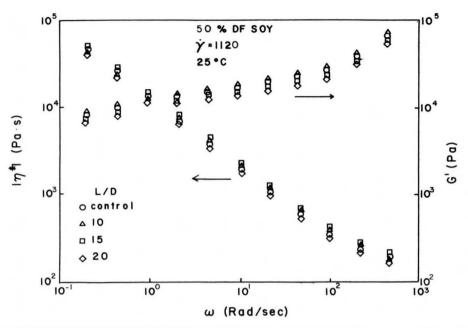


FIG. 3. LINEAR VISCOELASTIC RESPONSE OF EXTRUDED 50% DEFATTED SOY FLOUR DOUGH

protein by moisture. As the globular proteins expand they overlap increasing intermolecular contacts which raises the rheological properties of the dispersions. For the doughs, the concentration of flour is high enough so that intermolecular interactions are already present. Hence, increasing the temperature of the dough will only make the dough more easily deformable.

The next question to be considered was whether shear could have an effect on the rheological properties of the doughs and dispersions. The materials were extruded from the capillary rheometer at various shear rates at  $25^{\circ}$ C and the extrudates collected. Representative data is presented for a 50% dough in Fig. 3 and for a 40% dispersion in Fig. 4. Even at higher shear rates with strong entry flow effects possible no significant changes in the dough rheological properties were observed. This was true for all levels of flour (i.e., both the 30% and 60% flour doughs). Although we could measure no observable changes in the linear viscoelastic response of the doughs, the appearance of the extrudate was significantly different from those of the fresh sample. The chunks of dough emerged from the capillary as a continuous uniform extrudate.

The next point of consideration was whether the combined effect of shear and heat would affect the rheological properties of the dough. Defatted soy flour mixtures were heated at 50°C and 75°C for five min and then extruded at various shear rates. Representative data for the

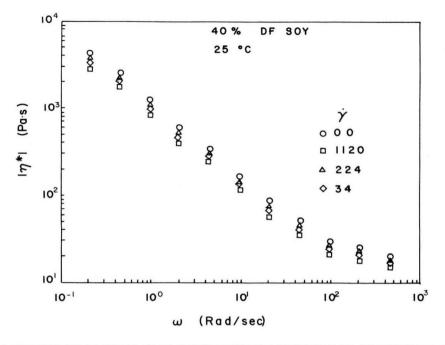


FIG. 4. THE EFFECT OF SHEAR RATE ON THE DYNAMIC VISCOSITY OF AN EXTRUDED SOY FLOUR DISPERSION

50% and 40% materials are presented in Fig. 5 and 6, respectively. First, we note a slight increase in G' and  $|\eta^*|$  when the dough is sheared at 75°C for the 50% dough whereas a very significant change occurs in the 40% dispersion. The effect of  $\dot{\gamma}$  at 75°C on the linear viscoelastic response of the doughs and dispersions is illustrated in Fig. 7 and 8. There may be a slight increase in values of G' and  $|\eta^*|$  for the 50% dough whereas a large effect is observed for the 40% sample. The fact that G' and  $|\eta^*|$  increase for the 50% dough is contrary to the results of the earlier experiment in which only heat was applied to the doughs. It may be that shear promotes diffusion of water into the protein bundles leaving them more swollen and increasing the degree intermolecular interactions.

#### **Cone-and-Plate Studies**

The flow and thermal history in the Instron rheometer are not well defined. Furthermore, a significant effect of shear and heat on the linear viscoelastic response of the doughs was observed. For these reasons it was believed that a system with a more well defined flow field should be used. We then turned to the cone-and-plate rheometer because of the uniform shear rate in the gap and better heating control

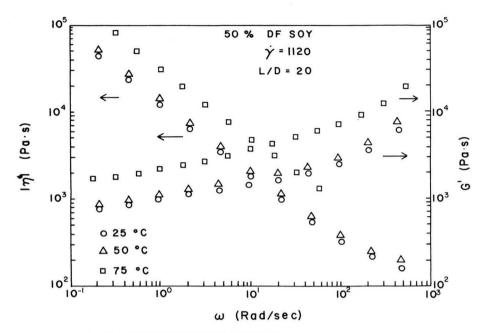


FIG. 5. LINEAR VISCOELASTIC RESPONSE OF EXTRUDED SOY FLOUR DOUGH AFTER BEING HEATED TO 25°, 50° AND 75°C

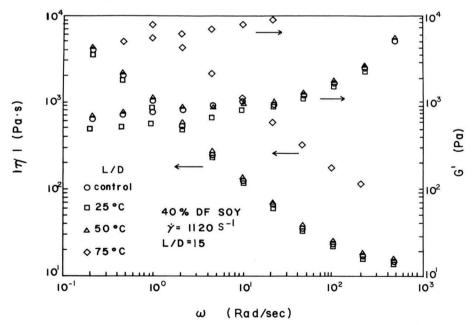


FIG. 6. LINEAR VISCOELASTIC RESPONSE OF A 40% SOY FLOUR DISPERSION AFTER HEATING TO 25°, 50° AND 75°C AND EXTRUDING IN A CAPILLARY RHEOMETER AT A SHEAR RATE OF 1120  $\rm S^{-1}$ 

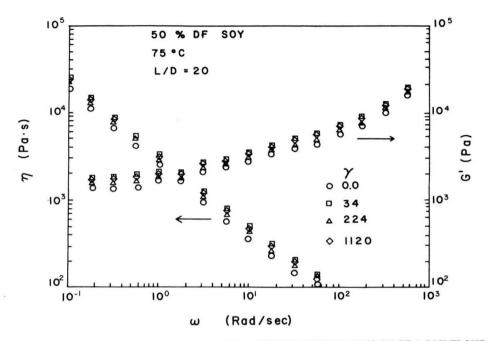


FIG. 7. EFFECT OF SHEAR RATE ON THE LINEAR VISCOELASTIC RESPONSE OF A SOY FLOUR DOUGH HEATED AT 75°C IN A CAPILLARY RHEOMETER

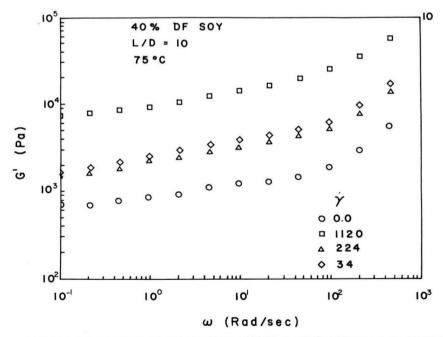


FIG. 8. EFFECT OF SHEAR RATE ON THE LINEAR VISCOELASTIC RESPONSE OF AN EXTRUD-ED 40% DEFATTED SOY DISPERSION

of the sample. The only difficulty with this geometry is the free sample edge which tends to dry during the heating cycle leaving a hard crust. This problem, however, was overcome as described earlier.

The most significant change in the rheological properties was observed in the dispersions. The question is whether there is a reaction kinetics which must be determined or do the changes occur spontaneously. Data is presented in Fig. 9 for 40 and 50% samples in which G'was measured every 30 s while the sample was heated to 75°C. It is observed that G' increases significantly for the 40% mixture over a period from about two to five minutes. The increase begins before a temperature of 75°C is reached. After this time there is a slight change in G' with time but this is probably due to the drying of the free edge of the sample. The 50% defatted soy flour dough exhibited no increase in G' with time. Hence the changes which occur must be associated with the moisture level. It is possible that the kinetics of this process are related to the diffusion of moisture into the globular protein rather than to a chemical reaction. These observations are the same for all of the higher moisture dispersions: i.e., the rheological properties increase

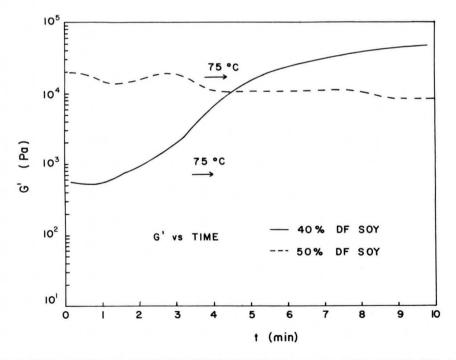


FIG. 9. TIME TRACES OF G' DURING THE HEATING TO 75°C OF A 50% DOUGH AND A 40% DISPERSION  $\omega = 1 \text{ rad/s}$ 

significantly above some critical temperature and time of heating. Hence, only with sufficient moisture available is there any indication of an increase in rheological properties.

Since changes are observed for the dispersions it is natural to ask if shear can affect the rate of change in G' (i.e. kinetics) or even impart changes in the doughs. Samples were heated to 75°C while simultaneously being sheared. The samples were heated and sheared for five minutes after reaching 75°C. G' and G'' and  $|\eta'|$  were determined before heating, at the end of the heating and deformation period and then after cooling the sample to 25°C. Results for the 40% dispersion are presented in Fig. 10 and 11. First, if we compare the results with those in Fig. 2 we see that shear increases the change in G' and  $|\eta'|$ . Furthermore, increasing  $\gamma$  to 100 s<sup>-1</sup> produces further increases in G'and  $|\eta'|$ . However, on cooling the sample to 25°C, the values of G' and  $|\eta'|$  return to the original values. For the 50% dough, for which data is presented in Fig. 12, we observe that G' and  $|\eta'|$  are still lower than the values at 25°C and that shear seems to have no effect at least for low

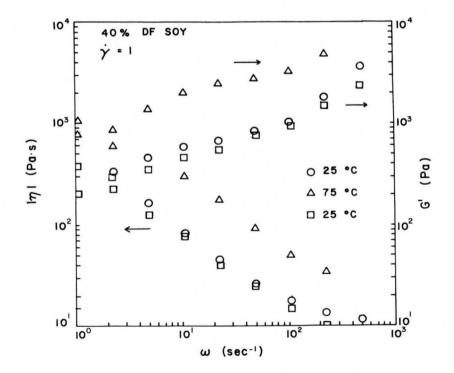


FIG. 10. EFFECT OF SHEAR AND TEMPERATURE PRODUCED BY A CONE-AND-PLATE RHEOM-ETER ON THE LINEAR VISCOELASTIC PROPERTIES OF A 40% DEFATTED SOY FLOUR DISPERSION

 $\gamma = 100 \text{ s}^{-1}$ . 0-before heat and shear.  $\Box$ -after heat and shear.

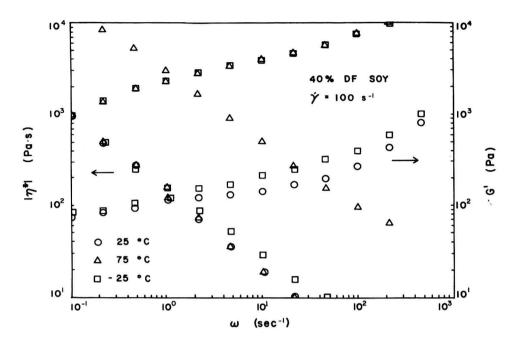


FIG. 11. EFFECT OF SHEAR AND TEMPERATURE PRODUCED BY A CONE-AND-PLATE RHEOM-ETER ON THE LINEAR VISCOELASTIC PROPERTIES OF A 40% DEFATTED SOY FLOUR DISPERSION

 $\gamma$  = 100 s<sup>-1</sup>. 0-before heat and shear.  $\Box$ -after heat and shear.

shear rates (e.g. compare data in Fig. 1 with those in Fig. 12). The effect of higher shear rates could not be investigated because the sample came out of the gap.

#### CONCLUSIONS

As a result of this work we have a better understanding of the effect of heating and shearing on the linear viscoelastic response and physicochemical changes of defatted soy flour doughs and dispersions. The phenomena of cooking which is associated with protein denaturation seems to occur only in the higher moisture dispersions (i.e., mixtures with more than 50% moisture). Even for the dispersions the data reported here indicate that the intermolecular interactions are noncovalent bonds. As long as sufficient moisture is available the changes which occur on heating are reversible. Heating the doughs to 75°C, which is commonly thought to be above the protein denaturation temperature, imparts no detectable changes in the linear viscoelastic response of the doughs. Shear is observed to significantly affect changes

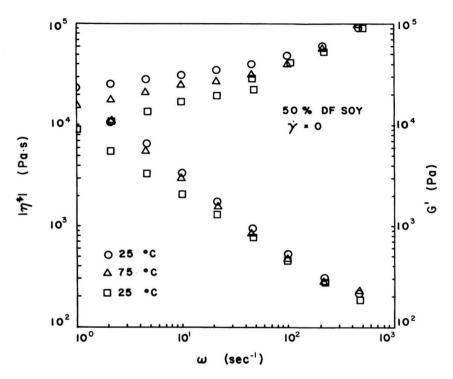


FIG. 12. EFFECT OF SHEAR AND TEMPERATURE PRODUCED BY A CONE-AND-PLATE RHEOM-ETER ON THE LINEAR VISCOELASTIC PROPERTIES OF A 50% DEFATTED SOY FLOUR DOUGH

 $\gamma$  = 1 s<sup>-1</sup>. 0-before heat and shear.  $\triangle$ -after heat and shear.

in the dispersion properties but not those of the dough. In modeling the rheological properties of doughs it does not seem necessary to have to use a model of the form proposed by Remsen and Clark (1978) which considers cooking to be a crosslinking process.

#### ACKNOWLEDGMENT

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#### LITERATURE ABSTRACTS

#### ABSTRACTS FROM TRANS. ASAE

#### DESIGN, EVALUATION AND EFFLUENT WATER QUALITY ANALYSIS OF THREE OYSTER SHELLSTOCK WASHERS. S. S. Chang, F. W. Wheaton. Trans. ASAE. 24FE, 234.

A rotary brush washer, a pressurized water spray washer and a rotary tumbler washer were designed, constructed, and their efficiency in washing oyster shellstock was determined. Washer effluent water quality was determined for the brush and spray washer. The brush and spray washer did an acceptable job of washing oyster shellstock. The tumbler washer damaged oysters enough that it could not be used in any commercial operation. Water quality analysis of washer effluent showed that effluent must be disinfected prior to discharge, and in some instances, the pH must be lowered to meet existing Maryland and EPA limitations.

#### PREDICTION OF BRUISING IN IMPACT MULTILAYERED AP-PLE PACKS. J. E. Holt, D. Schoorl, C. Lucas. Trans. ASAE. 24FE, 242.

A method for predicting total bruising and bruise distributions in apple packs under impact loads is given. The method takes into account the variables to be expected in distribution systems such as bruise resistance, numbers of layers, package rebound and impact conditions. It is based on the correlation between bruising and absorbed energy and calculates the absorbed energy at each interface in a multilayered arrangement. It is postulated that the behavior of apples in commercial multilayered packages is similar to that of apples in single columns.

Results calculated for Jonathan and Granny Smith apples in columns up to 10 deep for a variety of package and impact conditions are in good agreement with experimental values.

The method can be used to evaluate packaging and handling systems and should have wide application to other products transported in multilayered packs.

# PRODUCTION FUNCTIONS FOR THE MARYLAND OYSTER FISHERY. R. A. Cabraal, F. W. Wheaton. Trans. ASAE. 24FE, 248.

Using seed planting, shell planting, spatfall and fishing effort data, production functions were developed for the Chesapeake Bay oyster fishery. The oyster producing areas of the Maryland portion of the Chesapeake Bay and tributaries were divided into four regions and a production function was developed for each region. Results from these functions were then used to analyze the effectiveness of State operated seed and shell planting programs. The effectiveness of fresh shell versus seed planting in terms of both dollar and harvestable bushels of oysters returned was assessed.

#### ATMOSPHERIC PRESSURE EFFECT ON VAPOR PRESSURE DEFICIT AND POTENTIAL MOISTURE LOSS FROM HORTICUL-TURAL COMMODITIES. L. R. Erickson, R. E. Garrett. Trans. ASAE. 24FE, 252.

An analytical investigation of the effect of atmospheric pressure level on air-water psychrometric relationships is reported. Vapor pressure deficit (VPD) is shown to be essentially independent of atmospheric pressures normally encountered in postharvest handling of horticultural commodities. Water loss potential from horticultural commodities is shown to exist at low atmospheric pressures and negligible VPD since water vapor diffusivity into air increases exponentially as pressure is reduced.

#### ROUGH-RICE BREAKAGE IN RELATION TO KERNEL THICK-NESS FOR HAND AND COMBINE HARVESTED RICE. J. Matthews, J. I. Wadsworth, J. J. Spadaro, Trans. ASAE. 24FE, 255.

Five lots of Starbonnet rough rice harvested both by hand and by combine were dried under ambient conditions and separated into four fractions according to the thickness of the kernels. The distribution of kernel thickness was related to the moisture content of the rice at harvest. X-ray photographs were used to detect cracked and broken kernels in the rough rice. In both the hand- and the combineharvested samples, the thickest and thinnest grain had higher percentages of cracked and broken kernels than did kernels of intermediate thickness.

## A MACHINE TO EVISCERATE AND SKIN SQUID. D. E. Brown, R. P. Singh. Trans. ASAE. 24FE, 259.

Design features of a new squid eviscerating and skinning machine are presented. Physical properties of the squid were used in design of an orienting and feeding device. Parts of squid anatomy help in alignment before separating the tentacles from the body. The conical body is automatically conveyed to and lodged on an evisceration/skinning peg. Fan-shaped water spray is used to remove the skin. Additional water jets allow evisceration. The output from the machine is a clean white mantle ready for further processing. Performance trials of a pilot-scale unit and scale-up design aspects indicate that the system is feasible for a large-scale processing operation.

#### STEAM INJECTOR NOISE REDUCTION AND HEATING EFFI-CIENCY. S. A. Waggoner, P. C. van Alderwerelt, W. J. Salesky, M. O'Brien, R. E. Garrett. Trans. ASAE. 24FE, 514.

The use of steam injectors is a practical and effective method for noise control in hot water applications of the food processing industry. This study examined the noise reductions of nine types of commercial steam injectors as compared to a drilled pipe configuration. Relative heating times and cost effectiveness were evaluated. Insertion losses of up to 30 dBA were realized by installation of steam injectors in place of the drilled pipe configuration. Relative heating times of all injectors tested were twice as good as the drilled pipe configuration. Considering noise and heating performance along with cost, the Noiseless Water Heater would be the optimum injector for heating water to 100°C (212°F). For applications below 77°C (170°F) the Suction Tee would be the best choice.

#### ESTIMATING MARYLAND CHESAPEAKE BAY OYSTER POPU-LATION USING LESLIE AND DELURY EQUATIONS. R. A. Cabraal, F. W. Wheaton. Trans. ASAE. 24FE, 519.

The Leslie and Delury equations were used with historical oyster catch per boatday and harvest data to estimate the existing oyster population and resource exploitation rate in the Maryland portion of the Chesapeake Bay and tributaries. The area was divided into five regions and population estimates were obtained for each region. The 1975 population was estimated at about 10 million bushels and the exploitation rate of oysters over the total area averaged about 23%.

APARATUS AND TECHNIQUES FOR MEASURING THE PHYSI-CAL CLEANING PERFORMANCE OF SPRAY NOZZLES. J. M. Scott, R. N. Barnes, D. G. Dunsmore. Trans. ASAE. 24FE, 524.

Apparatus is described which permits the examination of spray nozzles for their efficiency in cleaning food-soiled surfaces. A pressurized reservoir pumps test solution through a variable geometry spray nozzle onto a test grid. The distribution of solution throughout the spray impact area can be measured using receptor vessels. The distribution of cleaning effect throughout the spray impact area is determined gravimetrically on pre-soiled test surfaces.

#### SPRAY NOZZLE PERFORMANCE IN CLEANING FOOD EQUIP-MENT. J. M. Scott, D. G. Dunsmore, M. D. Keegan. Trans. ASAE. 24FE, 526.

The lack of applied and fundamental information on spray cleaning has retarded improvements in performance. The performance of five nozzles with differing characteristics was examined in detail, using cold water as the spray liquid. The spatial distributions of spray volume and cleaning efficiency were determined. The effects on cleaning efficiency of the temperature, time, angle and distance of spraying, and also intermittent spraying were examined.

The data were used to select nozzles for cleaning dairy equipment. Substantially more effective nozzles than those currently used were identified; these were flatfan type nozzles of medium to high flow rates. Recommendations are made concerning choice of nozzles and improvement of design for spray cleaning systems.

#### ENERGY ACCOUNTING AND CONSERVATION IN THE MANU-FACTURE OF YOGURT AND SOUR CREAM. G. H. Vrusewitz, R. P. Singh. Trans. ASAE. 24FE, 533.

An energy accounting study was conducted and the energy intensive operations were identified in yogurt and sour cream manufacturing. Heating of the yogurt base by injecting steam directly into the product required 80% of all the energy. When yogurt was heated by recirculated hot water the energy consumption was reduced by 59%. All handling and packaging operations used only 6% of the total energy with one-half of this being used by the carton shrink-wrap machine for yogurt processing. Because of the need for less heating, the manufacture of sour cream, excluding pasteurization and cold storage, used 287 kJ/kg product or only one-fourth the energy, 1224 kJ/kg product used for yogurt manufacture.

AIR MUFFLER EVALUATION FOR THE FOOD PROCESSING INDUSTRY. S. A. Waggoner, S. Humpert, M. O'Brien. Trans. ASAE. 24FE, 778.

Unmuffled impulse air exhausts from air-cylinders produce sound pressure levels that exceed limits set by O.S.H.A. To comply with O.S.H.A., food processors can easily install air exhaust mufflers to reduce their plant noise levels. This article tests the effectiveness of eighteen types of commercial silencers in <sup>1</sup>/<sub>8</sub>, <sup>1</sup>/<sub>4</sub>, <sup>3</sup>/<sub>8</sub>, and <sup>1</sup>/<sub>2</sub> in. N.P.T. The article provides the food processor with comparative data to pick the most cost-effective muffler for their application.

#### **AUTHOR INDEX**

- AMUNDSON, C.H. See GAROUTTE, C.A. et al.
- AMUNDSON, C.H. See BLOCK, J.E. et al.
- ARBOLEDA, J.R. See KEPPLER, R.A.
- BAGLEY, E.B. See JASBERG, B.K. et al.
- BAIRD, D.G. The effect of Heat and Shear on the Viscoelastic Properties of Soy Flour Dough 231
- BLOCK, J.E., AMUNDSON, C.H. and von ELBE, J.H. Energy Requirements of Beet Colorant Production 67
- BRESNAHAN, D.P., WOLF, J.C. and THOMPSON, D.R. Potential for Utilizing 11S Soy Globular Protein to Study Texture Formation 89
- BUHLERT, J. See SCOTT, E.P. et al.
- CARROAD, P.A. See SCOTT, E.P. et al.
- CHERYAN, M. See CUEVAS, R.
- CUEVAS, R. and CHERYAN, M. Heat Transfer in a Verticle, Liquid-Full Scraped-Surface Heat Exchanger. Application of the Penetration Theory and Wilson Plot Models 1
- CORRIEU, G. see GALLOT-LAVALLE, T. et al.
- DEILY, K.R. and RIZVI, S.S.H. Optimization of Parameters for Packaging of Fresh Peaches in Polymeric Films 23
- DICKIE, A.M. and KOKINI, J.L. On the Use of the Bird-Leider Equation in Food Rheology 157
- GALLOT-LAVALLE, T., LALANDE, M. and CORRIEU, G. An Optical Method to Study the Kinetics of Cleaning Milk Deposits by Sodium Hydroxide 131
- GAROUTTE, C.A., AMUNDSON, C.H. and HILL, JR., C.G. Ultrafiltration of Whole Milk with Hollow Fiber Membranes 191
- HILL, JR., C.G. See GAROUTTE, C.A. et al.
- HORN, J. See SCOTT, E.P. et al.
- JASBERG, B.K., MUSTAKAS, G.C. and BAGLEY, E.B. Effect of Extruder Retention Time on Capillary Flow of Soy Dough 43
- KEPPLER, R.A. and ARBOLEDA, J.R. The Thermal Properties of Frozen Invert Sugar Solutions 113
- KNORR, D. Effects of Recovery Methods on the Functionality of Protein Concentrates from Food Processing Wastes 215
- KOKINI, J.L. See DICKIE, A.M.

- KUSTERMAN, M. and SCHERER, R. Respiration Activity of Shelled Corn 145
- LALANDE, M. See GALLOT-LAVALLE, T. et al.
- LOPEZ-SORIANO, E.M. See NORBACK, J.P.
- MATUSZEK, T. Effect of Time and Fish Temperature on the Blunting Rate of Filleting Knives 203
- MUSTAKAS, G.C. See JASBERG, B.K. et al.
- NORBACK, J.P. and LOPEZ-SORIANO, E.M. Cost Optimal Demand Forecasting for a Multi-Label Inventory Environment 175
- RIZVI, S.S.H. See DEILY, K.R.
- ROSE, W.W. See SCOTT, E.P. et al.
- RUMSEY, T.R. See SCOTT, E.P. et al.
- SCHERER, R. See KUSTERMAN, M.
- SCOTT, E.P., RUMSEY, T.R., CARROAD, P.A., BUHLERT, J., HORN, J. and ROSE, W.W. Energy Consumption in Steam Blanchers 77
- THOMPSON, D.R. See BRESNAHAN, D.P. et al.
- von ELBE, J.H. See BLOCK, J.E. et al.
- WOLF, J.C. See BRESNAHAN, D.P. et al.

#### SUBJECT INDEX

Angular frequency, 236 Apparent heat capacity, 92, 96, 98, 99, 100, 101.

Beet colorant, 67 Blunting rate, 203, 209 Bird-Leider equation, 157, 158, 170, 172

Calorimetry, 90 Capillary flow, 43 Cleaning, 131, 138, 143 Coagulation, protein, 218, 223 Concentration, protein, 194, 197 Cone and plate rheometer, 238 Cost optimal demand, 175 CO<sub>2</sub> equilibrium, 38

Density, 92, 104, 106, 107, 108, 109 Dewatering, 218

Elastic energy, 44 Energy consumption, 77 cost, 71, 73 efficiency, 87 measurement, 70 requirements, 67 Extraction, 69, 71 Extrusion, 117, 119, 122 Extruder retention, 43

Filleting knives, 203, 204, 208 Flow behavior, 157 Forecasting, 175 Fouling, 131, 135 Frozen solutions, 89

Gas transmission, 35

Heat transfer, 1 coefficient, 1, 4 Heat exchanger; milk pasteurizer, 138 scraped-surface, 1, 2 Infrared absorption spectrometer, 151 Instrom rheometer, 234 capillary rheometer, 235 Invert sugar, 89 Kinetic; cleaning, 131, 132, 133 loss modulus, 235 Mass flux, 194, 195, 200 Membranes, 191, 193 fiber length, 194, 198 pressure, 194, 195 Milk, 191 deposits, 131 Multi-label inventory, 175 Optical density, 136 method, 131, 132, 133 sensor, 138, 139, 143 Oxygen equilibrium, 38 Packaging, 23 machinery, 57 Penetration theory, 1, 8, 20 Plasticorder, 47 Polymeric films, 23, 25 Power law, 158, 159 Protein functionality, 215 potato, 221 solubility, 224 soy, 113, 222

Respiration, 29 activity, 145 heat, 147, 149, 152, 154 Retention time, 50 Rheology, 157 Rice bran, 222 Shear modulus, 44 rate, 44, 51, 158, 162, 238 stress, 157, 160, 163, 165, 167, 169 Shelled corn, 145 Soy dough, 43 extracts, 3, 11, 17 flour, 231 Steam blancher, 77 Storage modulus, 235

Texture, 30 formation, 113 Thermal energy balance, 78, 82 Thermal properties, 89 conductivity, 92, 104, 106, 108 diffusivity, 92, 94, 95 UHT, 1 Ultrafiltration, 191, 193 Viscoelastic fluid, 44 properties, 161, 231, 237, 239 Viscosity, 52, 162 Water absorption, 222 binding, 224, 225, 226 Wilson plots, 1, 14, 18

254

# P JOURNALS AND BOOKS IN FOOD SCIENCE AND NUTRITION

#### Journals

JOURNAL OF FOOD SERVICE SYSTEMS, G.E. Livingston and C.M. Chang JOURNAL OF FOOD BIOCHEMISTRY, H.O. Hultin, N.F. Haard and J.R. Whitaker

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HASSON, E. P. and LATIES, G. G. 1976. Separation and characterization of potato lipid acylhydrolases. Plant Physiol. 57, 142–147.

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### JOURNAL OF FOOD PROCESS ENGINEERING VOL. 5, NO. 4

#### CONTENTS

Meetings vii
Ultrafiltration of Whole Milk with Hollow Fiber Membranes
C. A. GAROUTTE, C. H. AMUNDSON and C. G. HILL, JR., University of Wisconsin, Madison, Wisconsin
Effect of Time and Fish Temperature on the Blunting Rate of Filleting Knives
<b>TADEUSZ MATUSZEK</b> , The Technological University, Gdańsk,Poland
Effects of Recovery Methods on the Functionality of Protein Concentrates from Food Processing Wastes
D. KNORR, University of Delaware, Newark, Delaware 215
The Effect of Heat and Shear on the Viscoelastic Properties of Soy Flour Dough
<b>D. G. BAIRD</b> , Virginia Polytechnic Institute and State University, Blacksburg, Virginia
Literature Abstracts
Author Index
Subject Index

