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# EFFECT OF MUSCLE FIBER ORIENTATION ON *PECTORALIS SUPERFICIALIS* MUSCLE EXPANSION IN NA<sub>2</sub>CO<sub>3</sub> SOLUTIONS<sup>1</sup>

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## ABSTRACT

*Cubes (.75, 1.00 or 1.25 cm<sup>3</sup>) cut from pectoralis superficialis muscles of broiler chickens were used to study muscle expansion in NaCl solutions. The cubes were submerged in NaCl concentrations ranging from .17M to .85M for up to 12 h at 22°C.*

*Length, width, depth, and volume of the muscle cubes increased ( $P < .05$ ) as NaCl concentration increased. Expansion was less ( $P < .05$ ) when measured along the longitudinal axes of the muscle fibers than when measured perpendicular to the longitudinal axes. Expansion of the cubes was different ( $P < .05$ ) when the longitudinal axes of the muscle fibers were rotated from horizontal to vertical with respect to the surface of the solution. Measurements made perpendicular to the long axes of the muscle fibers were 7 – 8% larger when the fibers were oriented horizontal to the surface. This research demonstrated that muscle fiber orientation must be considered when designing experiments that involve muscle tissue expansion and absorption characteristics.*

## INTRODUCTION

Marketing of poultry products as value added convenience items has increased the need for information on physical changes of chicken muscle tissue in a complex mixture of ingredients. The ability of muscle tissue to absorb and retain water soluble compounds during processing of value added products is often necessary to the development of unique products. Absorption and retention must be within specific limits and evenly distributed throughout the tissue to provide uniform organoleptic and functional properties.

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Salt (NaCl) is routinely added to meat products to retard bacterial growth (Sofos 1983 a,b; 1984;1985), increase shelf-life (Duncan and Foster 1968; Doyle and Roman 1982), alter flavor (Gardze *et al.* 1979; Kare *et al.* 1980; Bertino *et al.* 1981), add variety (Abu-Baker *et al.* 1982), and tenderize (Hamm 1960; Janky *et al.* 1978). One result of adding salt in an aqueous media to muscle tissue is increased water absorption and the consequent increase in volume of the tissue (Hamm 1975). Water is held in the muscle by capillarity, mostly in the interfibrillar spaces within the myofibrils and in the extracellular space (Offer and Trinick 1983). The concentration of NaCl required to cause the maximum increase in myofibril absorption is .8M (Hamm 1960; Offer and Trinick 1983) to 1M (Hamm 1960).

Tissue absorption characteristics depend, in part, on the ability of the tissue to expand (Hamm 1960, 1975; Offer and Trinick 1983). Resistance of muscle structure to expansion pressures is a major determinant in the location and amount of expansion (Lawrie 1981). Microscopic examination showed that chicken pectoralis muscle fibers are distorted after absorption and these changes in fiber shape and size alter the size of interstitial spaces (Tasker *et al.* 1959). Expansion characteristics of chicken pectoralis muscles have not been measured.

The objectives of this research were to: (1) measure the expansion characteristics of *pectoralis superficialis* muscle exposed to different concentrations of NaCl; (2) determine if the expansion was affected by muscle fiber orientation in NaCl solutions, and; (3) determine if the expansion was uniform in all directions. This research will provide information that will facilitate the design of experiments that involve muscle expansion in chicken tissue.

## MATERIALS AND METHODS

### Tissue Preparation

The *pectoralis superficialis* muscles were removed from eviscerated broiler chicken carcasses 20 – 24 h after slaughter and were stored for 18 h at -20C in sealed polyethylene bags. Cubes were cut from carefully selected locations near the center of the frozen *pectoralis superficialis* to provide tissue with muscle fibers oriented in the same direction. The tissue was frozen before the cubes were removed to prevent compression and movement of the tissue and improve the accuracy of the cut. Cube dimensions (.75 – 1.25 cm<sup>3</sup>) depended on the requirements of individual studies. The cubes were allowed to equilibrate to 22C in a sealed glass bottle. Each muscle cube was placed on a histostat embedding cassette base (Cat. # 15158-100, VWR Scientific, Philadelphia, PA 08014) and carefully lowered to the vertical and horizontal center of the appropriate NaCl solution.

## Solution and Measurements

Reagent grade NaCl was mixed with distilled, deionized water (wt/vol) to produce .17M, .34M, .51M, .68M, and .85M solutions. The osmolality of these solutions was .32, .64, .93, 1.26, 1.57 Os/kg, respectively. Solution concentration was verified with a Corning Combination Sodium X-EL electrode (Corning Glass Works, Corning, NY 14830) connected to a Beckman 2000 ion analyzer (Beckman Instruments, Inc., Fullerton, CA 92631).

Three sides, width (W), Length (L), and depth (D), of each cube were measured with calibrated calipers before and after submersion in NaCl solutions (200 mL). Measurements L and W were perpendicular to fiber length and measurement D was parallel to fiber length in all studies.

Muscle fibers were counted after submersion in NaCl solutions at 22C (Study 5). The cubes were removed from the NaCl solutions, submerged in buffered neutral formalin at 22C for 96 h (Luna 1968), and placed at -20C for 12 h. Slices were removed from each cube perpendicular to muscle fiber length. Eosin Y stain (Fisher Scientific Co., Pittsburgh, Pa 15122-23) was applied to the surface of each tissue slice. Muscle fibers were counted at six different locations on each slice using a light microscope with 10X eyepieces and a 40X objective. The microscopic field was projected onto a one square centimeter grid, copied, enlarged, and the number of fibers were expressed as the number per square millimeter of the grid. The number of fibers from each of the six locations were averaged.

## Experimental Design

**Study 1.** Muscle tissue was cut into .75, 1.00, and 1.25 cm<sup>3</sup> samples, weighed, and submerged in .51M NaCl for 5 h at 22C. The samples were removed from the NaCl solutions and allowed to drip for 1 min before they were reweighed and the percent change in weight calculated. Twenty cubes of each size were used to determine the effect of cube size on the amount of absorption and to choose a cube size for subsequent experiments.

**Study 2.** Forty-five, one cubic centimeter muscle samples were weighed and randomly assigned to one of three treatment groups. The cubes were submerged in either deionized water, .34M, or .68M NaCl for 5 h at 22C. The cubes were removed from the solutions and allowed to drip for 1 min before they were reweighed and the percent change in weight calculated. The purpose of this experiment was to determine if absorption by the 1.00 cm<sup>3</sup> cubes would increase significantly ( $P < .05$ ) when exposed to the range of NaCl concentrations to be used in the remaining studies.

**Study 3.** Thirty-six, one cubic centimeter samples were randomly assigned to one of four treatment groups to determine if muscle cubes expanded uniformly in NaCl solutions. Treatments consisted of one of three NaCl solutions (.17M, .51M, and .85M) or a distilled water control for 5 h at 22C. L, W, and D of each cube

were measured before submersion and again after suspension above the solution for one minute. The volume of each cube was calculated and the individual measurements were analyzed to identify any differences that may occur in expansion.

**Study 4.** Breast tissue cubes ( $1.00 \text{ cm}^3$ ) were submerged in deionized water or in .17M, .34M, .51M, .68M and .85M NaCl solutions to determine if orientation of fibers either horizontally or vertically with respect to the surface of the solution would have an effect on expansion characteristics. Twenty samples were removed from each of the solutions at hourly intervals for 12 h. Ten of the samples removed from each solution had been oriented with the longitudinal axes of the muscle fibers horizontal to the surface of the solution. The remaining ten cubes had been oriented with the longitudinal axes of the muscle fibers vertical to the surface of the solution. L, W, and D of each cube were measured to identify any differences in expansion characteristics and these measurements were used to calculate the volume of each cube. After measurement at each hourly interval, the samples were returned to the solution and continued on the experiment.

**Study 5.** Five muscle cubes with the longitudinal axes of their muscle fibers oriented horizontal to the surface of the NaCl solutions were suspended in .68M NaCl for 12 h at 22C. These conditions were repeated with five muscle cubes oriented with the longitudinal axes of their muscle fibers vertical to the solution surface. Tissue slices were removed from the top surface, bottom surface, and center of each cube. The first tissue slice from the top and bottom surface was discarded. The number of fibers were counted as previously described to determine differences in expansion characteristics resulting from horizontal and vertical orientation of the muscle fibers. This study was designed to measure the same expansion characteristics as study 4 using a different method of measurement.

### Statistical Analysis

Studies 1, 2, 3 and 5 were a randomly assigned, factorial arrangement of treatments and were statistically analyzed with the Analysis of Variance procedure (SAS Institute 1985). A linear regression analysis was conducted on the data from Study 3 (SAS Institute 1985). Study 4 was statistically analyzed with a split-plot repeated measures design (SAS Institute 1985). The main plot effects of trial and NaCl concentration were tested for significance ( $P < .05$ ) with the mean square calculated for the trial by NaCl concentration interaction. The subplot effect of time of submersion was tested with the mean square calculated for the trial by NaCl concentration by time of submersion interaction. Where appropriate, significantly different ( $P < .05$ ) means were separated using Duncan's multiple range test (SAS Institute 1985). Each study was repeated and data from the two trials were combined for presentation in the tables when no significant differences ( $P > .05$ ) were found attributable to the main effect of trial.

## RESULTS AND DISCUSSION

### Study 1.

The percent change in weight due to absorption of the NaCl solution decreased as cube size increased from .75 to 1.00 cm<sup>3</sup>. (Table 1). This was because the surface area to weight ration of the cube decreased as size increased. No difference in percent weight change was found when size was increased from 1.00 to 1.25 cm<sup>3</sup>. The 1.00 cm<sup>3</sup> sample size was chosen for use in the remaining studies.

TABLE 1.  
EFFECT OF INITIAL MUSCLE CUBE SIZE AND AND NAACL CONCENTRATION ON  
WEIGHT CHANGES AFTER SUBMERSION IN NaCl SOLUTIONS

Treatment	Weight change
Cube size (cm <sup>3</sup> )	%
.75	27.03 <sup>a</sup>
1.00	21.51 <sup>b</sup>
1.25	19.90 <sup>b</sup>
SEM = .95	
n = 20	
NaCl concentration	
0	2.47 <sup>c</sup>
.34M	24.88 <sup>b</sup>
.68M	41.09 <sup>a</sup>
SEM = .97	
n = 30	

<sup>a-c</sup>Means for each treatment with no common superscripts are significantly (P<.05) different.

### Study 2.

The weight of the muscle cubes increased as NaCl concentration in the solutions increased (Table 1). The NaCl concentrations used in this study were below the minimum concentration reported by Hamm (1960) to cause maximum expansion of muscle tissue. The results showed that the 1.00 cm<sup>3</sup> cube size and the NaCl concentration range used in Studies 1 and 2 would be appropriate to study the expansion characteristics of the *pectoralis superficialis* muscle.

### Study 3.

Measurements L, W, and D increased (P < .05) as NaCl concentration increased (Table 2). The means calculated for L, W, and D were significantly (P

TABLE 2.  
EFFECT OF NaCl CONCENTRATION ON CHANGES IN LENGTH, WIDTH  
AND DEPTH MEASUREMENTS OF TISSUE CUBES SUBMERGED IN NaCl SOLUTIONS

Measurements	NaCl concentration				SEM	b <sup>1</sup>
	0	.17M	.51M	.85M		
Length (mm)	10.1 <sup>c</sup>	11.0 <sup>c</sup>	12.5 <sup>b</sup>	12.9 <sup>a</sup>	.12	.559
% increase	1	10	25	29		
Width (mm)	10.1 <sup>d</sup>	10.8 <sup>c</sup>	12.0 <sup>b</sup>	13.2 <sup>a</sup>	.14	.615
% increase	1	8	20	32		
Depth (mm)	10.0 <sup>c</sup>	10.2 <sup>b</sup>	10.7 <sup>a</sup>	10.8 <sup>a</sup>	.05	.164
% increase	0	2	7	8		
Volume (mm)	1019.6 <sup>d</sup>	1219.1 <sup>c</sup>	1604.5 <sup>b</sup>	1842.9 <sup>a</sup>	35.3	

<sup>a-d</sup>Means in rows with no common superscripts are significantly different ( $P < .05$ ).  $n = 9$

<sup>1</sup>Slope of the line when the means in the same row are plotted.

< .05) different from each other and were not separated with Duncan's multiple range test because a significant ( $P < .05$ ) measurement by NaCl concentration interaction was found. The effect of NaCl concentration on the three measurements was investigated with a linear regression analysis. The slope (b) from the regression analysis showed that as NaCl concentration increased, measurements L and W increased more than did measurement D. Measurements L and W were made perpendicular to fiber length and the expansion in .85M NaCl was 29 and 32%, respectively. Measurement D was made parallel to fiber length and showed expansion of 8% in .85M NaCl.

The differences between measurements made perpendicular and parallel to fiber length were caused by physical constraints of the tissue and location of interstitial spaces relative to the muscle fibers. The smaller expansion of measurement D was not caused by contact with the cassette base because the side measured was on the opposite side of the cube and not in contact with the cassette base. The cubes were placed in the solutions without regard to orientation of the longitudinal axes of the fibers.

#### Study 4.

Muscle cubes were positioned in the NaCl solutions with the longitudinal axes of the muscle fibers either horizontal (Fig. 1) or vertical (Fig. 2) to the surface of

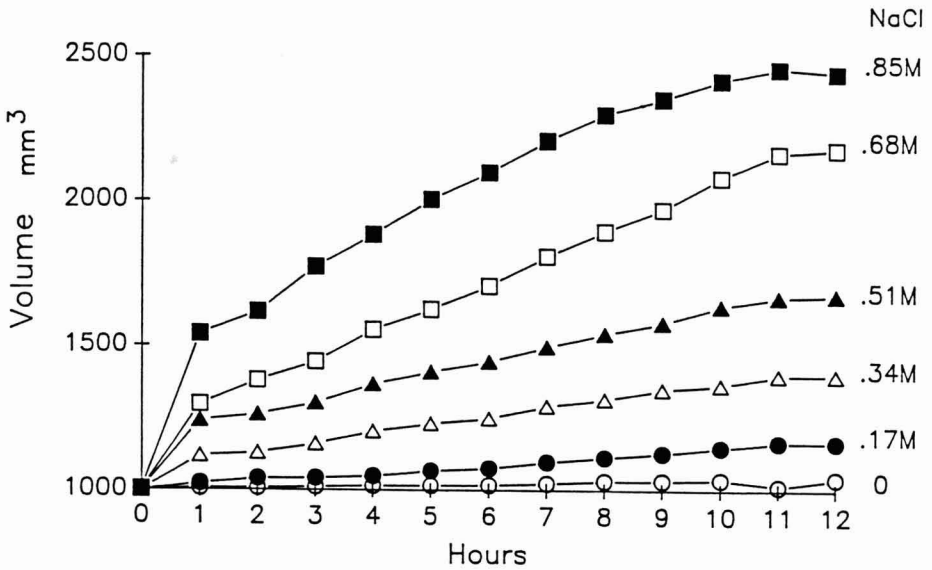


FIG. 1. EXPANSION OF *PECTORALIS SUPERFICIALIS* MUSCLE CUBE VOLUME MEASURED EACH HOUR DURING SUBMERSION IN .17M, .34M, .51M, .68M, AND .85M NaCl FOR 12 H

The longitudinal axes of the muscle fibers were oriented horizontal to the surface of the solution. n = 10, SEM = 65.3.

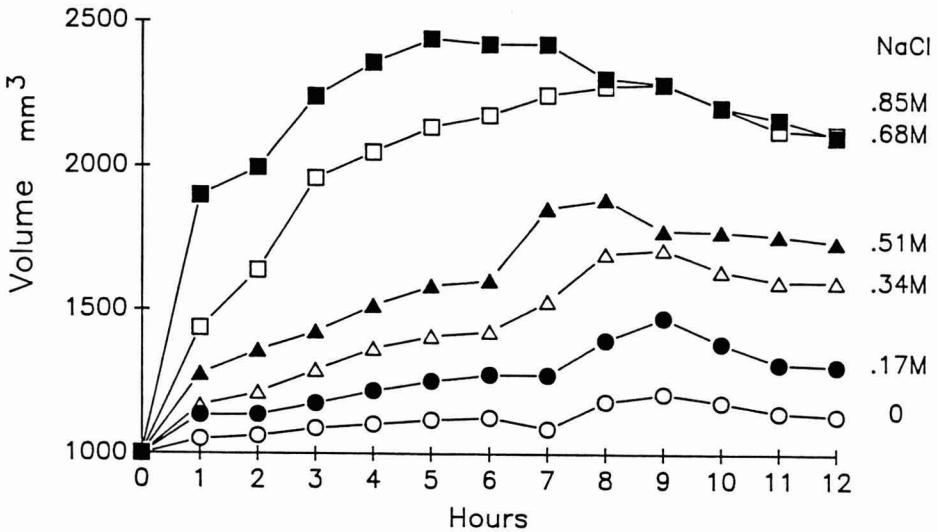


FIG. 2. EXPANSION OF *PECTORALIS SUPERFICIALIS* MUSCLE CUBE VOLUME MEASURED EACH HOUR DURING SUBMERSION IN .17M, .34M, .51M, .68M, AND .85M NaCl FOR 12 H

The longitudinal axes of the muscle fibers were oriented vertical to the surface of the solution. n = 10, SEM = 79.8.

the solution. Comparison of the lines plotted in the two figures show that orientation of the fibers resulted in different volume expansion characteristics. Both increased NaCl concentration and increased length of time the cubes were submerged resulted in increased ( $P < .05$ ) cube volume. Maximum volume was found after cubes oriented horizontally were submerged for 11 h. The cubes oriented vertically had maximum volume after submersion for 7–9 h in .17M, .34M, .51M and .68M NaCl solutions and after submersion for 5 h in .85M NaCl solutions. The coefficients of variation (SAS Institute 1985) were calculated for the data in Fig. 1 and 2, and it was found to be less than 10 in each calculation.

The sides of the cubes were measured to determine if different expansion rates caused the difference in volume expansion characteristics (Fig. 3 and 4). When the cube was oriented with the longitudinal axes of the muscle fibers horizontal to the surface of the solution, measurement D was the smallest ( $P < .05$ ) of the three measurements. Measurement D was restricted by the physical constraints of muscle anatomy because it measured the lengthwise expansion of fibers. Measurement L was made on one of the vertical faces of the cube perpendicular to fiber length and was larger ( $P < .05$ ) than measurement D. Measurement W was made across one of the horizontal faces of the cubes perpendicular to fiber length and was larg-

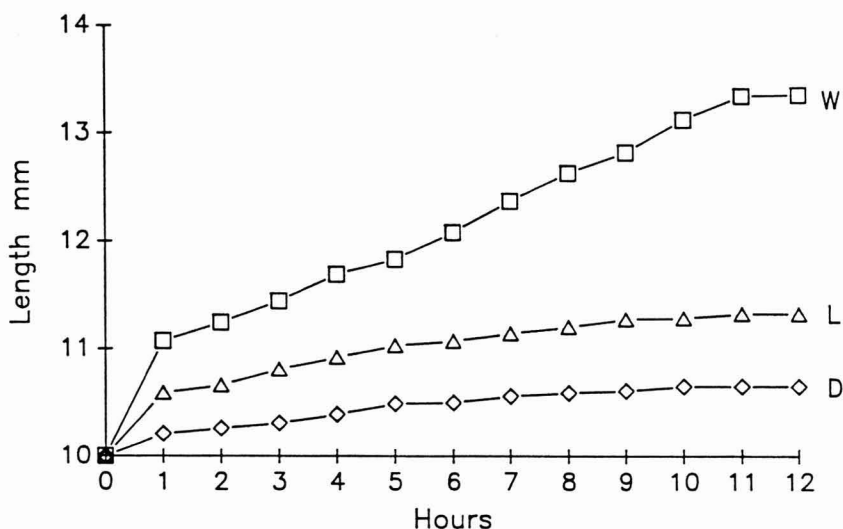


FIG. 3. LENGTH (L), WIDTH (W), AND DEPTH (D) MEASUREMENTS MADE ON *PECTORALIS SUPERFICIALIS* MUSCLE CUBES DURING 12 H OF SUBMERSION IN .17M, .34M, .51M, .61M AND .85M NaCl SOLUTIONS

The muscle fibers in each cube were oriented horizontal to the surface of the solution. L, W, and D measurements were combined for all NaCl solutions to facilitate graphing.  $n = 50$ , SEM = .3

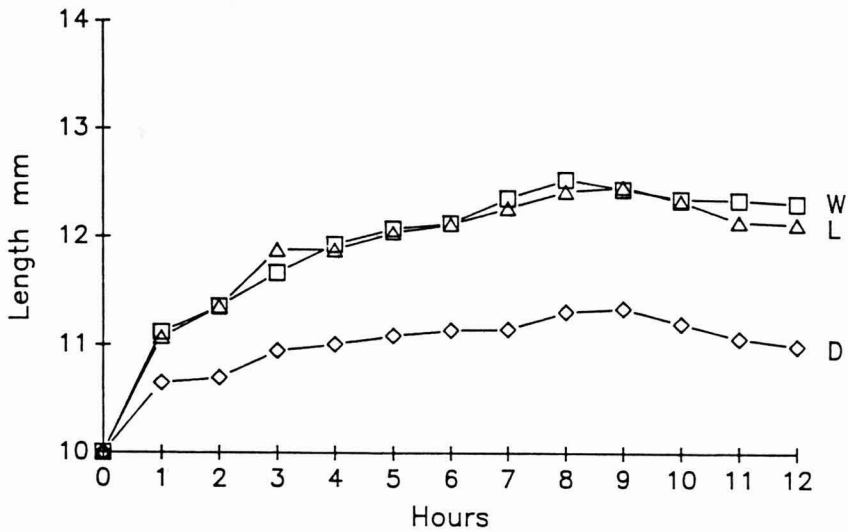


FIG. 4. LENGTH (L), WIDTH (W), AND DEPTH (D) MEASUREMENTS MADE ON *PECTORALIS SUPERFICIALIS* MUSCLE CUBES DURING 12 H OF SUBMERSION IN .17M, .34M, .51M, .68M AND .85M NaCl SOLUTIONS

The muscle fibers in each cube were oriented vertical to the surface of the solution. L, W, and D measurements were combined for all NaCl solutions to facilitate graphing.  $n = 50$ ,  $SEM = .4$

er ( $P < .05$ ) than the other two measurements. The measurement made across the horizontal face of the cube could have been larger because the fibers were compressed in this orientation decreasing the vertical measurement and increasing the horizontal measurement.

Muscle cubes were placed in the NaCl solutions so that fiber length was vertical to the surface of the solution (Fig. 4). Measurements W and L measured two of the top, horizontal faces of the cube and D measured one of the vertical faces. As before, measurement D was the smallest ( $P < .05$ ) of the three measurements. Measurements W and L were not different ( $P > .05$ ) at any sampling period. Changing fiber orientation from horizontal (Fig. 3) to vertical (Fig. 4) with respect to the solution surface reduced measurement W by 7.8% after 12 h submersion and increased measurement L by 7.1%.

### Study 5.

This experiment studied the effects of orienting muscle fibers horizontally or vertically on expansion of *pectoralis superficialis* muscle cubes during submersion in NaCl solutions (Table 3). The number of fibers within a given area of tissue were counted instead of measuring the sides of a cube. The number of fibers within the areas studied would decrease as the tissue expanded.



TABLE 3.  
EFFECT OF ORIENTATION OF TISSUE CUBES SO THAT THE FIBERS  
ARE HORIZONTAL OR VERTICAL TO THE SURFACE OF THE SOLUTION  
ON THE NUMBER OF FIBERS IN A GIVEN AREA<sup>1</sup>

Tissue slice	Muscle fiber orientation	
	Horizontal	Vertical
	-----Fibers/mm <sup>2</sup> -----	
Top surface of cube	2.7 <sup>c</sup>	2.3 <sup>d</sup>
Center of cube	3.3 <sup>a</sup>	2.9 <sup>c</sup>
Bottom surface of cube	2.7 <sup>c</sup>	3.1 <sup>b</sup>

<sup>a-c</sup>Means in rows and columns with no common superscripts are significantly different ( $P < .05$ ).  $n = 10$

<sup>1</sup>The microscopic field was projected onto a 1 cm<sup>2</sup> grid and enlarged for counting purposes. The numbers reported are counts based on the dimensions of the grid.

Slices from the top surface and middle of the cubes with fibers oriented vertical to the surface of the solution had fewer fibers per square millimeter than slices from the same location on cubes with fibers oriented horizontally. These differences were statistically significant ( $P < .05$ ). The slice from the bottom surface had more fibers per millimeter when the cubes were oriented vertically and compared to cubes oriented horizontally. A difference between the bottom and top slices was found because the bottom slice was resting on the cassette base while submerged. As expected the top of the cube absorbed more of the solution than the middle of the cube and had fewer fibers per square millimeter. This study verified the effect of fiber orientation relative to the surface of the solution on muscle tissue expansion.

These studies showed that muscle cubes can be successfully used to measure expansion characteristics of *pectoralis superficialis* muscles with possible application to other muscle systems. Muscle fiber orientation during submersion in the solution was found to be an important variable and it must be considered when designing experiments that involve tissue expansion and absorption characteristics. Expansion of the cubes when the fibers were horizontal to the surface was more uniform and allowed easier prediction of expansion and absorption than did the cubes oriented vertically. Muscle tissue expanded to a greater extent perpendicular to fiber length than it did parallel to fiber length.

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# EXTRUSION PERFORMANCE OF N-PROPANOL DENATURED SOYBEAN PROTEIN

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## ABSTRACT

*The texturization performance of alcohol-modified soybean flours plus an unmodified minimally processed flour was evaluated using a Werner & Pfleiderer twin-screw extruder under steady-state conditions. Prior to extrusion, the modified soybean flours were treated with various concentrations of aqueous n-propanol and spray dried. Evaluation of these feed materials indicated greater functionality for the n-propanol-modified proteins treated with 10% alcohol level as compared to other alcohol treatments. Following extrusion, the textural, functional and microstructural properties of the products were examined. In general, alcohol modification resulted in significant variation in the quality of the texturized soy proteins. Overall, the high quality of extruded low solubility alcohol-modified soy flour at high feed rates suggests possible renaturation mechanisms. Scanning electron microscopy examination of dried alcohol-modified products revealed microstructural features which corresponded to other extrudate properties.*

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## INTRODUCTION

The insolubilization of soybean proteins with organic solvents is a common extraction technique used in the manufacture of concentrates and isolates. Alcoholic treatments have also found limited usage in the denaturation of enzymes which are detrimental to flavor (Borhan and Snyder 1979). Since alcohol modified proteins are used extensively for commercial production of texturized vegetable proteins (TVP) via extrusion, it is essential that a basic understanding of the extrusion performance of these materials be attained.

To date, no previous studies have examined texturization of alcohol-denatured proteins. However, many studies have been conducted on the denaturation of soybean proteins with various organic solvents (Mann and Briggs 1950; Smith *et al.*, 1951; Wolf *et al.*, 1963). These studies have shown that the denaturing action of solvents is influenced by many factors (i.e., chain length, position of branching, concentration, temperature, time, etc.). In a systematic investigation by Fukushima (1969), it was determined that n-propyl alcohol had the strongest denaturing power of the water-miscible organic solvents. The author postulated some basic mechanisms involved in alcohol denaturation. He suggested that the hydrophilic groups on water soluble solvents can disrupt the hydrophilic shell of soy protein which allows the hydrophobic radicals to penetrate the hydrophobic regions. Subsequent disruption of the hydrophobic portion was viewed as the critical step necessary for denaturation of the molecule.

In the present study, a minimally processed soybean flour was denatured in aqueous n-propyl alcohol for predetermined periods of time and spray-dried. The alcohol-treated flours plus the minimally processed flour were evaluated and texturized by twin-screw extrusion. Extruded products were analyzed by rheological, functional and microscopic methods.

## MATERIALS AND METHODS

### Feed Preparation

Alcohol denatured soybean flour was prepared as follows: Soybean flour (ADM "Nutrisoy 7B", Decatur, IL) was dispersed in aqueous n-propyl alcohol solutions (0-70%) at a weight ratio of 7:1 (aqueous alcohol:soy flour) followed by mixing for 1 h at 35C. After agitation, the feed temperature was increased to 43C to facilitate dehydration. Spray dehydration of the alcohol-modified soy flour slurries was accomplished using an Anhydro Compact Spray Drier (Anhydro, Inc., Attleboro, MA) with an inlet temperature of 230C and an outlet temperature of 105C. Feed was atomized at 19,000 rpm.

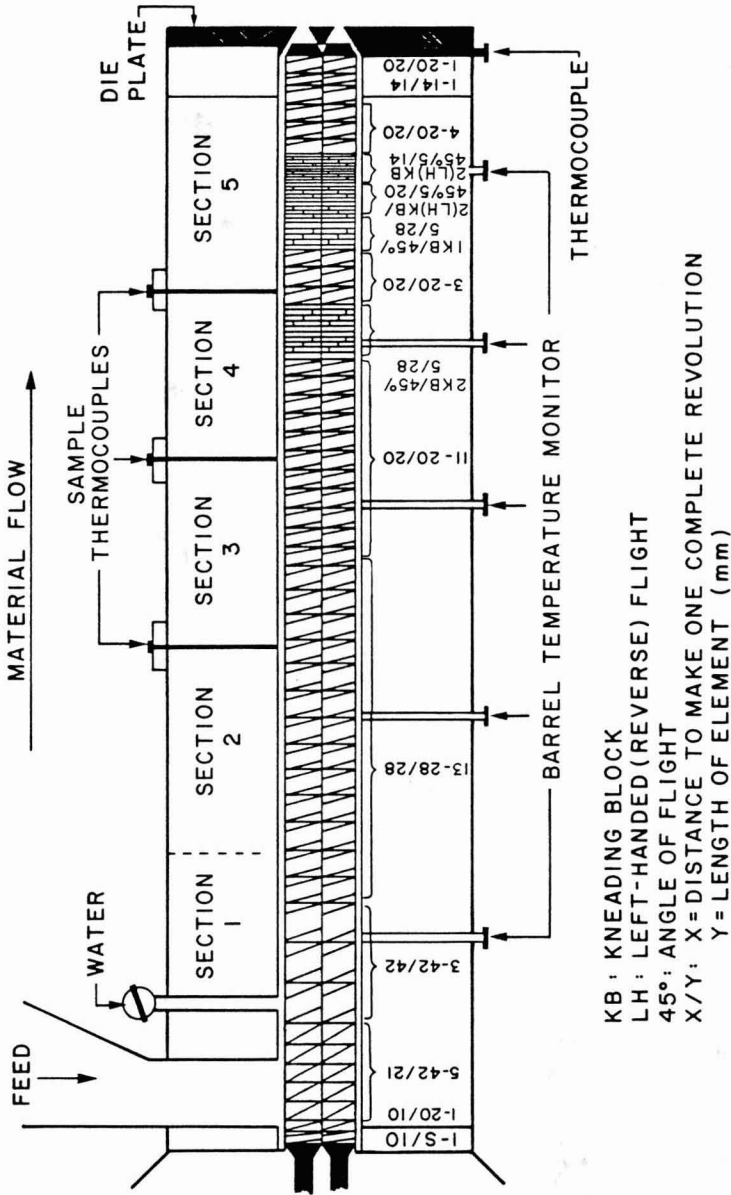


FIG. 1. SCHEMATIC DIAGRAM OF THE WERNER & PFLEIDERER ZSK-30 TWIN-SCREW EXTRUDER BARREL SECTIONS AND THE HIGH-SHEAR SCREW CONFIGURATION (From Dahl and Villota 1991)

## Functional Tests on Feeds

Water holding capacity (WHC) of feed materials was measured according to Quinn and Paton (1979) with duplicate measurements made for each sample. Water vapor adsorption (WVA) was determined by a modified method of Lang *et al.* (1981). Water uptake by the dry protein samples was expressed as g water/100 g solids after equilibration of samples exposed to 86% relative humidity (saturated KCl) at 20C (approximately 10 days). Each analysis was performed in triplicate.

Nitrogen solubility index (NSI) and macro-Kjeldahl determination of feed materials were performed by standard AACC methods (1982) on duplicate samples. Viscosity was monitored in 10% and 15% dispersions of soybean flours using a Haake Rotovisco RV-3 (Haake Buchler Instr., Inc., Saddlebrook, NJ) equipped with an NV sensor at 100 rpm and 25C. Duplicate measurements were made for each sample.

## Extrusion Processing

Extrusion-texturization of feed materials (0–20 % alcohol-treated soy flours) was performed using a Werner & Pfleiderer ZSK-30 twin-screw extruder (Werner & Pfleiderer Corp. Ramsey, NJ) equipped with a K-Tron Model T-35 twin-screw volumetric feeder and a Series 6300 digital speed controller (K-Tron Corp., Glassboro, NJ). Extrusion was carried out under the following conditions: screw speed, 400 rpm; water feed rate, 2.95 and 4.77 kg/h; dry feed rate, 11.01, and 13.85 kg/h; temperature profile in the barrel zones towards the die plate, 50°, 100°, 150°, 200°, 175°; and die diameter, 6.5 mm (dual orifice). All extrudates were produced under steady-state conditions using a high-shear screw design as shown in Fig. 1 (Dahl and Villota 1991). Extruded samples were oven dried at 50°C for 24 h, a portion of which was ground in a Burr Mill (Bauer Bros. Co., Springfield, OH) and screened to obtain a particle size range of 3.00 - 4.77 mm.

## Functional Tests on Extrudates

Rheological testing of products was carried out on one-inch segments of rehydrated extrudate (5 parts water/1 part extrudate, 1 h at 20C) in an FTC Texture-Testing System, Model T-2100-C (Rockville, MD) equipped with a standard shear compression cell, Model CS-1, using a 100 g sample per measurement. The maximum height of the resulting texturegram (kg force) was interpreted as peak shear force, while the area under the texturegram (cm<sup>2</sup>) indicated relative work of shearing. Duplicate determinations were made on each sample.

Water absorption capacity (WAC) of dried extrudates was measured as follows: 20 g ground extrudate were rehydrated in 100 mL water for 1 h, stirred at 10 min intervals, and drained of excess water for 2 min using a #20 sieve. Percent

WAC was reported as the average of duplicate samples and calculated as (weight gain upon rehydration/dry weight) X 100.

Product bulk density (BD) was determined by placing a given weight of ground extrudate in a 100 mL graduated cylinder, tapping 10 times at the 20, 40, 60, 80, and 100 mL marks and recording the resulting volume. Bulk density was expressed in g/cm<sup>3</sup> and determinations were conducted on duplicate samples.

Expansion ratio was calculated as the cross-sectional diameter of undried extrudates divided by the diameter of the die opening and reported as an average of ten replicates for each sample. Residual moisture content of undried extrudates was measured as the change in weight after vacuum drying at 60C for 24 h. Each sample analysis was performed in triplicate.

Microstructure of the extrudates was analyzed by scanning electron microscopy following a sample preparation procedure of Aguilera *et al.* (1980). Samples were examined in the lower stage of an ISI DS-130 scanning electron microscope using an accelerating voltage of 17 KV and a 30° stage tilt.

TABLE 1.  
FEED PROPERTIES

% Alcohol	Apparent Viscosity (cp) at		% NSI	% Protein*	Water	Water
	10% solids	15% solids			Holding Capacity (g H <sub>2</sub> O/g solids)	Vapor Adsorption (g H <sub>2</sub> O/100g solids)
0	102	268	85.0	52.0	2.66	19.97
5	63	247	81.0	52.4	2.14	25.46
10	85	391	42.9	51.8	2.87	25.48
15	79	325	31.5	51.3	2.68	25.63
20	65	288	20.4	51.7	2.45	24.28
50	44	151	14.8	50.1	2.13	23.04
70	32	118	17.4	48.7	1.95	19.23

\* % protein was calculated as N x 6.25

## RESULTS AND DISCUSSION

Table 1 summarizes results on feed properties. Nitrogen solubility (Fig. 2) showed a progressive decrease with increasing concentrations of alcohol up to the 20% level with a subsequent gradual leveling off. Rapid insolubilization occurred at the 10% n-propanol level, marked by a decrease to 42.1% NSI as compared to the control.



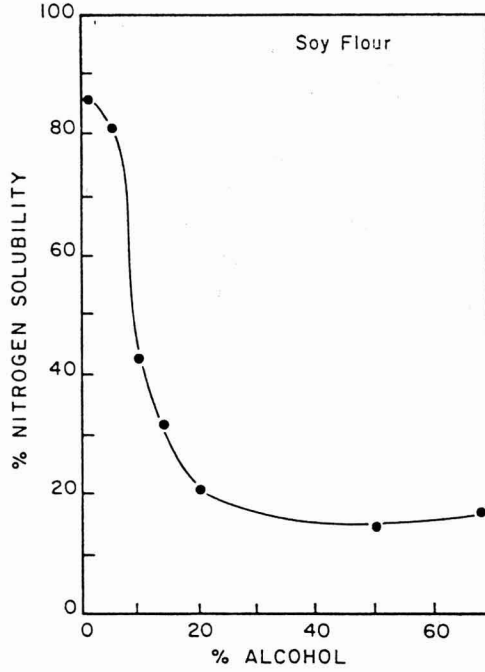


FIG. 2. DENATURATION OF DEFATTED SOYBEAN FLOUR PROTEIN WITH N-PROPANOL FOR 60 MIN AT 35C

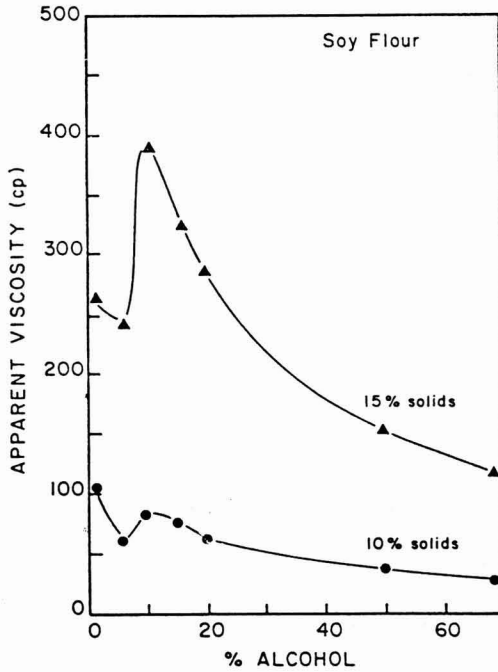


FIG. 3. EFFECT OF N-PROPANOL TREATMENT ON APPARENT VISCOSITY OF THE FEED

Soybean protein solutions exhibited an initial decrease in apparent viscosity when treated with 5% n-propanol concentration, followed by a maximum in viscosity observed in samples exposed to 10% n-propanol (Fig.3). Treatment of soy proteins with higher concentrations of alcohol (50–70%) resulted in substantially lower viscosity measurements. Some of these findings are in general accordance with viscosity studies of Shibasaki *et al.* (1972). Their investigation had shown that viscosity of globulins increased when exposed to ethyl alcohol. In addition, ultracentrifugation of the 11S unit in 30% ethanol revealed the existence of 11, 19, and 63S peaks. The authors concluded that polymerization rather than unfolding might have influenced the degree of viscosity.

Data obtained from water holding capacity (WHC) measurements showed similar trends to apparent viscosity (Table 1, Fig. 3). Linear correlations observed (WHC vs 15% solids viscosity,  $r = 0.917$ ; WHC vs 10% solids viscosity,  $r = 0.879$ ) indicate that protein-water interaction is a possible important criterion for the development of viscosity. Water vapor sorption showed slight variation from WHC data. Low water binding by unmodified soy flour may be indicative of protein conformation which is not favorable for extensive water vapor sorption. However, in aqueous systems (WHC), water binding is more prominent since slight alterations in structure induced by the liquid medium may provide a microenvironment favorable for greater water interaction. High water sorption by 5% alcohol-treated flours appears to be without a clear explanation.

Protein content of the alcohol-modified flours after spray drying remained relatively constant up to 20% n-propanol. Further alcohol addition resulted in somewhat lower values. Slightly decreased protein content may be a consequence of dilution (Table 1). Mustakas *et al.* (1962) indicated that alcohol can be quite strongly bound within the soybean solid matrix after extraction. Chung and Vilotta (1989) demonstrated that a large number of binding sites, with a low energy level, exist on soy protein. The protein becomes partially unfolded upon binding of alcohols exposing new binding sites easily accessible to alcohols. Possibly at high n-propanol concentrations, sufficient alcohol is bound within the soy flour, thus effectively decreasing the protein content by dilution. Subsequent reduction in protein available by weight will result in less protein-water interaction. Such modifications may have contributed to the reduced WHC, WVA and viscosity measurements noted at 50% and 70% n-propanol concentrations.

Extrudate properties are listed in Table 2. Peak force and work of shear measurements of products from alcohol denatured feeds (low feed rate) were found to decrease in samples treated with 5% n-propyl alcohol, followed by an increase to a maximum for samples exposed to 20% alcohol (Fig. 4). Linear intercorrelations were observed between extrudate textural properties (peak force versus work of shear,  $r = 0.953$ ). At higher feed rates, peak force of the highly alcohol denatured extrudate increased substantially more than the control.

Evidently, the denser, less expanded structure of the n-propanol modified extrudate contributed to notably higher values. Overall, textural properties did not

TABLE 2.  
EXTRUDATE PROPERTIES

% Alcohol	Expansion Ratio	Bulk Density (g/cm <sup>3</sup> )	Water Absorption Capacity (%)	Residual Moisture Content (%)	Texture	
					Maximum Force (kg)	Work of Shearing (cm <sup>2</sup> )
0 (a) <sup>1</sup>	1.40	0.476	185	16.8	351.6	10.3
0 (b)	1.73	0.323	250	13.9	201.9	11.3
5 (a)	1.10	0.495	164	7.3	313.1	6.6
10 (a)	1.07	0.550	133	15.2	342.6	8.4
15 (a)	1.05	0.570	124	14.6	352.4	10.2
20 (a)	0.96	0.591	121	11.1	353.2	9.6
20 (b)	1.49	0.461	171	13.7	274.5	13.1

<sup>1</sup> (a) Extruded at low dry feed and water feed rates.

(b) Extruded at high dry feed and water feed rates.

correspond well to functional studies performed on feed components.

Expansion properties of the extruded products decreased at both feed rates as higher concentrations of n-propanol were used to modify the protein samples. The observed linear correlations between bulk density (BD) and expansion ratio (ER) ( $r = -0.845$ ) may suggest that expansion and porosity of the extrudates are somewhat related. Other relationships were observed between feed nitrogen solubility index (NSI) and expansion properties (NSI versus BD,  $r = -0.997$ ; NSI versus ER,  $r = 0.796$ ). Such correlations indicate that the degree of alcohol denaturation as evidenced by NSI may be influential in the expansion of extruded products manufactured with alcohol-treated feeds. It should be emphasized that at higher feeding rates changes in expansion as determined by ER were substantially less than noted changes at lower feed rates. In addition, SEM showed that the microstructure was less affected. These issues will be addressed later in greater detail. Water absorption capacity (Fig. 5) as expected linearly corresponded to bulk density of products extruded at low feeding rates (WAC versus BD,  $r = 0.979$ ). Apparently, the denser, less porous n-propanol denatured samples could not imbibe large quantities of water like the control.

Product residual moisture was found to linearly relate to WHC measurements ( $r = 0.910$ ). Such correlations suggest that the protein-water interaction significantly affects the amount of water released by the extrudate upon exiting the die

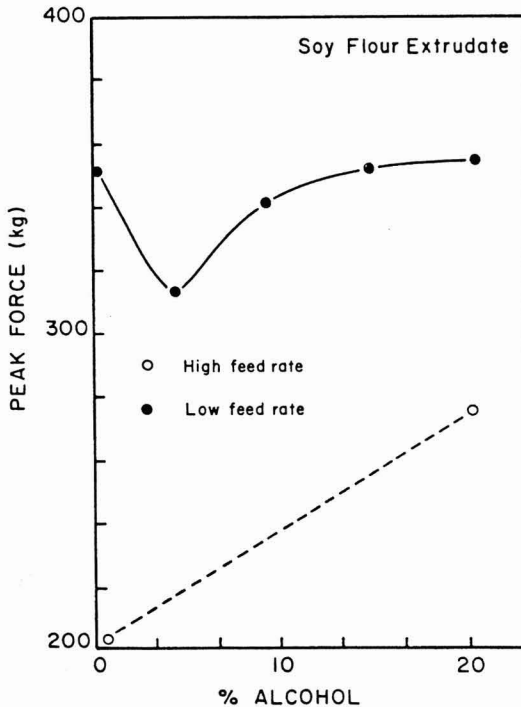


FIG. 4. EFFECT OF N-PROPANOL TREATMENT ON EXTRUDATE PEAK FORCE

orifice. Feed materials that demonstrated low water binding appeared to easily relinquish moisture while feeds with strong water attraction retained significantly more. However, subsequent loss of moisture of the material at the die did not affect the expansion of the extruded product.

Scanning electron micrographs of extrudates manufactured at low feed rates are presented in Fig. 6 and 7. Microstructural analysis of control samples (Fig. 6a) reveals thick cell wall structure and rather large air cells (up to 2,000  $\mu\text{m}$  diameter). Such features correspond to the intermediate water absorption capacity and bulk density noted for these products. Extrudates produced with 5% n-propanol-treated feed (Fig. 6b) exhibited reduced cellularity. Air cell configuration appeared to have a more circular nature as opposed to control specimens. Cell septums were quite thick (100–1,000  $\mu\text{m}$ ); however, definition of cell walls was reduced. Instead of a smooth continuous surface, alcohol-modified products yielded pores with a fibrous, rough topography. Products manufactured with 10% n-propanol-treated protein (Fig. 6c) exhibited very thick cell wall structure and decreased porosity. These results relate to the decreased WAC for these samples. Extrudates obtained with the 15% n-propanol-modified feed (Fig. 6d) lacked air cell formation and were extremely dense. Only large cracks were observed within

the protein matrix. Dense structure exhibited in these extrudates corresponded to the very low expansion ratio measurements noted. Soy flours modified with 20% alcohol (Fig. 7) resulted in extrudates showing similar trends; however, several pits were scattered throughout the matrix. High magnification of surface sections revealed fibrous protrusions arising from the exterior of the product. Similar fibrils were observed in fissures and cracks of the protein matrix. Evidently, uniform lamination of the protein filaments was decreased as compared to the control.

Under high feeding rates, ultrastructural features were quite different. The alcohol modified extrudate (Fig. 8b) showed rather thick cell wall structure (avg. 150  $\mu\text{m}$ ) and variable air cell dimensions (50–1,000  $\mu\text{m}$  diameter). However, air cells were predominantly small and circular. Unlike low feeding rates, definition of cell walls was apparent which may indicate greater uniformity in melting of carbohydrate and protein fractions and lamination of protein fibers. Control samples (Fig. 8a) yielded thinner cell walls (avg. 100  $\mu\text{m}$ ) and exhibited large, flattened air cells with increased surface area. Surface morphology of both modified and unmodified extrudates showed rough topographical features.

Apparent changes in extrudate rheology, functionality and ultrastructure appear to be related to chemical modifications introduced into the protein during alcohol

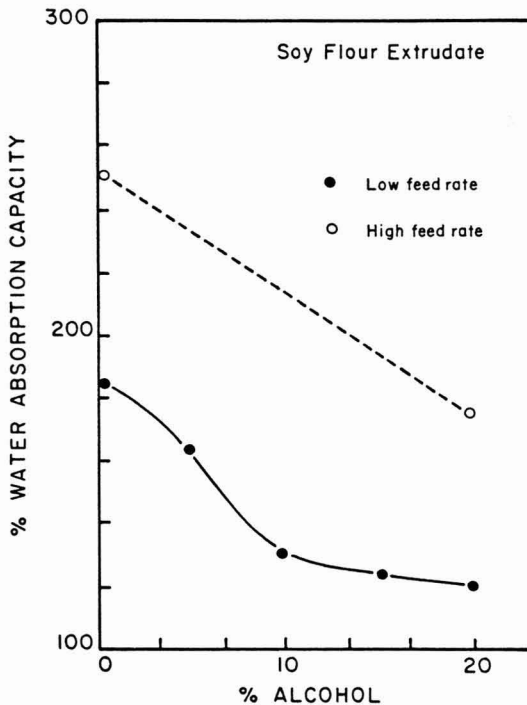


FIG. 5. EFFECT OF N-PROPANOL TREATMENT ON EXTRUDATE WATER ABSORPTION CAPACITY

treatments. Although feed functionality studies do not extensively correlate to extrudate properties, basic information on protein performance and extrudate characteristics can provide further information on texturization mechanisms. High water binding and viscosity of samples exposed to 10% n-propanol may suggest conformational changes. Soy flour prepared with higher concentrations of alcohol exhibited decreased functionality. Presumably, interaction between the unfolded polypeptide chains had occurred. At very high concentrations of n-propyl alcohol, similar trends were observed. Previous studies conducted at high alcohol concentrations (Herskovits *et al.* 1970) have discussed the mechanisms involved. Apparently, the denaturation process involves two distinct transitional stages. The initial step involves penetration of the hydrophobic region and unfolding of the protein molecule. Following disruption of the internal structure, a refolding of the molecule occurs which favors an ordered helical form.

Data from extrudate properties has demonstrated the influence of n-propanol denaturation on extrusion performance. Low feed rates showed a drastic decrease in expansion and water absorption capacity. Peak force increased at high feed rates while lower rates exhibited less alteration. Ultrastructural analysis has revealed decreases in porosity with lower NSI flours. Even small additions of n-propanol appear to have a pronounced effect on protein chemistry which may influence extrusion performance. Previous investigations from Roberts and Briggs (1963) clearly demonstrated that various components of soybean globulins are denatured at different rates when brought into contact with ethanol solutions; thus, implying that each component (subunit) behaves as a discrete species with respect to ethanol denaturation. High functionality of feed proteins in our investigation at 10–15% alcohol concentrations may indicate that some species have been favorably modified for increased water binding. However, poor extrudate functionality (low WAC and ER) of these materials may suggest detrimental alteration to certain portions of the protein fraction. From these observations, it may be possible that protein species within different stages of denaturation may improve certain feed functionality, while reducing extrusion-texturization ability.

Studies from high dry feed and water feed rates do present another interesting phenomenon, however. Extrusion of low solubility flours under high dry feed and moisture rates resulted in products with adequate texture, expansion and rehydration properties. Furthermore, scanning electron micrographs of these products demonstrated good plated sheet development and moderate expansion. Such findings may indicate that the denaturation process is not totally irreversible. Studies from Wolf *et al.* (1964) have implied that aggregation through a sulphydryl-disulfide interchange reaction may not be an important factor in alcohol denaturation of soybean globulins. If noncovalent interactions are more prominent during alcohol denaturation, renaturation may be possible. It is also possible that certain protein fractions can be renatured, and thus, proper selection of extrusion parameters may enhance this mechanism of action. Apparently, the higher feed and water

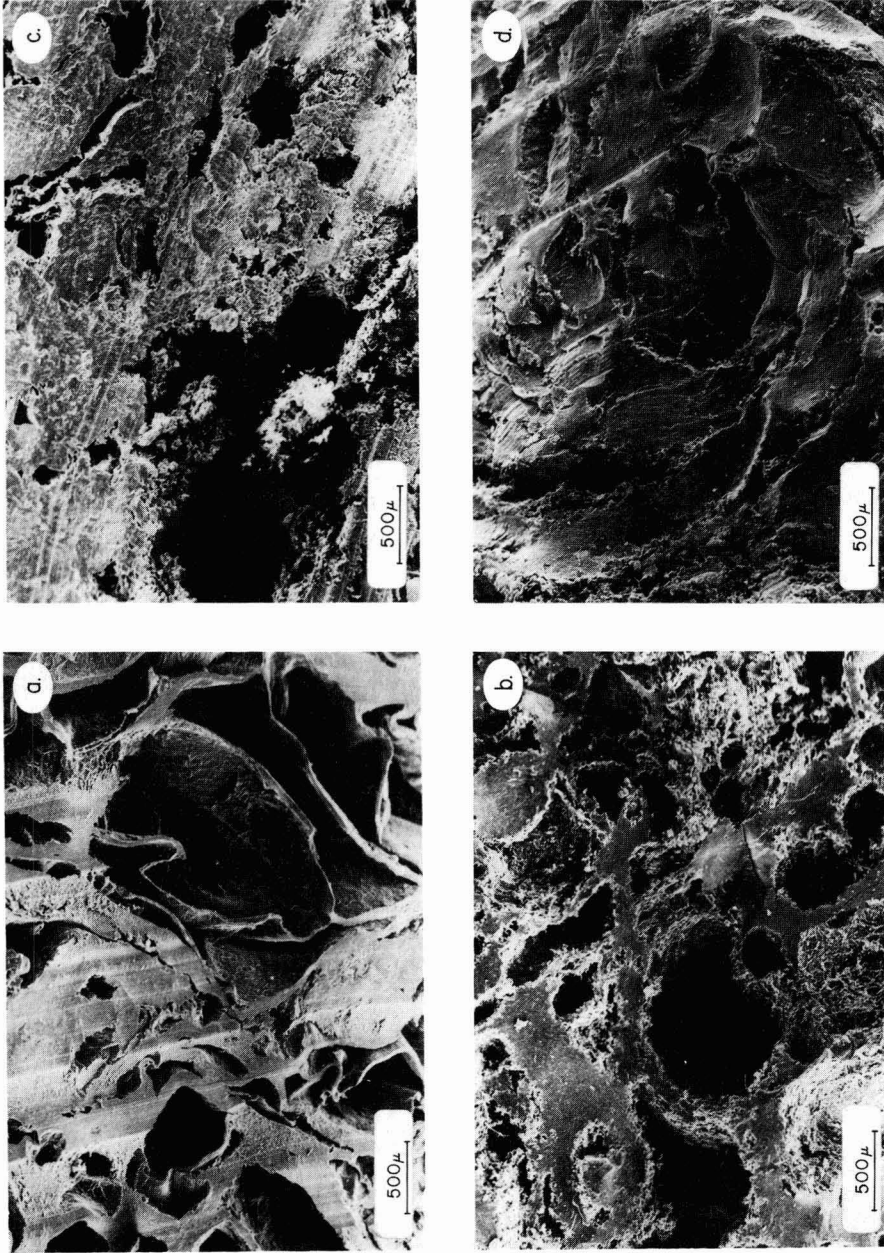


FIG. 6. SEM OF CROSS-SECTIONAL VIEW OF SOY FLOUR MODIFIED WITH DIFFERENT CONCENTRATIONS OF N-PROPANOL, AFTER EXTRUSION AT LOW FEED RATE  
 (a) 0% n-Propanol (24.7 X magnification); (b) 5% n-Propanol (27.4 X magnification);  
 (c) 10% n-Propanol (26.3 X magnification); (d) 15% n-Propanol (26.2 X magnification).

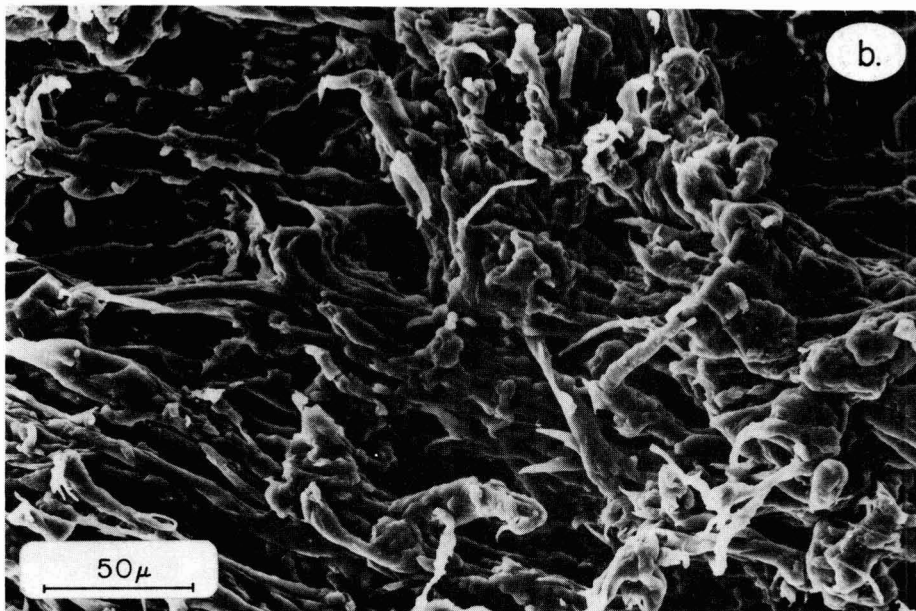
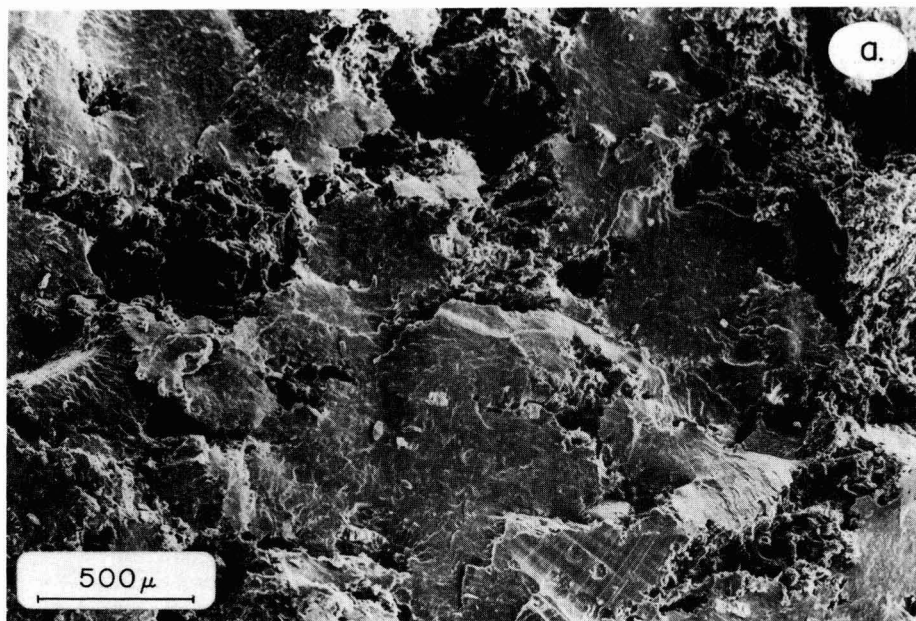


FIG. 7. SEM OF 20% N-PROPANOL MODIFIED SOY FLOUR EXTRACT EXTRUDED AT LOW FEED RATE  
(a) Cross-section (34.5 X magnification); (b) Surface (338 X magnification).



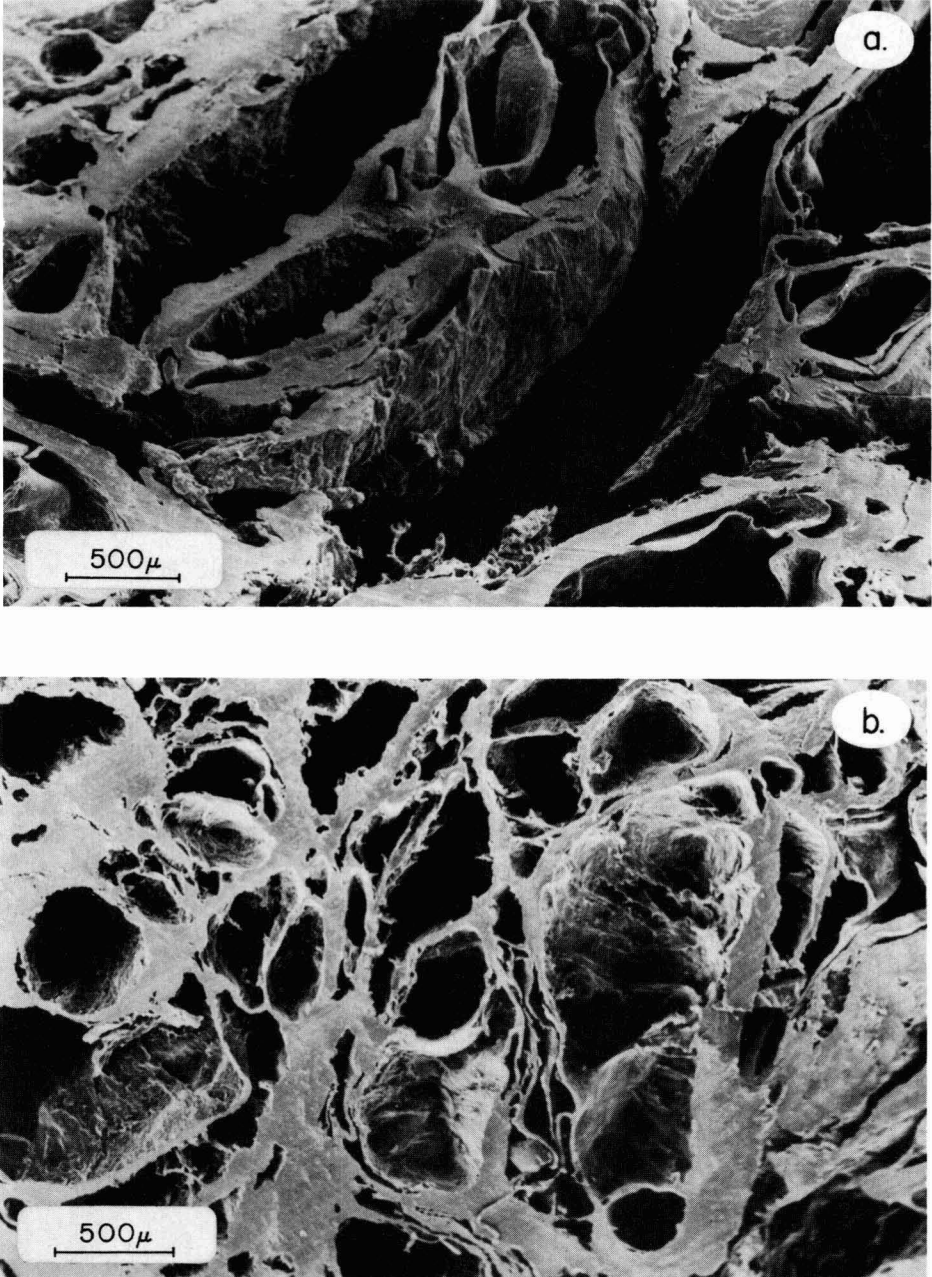


FIG. 8. SEM COMPARISON OF CROSS-SECTIONAL VIEW OF UNMODIFIED AND 20% N-PROPANOL-MODIFIED SOY FLOURS EXTRUDED AT HIGH FEED RATE (a) Unmodified soy flour (24.6 X magnification); (b) 20% n-Propanol modified soy flour (25.5 X magnification).

rates used in our study were more favorable for the production of adequately texturized products. Such increases in production rates evidently influenced the degree of shear generated within the extruder barrel. Increased shear ultimately will affect protein molecule unfolding, stretching and proper development of a laminated protein structure.

## CONCLUSIONS

The results presented in this paper have provided new information on the texturization of alcohol-modified proteins. From this investigation, it can be stated that alcohol denaturation phenomena are extraordinarily complex. Observations from feed property studies showed that even a small addition of alcohol can affect protein functionality. Although feed functional measurements did not correspond well to extrudate properties, such characteristics as water holding capacity may be influential in determining how much water is retained by the extrudate.

Overall, extrudates manufactured from alcohol-modified proteins demonstrated less expansion and lower rehydration as compared to the control. High feed rates were found to produce acceptable products with low solubility flours. Such high functionality may indicate possible renaturation mechanisms occurring. Scanning electron microscopic analysis of n-propanol modified extrudates showed decreased porosity with increasing alcohol content at low feed rates which corresponded to extrudate properties. Higher feed rates showed less variation in cellularity.

Obviously, this investigation has briefly touched upon an area in need of considerable research. It would appear essential that behavior of individual component species (2, 7, 11, and 15S) denatured by alcohol be thoroughly examined in order to fully understand the extrusion performance of these materials.

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# DESIGN AND CALIBRATION OF A CONTINUOUS TEMPERATURE<sup>1</sup> MEASUREMENT SYSTEM IN A MICROWAVE CAVITY BY INFRARED IMAGING

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## ABSTRACT

*A method for continuous measurement of surface temperature of food materials in a microwave oven during heating was developed. The method involved using a standard 2450 MHz microwave oven modified to allow for continuous measurement of surface temperature of samples using an infrared (IR) imaging system. The oven was modified by removing the top section of the microwave cavity and replacing it with 1/8 in. square hardware cloth to allow for direct thermal imaging of the sample. It was found that the wire mesh interfered with the IR measurement such that the temperature that the IR system measured differed from the actual sample temperature. The difference was also found to be dependant upon wire screen temperature. To determine the relationship between IR temperature and actual sample temperature a calibration procedure was performed. The relation between actual temperature and temperature as measured by the IR was found to be linear, and dependant upon the temperature of the wire screen.*

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## INTRODUCTION

With the increased use of microwave ovens for heating and thawing of food-stuffs both in the home and in industry much interest has been given to the study of heating patterns within the food (Ohlsson 1976, 1978; Risman *et al.* 1987). Uneven and runaway heating has been cited as one of the major problems encountered when using a microwave oven to heat or thaw food materials (Kirk and Holmes 1975; Ohlsson and Risman 1978). Typically temperature probes, such as fiber optic probes, have been used to measure temperature. However, these probes only provide information for discrete points within the sample. In order to gain information on temperature distribution a series of probes must be used, which due to cost and practicality often is not feasible. Therefore, an alternative method for measuring temperature distribution is needed.

The use of infrared imaging to measure temperature distribution in foods is a good alternative to temperature probes. Even though the IR system only has the ability to measure surface temperature it is a useful tool in determination of the uniformity of heating. Several studies have been done using IR systems to measure temperature distribution in food when heated in a microwave oven (Ohlsson and Risman 1978; Risman *et al.* 1987). However, in these experiments the oven was stopped and the sample removed before taking temperature measurements. This does not allow for continuous measurement during heating and may lead to errors due to temperature changes in the sample before the measurement can be taken. Monitoring the performance of susceptors during microwave heating is another example of how the IR imaging system along with the modified oven would be useful to monitor temperature and temperature distribution (Lentz and Crosset 1988). Temperature of a susceptor drops immediately when the microwave power is turned off because of its low thermal mass. Therefore, the objective of this work was to design and build a system which would allow for continuous and accurate temperature measurement of food being heated in a microwave oven using an IR system.

## EXPERIMENTAL METHODS

### System Setup

A Toshiba (Model ERS-6831B) 720 watt microwave oven was modified to allow for direct measurement of sample surface temperature during heating using an IR imaging system (Model 7300, Flir Systems, Portland, OR). A 27cm x 22cm rectangular section of the oven cavity top was cut away and replaced by a 1/8 in. square hardware cloth screen, which was riveted into place with pop-rivets spaced approximately 2–2.5 cm apart around the perimeter. This allowed for a clear view

of the sample through the top of the oven with the IR camera.

Note that the Food and Drug Administration/Center for Devices and Radiological Health (FDA/CDRH) stated in the 1971 Occupational Safety and Health Act (OSHA) that leakage should not exceed  $5 \text{ mW/cm}^2$  (Lambert 1980). Maximum leakage for the modified oven described here, measured with a microwave survey meter (Model HI-1501, Holaday Industries, Inc., Eden Prairie, MN), was  $3 \text{ mW/cm}^2$ . Modifications such as this should be done with care by trained technicians only, and all persons using the oven should be aware of the potential dangers and trained in the safe operation of the oven. It is, for safety reasons also, extremely important not to put a conducting material through the screen.

### **Temperature Measurement**

Sample temperature was measured using an Infrared Imaging System (Model 7300, Flir Systems, Portland, OR) by focusing the camera through the screen onto the sample during heating. Initial tests were done using 200 mL water loads. Water was used because it has an emissivity similar to most foods. The IR system was set up to measure the average temperature of a specified area of interest. Actual water temperature and the screen temperature were measured with a Luxtron (Model 755) fluoroptic temperature sensing unit. The screen temperature was measured by placing the probe tip directly adjacent to the screen at several locations. This procedure measures the air temperature surrounding the screen, and it was assumed that the screen would be at the same temperature as the air surrounding it.

To test whether the surface temperature that the IR system measured was the actual water temperature, measurements were taken outside of the microwave oven on water of known temperature ranging from 0C to 100C. It was found that for both stirred and unstirred water, the surface temperature, as measured by the IR system, was within 2C of the actual temperature of the bulk water as measured by the Luxtron unit. Note, that to get an accurate temperature with an IR system one must know the emissivity of the material that is being tested. For water the emissivity ranges from 0.96 to 0.98. In all measurements 0.96 was used as the emissivity of water.

### **Calibration**

Upon doing several test runs using water loads inside the microwave oven, it was discovered that the temperature reading through the screen by the IR system and the actual temperature of the water, measured using the Luxtron fluoroptic temperature probe, were not equal. Since the IR system accurately measured water temperature when no screen was present it was deduced that the screen must be interfering with the IR measurement. Therefore, a calibration needed to be performed to relate IR temperature to actual water temperature.

It was hypothesized that the reason for the incorrect temperature readings through the screen was caused by the IR camera sensing a combination of the temperature of the water and the temperature of the screen. Given this hypothesis, then the relationship between IR temperature reading and actual water temperature would be a function of the screen temperature. During heating of a material in a microwave oven the temperature within the cavity increases due to heat given off by the food as well as the oven itself. Therefore, the temperature of the wire screen is likely to increase during the heating process. To test this theory, a series of experiments were run where water was heated from 5C to 100C while air, at several different temperatures, was blown across the screen to maintain it at a desired temperature. The air was supplied by a hot air blower to maintain screen temperatures at 30, 50, 70, and 100C. Actual water temperature, as measured by the Luxtron, as well as IR water temperature, was recorded during the heating process to determine the relationship between the two. A linear regression was performed at each screen temperature to determine an equation to convert IR temperature to actual temperature. A multivariable regression was also done to relate actual water temperature to IR temperature and screen temperature.

## RESULTS AND DISCUSSION

After completion of the oven modification, the system was set up with the IR camera in place above the oven focused through the mesh onto the sample. Heating experiments using water loads heated from 0 to 100C at several screen temperatures revealed that at low water temperature (below ambient) the temperature reading that the IR system measured was higher than the actual temperature of the water, as seen in Fig. 1. At a temperature somewhere above ambient, the IR temperature became equal to the actual temperature and crossover occurred between the two temperatures shown in Fig. 1 and 2. At temperatures above the crossover point the actual temperature of the water was higher than the IR temperature. The temperature where crossover occurred was a function of screen temperature and varied from 36C for a screen temperature of 30C to 68C for a screen temperature of 100C.

The relationship between actual water temperature and IR measured temperature for all screen temperatures can be seen in Fig. 2. The data were fit with linear regression equations for each screen temperature as seen in the figure. The regression coefficients for these curves are given in Table 1. A multivariable regression

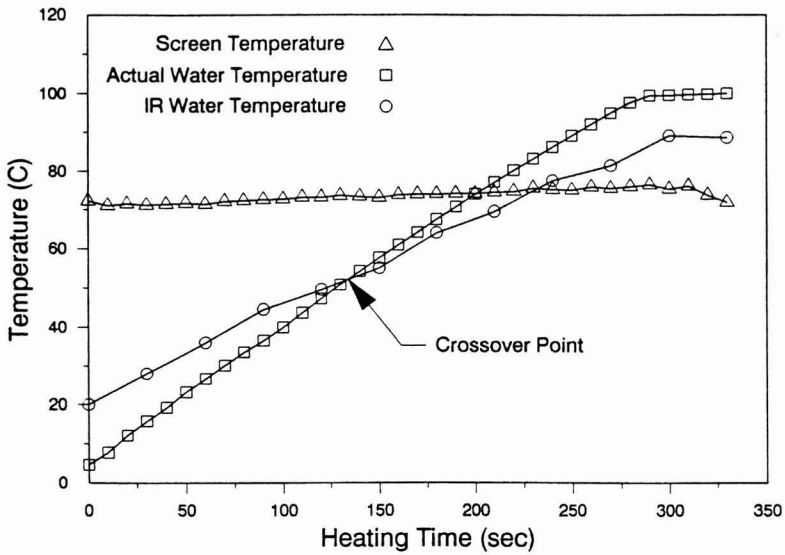


FIG. 1. INFRARED IMAGING MEASURED TEMPERATURE AND ACTUAL WATER TEMPERATURE HEATED FROM 3 TO 100C IN A MICROWAVE OVEN WITH THE SCREEN TEMPERATURE EQUAL TO 70C

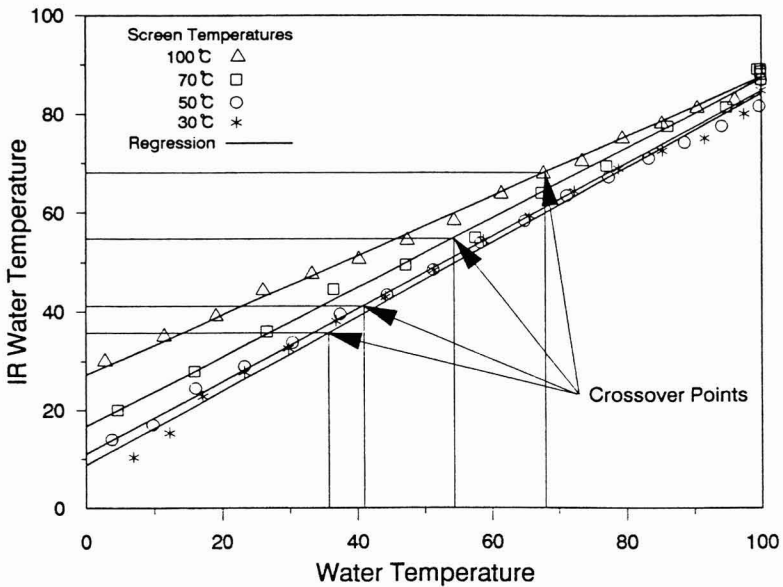


FIG. 2. RELATIONSHIP BETWEEN ACTUAL WATER TEMPERATURE AND IR MEASURED TEMPERATURE, AT SEVERAL SCREEN TEMPERATURES



was also done relating actual water temperature to IR temperature and screen temperature. This equation had the form as follows:

$$T = 5.402 + 1.151 * IRT - 0.483 * ST + 0.00472 * IRT * ST$$

where T = actual water temperature  
 IRT = IR measured temperature  
 ST = screen temperature  
 R-squared > 0.99

TABLE 1.  
 REGRESSION COEFFICIENTS FOR CALIBRATION CURVES AS SEEN IN FIG. 2

Screen Temperature (°C)	Slope	Y-intercept	R-squared
30	0.754	8.81	0.996
50	0.737	11.07	0.995
70	0.704	16.74	0.996
100	0.603	27.26	0.995

The results from these tests support the initial hypothesis that the IR camera is reading a combination of the sample temperature and the screen temperature. As the temperature of the screen was increased at a given water temperature, the temperature that the IR system measured increased, as seen in Fig. 2. However, crossover did not occur at the point where screen temperature was equal to water temperature, as might be expected. This is likely due to the difference in emissivity between the water and the screen. Water has an emissivity of 0.96 which is the value that was used in the tests. Emissivity for polished metal surfaces, which best describes the screen, ranges from 0.05 to 0.50, depending on surface roughness and degree of oxidation.

Although not shown here, the relationship between the IR measured temperature and the actual temperature was also dependant upon the food material's emissivity. If accurate temperature measurements are required for a particular food with a different emissivity than water, a calibration curve has to be determined in advance. Since the calibration varies for different screen temperatures it is best if a fan (or blower) is used to blow constant temperature air across the screen to keep it at a constant temperature during experiments. Calibration coefficients obtained in this study are unique to the particular setup and are likely not applicable to other setups. For this setup the calibration may need to be reverified at a later date if oxidation occurs on the screen causing it to have a different emissivity value.

## CONCLUSIONS

A system was designed and built whereby temperature of a food material could be measured continuously during heating in a microwave oven using an IR imaging system. Calibration parameters were found to relate IR temperature to actual sample temperature over a range of screen temperatures. A multivariable regression equation was found that would calculate sample temperature given IR measured temperature and screen temperature with an R-squared value of greater than 0.99.

## ACKNOWLEDGMENTS

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# EFFECT OF FILLING MATERIAL ON THE TEMPERATURE DISTRIBUTION IN A THERMAL CONDUCTIVITY PROBE AND THERMAL CONDUCTIVITY MEASUREMENTS: A NUMERICAL STUDY<sup>1</sup>

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## ABSTRACT

*A numerical investigation taking into account the composite nature of a line thermal conductivity probe was carried out using a finite-difference technique, to study the temperature distribution inside a thermal conductivity probe for different filling materials. Air, mercury and a high thermal conductivity paste were used as the filling materials. The radial temperature gradient in a mercury filled probe was small, while a large temperature gradient was found in an air filled probe. It was found that the location of the thermocouple in the probe has very little or no effect on the calculated thermal conductivity values, for all three filling materials tested. The plots of temperature rise versus natural logarithm of time*

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were piecewise linear. Correct thermal conductivity could be obtained only when an appropriate time interval was selected for calculation of slope. The time interval depended upon the type of filling material and the thermal capacity of the sample.

## INTRODUCTION

Knowledge of thermophysical properties including thermal conductivity is essential in prediction of heat transfer process and the performance of a heat transfer equipment. The requirement for more accurate thermal conductivity data for food material is greatly increased as the numerical modeling techniques become more powerful and sophisticated.

There are many techniques used for measuring thermal conductivity of food materials. Descriptions of these techniques can be found in the literature (Reidy and Rippen 1971; Choi and Okos 1986; Wallapapan *et al.* 1986; Murakami and Okos 1988). In general, thermal conductivity measuring techniques can be divided into two categories, steady-state and unsteady-state techniques. Unsteady-state methods are more popular for measuring thermal conductivity of food materials because much less time is require, which eliminates the complications of moisture migration an property changes. Among the unsteady-state methods, the line heat source thermal conductivity probe method is probably most frequently used and is recommended for most food applications because the method is convenient, rapid, relatively inexpensive, suitable for small samples, and relatively independent of the geometry (Mellor 1983; Ohlsson 1983; Sweat 1986; Murakami and Okos 1988).

The theory of the thermal conductivity probe method is based on the line heat source analysis (Nix *et al.* 1967). In the line heat source technique, electric power is applied at a constant rate to an infinitely long resistive wire with an infinitesimal diameter that is embedded in the material of an unknown thermal conductivity. The sample is initially at a uniform temperature. When the power is turned on, the temperature rise at a location adjacent to the line heat source is recorded. After the initial transient period, the variation of temperature with the natural logarithm of time is linear. The thermal conductivity of the sample can them be calculated as:

$$k = \frac{q}{4\pi S} \quad (1)$$

where S is the slope of the linear region given by

$$S = \frac{T_2 - T_1}{\ln(t_2) - \ln(t_1)} \quad (2)$$

In practice, the probe method deviates from the line heat source analysis for the following reasons:

- (1) finite length of the line heat source;
- (2) finite size of the sample;
- (3) finite radius of the line heat source
- (4) the temperature is measured in the filling material surrounding the heater wire, inside a thermal conductivity probe and the fillign material, generally, has different thermophysical properties than that of the sample.

Different filling materials such as air, oil (Sweat 1986), epoxy (Morley 1966), and mercury (Tong and Lund 1989) have been used in construction of a thermal conductivity probe. These materials differ significantly in their thermophysical properties. Limited information is available in the literature describing the influence of filling materials on thermal conductivity measurements by the probe method. Therefore, the objective of this work was to carry out numerical simulation to study heat transfer in a thermal conductivity probe with different filling materials and their effect on the accuracy of thermal conductivity measurements.

## THEORETICAL CONSIDERATIONS

The simplified geometry of a composite thermal conductivity probe is illustrated in Fig. 1, which shows the central heater wire (1), the insulation (2), the filling material (3), the stainless steel tubing (4), and the sample (5) in which the thermal conductivity is of interest. It is assumed that the length to diameter ration of the probe is very large and the temperature field is axisymmetric. Thus, the temperature is a function of time and radial location only. For constant material properties, the governing transient heat conduction equation is:

$$\rho_i C_i \frac{\partial T_i}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left[ r \left( k_i \frac{\partial T_i}{\partial r} \right) \right] + Q_i \quad (3)$$

where

i = 1 : heater wire (constantan)

i = 2 : insulation (Teflon)

i = 3 : filling material

i = 4 : stainless steel tubing

i = 5 : sample of unknown k

$Q_i = Q$  for i = 1

$Q_i = 0$  for i  $\neq$  1

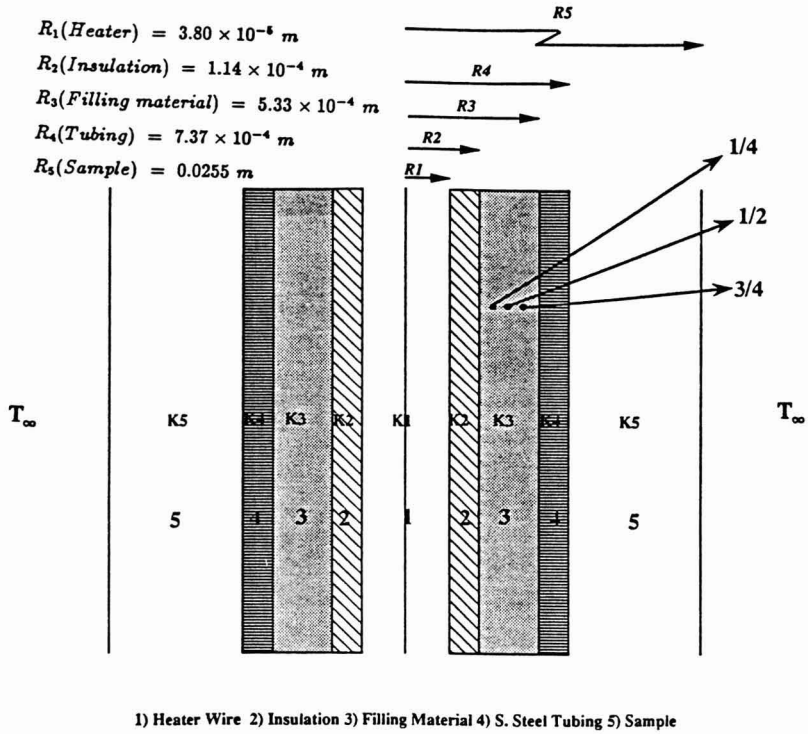


FIG. 1. SIMPLIFIED GEOMETRY OF THE COMPOSITE LINE HEAT SOURCE PROBE

**Boundary Conditions**

for  $t > 0$

at  $r = R_i, 1 < i < 5$ :

$$-k_i \frac{\partial T_i}{\partial r} = -k_{i+1} \frac{\partial T_{i+1}}{\partial r} \tag{4}$$

$$T_i = T_{i+1} \tag{5}$$

at  $r = 0, (i = 1)$ :

$$\frac{\partial T_i}{\partial r} = 0 \tag{6}$$

$$T_5(t, R_5) = T_\infty \quad (7)$$

### Initial Conditions

$$T_i(0, r) = T_\infty, \quad i = 1, \dots, 5 \quad (8)$$

Various symbols appearing in the above equations are defined in the nomenclature. The temperature distribution at any time  $t$  is governed by the rate of heat input  $Q$ , thermal conductivity  $k$ , and thermal diffusivity  $\alpha (= \frac{k}{\rho C})$ .

The analytical solution to the above equations for constant material properties would be extremely tedious involving Bessel functions. It would be even more difficult and cumbersome to obtain an analytical solution for variable material properties. Therefore, a numerical method of solution was adopted.

Governing Eq. 3 was solved by a finite difference technique. Because of axial symmetry, the solution was obtained along the radial coordinate  $r$  only. The computational domain  $0 \leq r \leq R_5$  was divided into 2800 grid points giving the grid spacing  $\Delta r = 0.01$  mm. The time step  $\Delta t$  was 0.01 s.

The finite difference equations obtained after discretization of Eq. 3 were solved by means of the Crank-Nicolson (Jaluria and Torrance 1986) scheme. The accuracy of the numerical scheme is of the order of  $(\Delta t)^2$  and  $(\Delta r)^2$ . The numerical solution was obtained for a given amount of time, which typically was about 5 min.

The numerical scheme was applied for three different filling materials: (1) air, (2) mercury and (3) high thermal conductivity silicon paste (Omegatherm 201, Omega Engineering, Inc., Stamford, CT). The materials of the other parts of the probe were kept unchanged. The sample material was glycerin. The properties of all materials used in this simulation are given in Table 1 and Table 2 which were taken from the literature (Perry *et al.*, 1984). In all cases, power levels were chosen such that the temperature rise in the filling material was less than 12C in 300 s.

TABLE 1.  
PROPERTIES OF THE FILLING MATERIALS

	$k$	$\rho$	$C$	$\alpha$
	$(\frac{W}{mK})$	$(\frac{kg}{m^3})$	$(\frac{J}{kgK})$	$(\frac{m^2}{s})$
Air	0.026	1.165	1172	$1.9 \times 10^{-5}$
Omegatherm 201*	2.38	2380	3830	$2.6 \times 10^{-7}$
Mercury	8.0	13,600	137	$4.49 \times 10^{-6}$

\* Properties supplied by Omega Engineering, Inc., Stamford, CT.



TABLE 2.  
PROPERTIES OF THE PROBE AND THE SAMPLE MATERIALS

	$k$	$\rho$	$C$	$\alpha$
	$(\frac{W}{mK})$	$(\frac{kg}{m^3})$	$(\frac{J}{kgK})$	$(\frac{m^2}{s})$
Heater (Constantan)	21.8	8900	410	$5.97 \times 10^{-6}$
Insulation (Teflon)	0.26	2180	993	$1.2 \times 10^{-7}$
Tubing(S. Steel)	13.8	7917	455	$3.83 \times 10^{-6}$
Sample (Glycerin)	0.284	1252	2510	$9.0 \times 10^{-8}$

Heater Resistance = 213.8 Ohms/m

PROPERTIES OF THE PROBE AND THE SAMPLE MATERIALS

## MATERIALS AND METHODS

Several experimental studies were performed to verify the results of the numerical model. It is important to point out that even though the model took into account the composite nature of a thermal conductivity probe, as shown in Fig. 1, it only represented a simplified and ideal probe design that is almost impossible to duplicate experimentally. It was difficult to locate a single straight heater wire at the geometrical center of the probe, and the addition of thermocouple wires for temperature measurements would also change the thermophysical properties of the probe. Furthermore, it is difficult to know the exact location of the thermocouple tip in the filling material. Therefore, experimental data only served as indirect verifications of the computer simulation. No attempts were made to superimpose the experimental time-temperature data with calculated values.

The construction of a thermal conductivity probe, the experimental apparatus, and the data acquisition system have been previously described by Tong and Lund (1989). Thermal conductivity of glycerin sample (Fisher Scientific Co., Fair Lawn, NJ) at 30C was measured using an air-filled probe.

## RESULTS AND DISCUSSION

Results from numerical calculations are presented in temperature versus time plots at different locations in the filling material of the probe and in radial temperature profiles. Results are also presented for calculated values of thermal conductivity of the sample at different times, using Eq. (1) and (2).

Figure 2 shows variation of temperature with natural logarithm of time at three equally spaced locations ( $\frac{1}{4}$ ,  $\frac{1}{2}$  and  $\frac{3}{4}$ ) inside an air filled probe with glycerin as the sample material. Also shown in Fig. 2 is the variation of temperature 0.5 mm away from the probe inside the sample material. A large thermal gradient was predicted in the filling material. As expected, the temperature nearer to the heater is higher. However, as one can see from Fig. 2, at all three locations inside the filling material as well as at the location outside the probe, the temperature versus  $\ln$

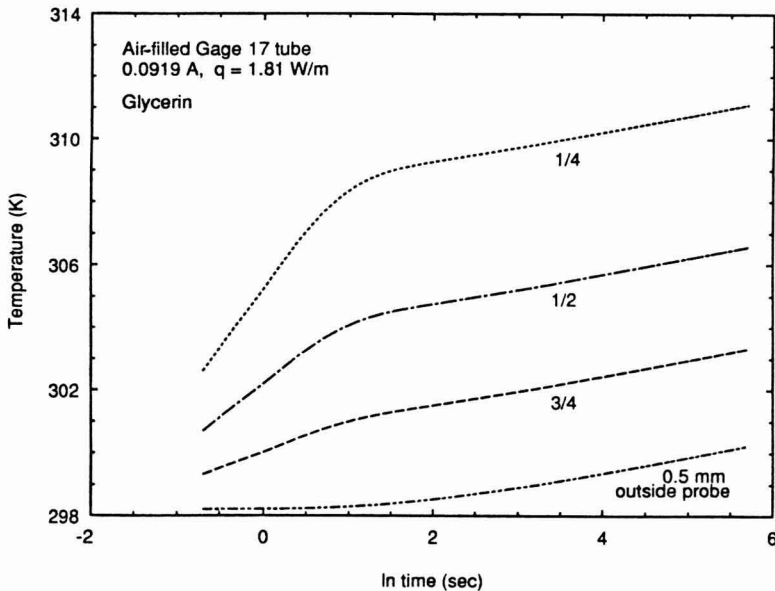


FIG. 2. TEMPERATURE VARIATION WITH  $\ln(\text{TIME})$  FOR AIR AS THE FILLING MATERIAL

(time) plot has a linear or nearly linear region. The same observation was also reported Lentz (1952).

Although it appears that all the plots have reached a linear region of constant slope. Upon close examination of the calculated values, it can be seen that the values of the slopes differ slightly from each other and are gradually changing with time as shown in Table 3. It can be seen from Table 3 that the location of the thermocouple inside the filling material does not have a significant effect on the slope and hence the estimation of thermal conductivity, which has also been reported by others (Hooper and Lepper 1950; Lentz 1952; D'Eustachio and Schreiner 1952).

The plot of temperature versus natural logarithm of time was piecewise linear which is not in accordance with theory. Figure 3 shows experimentally obtained temperature response for an air-filled probe that has the same geometry and dimensions used in the computer simulation. The experimental data also shows

TABLE 3.  
VALUES OF SLOPES AT DIFFERENT LOCATIONS

t(s)	slope at 1/4	slope at 1/2	slope at 3/4	slope at 0.5 mm outside probe
10.00	2.124249	1.303402	0.714131	0.121790
20.00	0.464704	0.457532	0.452373	0.394574
30.00	0.482550	0.478382	0.475383	0.438863
40.00	0.493596	0.490583	0.488416	0.461285
50.00	0.501051	0.498676	0.496969	0.475277
60.00	0.506466	0.504502	0.503088	0.484977
70.00	0.510605	0.508927	0.507720	0.492154
80.00	0.513886	0.512420	0.511366	0.497707
90.00	0.516561	0.515259	0.514322	0.502148
100.00	0.518789	0.517618	0.516775	0.505790
110.00	0.520678	0.519613	0.518847	0.508837
120.00	0.522303	0.521326	0.520623	0.511428
130.00	0.523717	0.522815	0.522166	0.513661
140.00	0.524960	0.524122	0.523519	0.515607
150.00	0.526063	0.525281	0.524718	0.517320
160.00	0.527049	0.526315	0.525787	0.518841
170.00	0.527937	0.527245	0.526748	0.520201
180.00	0.528740	0.528087	0.527616	0.521425
190.00	0.529471	0.528852	0.528406	0.522534
200.00	0.530140	0.529551	0.529127	0.523542
210.00	0.530754	0.530193	0.529789	0.524464
220.00	0.531320	0.530784	0.530398	0.525310
230.00	0.531843	0.531331	0.530962	0.526090
240.00	0.532329	0.531838	0.531484	0.526811
250.00	0.532782	0.532310	0.531970	0.527480
260.00	0.533205	0.532750	0.532424	0.528103
270.00	0.533600	0.533162	0.532848	0.528684
280.00	0.533971	0.533549	0.533245	0.529228
290.00	0.534320	0.533912	0.533619	0.529737
300.00	0.534649	0.534254	0.533971	0.530216

that the variation of temperature with logarithm of time is piecewise linear which is consistent with the numerical result. Similar observations have been reported by Baghe-Khandan *et al.* (1981). This also may help to explain why different values of  $r^2$  ranging from 0.8 to 0.99 have been reported for the linearity of temperature versus  $\ln(\text{time})$  plot (Wang and Kolbe 1990). It should be pointed out that direct comparison of experimental results with numerical predictions is not possible because the exact location of the thermocouple in the probe is not known. The introduction of thermocouple leads in the filling material make the geometry of the probe different than the simplified geometry shown in Fig. 1. The computer model was used to study the other effects without further direct validation.

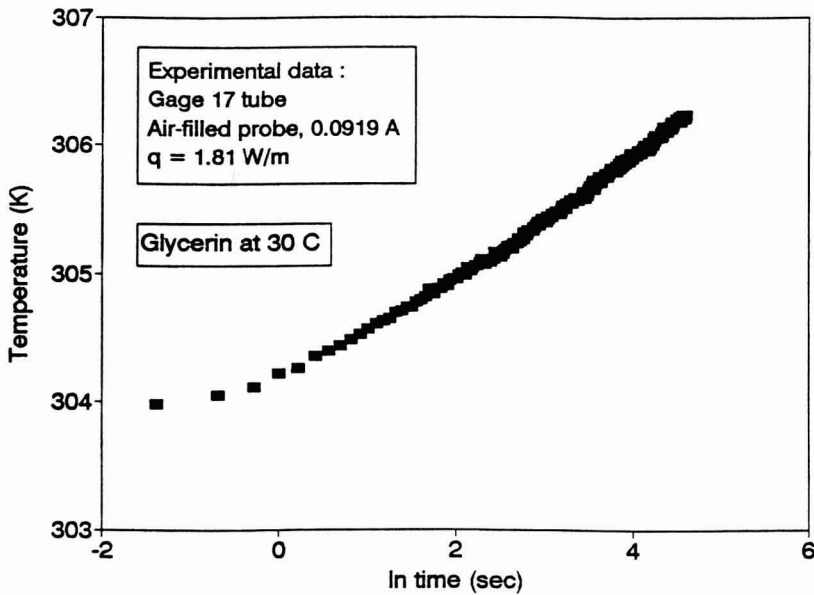
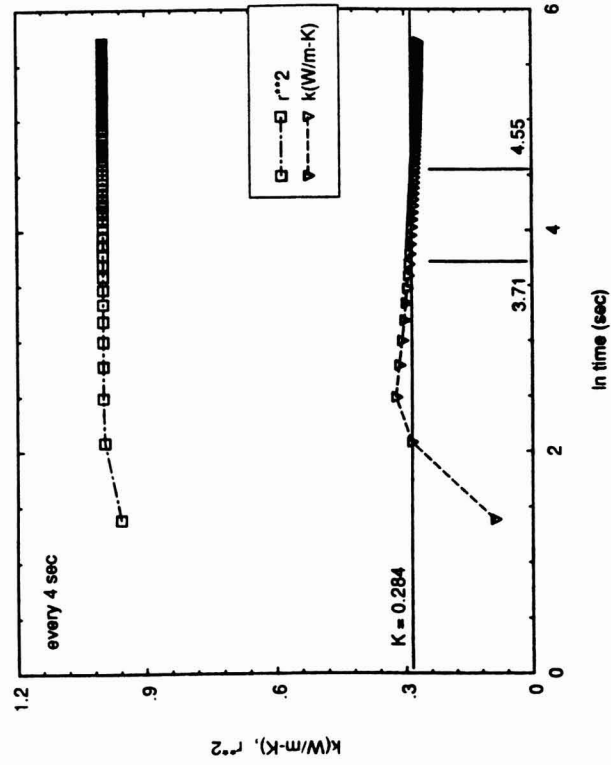


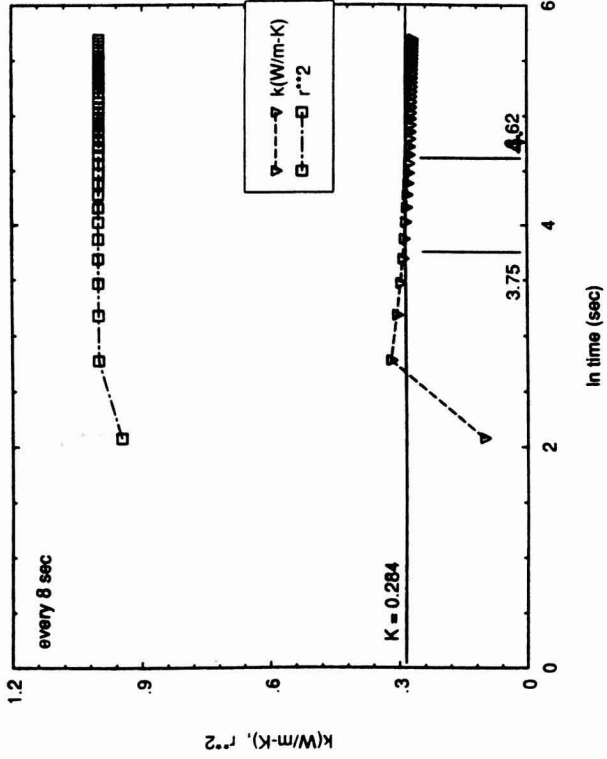
FIG. 3. EXPERIMENTALLY OBTAINED TEMPERATURE RESPONSE OF AN AIR-FILLED THERMAL CONDUCTIVITY PROBE

The data indicate that depending upon the time-temperature region chose to perform linear regression to get the slope and hence the value of the thermal conductivity, using Eq. 1, the result may vary. In this paper, the results are presented based upon the temperature gradients obtained at the middle location ( $\frac{1}{2}$ ) inside the filling material.

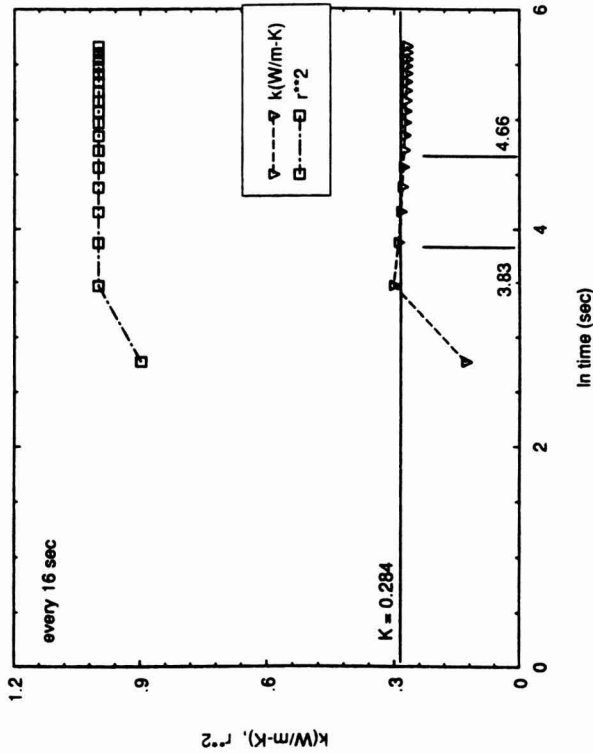
In Fig. 4(a-d) results are presented showing the effect of the time interval chosen to carry out the linear regression analysis. The time intervals chosen were 4, 8, 16 and 32 s. Also shown on the figures is the 97.5% accuracy interval which corresponds to the conductivity range of 0.276–0.291 W/mK. It can be seen that



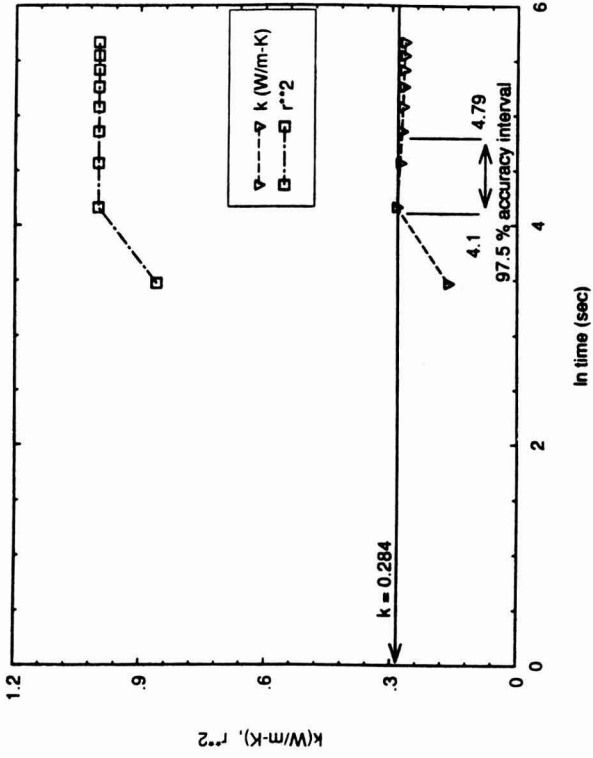
(a)



(b)



(c)



(d)

FIG. 4. CALCULATED VALUES OF THERMAL CONDUCTIVITY OF GLYCERIN USING DIFFERENT TIME INTERVALS, AIR AS THE FILLING MATERIAL.

a = 4 s; b = 8 s; c = 16 s; d = 32 s.

the 97.5% accuracy interval shifts slightly for higher time intervals corresponding to about 40–90 s for Fig. 4 (a) and 60–120 s for Fig. 4 (d).

### Effect of Filling Material

Figures 5 and 6 and 7 and 8 show the effect of Omegatherm 201 and mercury, respectively, instead of air as the filling materials on the calculation of the sample conductivity.

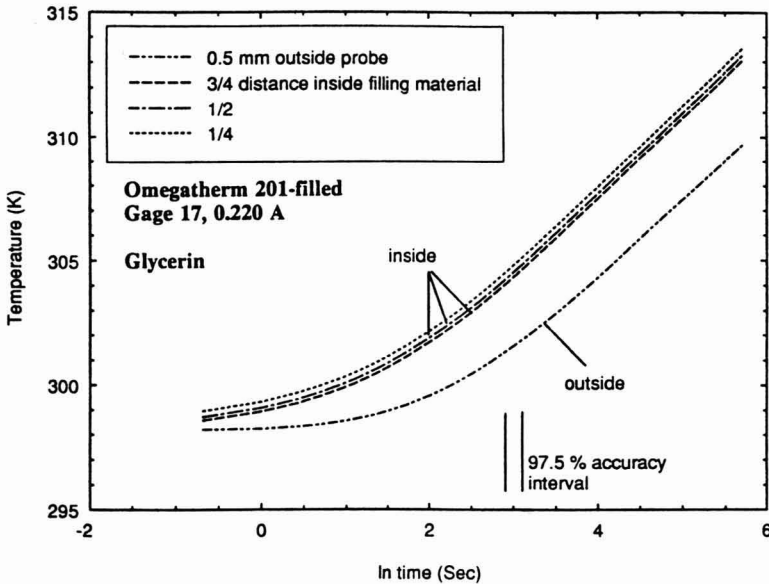


FIG. 5. TEMPERATURE VARIATION WITH  $\ln(\text{TIME})$  FOR OMEGATHERM 201 AS THE FILLING MATERIAL

For Omegatherm 201 as the filling material, the temperature variation at three locations inside the filling material as well as outside the probe is shown in Fig. 5. Figure 6 shows the corresponding calculated values of the thermal conductivity of the sample (glycerin). From Fig. 5, it can be seen that temperature difference at three locations inside the filling material is smaller than that in the case of air because of higher thermal conductivity of the epoxy. In Fig. 5 the variation of temperature with  $\ln(\text{time})$  at a point 0.5 mm outside the probe also indicates that it approaches a linear region, through at a later time. Also, it can be seen from Fig. 5 and 6 that the 97.5% accuracy interval is quite narrow (18–22 s) in the case of Omegatherm 201.

Figure 7 shows the temperature variation with  $\ln(\text{time})$  for mercury as the filling material and Fig. 8 shows the corresponding calculated values of the thermal

conductivity of the sample. Again, the slope is found to be time dependent. Although the experimental data are not shown here this phenomena has been observed in our laboratory. Because of even higher thermal conductivity of mercury, the temperature difference at the three locations inside is very small. Figure 8 shows that the 97.5% accuracy interval is much wider than that for the epoxy filled probe.

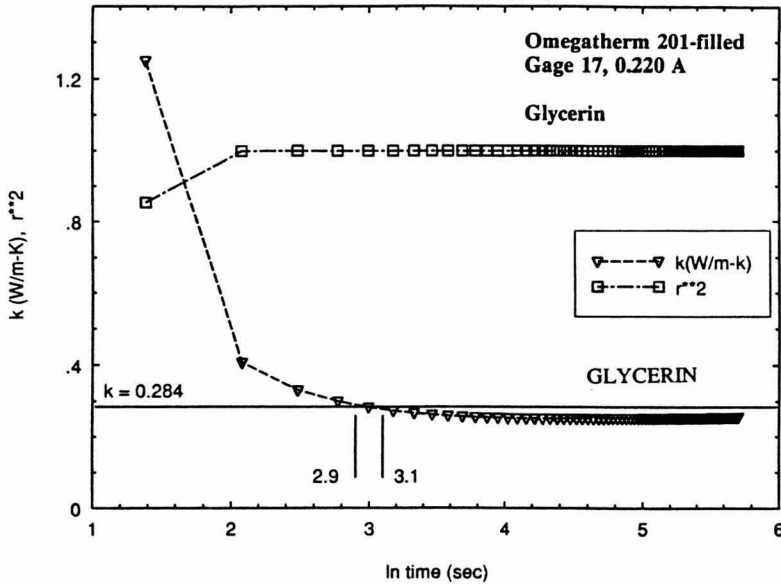


FIG. 6. CALCULATED VALUES OF THERMAL CONDUCTIVITY OF GLYCERIN USING OMEGATHERM 201 AS THE FILLING MATERIAL

Figure 9 shows the radial temperature profile inside the probe at  $t = 300$  s. As expected the temperature gradient in the filling material is steepest for the case of air compared to the other two. On the other hand, the temperature rise in the sample is smallest in the case of air. In the case of measurement of the thermal conductivity of frozen materials, it would be desirable to have a minimum temperature rise in the sample in order to minimize melting. Thus an air-filled probe would be most suitable for measuring thermal conductivity of frozen foods because of the small thermal gradient in the food with appropriate selection of time interval. However, steep temperature gradients inside the probe violates the assumption that there is no temperature gradient between the point near the heat source, where the temperature is measured, and the sample itself. On the other hand, thermal gradients in both mercury-filled and Omegatherm-filled probes are small but mercury-filled probe would be more suitable than Omegatherm 201 filled probe because of its wider accuracy interval.



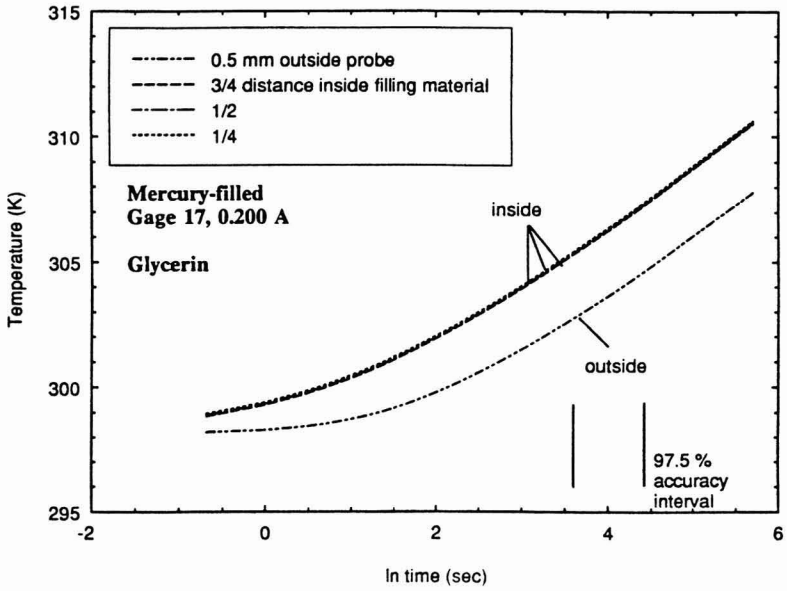


FIG. 7. TEMPERATURE VARIATION  $\ln(\text{TIME})$  FOR MERCURY AS THE FILLING MATERIAL

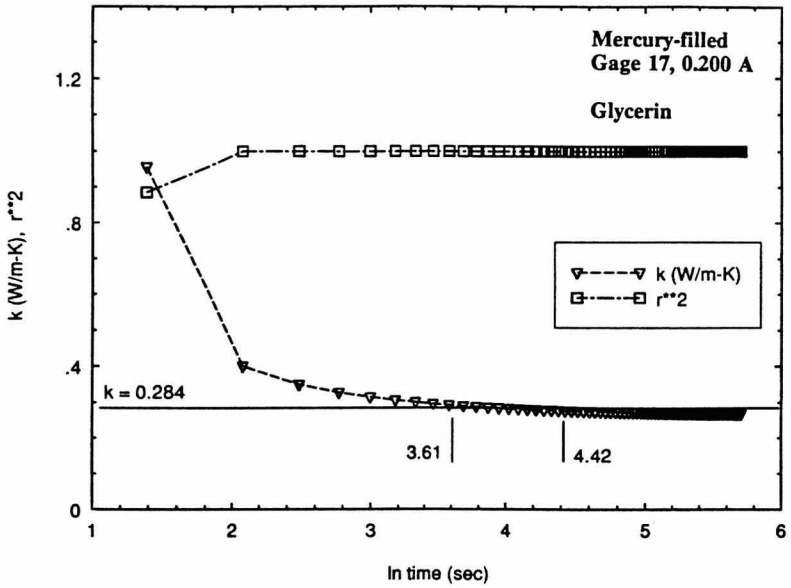


FIG. 8. CALCULATED VALUES OF THERMAL CONDUCTIVITY OF GLYCERINE USING MERCURY AS THE FILLING MATERIAL

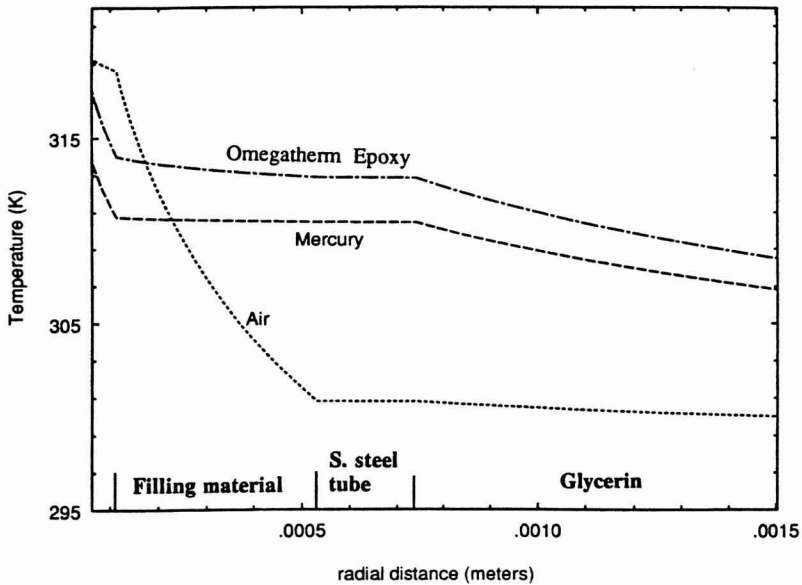


FIG. 9. RADIAL TEMPERATURE DISTRIBUTIONS NEAR THE CENTER OF THE PROBE, FOR THE THREE DIFFERENT FILLING MATERIALS AT  $t = 300$  s

### Effect of Variation of Thermal Capacity of the Filling Material

Figure 10 shows the temperature variation for the cases when the thermal capacity ( $\rho C$ ) of the filling was changed, keeping its thermal conductivity constant at the value of Omegatherm 201. It can be seen from Fig. 10 that slopes of the linear region vary with the thermal capacity of the filling material. The corresponding calculated values of the thermal conductivity of the sample are shown in Fig. 11. It can be seen that as the thermal capacity of the filling material increases, the range of time interval over which the calculated value of the thermal conductivity of the sample is within a desired accuracy becomes narrower. Thus, for a filling material with smaller value of thermal capacity, it takes longer time to reach the linear region of given accuracy. However, the extent of this region is much larger which is better from experimental point of view.

It should be pointed out that the results obtained here may be further improved with a time correction factor which has been introduced by Van der Held and Van Drunen (1949) to correct for possible errors due to the position of the thermocouple, the contact resistance between the probe and the sample, the finite radius of the heater wire, and the thermal properties of the probe materials. The time correction factor has not been popularly used by investigators. The significance of time correction factor in the possible improvement of the accuracy of the results is under investigation.

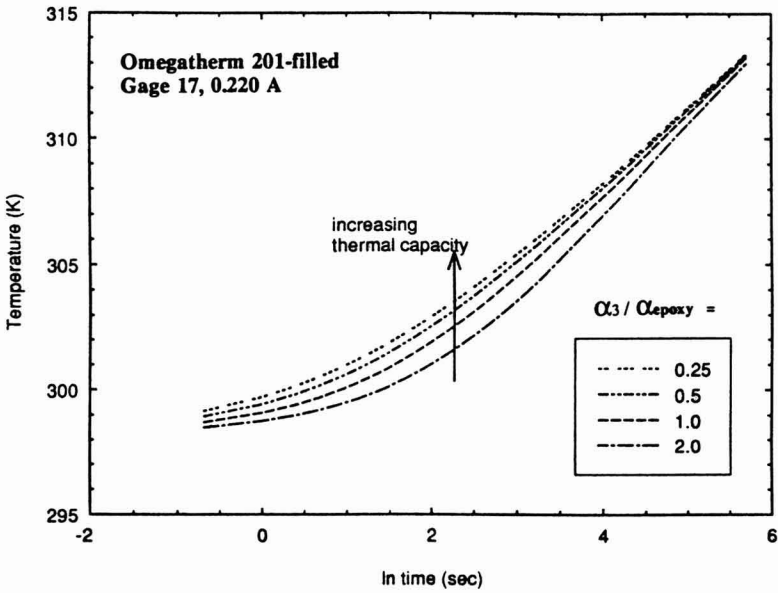


FIG. 10. EFFECT OF THE THERMAL CAPACITY OF THE FILLING MATERIAL ON THE RESPONSE OF THE PROBE

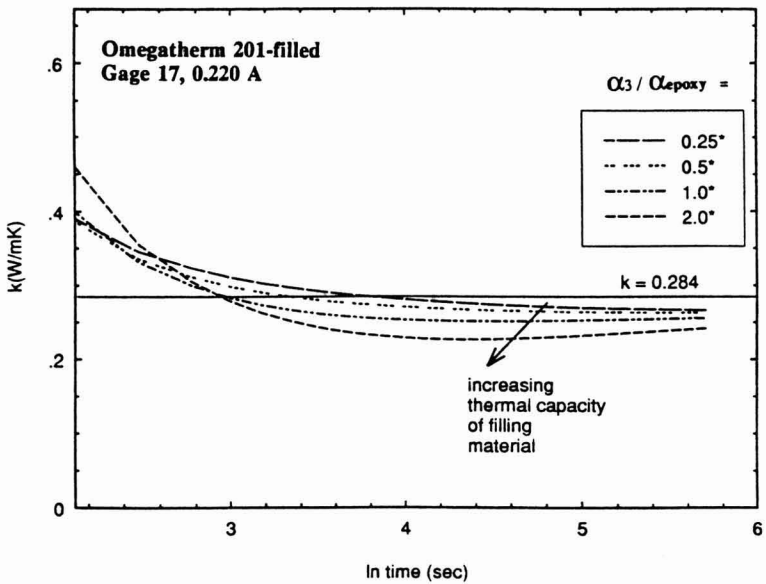


FIG. 11. EFFECT OF THE THERMAL CAPACITY OF THE FILLING MATERIAL ON THE CALCULATED VALUES OF THE THERMAL CONDUCTIVITY OF GLYCERIN

## CONCLUSIONS

A numerical simulation of the line of the line thermal conductivity probe was carried out taking into account the composite geometry of the probe. In particular, the effect of the filling material on the temperature response of the probe and thus, predicted value of the thermal conductivity of the sample, was investigated.

The results indicated that large temperature gradients may exist in the filling material, which may violate some of the assumptions. However, by choosing the right range of time interval over which linear regression is carried out, the value of  $k$  can be calculated accurately. It was found that a wide range of time exists, typically between 30–120 s, which can be used for calculations. Thermal capacity of the filling material should be as small as possible to obtain an accurate estimate of the unknown  $k$ .

Numerical simulation indicated that the probe is accurate to within 5–8%. However, for more accuracy, a proper choice of filling material and the range of time interval should be made. The numerical simulation can be helpful in the design or selection of a line probe for a particular application.

## NOMENCLATURE

C	heat capacity [ $\frac{J}{kgK}$ ]
k	thermal conductivity [ $\frac{W}{mK}$ ]
q	heater input per unit length [ $\frac{W}{m}$ ]
Q	heater input per unit volume [ $\frac{W}{m^3}$ ]
r	radial coordinate [m]
R	Radius [m]
t	time [s]
T	temperature [K]

### Greek symbols

$\alpha$	thermal diffusivity [ $\frac{m^2}{s}$ ], $\alpha = k/\rho C$
$\rho$	density [ $\frac{kg}{m^3}$ ]

### subscripts

1	heater wire
2	insulation
3	filling material
4	tubing
5	sample
$\infty$	ambient temperature

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# USE OF CHITOSAN COATING TO REDUCE WATER LOSS AND MAINTAIN QUALITY OF CUCUMBER AND BELL PEPPER FRUITS

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## ABSTRACT

*The effect of chitosan coating (1.0 and 1.5% w/v) on the storability of bell pepper and cucumber fruits stored at 13 and 20C (RH 85%) was determined by monitoring the weight loss, respiration and quality. Chitosan coating markedly reduced the weight loss in both bell pepper and cucumber at both temperatures. Increasing the concentration of chitosan from 1.0 to 1.5% (w/v) resulted in a significantly greater weight retention in both fruits. In addition, coating cucumber and bell pepper with chitosan reduced the respiration rate, loss of color, wilting and fungal infection. The mechanism by which chitosan coating delayed senescence in bell pepper and cucumber is most likely due to its ability to alleviate water stress.*

## INTRODUCTION

Dessication and decay are the two major causes of the termination of commercial life span of cucumber and bell pepper (Kader 1983). The water loss resulting from transpiration causes not only shrinkage, wilting and softening of cucumber and bell pepper, but also accelerates senescence leading to deterioration. Gen-

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erally, transpiration of the fresh produce can be reduced by storage at low temperatures and high relative humidity. But storage of these crops below 7°C is detrimental since they are chilling sensitive (Wang 1989).

Prevention of transpirational water loss can also be achieved by individual seal-packaging of fruits with high density polyethylene film (Ben-Yehoshua *et al.* 1979, 1983, 1985; Risse *et al.* 1987) and by waxing (Segall *et al.* 1974; Ben-Yehoshua *et al.* 1985). Even though these methods are effective in alleviating water stress and retarding senescence, they have several disadvantages. The drawback of seal-packaging is that the storage temperature fluctuations cause condensation of water which promotes the fungal growth (Miller *et al.* 1983). Waxing when applied without fungicides usually increase the incidence of decay and also can cause anaerobic off-flavor production (Segall *et al.* 1974; Ben-Yehoshua *et al.* 1985).

Coating fruits with substances that form semi-permeable film is yet another method used to delay ripening and prolong the storage life of produce (Lowings and Cutts 1982). Chitosan [poly- $\beta$ -(1,4)-glucosamine] appears to possess several biological properties in addition to its ability to form a semi-permeable film (Bai *et al.* 1988): it possesses antifungal activity (Allan and Hadwiger 1979; Stössel and Leuba 1984; Hirano and Nagao 1989), elicits fungal cell wall degrading enzymes such as chitinase and  $\beta$ -1,3-glucanase (Mauch *et al.* 1984), and phytoalexins (Hadwiger and Beckman 1980; Walker-Simmons *et al.* 1983). Furthermore, chitosan coating retarded ripening and prolonged the storage life of tomatoes without affecting their ripening characteristics (El Ghaouth *et al.* 1990).

The objective of this study was to determine the effect of chitosan coating on weight loss, respiration rate, appearance, and decay of cucumber and bell pepper stored at 13 and 20°C.

## MATERIALS AND METHODS

### Fruits and Chemicals

Cucumber (*Cucumis sativus* L. cv marketmore) and green bell pepper (*Capsium annuum* L. cv bell boy) were hand harvested from field plots of Université Laval, sorted on the basis of size, color and absence of external injuries. The selected fruits were randomly divided into lots of ten fruits. Crab-shell chitosan was obtained from ICN Biochemical Inc. (Cleveland, OH). All the other chemicals were of analytical grade.

### Treatment and Storage Conditions

Chitosan (1.0 and 1.5% w/v) was dissolved in 0.25 N HCl and its pH was adjusted to 5.4 with 1.0 N NaOH. To improve the wettability, 0.1% Tween 80 was added to chitosan solutions. Cucumber and bell peppers were sur-

face sterilized with 1.0% sodium hypochlorite solution (prepared from 6% commercial bleach), and subsequently rinsed in sterile deionized water and allowed the surface to dry. Finally the fruits were individually hand-dipped in chitosan solutions (1.0 and 1.5% w/v) or in deionized water containing 0.1% Tween 80. After coating the fruits were drained, dried in an oven by moving ambient air (20C and 60% RH) for 2 h, and subsequently stored at 13 or 20C in plastic containers. The containers were flushed continuously with charcoal filtered-humidified air (85% RH). Control fruits were stored under the same conditions of storage. Each treatment consisted of 4 replicates of 10 fruits (a total of 40 fruits) in a randomized complete back design.

### **Weight Loss and Appearance**

Four replicates of ten fruits per treatment were weighed at the beginning of the experiment and at various times during the course of the storage period, and the results were expressed as the percentage loss of initial weight. The fruits were inspected daily for decay and the number of infected fruits was determined and was expressed as percentage of the total. In addition, cucumber and bell pepper were rated for visual quality, color, wilting and shriveling and a grading was given from 6 to 1 on the basis of external appearance including color: 6, dark green–excellent; 4, green–good; 2, moderate; and 1, poor external appearance. The final grade of a treatment lot was the mean of the gradings of the individual fruits.

### **Ethylene and CO<sub>2</sub> Production**

The effect of coating on the ethylene production and respiration rate of cucumber and bell pepper at 13C and 20C was determined by sealing a single bell pepper or a cucumber in a 1.35 or 2.28-L container for 1 or 3 h, respectively, at which time 5 mL sample of headspace gas was withdrawn with a gas-tight syringe and analyzed for ethylene and CO<sub>2</sub> using a gas chromatograph (Perkin-Elmer, model 8500). For each treatment four single-fruit replicates were analyzed. Because some of the fruits used for respiration measurement at 20C showed sign of infection after 8 days of storage, only the results from the measurement at 13C are presented.

### **Internal Gas Composition**

To assess the effect of chitosan (1.0 and 1.5% w/v) coating on the internal atmosphere of bell pepper fruits, one-mL samples of internal gas were withdrawn from the cavity of the fruit on day 2 and 12 of storage using a syringe (Banks 1984) and analyzed for CO<sub>2</sub> and O<sub>2</sub>. Gas samples were drawn from 4–6 fruits per treatment.

## RESULTS AND DISCUSSION

Weight loss during storage was different between the fruits, chitosan concentrations, and storage temperatures (Fig. 1 and 2). The weight loss of chitosan-coated cucumber, stored for 13 days at 13° and 20°C was significantly ( $P \leq 0.05$ ) lower than that of noncoated cucumber held at respective temperatures (Fig. 1). Increasing the concentration of chitosan from 1.0 to 1.5% (w/v) resulted in a significantly greater weight retention of cucumber stored at both the temperatures. The pattern of weight loss in bell pepper was similar to that of cucumber stored under similar conditions (Fig. 2). At 13C, the weight loss was lower in bell pepper than in cucumber regardless of the treatment. However, at 20C the weight loss in bell pepper was slightly higher than in cucumber especially after 8 days of storage (Fig. 1B vs Fig. 2B). This difference could be a result of differences between

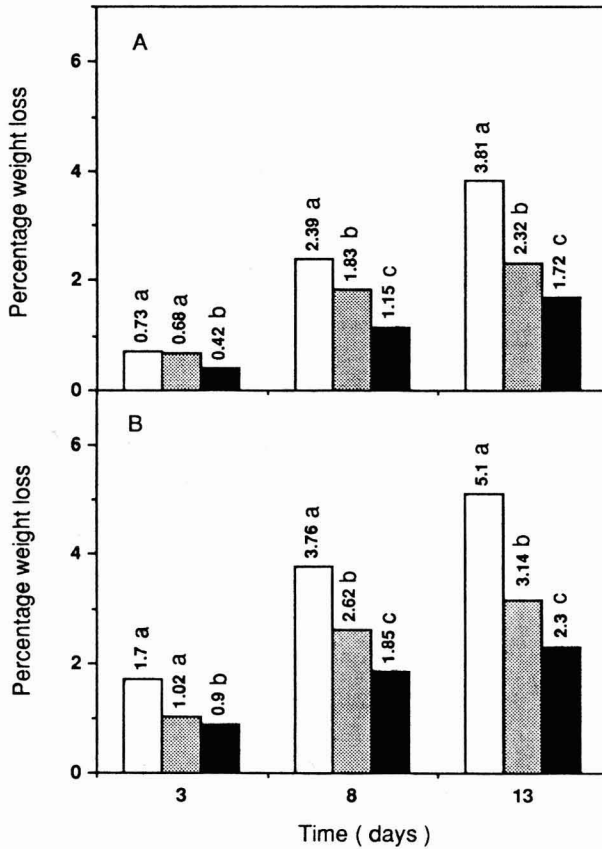


FIG. 1. WEIGHT LOSS OF CUCUMBER STORED AT 13 C (A) AND 20 C (B) □, control; ▨, 1.0% chitosan; ■, 1.5% chitosan coated. Means among each set of data labeled by the same letter are not significantly different ( $P \leq 0.05$ ) by Duncan's multiple range test.

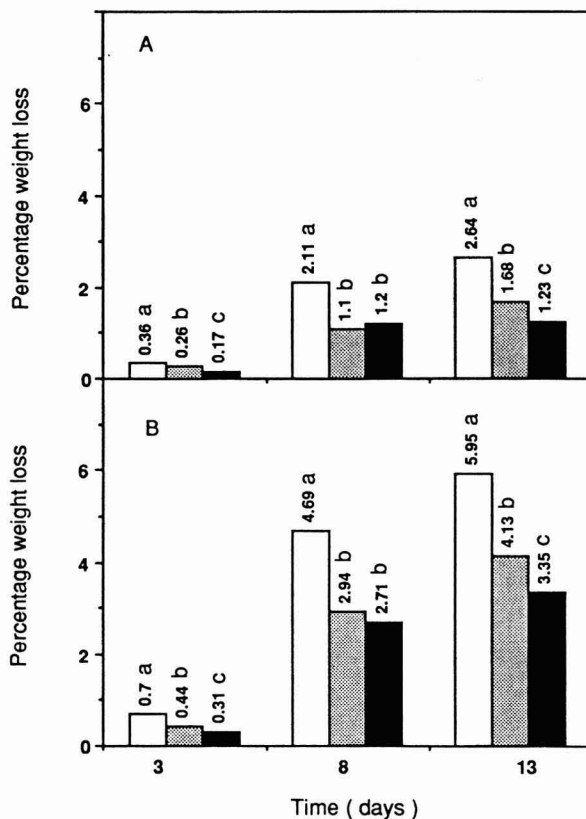


FIG. 2. WEIGHT LOSS OF BELL PEPPER STORED AT 13 C (A) AND 20 C (B) □, control; ▨, 1.0% chitosan; ■, 1.5% chitosan coated. Means among each set of data labeled by the same letter are not significantly different ( $P \leq 0.05$ ) by Duncan's multiple range test.

the crops in their inherent characteristics, such as the structure and chemical composition of the cuticle, as well as the effect of temperature on the rate of transpiration and respiration of the two crops. In general the weight loss of fruits was lower at 13C than at 20C and this was presumably due to the effect of temperature on water vapor pressure potential and on the respiration rate of the produce.

### Ethylene Production, Respiration Rate and Internal Atmosphere

Ethylene production of cucumber and bell pepper during the 13-day storage period at 13C was very small. There was a slight decrease in ethylene production in chitosan-coated fruits in comparison to the noncoated fruits (data not shown). However, the effect of chitosan coating in slowing down the respiration rate of

cucumber and bell pepper was more evident at 13C (Fig. 3 and 4). Chitosan coating markedly reduced the respiration rate of both cucumber and bell pepper. The effect of chitosan coating on respiration rate was greater at the higher concentration. Although coating reduced the respiration rate of cucumber, the rate of reduction in the respiration of the coated fruits was similar to those of the noncoated fruits. In bell pepper, however, the decrease in the rate of respiration achieved by coating was markedly higher than that of the control fruits. This suggests that coating might have affected the course of senescence (as indicated by respiration rate) more effectively in bell pepper than in cucumber; a significant reduction in respiration occurred earlier in the storage of chitosan-coated bell pepper than the cucumber.

Chitosan coating did not have any marked effect on the internal atmosphere of bell pepper. After 12 days of storage at 13C, the internal atmosphere of 1%-coated fruits (2% CO<sub>2</sub>; 19% O<sub>2</sub>) was essentially the same as that of noncoated fruits (1.7% CO<sub>2</sub>; 19.3% O<sub>2</sub>). Also there was no difference in the internal gas composition between fruits coated with 1.0% and 1.5% chitosan solutions.

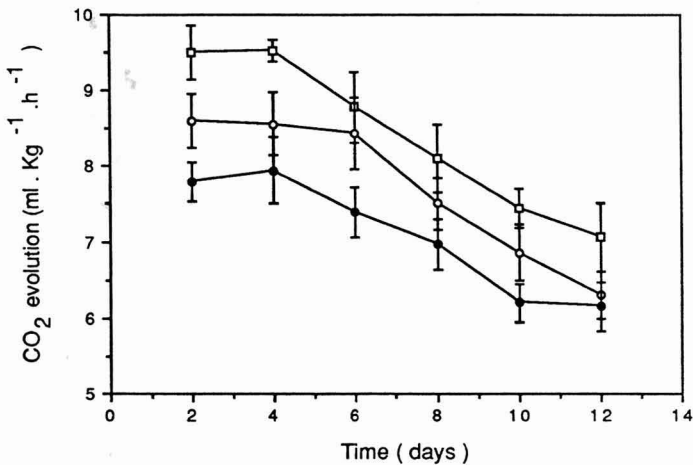


FIG. 3. EFFECT OF CHITOSAN COATING ON THE RESPIRATION RATE OF CUCUMBER STORED AT 13 C

□, control; ○, 1.0% chitosan; ●, 1.5% chitosan coated. Bars on data points represent S.E. value.

### Effect of Chitosan Coating on Quality

Chitosan coating delayed the deterioration of cucumber and bell pepper as judged by the external appearance and the level of infection (Table 1). The loss of color, wilting and decay were greater in noncoated than in coated fruits held at

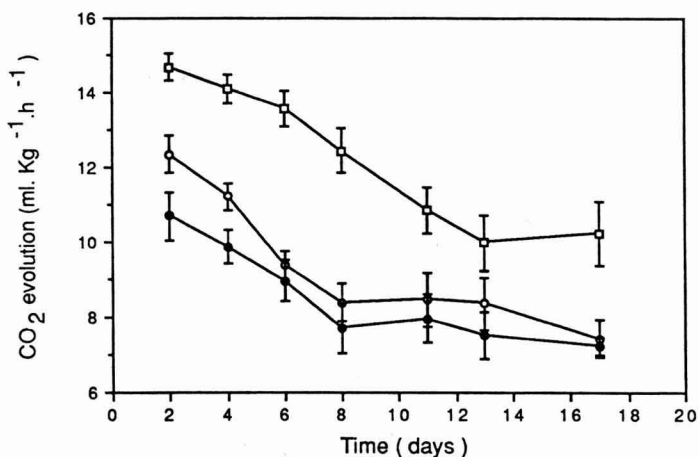


FIG. 4. EFFECT OF CHITOSAN COATING ON THE RESPIRATION RATE OF BELL PEPPER STORED AT 13 C

□, control; ○, 1.0% chitosan; ●, 1.5% chitosan coated. Bars on data points represent S.E. value.

both 13 and 20C. Cucumber and bell pepper coated with chitosan were greener, less wilted and wrinkled than the controls after 13 days of storage at 13C and 20C (Table 1). There was no added benefit on the external appearance of fruits, when the concentration of the coating solution was increased from 1.0 to 1.5% (w/v).

In addition to improving the appearance of the fruits, chitosan coating controlled the decay caused by *Botrytis cinerea*, species of *Erwinia* and *Alternaria*. Cucumber and bell pepper coated with chitosan were markedly less decayed than the control (Table 1). Increasing the concentration of coating solution did not improve the control of decay any further. A delay in the spoilage was also observed in chitosan-coated strawberries and it was attributed to the fungistatic property of chitosan (El Ghaouth *et al.* 1990).

The ability of chitosan to form a film barrier to water vapor transmission and retain the surface moisture, which alleviates water stress, appears to be the dominant factor in improving appearance and in reducing weight loss and respiration rate rather than the modification of internal atmosphere of the tissue. Although the reduction of C<sub>2</sub>H<sub>4</sub> and CO<sub>2</sub> production and the retention of firmness of fruits as a result of coating is generally explained by the modification of the endogenous levels of O<sub>2</sub>, CO<sub>2</sub> and C<sub>2</sub>H<sub>4</sub>, this is not supported by our data from the internal gas measurements in coated bell pepper. The internal levels of O<sub>2</sub> and CO<sub>2</sub> were essentially the same for both coated and noncoated fruits. Furthermore, the O<sub>2</sub> level (19%) in coated fruits is more than adequate to saturate the low affinity oxi-

TABLE I.  
EFFECT OF CHITOSAN COATING ON QUALITY AND DECAY OF CUCUMBER AND BELL PEPPER STORED FOR 13 DAYS AT 13 °C AND 20 °C

Treatment	Storage temperature (°C)	Cucumber		Bell pepper	
		Infection <sup>1</sup> (%)	Visual quality <sup>2</sup>	Infection <sup>1</sup> (%)	Visual quality <sup>2</sup>
Control	13	36.20±2.10	2.05	57.69±2.05	2.50
	20	74.37±3.74	1.40	82.11±4.32	1.61
1% chitosan	13	12.70±0.70	3.90	19.23±1.67	4.10
	20	23.15±1.13	2.84	37.43±2.43	3.10
1.5% chitosan	13	10.50±0.96	4.36	15.38±0.92	4.60
	20	19.31±1.41	3.20	33.22±2.03	3.30

<sup>1</sup> Mean ± S.E.

<sup>2</sup> Visual quality rating: 6= excellent; 4= good; 2= moderate; 1= poor.

dases in the respiratory chain as well as polyphenol oxidase lipoxygenases involved in the deterioration of tissue (Burton 1974).

The control of deterioration of fruits by seal-packaging with HDPE film (Ben-Yehoshua 1979, 1983, 1985) or coating with "Pro-Long" (Dhalla and Hanson 1988) has been previously demonstrated. Ben-Yehoshua *et al.* (1983) showed that seal-packaging delayed deterioration of lemon and bell pepper in HDPE film reduced not only their weight loss, but also reduced the leakage of amino acids and electrolytes which are indicators of membrane deterioration and senescence. Thus it is likely that the observed delay in ripening process achieved by coating was mediated by its ability to reduce transpirational losses rather through modification of internal atmosphere.

In conclusion, this study demonstrates the potential of chitosan as preservative coating for bell pepper and cucumber in reducing weight loss and respiration, and

improving the appearance. Further investigations are necessary to explore the potential of chitosan coating in controlling postharvest decay and in understanding its effect on the physiological processes associated with ripening and senescence.

### ACKNOWLEDGMENT

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# YIELDS AND BREADING DISPERSION OF CHICKEN NUGGETS DURING DEEP-FAT FRYING AS AFFECTED BY PROTEIN CONTENT OF BREADING FLOUR

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## ABSTRACT

*The effects of breading flour protein level on the yield, coating adhesion, and breading dispersion of chicken nuggets during deep-fat frying were investigated. Battered and breaded chicken nuggets with low protein breading flour had a higher ( $P < 0.05$ ) breading pickup than those with high protein flour. However, frying yields and batter and breading "drop" in the shortening were not affected ( $P > 0.05$ ) by the protein content of breading flour. The protein content of the flour affected ( $P < 0.05$ ) the dispersion of breading particles in shortening during deep-fat frying. The lower the protein content of the flour, the greater the concentration of suspended breading in the shortening.*

## INTRODUCTION

In recent years, considerable attention has been focused upon the development of further-processed poultry products as a means of providing alternative food sources of high nutritional quality. Battered and breaded products comprise the largest segment of the further-processed poultry market with parts, nuggets and patties being predominant. The acceptance of battered and breaded foods depends on coating characteristics such as overall appearance, color, flour texture, and crispness.

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Wheat flour is one of the major ingredients in a batter/breading system. Flour quality is related primarily to the gluten portion of the flour protein (Bushuk 1985; Kulkarni *et al.* 1987). Gilmour and Biggs (1976) reported that various levels of available gluten in flour can be used to obtain a desired batter viscosity. Gluten is made up of two groups of large protein molecules, which when hydrated will interact to form a cohesive matrix (Davis 1983). Flours with high protein content yield more gluten than those with low protein content. The level of flour protein affects the characteristics of fried products. Hanson and Fletcher (1963) demonstrated the effect of flour type on the color of deep-fat fried chicken parts. Recently, Rudd *et al.* (1988) reported that high levels of flour protein resulted in a darker color, increased crispness and rough texture in fried coatings.

In general, hard wheat flour tends to have higher protein levels than soft wheat flour (Davis 1983). When a flour type is mentioned, batter and breading formulas usually call for all-purpose flour. Occasionally, soft wheat flour is specifically indicated. The data to support such a choice are lacking in the literature. Davis (1983) indicated that flour with moderate protein levels of 9–10% produced more acceptable breading than did those with higher levels of 12–13% or lower levels of 7–9%.

Cooking losses and product yields are of great concern to fast food operations and poultry product companies. Gilmour and Biggs (1976) indicated that a small change in concentration of water in a high viscosity batter affects the batter pick-up and product yield. Hard wheat flour tends to have a greater proportion of its starch damaged in the milling process than soft-wheat flour and will require more water than that from soft wheats to reach the same viscosity in a batter (Davis 1983). Yang and Chen (1979) reported that batter with higher batter mix to water ratio resulted in a higher yield of deep-fat fried chicken parts. Chiu (1986) indicated that breading pickup by chicken patties was affected by batter density. Chiu showed that (1) patties prepared with a 33% (33 g dry batter mix per 100 mL of water) batter suspension had a higher breading pickup when compared to those prepared with 26.5 and 20% batters and (2) the amount of breading that fell off during deep-fat frying not only affected the yield of the product, but also affected its flavor due to the prolonged contact of the breading with the heating coils.

The objectives of this study were to determine the effects of breading flour protein concentration on the yields, breading dispersion, and coating adhesion of fried chicken nuggets during deep-fat frying.

## MATERIALS AND METHODS

### Nugget Sample

Raw chicken nuggets without batter or breading were obtained from a commercial poultry company. The composition of the nuggets was 39.9% breast meat

with skin (including the tenderloin), 26.6% water, 19.9% skin, 12.3% soy protein, 1.0% seasonings, and 0.3% polyphosphate. The nuggets were oval in shape, and averaged about 10 g in weight and 1.0 cm in thickness. They were frozen with dry ice and transported to the laboratory where they were stored in a  $-19^{\circ}\text{C}$  freezer.

### Determining the Particle Size Distribution of A Commercial Breeding

A commercial flour-based breeding (#1784601 Milwaukee Seasonings, WI) was placed on standard wire sieves in a RO-TAP Testing Sieve Shaker (The W.S. Tyler Company, OH) and shaken for 10 min to separate it into different particle sizes. The particle size distribution of the breeding was unknown, therefore, mesh No. 8, 12, 16, 20, 30, 40, 60, and 80 sieve sizes were used (Table 1).

TABLE 1.  
PARTICLE SIZE DISTRIBUTION OF A COMMERCIAL FLOUR-BASED BREADING

Particle size (U.S. Sieve #)	%
8	8.07
12	5.50
16	5.83
20	5.27
30	5.14
40	4.40
60	9.62
80	37.52
>80	18.63

### Preparation of Experimental Breeding Mixtures

Based on the particle size distribution data (Table 1), the flour-based breeding (#1784601) was separated into two portions. One portion was that which would not pass through a No. 60 screen and the other portion passed through the No. 60 screen. Three experimental breeding mixtures containing flour with a high, medium, and low protein content were prepared by mixing the bread in portions possessing particle sizes greater than mesh No. 60 with three wheat flour portions having protein contents of 14.0–15.5%, 11.5–13.5% and 8.0–9.5% (Newly Weds

Foods, IL., #20131, #21094, and #21078, respectively). The weight ratio of the flour-based breading portion and the wheat flour portion was 43.85:56.15.

### **Coating Preparation and Frying**

Prior to each frying study, ten frozen raw nugget samples were tempered at room temperature (25°C) to achieve an internal temperature of approximately 10°C. The nuggets were first dipped into a batter suspension (28g dry batter mix per 100 mL water) (NW2184, Newly Weds Foods, IL) then breaded with the above prepared breading mixtures. These battered and breaded samples were fried in preheated shortening (Crystal Wesson, Heavy-Duty clear shortening, Beatrice/Hunt Wesson CA) at 190.5°C for 30 s in a Kitchen Kettle fryer (National Presto Industries, Inc., WI). Clear, prefiltered shortening (1 L) was used for each frying study. The time between coating and frying was approximately 10 min.

### **Coating Pickup and Yields**

Batter and breading pickup and breading pickup alone were calculated from the difference between battered and breaded weight and raw sample weight and the amount of breading used. Percentage of overall yield was obtained by dividing the fried nugget weight by the raw nugget weight before coating and multiplying by 100. Percentage of frying yield was obtained by dividing the fried nugget weight by the raw nugget weight after coating and multiplying by 100.

### **Measurement of Breading Dispersion**

**Optical Density.** After each frying study, the shortening was stirred and placed in 1/2-in. diameter test tubes (Spectronic 20, Bausch & Lomb Co., NY). The optical density of the shortening was measured at 600 nM against a filtered shortening blank. Optical densities were measured immediately after sampling and at 15 min intervals up to 105 min in a Spectronic 20 Spectrophotometer (Bausch & Lomb Co., NY).

**Filtration.** After the frying of ten nuggets, the shortening in the fryer was stirred and filtered through a glass fiber filter disc (Type A-E Pore size 0.2–10 µm, Gelman Instrument Co., MI) with the aid of a vacuum. Residue on the disc was rinsed with 500 mL ethyl alcohol to remove any adsorbed shortening on the breading particles. The particles collected on the filter disc were dried and weighed as breading “drop.”

### **Coating Adhesion of Finished Product**

The method for coating adhesion as described by Suderman and Cunningham (1979) was used. Ten pieces of finished nuggets were placed in a standard wire sieve (No. 4 U.S. sieve) and shaken in a RO-TAP Testing Sieve Shaker for 1 min.

The bread crumbs that accumulated in the catch pan were weighed and the percentage of coating loss calculated.

### Data Analysis

The variables analyzed were coating pickup, yields, breading "drop" and coating adhesion. Each variable was analyzed statistically using analysis of variance in a completely random design with four replications (Steel and Torrie 1980). When a significant difference occurred, the means were separated by Duncan's New Multiple Range Test (Duncan 1955).

## RESULTS AND DISCUSSION

The batter and breading pickup of the chicken nuggets before frying ranged from 19.43% to 20.88% and most of this pickup was the breading component (Table 2). Flour-based battering and breading, which contained low protein

TABLE 2.  
BREADING PICKUP, FRYING YIELD, AND BREADING "DROP"  
DURING DEEP-FAT FRYING OF CHICKEN NUGGETS AS AFFECTED  
BY THE FLOUR PROTEIN CONTENT OF BREADING

	Flour protein level (%)		
	8.0-9.5	11.5-13.5	14.0-15.5
Batter and Breading Pickup (%)	20.88 <sup>b</sup>	20.44 <sup>b</sup>	19.43 <sup>a</sup>
Breading Pickup (%)	15.55 <sup>b</sup>	15.19 <sup>b</sup>	14.21 <sup>a</sup>
Frying Yield (%)	98.20 <sup>a</sup>	98.01 <sup>a</sup>	97.85 <sup>a</sup>
Overall Yield (%)	118.18 <sup>a</sup>	117.27 <sup>a</sup>	116.23 <sup>a</sup>
Batter and Breading "drop" in Frying Shortening (g/10 nuggets)	2.04 <sup>a</sup>	2.24 <sup>a</sup>	2.03 <sup>a</sup>

1

Mean of 4 observations.

Means within a row not followed by the same letter are significantly different (P<0.05).

(8.0–9.5%) and medium-protein (11.5–13.5%) flour, resulted in a higher ( $P < 0.05$ ) batter and breading pick-up than those containing high-protein (14.0–15.5%) flour components (Table 2). According to Gilmour and Biggs (1976), different levels of available gluten in batters can be used to give the desired viscosity. Lane and Abdel-Ghany (1986) indicated that the protein content of batter flour not only affected the viscosity of the batter, but also affected breading pickup.

Although the flour protein components affected the batter and breading pickup and breading pickup alone, there was no effect ( $P > 0.05$ ) on the frying yield, overall yield, or breading “drop” in the shortening (Table 2). The small difference between the coating pickup as well as the possible difference in frying fat pickup might be responsible for these results.

Regardless of the flour protein level, less than 0.5% of the crumb was collected when the finished product was shaken in a RO-TAP shaker for 1 min for all treatments. The protein content of the flour in a flour-based breading affected ( $P <$

TABLE 3.  
EFFECT OF STANDING TIME AND DIFFERENT FLOUR PROTEIN LEVELS OF BREADING  
ON PARTICLE DISPERSION IN FRYING SHORTENING

Standing Time (min)	Flour protein (%)		
	8.0–9.5	11.5–13.5	14.0–15.5
0	0.228 <sup>Cc</sup>	0.169 <sup>Bf</sup>	0.139 <sup>Af</sup>
15	0.178 <sup>Cb</sup>	0.128 <sup>Be</sup>	0.095 <sup>Ae</sup>
30	0.169 <sup>Cab</sup>	0.111 <sup>Bd</sup>	0.091 <sup>Ade</sup>
45	0.163 <sup>Cab</sup>	0.100 <sup>Bcd</sup>	0.080 <sup>AcD</sup>
60	0.159 <sup>Bab</sup>	0.092 <sup>Abc</sup>	0.076 <sup>Abc</sup>
75	0.148 <sup>Ba</sup>	0.082 <sup>Aab</sup>	0.073 <sup>Aabc</sup>
90	0.148 <sup>Ba</sup>	0.079 <sup>Aa</sup>	0.065 <sup>Aab</sup>
105	0.140 <sup>Ba</sup>	0.078 <sup>Aa</sup>	0.061 <sup>Aa</sup>

<sup>1</sup>  
Mean O.D. reading of 4 observations.

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Means within a row not followed by the same letter are significantly different ( $P < 0.05$ ).

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0.05) the dispersion of breading particles in the shortening (Table 3). Butaki and Dronzek (1979) reported that flour with a high protein content yields more gluten than low protein flour. The gluten of high protein flour consists of larger aggregated proteins that are more compact and more heavily cross-linked (Muller 1969). When the protein content of the flour in a flour-based breading was lower, a greater quantity of breading was suspended in the frying oil. This breading suspension was relatively stable and the suspended material may have caused a burnt odor in the shortening. Regardless of the protein content of the flour, due to the sedimentation of the particles, a slight decline in the optical density was observed after the samples stood for 15–105 min (Table 3). The fine coating ingredients that are suspended in the frying medium may have contacted the heating coil and burned, affecting the overall quality of the shortening.

In summary, the protein or gluten content of flour in flour-based breading plays an important role in the quality of deep-fat fried chicken nuggets. Low protein flour in flour-based breading released a high amount of suspended particles into the shortening, which may have caused the shortening to have a burnt odor after having come into contact with the heating coil of the fryer.

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## BOOK REVIEW

**COLONIZATION CONTROL OF HUMAN BACTERIAL ENTEROPATHOGENS IN POULTRY.** Edited by Leroy C. Blankenship. Academic Press, 393 pages.

Reducing the presence of human enteropathogens in poultry products has become a high research priority for animal agriculture in the North America and Europe. Consequently, there has been an explosion of new information on the topic. This book presents the proceedings of an international conference on control of enteropathogens in poultry. Emphasis is primarily on *Salmonella* and to a lesser extent *Campylobacter jejuni*. The book presents a comprehensive, in-depth look at current research in the area. Sixteen full length papers and 16 extended abstracts are included, and there are 75 contributing authors. The full length papers are divided into four groups: environmental factors and sources, competitive exclusion, mechanisms of colonization, and immunization. Four papers within each group combine to thoroughly cover the topic while avoiding redundancy. Papers are uniformly well written, and tables and figures are of high quality. Although there are some printer's errors, the quality of publication is quite high compared to most rapidly published symposium proceedings.

This book will be a valuable source of information for academic and industry scientists involved in pathogen control in poultry. In addition, it provides an excellent overview for scientists requiring an introduction to the subject.

JOSEPH F. FRANK

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