Response of Peanut Protein Spun Fibers to Applied Stresses[1]

E. M. Ahmed and D. L. Fletcher[2]

ABSTRACT

Dope solutions prepared from Altika peanuts protein concentrate were used for the spinning of fibers. The effects of protein concentration in the dope, as well as NaC1 and acetic acid concentrations in the coagulating bath, storage duration at 1°C and orientation configuration on the responses of the spun fibers to applied stresses were studied. The Instron Universal Testing Machine model TM was used for all measurements. Spun fibers prepared from dope solutions containing 13.0% protein were more resistant to applied tensile and shear stresses than those prepared from 13.5% protein dope solutions. Fiber strength was maximal when acetic acid and NaC1 concentrations were 2 N and 20%, respectively. Minimum concentrations of 2 N acetic acid and 15 NaC1 in the coagulating bath were required for fiber formation. Fibers stored at 1°C for 3 weeks showed increased tensile strength, stretchability and shear strength than the non-stored fibers. Orientation test results showed that two fiber tows placed in a 45° orientation were more resistant to punch shear stresses than tows placed in 90°, random or parallel orientation. Two tows required higher punch shear forces than a single tow.

Keywords: Peanut spun-fibers quality, physical properties of peanut fibers, peanut fibers stretchability, shear resistance of peanut fibers, tensile strength of peanut fibers, fiber-tows orientation.

[1]Florida Agricultural Experiment Stations Journal Series No. 6171, University of Florida, Gainesville, FL 32611.

[2]Professor of Food Science and former graduate student, Food Sci. Dept.

Much of the literature on spun protein fibers is focused on the physical and chemical properties of the dopes (viscous NaOH-protein solutions used in spinning) as well as the techniques needed to achieve desired results through fiber manipulation after coagulation. The rheological properties of the dopes [have been] related to fiber spinnability and the chemical phenomena that occur upon coagulation have been extensively investigated (3, 8, 12). There seems to be little available information on the effects of varying some of these parameters on the physical properties of the spun fibers.

The food industry has attempted for many years to produce meat analogs to supply an acceptable protein-based food product to segments of the American population which do not consume meat for religious, ethical, health or more recently for economic reasons. Most of the meat analogs thus produced are of the comminuted type in which it is not as critical to simulate the fibrous structure of meat tissues (2, 5, 11, 14, 15). The major emphasis on simulated meat products must be on the development of better methods to produce fibrous structures. Odell (10) stated that textural characteristics of a fibrous tissue are controlled by selection of a combination of fiber diameter and fiber strength and by random or palisade orientation of the spun fibers. On the other hand, spun soy fibers arranged in a perpendicular network exhibited more resistance to compressive forces than either the random or paralled orientation (6). The purposes of the present study were to investigate (a) the effects of protein concentration in the dope as well as of NaC1 and acetic acid concentrations in the coagulating bath and of the effects of short term storage at 1°C on the physical properties of peanut spun fibers and (b) the effects of orientation of fiber tows on their mechanical strength.

Materials and Methods

Protein was extracted from raw blanched peanuts according to the method used by Fletcher and Ahmed (7). The extracted proteins were washed 3 times with water (pH 4.0) to remove excess buffering salts, mixed and freeze dried at a shelf temperature of 60°C. The proximate analysis of the dried proteins showed 85.5% protein (N X 5.46), 3.1% fat and 2.0% ash. This protein preparation was used in all the studies reported herein. Dope solutions (13.0 or 13.5% protein, pH 11.4) were prepared and fibers were spun using the spinning apparatus devised by Fletcher and Ahmed (7). The resulting spun fibers (tows) were examined visually and those areas that contained defects, due to bulb formation, were removed. The lengths of tows were loosely wound around a smooth wall glass beaker, placed in a 0.05 M phosphate buffer (pH 4.0) and held at 1°C until tested.

Experimental design. The effect of protein concentration in the dope on the physical properties of the spun fibers was studied using a 2x2x3 factorial experiment. Two protein concentrations (13.0 and 13.5%) were spun in coagulation baths with constant acetic acid and NaC1 concentration (2N-20%, respectively) in three replications each, and evaluated using three physical tests.

A 4x5x3x3 factorial experiment was conducted to investigate the effects of acetic acid and NaC1 concentration in the coagulation bath on the physical properties of the fibers produced. Four acetic acid (0, 1, 2 and 3N) and five (0, 10, 15, 20, and 30%) NaC1 concentrations were investigated using three replications each. The fibers were analyzed using three separate physical tests for each treatment.

A storage study was conducted using a 4x2x3 factorial experiment. Four storage times (0, 1, 2, and 3 weeks) and two replications were evaluated using three physical tests to determine the effects of storage time on the physical properties of the fibers for each treatment.

Experiments were conducted to determine the mechanical strength of the spun fiber tows as influenced by their orientations. A single tow, and pairs of tows in parallel, perpendicular and 45° orientations were evaluated using punce shear. Three replications were used for the punch shear test.

Statistical treatments of the collected data were limited to analysis of variance, Duncan Multiple Range test, mean, standard deviation, and mean standard error (13).

Fiber strength analysis. The Universal Testing Machine, model TM equipped with a tension load cell model B and compression load cell model CB (Instron Engineering Corporation, Canton, MA) was used to determine the tensile strength, stretchability and shear strength of the spun fiber tows. All tows were allowed to equilibrate to room temperature prior to testing.

A pair of knurled bars equipped with clamps (5 cm apart) were used to hold fiber tows for the tensile strength and stretchability tests. Tows were cut in 11 cm lengths, excess tows wrapped around the bars and held in place by the clamps and stretched until the tows snapped. The Instron crosshead and recorder chart speeds were set at 3 cm/min. The force (g) required to snap the tow was considered as the tensile strength and the distance the tow was stretched, prior to breakage, was divided by the initial distance between the bars and multiplied by 100 to determine percent stretchability. Shear strength was conducted using a Warner-Bratzler type shear attachment with 3 blades (Canners Machinery Limited, Simcoe, Ontario, Canada). Sections of tows 5 cm in length were placed on the v-shaped cutouts of the blades and the tow was pulled through the slots. The Instron crosshead and recorder chart speeds were set at 5 cm/min. The force (g) required to shear the tow was considered as resistance to shear. Punch shear was conducted using the compression load cell and a cylindrical punch (3.3 mm in diameter) that would pass through a hole of the same size. A single or a pair of tows were placed directly over the hole at the intersection of the desired orientations. The pair of tows was arranged in a parallel, random, perpendicular or 45° orientation. The Instron crosshead and recorder chart speed were set at 5 cm/min. The punch was allowed to punch shear the tow(s) and maximum force (g) obtained was considered as the shearing force.

Results and Discussion

Dope Protein Concentration

Spun fiber tows produced from 13.0% protein dope were stronger in tensile strength, more stretchable and stronger in shear strength than those produced from 13.5% protein dope (Table 1). The 13.0% dope was allowed to mature much

Table 1. Effect of protein dope concentration on the physical properties of the spun fiber tows.[1]

Physical Test	Protein Concentration	Means	Standard Deviation	Mean Standard Error
Tensile strength	13.0	67 a	5.8	0.75
(g)	13.5	50 b	5.7	0.74
Stretchability	13.0	235 a	27.1	3.50
(%)	13.5	186 b	20.3	3.91
Shear strength	13.0	486 a	84.8	10.95
(g)	13.5	323 b	46.7	6.03

[1]Coagulating bath conditions; 20% NaCl, 2N acetic acid.

Means, within each physical test, followed by the same letter are not different ($P < .05$).

longer (10-12 hrs) than the 13.5% dope (1-2 hrs) to reach optimum spinning viscosity. This longer holding period and the lower protein concentration could have allowed for more conformational changes and cross-bonding in the protein molecules of the 13.0% dope, which in turn allowed for a more closely packed structural arrangement upon extrusion and coagulation. The 13.5% dope had a relatively short maturation time, since it would have no doubt formed a gel within several hours, and therefore did not have the same conformationa results as did the 13.0% dope.

Coagulating Bath Variables

Combinations of NaCl and acetic acid capable of coagulating peanut protein spun fibers were found to be at concentrations of 15-30% for NaCl and 2-3 for acetic acid (7). The effects of varying NaCl and acetic acid concentrations on the physical properties of the formed fiber tows are shown in Tables 2 & 3 and Fig. 1. The interaction between

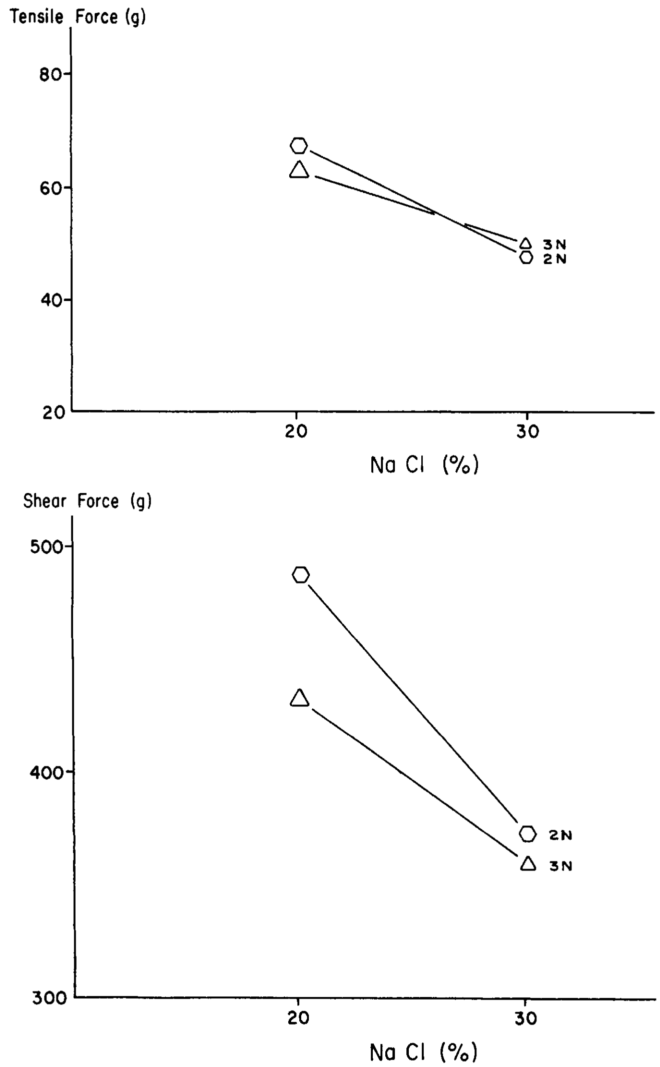

Fig. 1. Mean tensile strength (g) and mean shear force (g) of peanut spun fiber tows as influenced by sodium chloride (%) and acetic acid (N) concentrations. Protein concentration 13%.

NaCl and acetic acid concentrations was statistically significant for both tensile strength and shear forces (Table 2). The average tensile strength was more influenced with changes in NaCl concentration than with varying acetic acid concentration. Similar trend was found for average shear force. In both physical parameters, the decreases in average force values due to increasing NaCl concentration were greater than the decreases in force values due to increasing acetic acid concentration (Table 3). In addition, increasing NaCl

Table 2. Statistical summary of analysis of variance of tensile strength, shear force and stretchability of peanut spun fiber tows.

Source of variation	Degrees of freedom	Tensile strength		Shear force		Stretchability	
		Mean^2	F-value	Mean^2	F-value	Mean^2	F-value
Total	239						
Replicates	2	2170.4		385690.9		2272.3	
Treatments	3	5916.2		204357.2		36969.8	
Sodium chloride	1	16917.6	614.0	520056.6	105.0	45210.1	74.0**
Acetic acid	1	113.4	4.1	68411.3	13.8	64747.4	105.0**
Sod. chloride + acid	1	717.6	26.0**	24603.0	5.0*	952.0	1.5^{ns}
Reps. + treatments	6	395.7		128563.6		8728.1	
Samples within	228	17.9		1702.7		400.1	
Error	234	27.6		4955.5		613.6	

*,** is significant at the 5% and 1% level, respectively; ns is not significant.

Table 3. Mean degree of stretchability (%) of peanut spun fibers[1] as influenced by sodium chloride (%) and acetic acid (N) concentrations.

Sodium chloride		Acetic acid	
Conc.	Stretchability	Conc.	Stretchability
20%	215.8** a	2 N	218.5** z
30%	188.4 b	3 N	185.7 y

[1]Protein concentration 13%

**Significance at the 1% level.

Mean values not followed by the same letter are significantly different.

concentration from 20% to 30% at the same level of acetic acid (2N) resulted in the decrease of tensile strength forces by 20.3 g whereas with the 3 N acetic acid level, the increase in NaCl concentration resulted in a decrease of 13.3 g (Table 3, Fig. 1). Similarly, decreases in shear force values with increasing NaCl concentration were greater at the level of 2 N acetic acid than that for the 3 N acetic acid. These different rates of changes were reflected by the significant NaCl X acetic acid interaction (Table 2) and no definite conclusions could be drawn about the influence of the changes in the concentrations of the main effects on the tensile strength and shear forces of peanut protein spun fiber tows.

The α-helical portions of proteins are held together by H^+ bonds formed between R groups, by the hydrophobic interactions and by covalent disulfide linkages (1). Disulfide linkages are the strongest bonds maintaining the tertiary structure of the protein. In this type of protein, the hydrophobic amino acids tend to be folded to the interior of the protein molecule while most of the hydrophillic residues (polar) are on the surface. The presence of polar R groups on the surface of proteins usually accounts for their solubility in aqueous solutions (1). Under the conditions of the coagulating bath used in the present study, protein precipitates into a fiber which gets stronger with time due to the formation of disulfide bonds between chains. The effect of NaCl on fiber formation has been attributed to its ability to promote the formation of hydrophobic bonds during coagulation. It could be assumed that the minimum concentration of NaCl in the bath would be dependent on the concentration of protein in the dope solution. Accordingly, the decrease in fiber strength noted for the 15% NaCl versus the 20% NaCl (Fig. 2) could be attributed to insufficient NaCl to maximize fiber formation. This assumption could also explain the effect of protein concentration in the dope where the 13.5% protein dope showed less strength than the 13.0% protein (Table 1) when both were spun in 20% NaCl-2N acetic acid baths. Increasing NaCl concentration from 20 to 30% resulted in the formation of weaker fibers at both acetic acid concentrations (Fig. 2). This could be attributed to the fact that as the fibers emerge from the 30% salt bath, the drop in fiber temperature as being removed from the 50°C bath and the difference in vapor pressure deficit from bath to air, would cause the coagulation bath media within the fibers tow to become supersaturated. This would cause salt crystallization on and between the fibers in the tow result-

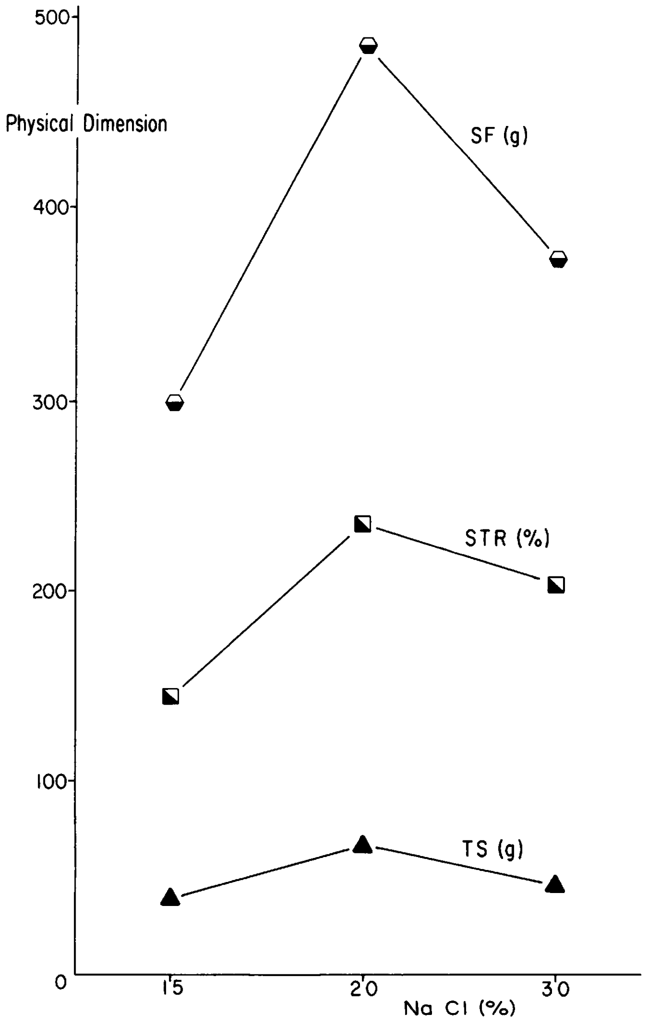

Fig. 2. Effect of NaCl concentration (%) on degree of stretchability (◪——◪), tensile strength (▲——▲) and shear force (⬣——⬣) values of peanut spun fiber tows.

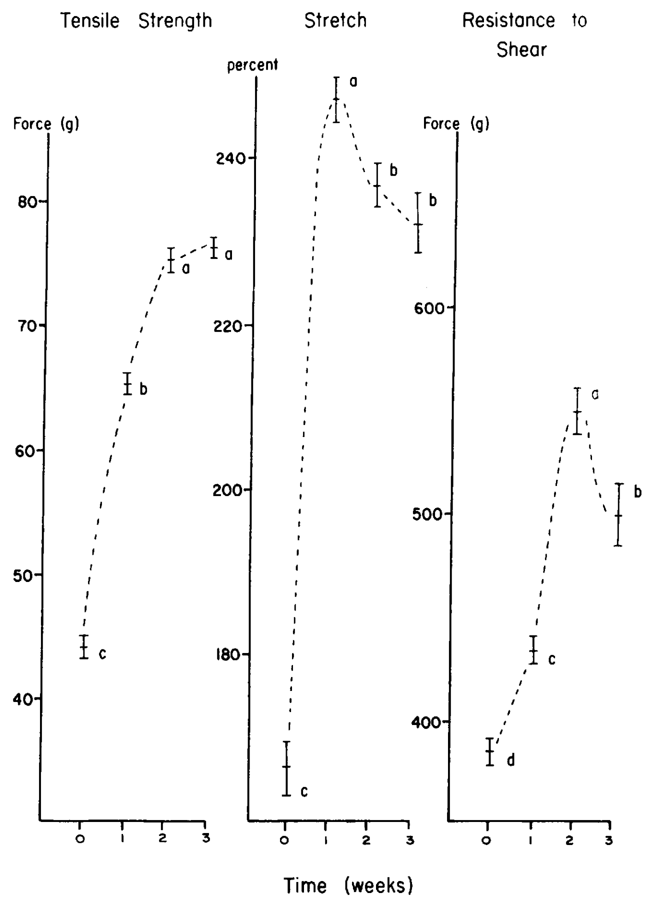

Fig. 3. Effect of storage time at 1°C on the physical properties of peanut spun fiber tows. Spinning conditions: 13% protein, 20% sodium chloride, 2N acetic acid. Each bar represents treatment mean ± standard deviation. Means, within each physical test not followed by the same letter are statistically different.

ing in a physical stress to the fibers as they were pressed between the drawing rollers. As the fibers are stored in the pH 4.0 phosphate buffer, the salt would go back in solution leaving voids or weak spots in the fibers. The decrease in fiber strength with increasing acetic acid concentration from 2N to 3N, at 20% NaC1, (Fig. 1 & Table 3), could be attributed to increased diffusion of acid into the coagulated fibers resulting in softening of the fibers since proteins are more soluble at a lower pH. The slower diffusion of the 2N acid bath would leave the protein in the fibers closer to its isoelectric point and thereby result in a stronger fiber formation. However, this effect did not seem to take place at the 30% NaC1 concentration for the tensile strength (Fig. 1).

STORAGE STUDY

The effects of storage duration at 1°C on the response of peanut protein fiber tows to applied stresses were varied for the different tests (Fig. 3). The tensile strength test showed that the fibers became stronger during the first 2 weeks of storage, which then leveled off by the third week. This would indicate that fiber toughness increased with storage. Storage of soybean protein fibers resulted in more toughness and loss of elasticity (4). The sharp increase in the stretchability during the first week indicated increased elasticity while the reverse was true for the ensuing 2 weeks.. Resistance to shearing forces increased during the first 2 weeks but the drop during the third week may be due to decreased elasticity in relation to a stable toughening of the fibers.

STRUCTURAL ORIENTATION

Method of orientation of peanut fiber tows influenced their resistance to punch shear test (Table 4). Orientation testing of tows stored for 3 weeks showed that tows arranged in the perpendicular orientation required a greater force to punch shear than those arranged randomly, parallel or in single tow. Results for fiber tows stored for 6 weeks showed that tows in the 45° angle configuration required more punch shear force than those in perpendicular, parallel or in single tow. Spun soy fiber arranged in a parallel net-

Table 4. Mean punch shear force (g) of peanut spun fiber tows[1] as influenced by orientation method.

Orientation Method	Storage time (wks) 3	6
Single	376 d	355 w
Parallel	565 c	441 x
Random	615 b	--
Perpendicular	726 a	490 y
< 15°	--	514 z

[1]Spinning conditions: 13% protein, 20% sodium chloride, 2 N acetic acid.

Means, within each orientation test, not followed by the same letter are statistically different.

work was most easily compressed, followed by a random arrangement, while a perpendicular network was the least compressable (6). It would be noted here that the lower punch shear values recorded for the respective orientations of tows stored for 6 weeks in comparison with the 3 weeks storage indicate an even greater decrease in fiber strength as a result of the increased aging time than was previously noted in the storage study.

Results obtained in the present study show that resistance to shearing forces was greatest while tensile strength was least in all cases. Tensile strength and stretchability tests were conducted to describe mechanical properties of the spun fibers. Resistance to shear tests were conducted since they provide the objective methods that most closely approximate consumer acceptance of fibrous food products (9).

Summary and Conclusions

Protein concentrates extracted from raw blanched Altika peanuts were used to produce dope solutions for the spinning of fibers. Protein concentrations in the dope, as well as NaC1 and acetic acid concentrations in the coagulating bath and fiber storage time were studied in relation to the physical properties of the fibers produced. The effect of differing orientation configurations between tow fiber tows was studied as to their mechanical properties. Results of these investigations show that:

1) 13.0% protein dope solutions produced stronger fibers than 13.5% protein dope solutions spun at the same relative viscosity.
2) Fiber strength was maximal when acetic acid and NaC1 concentrations were 2N and 20%, respectively. Fiber strength decreased when either of these variables increased or decreased.
3) Fibers stored at 1°C for 3 weeks showed greater tensile strength, stretchability and shear strength than the nonstored fibers. However, fiber strength decreased with lengthening the storage period to 6 weeks.
4) Orientation tests showed that two tows arranged in a 45° angle required the largest punch-shear force, followed by the perpendicular, random and parallel orientations, respectively. Two tows arranged in any orientation pattern required higher punch shear forces than a single tow.

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