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Chang, Hung Min
Department of Food Science, Faculty of Agriculture, Kyushu University

Hayakawa, Isao
Department of Food Technology, Faculty of Agriculture, Kyushu University

Omura, Hirohisa
Department of Food Science, Faculty of Agriculture, Kyushu University

Hoshi, Masakazu
Central R & D Institute, Taiyo Fishery Co., Ltd., Tokyo

他

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Effect of Hydrophilic Materials on the Prevention of Expressible Drip of Spun Fibers*

Hung Min Chang, Isao Hayakawa, Hirohisa Omura, Masakazu Hoshi† Yasuhiko Sasamoto† and Michio Nonaka†

Department of Food Science & Technology, Faculty of Agriculture, Kyushu University 46-09, Fukuoka 812

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Spun protein fibers prepared from antarctic krill muscle protein and Na-alginate were severely deteriorated during their storage in frozen state or upon heating in autoclave. Polysaccharides and protein, such as pectin, starch pastes, CMC and gelatin were added in spinning dopes and the effects of these hydrophilic materials on the prevention of weight loss were examined by measuring the weight change before and after their storage at -20°C for various periods of time or heating at 100°C for 10 min in an autoclave. Expressible drip decreased sharply, as the fibers spun from the dopes containing hydrophilic polysaccharides or protein were stored at −20°C overnight. No apparent changes in fiber appearance, diameter and strength were observed on spun fibers obtained from the spinning dopes added with 3 % or more of the hydrophilic polysaccharides or protein even after stored at -20°C for 3 months, Similar results were obtained in the case of heating when the hydrophilic materials were added in the spinning dopes.

INTRODUCTION

Frozen storage and thermal treatment are important procedures for meat preservation but these treatments carry along chemical and microstructural changes that modify the quality of final product.

One of the most remarkable effects of freezing and heating is the decrease in muscle water holding capacity after thawing. Such phenomenon is associated with the fact that during freezing and heating, water-protein associations are replaced by protein-protein associations or other interactions (Hamm, 1975). In meat, the changes occurred in texture as well as in the muscle water holding capacity relate to protein denaturation during freezing and heating (Matsumoto, 1980 ; Shenouda, 1980 ; Fukuda et *al.*, 1980). Only a few studies (Bito, 1978 ; Tanaka, 1969) have been performed on the modification of water holding capacity of meat and meat products during freezing or heating.

Wallingford and Labuza (1983) reported that xanthan gum was effective in preventing water loss from a low-fat meat emulsion. Fox *et al.* (1983) studied the effect of xantham gum and carrageenan on the texrure of frankfurters. Foegeding and Ramsey (1986) also reported the effects of adding carrageenan, guar gum, locust beam

* Reprint reguests to Dr. H. Omura.

† Central R & D Institute, Taiyo Fishery Co., Ltd., Tokyo.

gum, xanthan gum and methylcellulose to low-fat, high moisture meat batters. These reports showed that the hydrophilic polysaccharides are effective in preventing weight loss during thermal processing.

Spun protein fibers which could be taken up at a velocity of more than 35 m/min were manufactured successfully from the dope made with krill muscle proteins and Na-alginate (Chang *et al.*, 1987). But expressible drip was observed to be about 50 % by weight of spun fibers as they were stored at -20°C overnight. Thus, the handling of this problem is becoming serious and its prevention is necessary before any of the above products could be considered for commercial processes.

In this study, hydrophilic polysaccharides and protein such as pectin, starches, CMC and gelatin which are widely used in a number of food items as gelling, thickening and coating agents, were mixed with krill muscle proteins and Na-alginate to make spinning dopes. The objective of this study was to evaluate the effects of the above hydrophilic materials on the prevention of weight loss, change of fiber strength and fiber diameter of spun protein fibers.

MATERIALS AND METHODS

Raw materials

Materials used to prepare spun fibers from spinning dope were antarctic krill (*Euphausia superba*) tail meat which were obtained from Taiyo Fishery Co., Ltd. The general analysis of raw materials were 83.0 % moisture, 14.5 % crude protein, 0.8 % lipid, 0.3 % carbohydrate and 1.4 % ash, as described elsewhere (Chang *et al.*, 1987).

Saccharose, Na-alginate, pectin, CMC and gelatin were of guaranteed grade and were obtained from Ishizu Pharmaceutical Co., Ltd. Potato starch and corn starch were of chemical grade of the same pharmaceutical company.

Preparation of spinning dope and spun fibers

Spinning dopes and spun fibers were prepared essentially in accordance with the procedures described elsewhere (Chang *et al.*, 1987). Various amounts of Na-alginate and sufficient deionized water were added to the krill tail meat to give a final weight of 400 g. Then the mixture was blended to a fine suspension with a mixer (Polytron PT -45, Kinematica Co., Ltd., Switzerland) at 10,000 rpm for 10 min. Deaeration was achieved by centrifugation at 3000 rpm for 5 min, and the resulting viscous material was used as the dope. The prepared dope was poured into the hopper and then was pressed with N_2 gas through spinnerette into the coagulation solution containing 3.8 % (w/v) $CaCl_2$, at pH 8.5. The formed fibers were then pulled away from the spinnerette towards the take-up reel. Spinnerette used were with 49 holes, 0.5 mm in diameter.

The dope with 7 % antarctic krill muscle proteins and 1.75 % Na-alginate was used as the standard dope. Various levels of saccharose, hydrophilic polysaccharides and protein were individually mixed with standard dope to investigate their effect on the prevention of weight loss of spun protein fibers.

Measurement of spun fiber strength

Measurement of fiber strength was conducted with a rheometer R-UDJ-DM (Sun

Kagaku Co., Ltd.). The tensil strength of 50 pieces of each spun fiber were measured and the average value was expressed as fiber strength (g/fiber).

Measurement of expressible drip

Freshly prepared spun fibers were put in a stainless-steel centrifugal tube where a wire netting frame and a linen cloth filter were inserted to separated the fibers from water during centrifugation (Ohashi and Sugano, 1973). Fiber weight (A) was measured posterior to centrifugation at 2000 rpm for 5 min and then the fibers were packed with vacuum packing machine (Nippon Polisero Industrial Co., Ltd.). After their storage at -20°C for various periods of time or heating in an autoclave (Type 40, Kubota Co., Ltd.) at 100°C for 10 min, fibers were thawed in still water at 10°C. Then fiber weight (B) were measured posterior to centrifugation at 2000 rpm for 5 min. The difference of weight (A-B), before and after freezing or heating, was divided by the initial weight (A) and was expressed as expressible drip after freezing or heating, respectively. Measurement of expressible drip was performed in duplicate.

Measurement of spun fiber diameter

Measurement of fiber diameter was conducted with a microscope (Nikon Optical Co., ltd.). Fiber diameters of 10 pieces of each spun fiber were measured and the average value was expressed as the fiber diameter (mm).

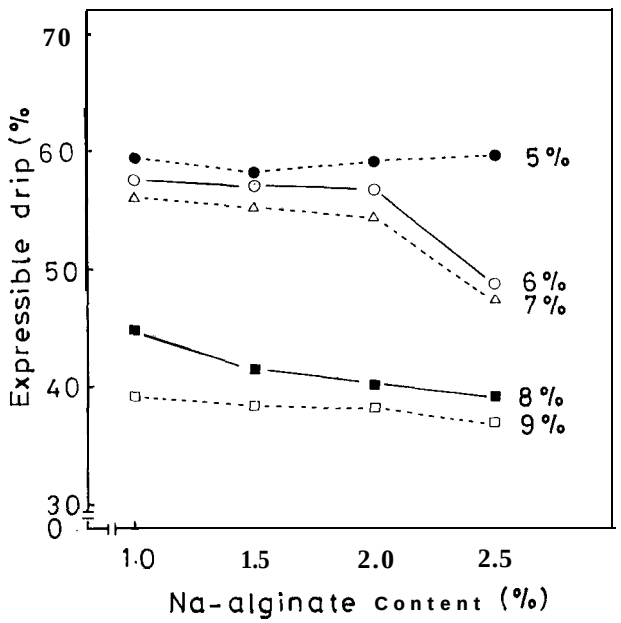

Fig. 1. Effects of protein and Na-alginate contents in spinning dopes on the expressible drip, Percentage in this figure shows protein content in spinning dope. Fibers were stored at −20°C overnight and thawed in still water at 10°C.

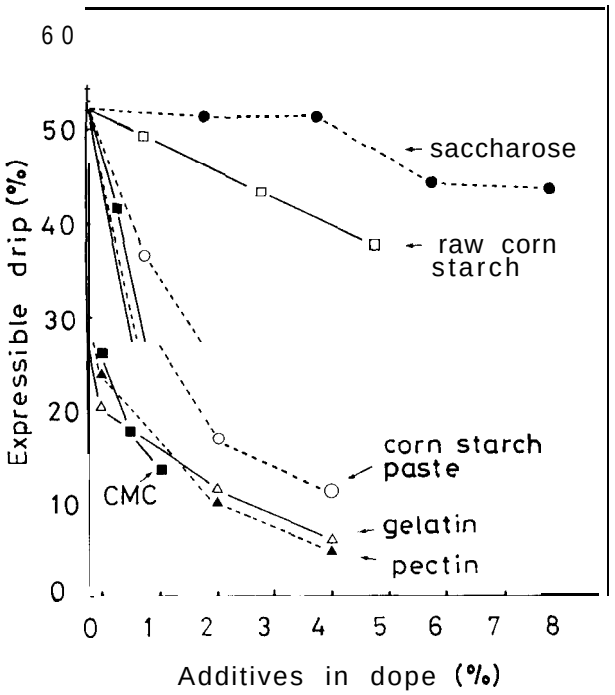

Fig. 2. Relationship between expressible drip and hydrophilic materials in spinning dopes. Experimental conditions were the same as in Fig. 1.

Preparation of starch pastes

Starch pastes (10 %, w/w) were prepared by heating potato starch and corn starch individually at 100°C for 20 min with continuous stirring (Hizukuri *et al.*, 1972).

RESULTS AND DISCUSSION

Effects of protein and Na-alginate contents on the expresseble drip

Dopes with various contents of antarctic krill muscle proteins and Na-alginate were prepared, and then extruded into the coagulation solution, containing 3.8 % $CaCl_2$, to form spun protein fibers.

The results presented in Fig. 1 confirmed that the expressible drip of spun fibers decreased with the increasing protein and Na-alginate contents in spinning dopes. It was observed that expressible drip of fibers spun from the dope containing 9 % protein was about 2/3 of those obtained from the dope containing 5 % protein when those two fibers were stored at -20°C overnight. Since higher solid content affected the spinnability of dope, a dope prepared with 7 % protein and 1.75 % Na-alginate was used as the standard formulation for the following experiments.

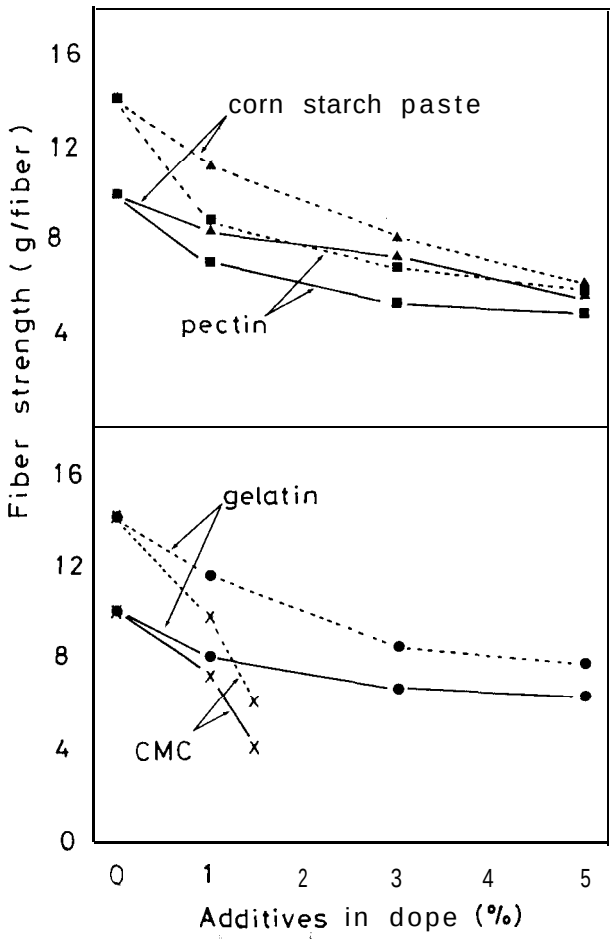

Fig. 3. Changes of spun fiber strength before and after storage at -20°C overnight. Other experimental conditions were the same as in Fig. 1. ——, before frozen storage; ···, after frozen storage.

Relationship between the expressible drip and hydrophilic materials

Hydrophilic polysaccharides and protein treatment were observed to have significant effects on the prevention of weight loss, as the results presented in Fig. 2. Pectin, CMC, corn starch paste and gelatin lowered the expressible drip of spun fibers which were stored at -20°C overnight. The overall influence of those hydrophilic materials on weight loss was significant, while no so much with saccharose and raw corn starch. Raw corn starch mixed directly to make spinning dope, without previous solubilization in boiling water, showed little effect. Addition of 5 % pectin or gelatin in spinning dope showed much satisfactory effect on prevention of weight loss, but higher content of those additives made the dopes more viscous and hardly pressed out through narrow spinnerette into coagulation solution. Standard dope added with 3 %

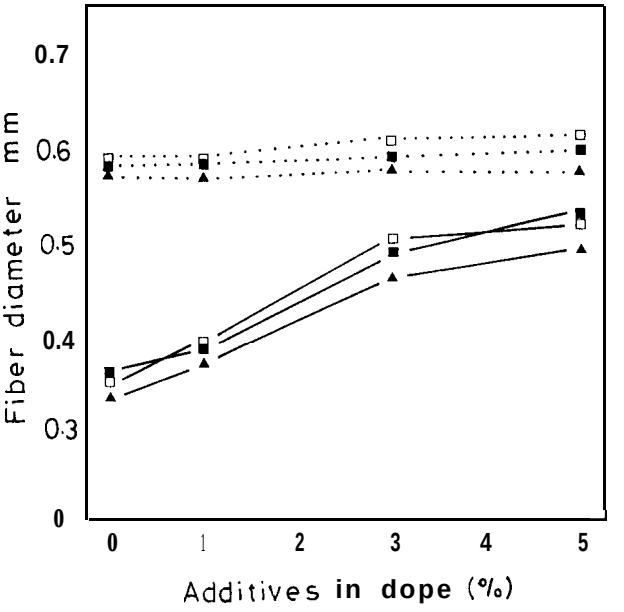

Fig. 4. Changes of spun fiber diameter before and after storage at -20°C overnight. Other experimental conditions were the same as in Fig. 1. ·····, before frozen storage ; ——, after frozen storage ; ■ , pectin ; □, gelatin ; ▲, corn starch paste.

gelatin or pectin showed good spinnability and was observed as the best dopes for this experiment.

Changes of fiber strength and fiber diameter before and after frozen storage

Fibers spun from the standard dope were severely deteriorated because of toughening and drying out during storage at frozen state. In Fig. 3, it was shown that the strength of spun fibers obtained from the standard dope increased, from 10 to 14 g/fiber as those spun fibers were stored at −20°C overnight. But the addition of 3 % pectin, corn starch paste or gelatin in the dope stabilized the fiber strength, before and after overnight storage at -20°C. But these spun fibers seemed a little weaker compared with those spun from standard dope. A mutual relationship was observed between the percentage of expressible drip and fiber strength. The higher percentage of expressible drip of spun fibers appeared, the stronger those spun fibers would become after their storage in frozen state.

Addition of hydrophilic materials also affected the fiber diameter. As the results shown in Fig. 4, the fibers obtained from the dopes containig 3 % or more pectin, corn starch paste or gelatin had less change in fiber diameter, before and after storage overnight at −20°C. On the other hand, the fiber diameter of the fibers spun from the standard dope change significantly. Diameters of all freshly prepared spun fibers were all about 0.75 mm. But when those spun fibers were centrifuged prior to freezing, diameters were decreased approximately to 0.6 mm, owing to a somewhat extent of dehydration. After frozen storage for overnight, fiber diameters with a variation of

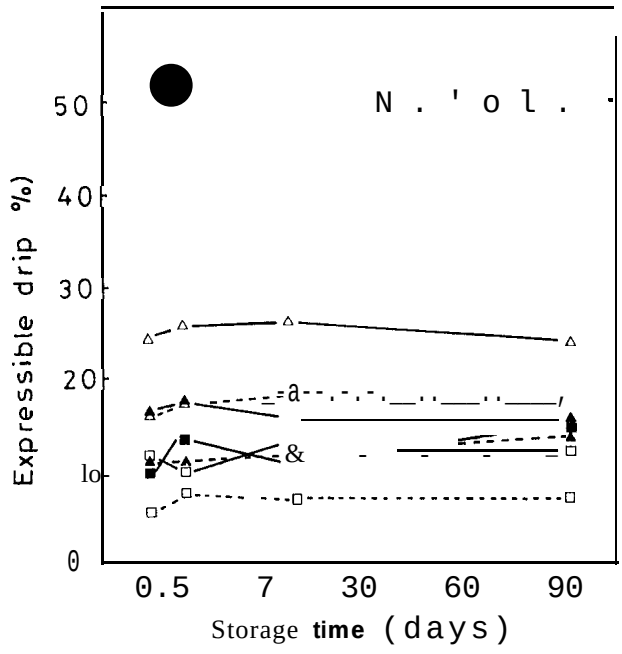

Fig. 5. Changes of expressible drip of spun fibers after storage at -20°C for various periods of time. Other experimental conditions were the same as in Fig. 1. —●—, control (no addition of additive) ; —□—, 3 % gelatin; ···□···, 5 % gelatin; —▲—, 3 % corn starch paste ; —■—, 3 % pectin ; ···▲···, 5 % corn starch paste ; —△—, 3 % potato starch paste ; ···△···, 5 % potato starch paste.

0.35-0.55 mm were observed posterior to centrifugation and the values were proportional to the levels of hydrophilic materials in the dopes.

Changes **of expressible drip and fiber diameter of various spun fibers stored for various periods of time**

As described in Figs. 2 and 3. expressible drip and fiber strength of spun fibers varied with the composition of spinning dope. Figs. 5 and 6 show similar results regardless of the storage period at -20°C.

After storage at -20°C for 3 months, fibers obtained from the dopes containing 3 % or more pectin, corn starch paste and gelatin remained acceptable and had the same appearance and textural strength prior to frozen storage.

Effects of thermal treatment on spun fibers

Freshly prepared spun fibers were vacuum packed and heated at 100°C for 10 min in an autoclave.

As shown in Table 1, after heating, fibers spun from the standard dope showed remarkable changes in fiber strength and fiber diameter, and the expressible drip was about 35 % by weight of spun fibers. But, similar to the results of hydrophilic materials

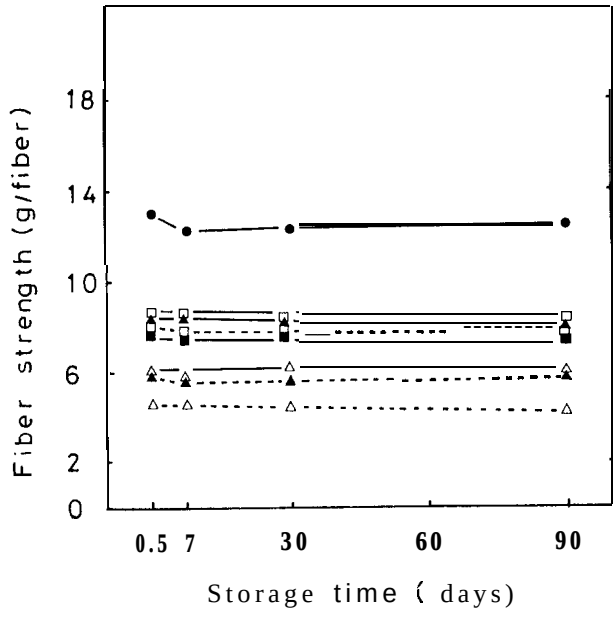

Fig. 6. Changes of spun fiber strength after storage at -20°C for various periods of time. Other experimental conditions were the same as in Fig. 1. The symbols were the same as in Fig. 5.

Table 1. Changes of expressible drip, fiber strength and fiber diameter after thermal treatment at 100°C for 10 min in autoclave.

Additives	Drip (%)	Fiber strength (g/fiber)	Fiber diameter (mm)
Control (unheated)		10.0	0.6
Control (heated)	35	14.0	0.4
Pectin 3 %	17	7.5	0.55
Gelatin 3 %	14	8.4	0.55
*Corn starch 3 %	20	9.2	0.5

Control : standard dope with no addition of additive.
*Corn starch paste

on the expressible drip in Fig. 2, the addition of those hydrophilic materials to the standard dope also showed significant effect on the prevention of weight loss, and on the change of fiber diameter and fiber strength. The addition of more than the 3 % of hydrophilic materials by weight of spinning dope showed much remarkable effect, but those dope seemed not acceptable in this experiment.

From above results; addition of highly hydrophilic polysaccharides and protein showed satisfactory effect on the prevention of weight loss, and on the change of fiber diameter and finer strength. Other pol,ysaccharides, such as karaya gum, arabic gum and carrageenan, were also added to the spinning dope in order to investigate their

effects on the prevention of weight loss, and similar results were obtained. Agar, which was hard to be homogenized in spinning dope during the mixing process, gave insignificant results. The increase in water holding ability of the spun fibers seen in hydrophilic polysaccharides or protein treatments could be due to a gum-water interaction or a gum-protein-water interaction. Whatever the mechanism, the hydrophilic materials appear to increase the water holding ability of spun protein fibers.

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