

## Soyprotein Fiber Formation

by

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### 대두 단백질섬유의 제조에 관한 연구

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#### Abstract

In our previous report (Korean J. Food Sci. Technol., 9, 123. (1977)), functional properties of soyprotein isolates prepared from defatted soybean meal were studied. Using those properties soyprotein fibers, which may be acceptable as meat analogs, were prepared with protein spinning apparatus. Soyprotein can be converted into the suitable form for the spinning by denaturation with alkali (0.6%) and continuous fibers were spun by extruding spinning solution into an 20% NaCl-1 N acetic acid coagulating bath. The process for producing soyprotein fibers on a bench scale was described and break strength, break elongation and textural parameters of the fibers formed were evaluated. The possible scheme of formation of soyprotein fibers was discussed.

#### Introduction

To overcome food protein shortage, specially to maintain the high level of meat consumption in the world, an intensive world-wide effort in institutional and industrial laboratories resulted in the stimulation of considerable research in the uses of soybean protein in foods<sup>(1-8)</sup>. Concurrent with these developments have been efforts to present the soybean proteins in acceptable textural forms resembling traditional animal protein foodstuffs.

Proteins are recognized as having two principal type of molecular configuration, fibrous and corpuscular. Since most of the animal proteins are fibrous in nature while soyprotein are globular, much of works has been dealt with aspects of modifying structure of soyprotein by solubilization, unfolding and mechani-

cal formation of fibers, together with orientation, in order to simulate animal proteins. Although a number of process have been described for preparing textured proteins, such as protein-lipid films<sup>(4)</sup>, acid precipitated textured soyprotein<sup>(5-6)</sup> and heated gel formation<sup>(7-8)</sup>, basically two processes-thermoplastic extrusion and fiber spinning-are employed for making meat analogs. Former is similar to that involved in making cereal based snack foods and uses less expensive soy flours (50-53% protein). The products by this method are currently being used as mainly extenders in the formulation of comminuted and other meat items<sup>(9-10)</sup>. Latter is, however, a modification of techniques in the production of synthetic fibers for the textile industry and gives better textured product (meat analogs in contrast to meat extenders) and yet should use rather pure form of protein solution.

Since Boyer<sup>(11)</sup> developed the first procedure of

producing synthetic meats from vegetable proteins, much of works were carried out to develop the technical processes<sup>(15-17)</sup>. But detailed studies with soyproteins are rather limited and remained as technical know-how. Furthermore, no such research was done in Korea.

This study is to establish the feasibility of spinning admixtures of the soyprotein isolates by using spinning apparatus designed in our laboratory. The process developed is based upon unfolding of peptide chains by alkali treatment and molecular orientation of the fibers formed by mechanical spinnings.

### Materials and Methods

**Spinning apparatus :** The bench scale proteinspinning apparatus (Fig. 1 ) was designed in our lab. with a modification of the method of Works *et. al.*<sup>(18)</sup> and manufactured by a commercial manufacturer. The protein dope tank is a 5 gal. capacity stainless steel tank intened for pressures up to 100 PSI(6 atm) applied with a compressed air. The dope was forced to a metering pump which consists of variable speed gear reducer and thus provides varying flow velocities. The dope solution leaves the metering pump and it passes through double filter assembly of spinneret. The stainless spinneret assembly is mounted in such a manner that rotation is possible around the filter pivot and can be immersed in the spin tank circulated by a pump. The dope solution thus passes 50 mesh of filter spinneret and the spinneret having 600 holes

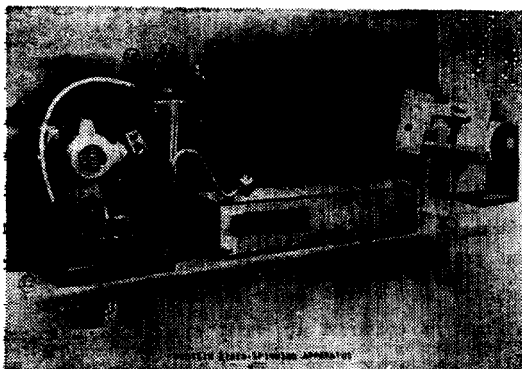


Fig. 1. Protein spinning apparatus. The apparatus was designed in our laboratory and manufactured by a commercial manufacturer

each of 0.008 cm diameter in the spin pack assembly. After precipitation, the fibers were drawn from the tank using collection winder which wound the fibers on rigid PVC plastic bobbin. The rotational speed of the winder wheel can be varied which in turn altered the degree of stretching of the fibers. The spinneret was immersed deeply in the tank to provide maximum dwell time of the precipitated fibers in the coagulating solution of 20 % NaCl in 1 N acetic acid. The fibers were removed from the bobbin and left to drain of excess coagulating solution and washed with excess of water. They were cut into bundles and stored at the refrigerator.

**Preparation of soyprotein dope :** Soyprotein isolates (SPI) were prepared by the method of previous report<sup>(9)</sup> from "Soy Flour F" provided by Dong Bang Co. Ltd. The dope was made by dissolving weighed amount of SPI in certain volume of dist. H<sub>2</sub>O and suspended with Waring blender and NaOH was added as final concentration of 0.6 % and workable viscosity was between 40 to 80 Poises. Apparent viscosities were measured with Brookfield viscometer at 28 °C<sup>(8)</sup>. After 60 min, the dope solution was subjected to fiber formation using the spinning apparatus.

**Physical properties :** Breaking strength (g) and elongation (g) of finished fibers were measured with an Instron universal test machine Model 1127 which was calibrated before each use. Fiber were conditioned in the Instron testing room for 48 hr at 70 % relative humidity and 25 °C before tests were made. Operating conditions were as follows : cross head speed, 20 cm/min ; chart speed, 20 cm/min ; sample length, 5 cm ; sample weight, 0.1 g. Breaking strength and elongation were calculated from the stress-strain curves obtained. Textural parameters including hardness, cohesiveness elasticity, gummness and chewiness were determined by using Zenken texturometer according to the method of Lee *et al.*<sup>(19)</sup>.

### Results and Discussion

In the fiber spinning the basic principle along the spinning line could be described as follows: the dispersion of soyprotein in an alkaline solution and forcing

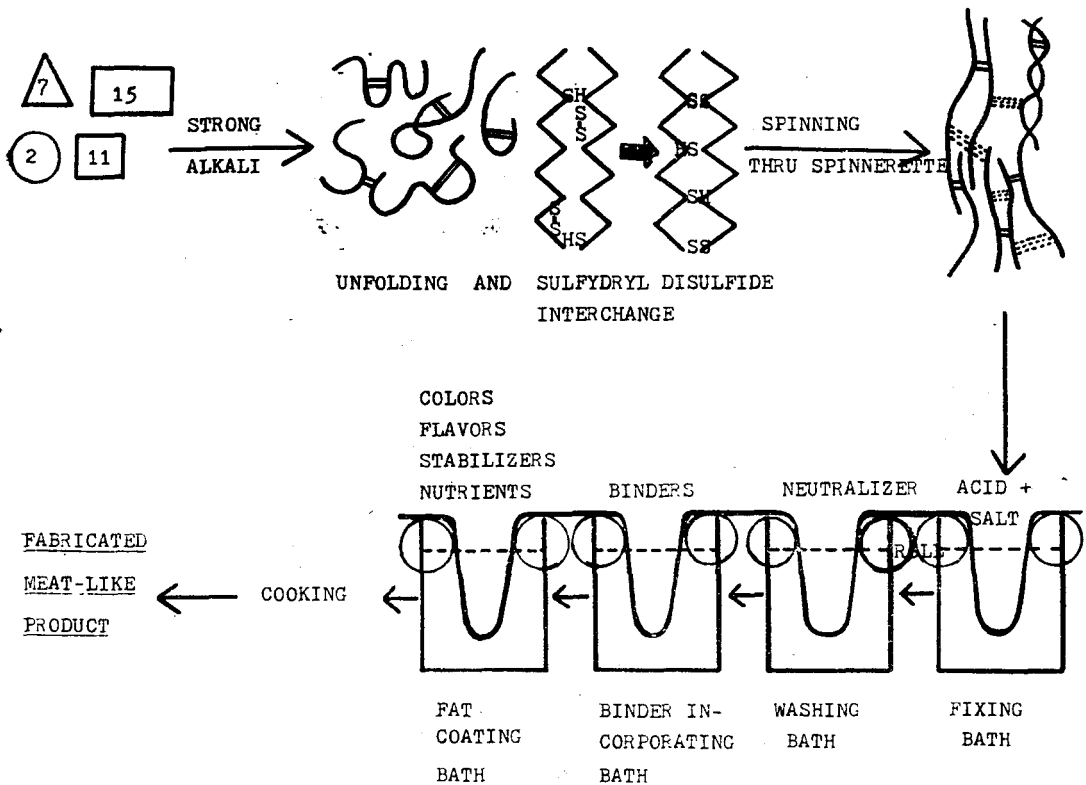


Fig. 2. Schematic representation of soyprotein fiber formation

it through a die (spinneret) into an acid salt solution forms fine protein fibers. As the filaments come in contact with the bath, diffusion of the solvent out of the dope and the coagulating medium into the dope takes place<sup>(20,21)</sup>. When the right concentration of the components is reached, structural rearrangement in the filament involving the solvent, the medium and the polymer occurs. A skin of precipitated polymer is then formed. The thickness of this skin grows as the filament travels along and more polymer is precipitated. At the same time the filament is being elongated by means of tensile force applied by the drawing roller. The diameter of the filament therefore decreases along the spinning line. A stable and coagulated fiber thus is formed which could be wound up with a certain velocity of drawing. Fig.2 shows the scheme of the fiber formation.

Since native soyproteins are globular whose ultracentrifugal components are 2, 7, 11 and S<sup>(6)</sup>, they are converted to unfolded polypeptide chains, as shown in the upper middle of the figure by strong alkali

treatment. This unfolding and dissociation is evidenced by the increase in relative viscosity as Kelley *et al.* reported<sup>(6)</sup>. Alkali treatment also favors sulfhydryl-disulfide interchange reactions. Subjection of these unfolded polypeptides to spinning gives mechanical formation of fibers, together with molecular orientation, forming new disulfide linkages (solid line in the figure) and hydrogen or ionic bonds (dotted line in the figure)<sup>(6)</sup>.

Table 1 illustrates those phenomena, indicating 34.7 % of SPI treated with alkali was soluble in 0.5 M phosphate buffer, pH 7.6 while 84.4 % was solubilized by treatment of 0.01 M mercaptoethanol-6 M urea which disrupts the disulfide and hydrogen bonding between polypeptide chains.

From Fig. 3 to Fig. 7 various spun fibers before treatment with binders are shown. As protein concentration and spinning press increase, the products become compacted and better textured in appearance. Twelve % of protein concentration which showed only 18 Poes of viscosity gave fluppy fibers and the

**Table 1. Solubility of various preparations of soyprotein**

Sample	Buffer	Amount soluble (%)
Acid precipitated* <sup>1</sup> protein(SPI)	P.B.* <sup>2</sup>	71.5
	P.B.+M.E.* <sup>3</sup>	88.7
	P.B.+6 M urea	96.1
	P.B.+M.E.+6 M urea	99.3
SPI treated at pH 12 for 30 min and reprecipitated* <sup>1</sup>	P.B.	34.7
	P.B.+M.E.	43.6
	P.B.+3 M urea	56.4
	P.B.+6 M urea	77.3
	P.B.+M.E.+6 M urea	84.4
Soyprotein fiber	P.B.	0
	P.B.+M.E.	4.2
	P.B.+6 M urea	12.6
	P.B.+M.E.+6 M urea	13.1

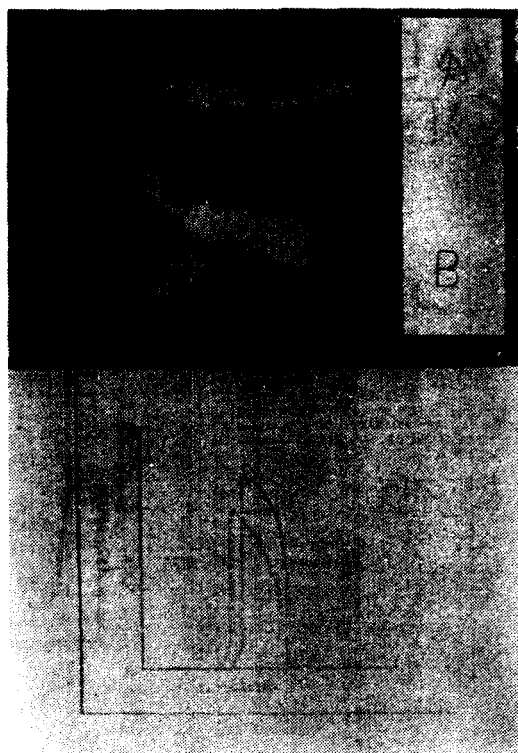
\*1 : prepared by the method of previous report (8)

\*2 : 0.5 M phosphate buffer, pH 7.6

\*3 : 0.01 M mercaptoethanol

products did not remained in shape (Fig. 3). Furthermore no fibers were formed from 12 % protein solution when 100 PSI of spinning press was applied. The products prepared from 15 % and 18 % protein solutions showed no clear differences in appearance and they are very resembled to crab meat in color and appearance.

To see any change of appearance in texture by the

**Fig. 3. Soyprotein fibers from 12 % soyprotein isolate**

A (○—○—○)·····spun fibers collected directly from coagulating bath  
 B (————)·····spun fibers fixed for 15 hrs. in coagulating bath

Code No. I-1: protein conc. 12 % and spinning press 50 PSI.

**Table 2. Elongation of soyprotein fibers by Instron universal test machine**

Code No.	Protein conc. (%)	Spinning Press(PSI)	Breaking strength (g)		Breaking elongation (g)	
			No fixation(A)	Fixation for 15 hr (B)	No fixation (A)	Fixation for 15 hr (B)
I-1	12	50	100	120	53	46
I-2*	12	100	—	—	—	—
II-1	15	50	205	207	107	117
II-2	15	100	209	225	114	120
III-1	18	50	212	290	157	120
III-2	18	100	250	300	120	107

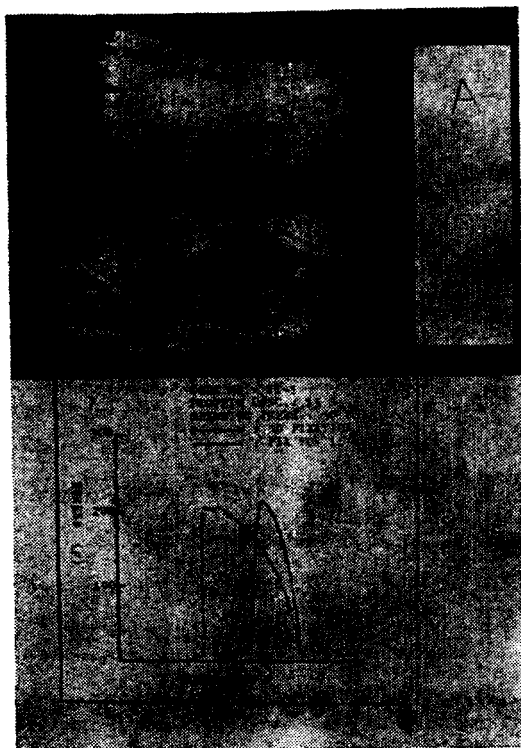
Experimental conditions : Crosshead speed 20 cm/min

Chart speed 20 cm/min

Sample length 5 cm

Sample weight 0.1 g

\* No measurements for product prepared from 12 % protein solution were carried out because of insufficient texture

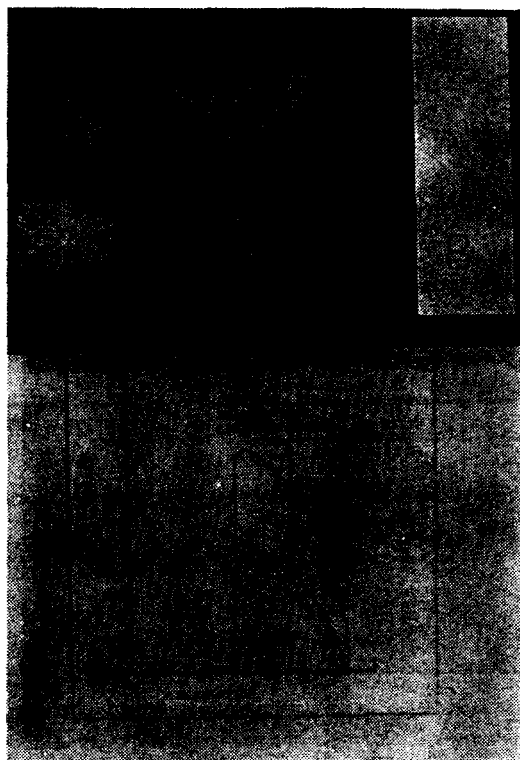


**Fig. 4. Soyprotein fibers from 15 % soyprotein isolate**

A (○—○—○)···spun fibers collected directly from coagulating bath

B (————)···spun fibers fixed for 15 hrs. in coagulating bath

Code No. II-1 : protein conc. 15 % and spinning press 50 PSI.



**Fig. 5. Soyprotein fibers from 15 % soyprotein isolate**

A (○—○—○)···spun fibers collected directly from coagulating bath

B (————)···spun fibers fixed for 15 hrs. in coagulating bath

Code No. II-2 : protein conc. 15 % and spinning press 100 PSI

**Table 3. Textural parameters of soyprotein fibers by texturometer**

Textural parameter	No fixation (A)				Fixation for 15 hr (B)			
	II-1	II-2	II-1	II-2	II-1	II-2	II-1	II-2
Hardness (Kg/Wt)	5.56	5.7	5.62	7.20	4.0	3.4	2.82	4.5
Cohesiveness	0.69	0.79	0.81	0.75	0.67	0.64	0.70	0.66
Elasticity (mm)	4.0	4.5	4.0	4.5	3.0	2.5	2.0	3.0
Gumminess	386	449	457	536	268	223	219	217
Chewiness	154	202	183	241	80	54	44	49

Experimental conditions : Chart speed 750 mm/min

Clearance 3 mm

Voltage 0.5 V

Plunger 18 mm lucite

Platform flat

No measurements for products prepared from 12% protein solutions (Code No. I-1 and I-2) were not carried out because of insufficient texture

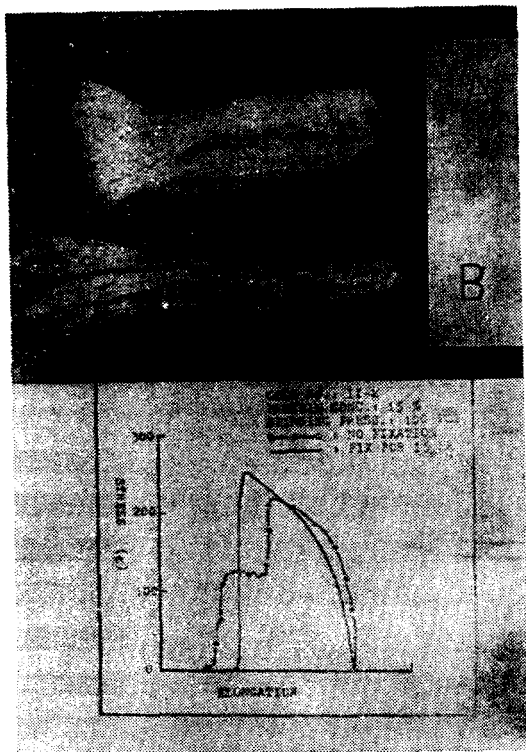


Fig. 6. Soyprotein fibers from 18 % soyprotein isolate

A (○—○—○)···spun fibers collected directly from coagulating bath

B (————)···spun fibers fixed for 15 hrs. in coagulating bath

Code No. III-1 : protein conc. 18 % and spinning press 50 PSI

dwel time in the coagulating bath, fibers formed were placed immersed in the bath for 15 hrs. Results indicated that no remarkable changes in texture were observed, but fibers are noticeably more brittle when the products were fixed in the coagulating bath for 15 hrs after winding the fibers on the plastic bobbins (B in the figures). This might be caused by the complete denaturation of fiber filaments extruded from spinneret and was evidenced by the measurements of textural parameters which will be discussed in the later part of this report(see Table 2 and 3), although 18 % protein solution formed tough fibers.

Table 2 and 3 show the effect of the variables employed on texture of fibers. Analysis of variance indicated that operating protein concentration of the dope solution was between 15-18 % and no remarka-

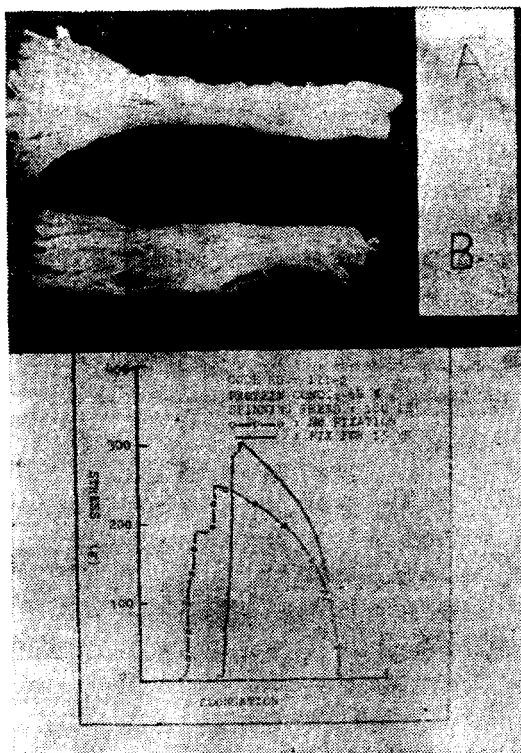


Fig. 7. Soyprotein fibers from 18 % soyprotein isolate

A (○—○—○)···spun fibers collected directly from coagulating bath

B (————)···spun fibers fixed for 15 hrs. in coagulating bath

Code No. III-2 : protein conc. 18 % and spinning press 100 PSI

ble differences in the textural parameters were observed in this range. In general, however, the fibers of 18 % protein solution break into pieces more easily to compare with those of 15% solution. This fact is more remarkable for the products fixed for 15 hrs at coagulating bath which is specially noticed in gumness and chewness as shown in Table 3. Furthermore 18 % protein dope solution turned gelation quickly before spinning and resulted fibers contained toughness. Increasing the spinning press and the dwell time in the coagulating bath generally showed a slight increase in textures. Protein concentration as expected has the greatest effect on these parameters. Up to 12% of protein concentration, no fibers were formed and that it showed only approximately 18 Poises of viscosity. Green *et al.*<sup>(22)</sup> reported that range of

viscosity for spinning protein fibers are between 28 to 300 Poises. Young *et al.*<sup>(17)</sup>, however, indicated that the viscosity rised 300 Poises rapidly and did not level off with time when plasma protein was treated with alkali. This caused the quick gelation of the dope and therefore, they stabilized within the range of 28-300 Poises by the addition of acetic acid such that pH of the dope was reduced to 11. By adjustment of pH of the dope resulting 28-300 Poises, alkali-treated soyprotein was easily spun by the process described in their experiment.

It is interesting to note that fibers formed did not possess off-flavors of soybean proteins after fixing step was completed in the coagulating bath. Beany off-flavors of soyprotein was one of the limiting factors for use of soyprotein in the certain foods<sup>(23)</sup>. The mean analysis of soyprotein fibers formed form 18% protein solutions was as follows: protein 93%, moisture 53%, ash 0.22% and lipid 0.7%. The composition of the spun fibers varied according to the methodology and raw materials and other process. The protein content of the final products can be varied by changing the component of dope solution or adjusting the component of binders inthe following step after spinning. Young<sup>(17)</sup> reported spun fibers prepared from blood plasma protein contained protein 17.3%, moisture 73. 3% and ash 8.6% while other investigators<sup>(24)</sup> showed protein 93 -98.5% and very small amount of ash and lipid when fibers were spun from cottage cheese whey.

Since focus was imposed on the fiber formation process in this research, spun fiber products were not processed in binder incorporating bath and fat coating bath (Fig. 2). To simulate proper finished meat analogs fibers formed should be treated with edible binders and seasonings including colors,flavors and other nutrients after spinning process was completed. Studies on these subjects are presently under way in our lab, together with the characterization of spun fibers such as microscopic analysis and manipulation of texture.

## 요 약

저자들은 전보(Korean J. Food Sci. Techno., 9,123

(1977)에서 분리 대두 단백질의 식품학적 성질을 조사하였다. 본 연구에서는 이 성질을 응용하여 대두 단백질 섬유를 제조하고자 분실 현상에서 고안 설계하여 의뢰제작한 protein spinning apparatus를 사용하여 분리 대두단백으로 대두단백섬유 제조실험을 행한결과 texture가 우수한 제품을 얻었다. 제조조건은 15-18% 단백질 용액을 알카리 (0.6%)로 처리하여 50-100 PSI spinning press로 사출한 후 12% NaCl-1 N acetic acid bath에서 응고시켰다. 12% 단백질 용액은 생성된 단백질섬유의 texture가 불량하여 일정한 모형을 유지하지 못하였다. 제품의 성질을 Instron기기와 texturometer를 사용하여 측정하였다. 아울러 대두 단백질섬유의 형성기작을 조사하였다.

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