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Comparative Analysis of the Physicochemical Properties of Sun-dried and Natural Cyclic Freeze-Thaw Dried Alaska Pollack

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Abstract The physicochemical properties of sun-dried and cyclic freeze-thaw dried Alaska pollack were analyzed to compare the 2 drying processes. The moisture content and water activity of sun-dried Alaska pollack were higher than cyclic freeze-thaw dried and 1 year-aged cyclic freeze-thaw dried Alaska pollack (*hwangtae*). The relatively low temperatures used in cyclic freeze-thaw drying retards lipid oxidation compared to sun drying based on the acid and peroxide values, and the levels of thiobarbituric acid-reactive substances (TBARS) in the dried fish. The water holding capacity of cyclic freeze-thaw dried Alaska pollack aged for 1 year (*hwangtae*) under ambient conditions at the drying location was higher than that of sun-dried Alaska pollack. The swelling of myofibrilar filaments during cyclic freeze-thaw drying may be responsible for the softening of the dried muscle protein. Aging the cyclic freeze-thaw dried Alaska pollack for 1 year contributed to an increased yellowish color of the *hwangtae*.

Keywords: Alaska pollack, drying, freezing, lipid oxidation, texture

Introduction

Before technology for food preservation and processing was widely available, animal and fish flesh were usually dried for long-term storage. Currently, most salted and dried animal flesh is used as snack food. Some types of animal and fish flesh are dried by smoking. Low temperature drying is typically used to reduce oxidation, and retain desirable color and textural qualities, taste enhancing components, and nutraceuticals (1-3). The sublimation of ice during freeze-drying imparts a porous structure to the dried material. Alaska pollack are caught during the winter in the East Sea of Korea (4), and traditionally most were dried for preservation and countrywide distribution until freezing technology became popular. Two different drying processes have been used to prolong the preservation of Alaska pollack in Korea. Sun drying usually takes 2-3 weeks to reduce the moisture content to below 19%(wt.) under normal conditions. Another method for drying Alaska pollack has been developed in regions where daily temperature differences are at least 15°C with maximum daytime temperatures of 2°C, and relatively low humidity, and high wind speeds create conditions suitable for food drying. The completion of drying to a final moisture content of approximately 10% (wt.) takes more than 3 months.

Hwangtae, a type of dried Alaska pollack with a yellowish-brown color, is made by aging for 1 year under ambient conditions without exposure to direct sunlight after cyclic freeze-thaw drying. Sunlight supplies radiant energy to the surface of the fish body to melt ice and evaporate water. Frequent snows during the drying period are critical to supply moisture to the fish flesh surface for cyclic freezing and thawing, creating physicochemical properties that differ from those of sun-dried fish. Relatively high wind speeds facilitate moisture transfer from the surface of the fish body to the air. Fish proteins are very sensitive to denaturing factors active during frozen storage. A major cause of the toughening of codfish muscle during frozen storage is the denaturation of tissues by the interaction with fatty acids produced during frozen storage (5). However, repetitive night freezing and daytime thawing at the initial stages of natural cyclic freeze-thaw drying ruptures the protein matrix, presumably as a result of volume increases associated with the freezing of water in the fish flesh. Salt-induced swelling of the meat occurs due to a combination of increases in the myofilament lattice spacing and a loss of myofilament order (6). Multiple cycles of freeze–thaw during processing have been shown to change the physicochemical properties of cuttlefish (7).

Numerous freeze–thaw cycles may induce an increase in the myofilament lattice spacing and a loss of myofilament order, which would eventually cause the swelling of fish meat. The resulting *hwangtae* is softer and more fragile than sun-dried Alaska pollack. Moreover, the low temperatures during the drying period from December to April prohibit the oxidation of lipids which is largely responsible for the unpleasant taste and odor of dried fish. The equilibration of moisture both at the surface and inside the fish facilitates moisture loss from inside the flesh to obtain a homogeneously rigid and porous structure in the dried product (8). Finally, the open air-dried Alaska pollack was aged for another year in a storehouse under ambient conditions to finish the production of *hwangtae*. Therefore, *hwangtae* is completely different from sun-dried Alaska pollack in terms of its sensorial qualities.

Although the characteristics of the 2 types of dried Alaska pollack are clearly different from the standpoint of drying conditions, the physicochemical properties of sun-dried Alaska pollack and *hwangtae* have yet to be compared systematically. Thus, we compared 3 dried Alaska pollack products, i.e., sun-dried, cyclic freeze-thaw

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dried, and *hwangtae* obtained from independent processors.

Materials and Methods

Materials *Hwangtae*, cyclic freeze-thaw dried and aged Alaska pollack, was purchased from Yongdae-ri in Gangwon-do, Korea, in December 2004. Un-aged cyclic freeze-thaw dried Alaska pollack was also obtained from the same local supplier in April 2005. Sun-dried Alaska pollack was purchased from a local market in Gimhae Gyeongsangnam-do, Korea, in January 2005. All analyses were carried out immediately after the sample materials were obtained.

Moisture content, water activity, and pH measurements Moisture content was measured using the 105°C drying method (9). The water activity of each sample was analyzed using a water activity analyzer (BT-RS1; Rotronic AG, Bern, Switzerland). Samples weighing 3 g were homogenized with 30 mL of distilled and deionized water for 1 min. The homogenate was filtered through Whatman No. 2 filter paper and the pH of the filtrate was measured with a pH meter (730P; Istek Inc., Seoul, Korea).

Acid and peroxide measurements Lipid extraction was performed using the method described by Schreiber *et al.* (10). Dried fish was crushed in a blender (51BL30, 7010; Waring Products, Torrington, CT, USA) and shaken overnight with ether. After filtration, the ether was removed from the filtrate using a rotary vacuum evaporator in a water bath (40°C). The extracted lipid was refrigerated at 4 °C. Acid values were obtained using the method described by Schreiber *et al.* (10). Lipids extracted from each sample (5-10 g) were shaken well with 100 mL of ether:ethanol (2:1, v/v) and titrated with phenolphthalein indicator using 0.1 N KOH-ethanol solution (Sigma-Aldrich, St. Louis, MO, USA). Peroxide values were analyzed using AOAC methods (9). Lipids extracted from each sample (2-5 g) were dissolved in 250 mL of acetic acid:chloroform (3:2, v/v) and left for 30 min at room temperature. Saturated potassium iodide (KI, 1 mL, Sigma-Aldrich) was then added, and the mixture was shaken for 30 sec and left for 10 min in a dark room. The mixture was diluted with 30 mL of distilled deionized water and shaken vigorously. Titration was conducted with 1% starch solution indicator using 0.01 N sodium thiosulfate solution (Sigma-Aldrich). All chemicals used were of analytical grade.

Thiobarbituric acid-reactive substances Measurement of thiobarbituric acid-reactive substances (TBARS) was carried out using the method of Hiroko *et al.* (11), with slight modifications. Extracted lipid samples (3 g) were placed in a 50-mL separatory funnel and dissolved in 10 mL of benzene. TBA reagent (0.69% 2-thiobarbituric acid: acetic acid = 1:1; 10 mL, Sigma-Aldrich) was added and the mixture was shaken several times. The lower layer was transferred to a capped test tube after 4 min. The test tubes were boiled in a water bath (TS-1400; Young Ji Instrument Co., Busan, Korea) for 30 min and then cooled in running water. The absorbance was determined at 530 nm (V-2000; Hitachi, Tokyo, Japan). TBARS values were calculated as follows:

$$(A - B) \times 3 \times 100 / S$$

where A = absorbance of sample, B = absorbance of blank, and S = sample weight (g).

Color measurement Sample color was measured under dry and wet conditions by placing samples on a standard white plate to measure L (lightness), a (green to red), and b (blue to yellow) values using a chromameter (CR-210; Minolta, Osaka, Japan). The standard white plate had L value of 97.23, a value of -1.02, and b value of 2.16. All samples were measured in the middle for consistency and each value was the mean of 10 determinations.

Specific volume and water holding capacity measurements The volume of 3×5×1 cm dried samples was measured using hulled millet. The water holding capacity was analyzed by following the method of Chen *et al.* (12). Samples (5 g) were immersed for 1 hr at room temperature. The mixtures were centrifuged at 12,000×*g* for 10 min and then filtered through Whatman filter paper (No. 2). The pellets were weighed, and the weight of water bound per gram of sample was calculated as the water holding capacity.

Texture measurements The textural properties of dried Alaska pollack were evaluated by subjecting each sample to chewiness and shear strength tests. The resistance of individual samples to 30% compression was determined at room temperature using a computer-controlled Fudoh rheometer (RT-2010DD; Rheotech Co., Ltd., Tokyo, Japan) equipped with a 2 kg load cell. All samples were 10×10×10 mm. To test chewiness, the probe (adaptor No. 33) was placed both parallel and perpendicular to the direction of the fish backbone. The shear strength test was carried out using adaptor No. 5-3. A 5 mm diameter stainless steel cylinder probe at a crosshead table speed of 20 cm/min was used. Strength and shearing strength values for the test samples were obtained from the shear strength test. Each sample was tested in triplicate, and mean values were compared. All analyses were repeated 10-15 times.

Scanning electron microscopy Sections of dried Alaska pollack were peeled off and cut into small pieces. The surface of the dried Alaska pollack was gold-sputtered to increase resolution under a scanning electron microscope (S-2400; Hitachi).

Statistical analysis All experiments were carried out in triplicate, and the data are presented as the mean±standard deviation (SD). Statistical significance was determined using analysis of variance (ANOVA) and subsequent Duncan multiple range tests ($p<0.05$). Statistical analyses were performed using SAS (13).

Results and Discussion

Repeated freezing and thawing appeared to affect the textural properties of dried Alaska pollack, resulting in increased gaping and a softer structure in cyclic freeze-thaw dried Alaska pollack (CFD) and *hwangtae* than in

sun-dried Alaska pollack (SD). The softening of the dried fish muscle seemed to result from myofibrilar filament swelling caused by the volume expansion of ice during cyclic freeze-thaw drying and pore formation caused by the sublimation of ice. The lower temperatures experienced during cyclic freeze-thaw drying relative to sun drying contributed to a significant retardation of lipid oxidation based on acid and peroxide values, and TBARS levels in the various dried fish products. The greater lightness and lower color intensity of the cyclic freeze-thaw dried fish compared to the sun-dried fish is likely due to the lower temperatures involved in the cyclic freeze-thaw process. The aging of CFD for 1 year to produce *hwangtae* contributed to its yellowness, presumably through browning reactions, without any significant changes in the other physicochemical properties of CFD. The water activities of the 3 dried fish were low enough for the fish to be shelf stable.

Physicochemical properties The moisture content, water activity (a_w), and pH of *hwangtae*, SD, and CFD are shown in Table 1. The moisture content and water activity ($p<0.05$) of SD was the highest of the 3 types of Alaska pollack. No significant change in moisture content was found during the aging period of CFD. The water activity of each type of dried Alaska pollack was low enough to prevent microbial growth when preserved at ambient temperatures. The pH values of all samples (filtrates of homogenates diluted with 10 volumes of distilled water) were more or less neutral, and ranged from approximately 6.7 to 7.2 ($p<0.05$). Because the degree of hydrolysis is highly dependent on the drying temperature, the pH of the dried fish may reflect the degree of hydrolysis, not only due to the enzymatic hydrolysis of lipids and proteins, but also the oxidation of hydrolysates of fatty acids and amino acids. Therefore, pH can be an important indicator of the level of lipid and protein hydrolysis in dried fish products.

Lipid oxidation Fish lipids are high in polyunsaturated fatty acids such as eicosapentaenoic acid (EPA, 20:5) and docosahexaenoic acid (DHA, 22:6), which are associated with health benefits including the reduction of cardiovascular disease (14). However, polyunsaturated fatty acids are fairly susceptible to oxidation, leading to a number of complex chemical changes that eventually give rise to the development of off-flavors in foods, as well as the generation of harmful oxidation products (15, 16). For cuttlefish, 7 freeze–thaw cycles increased TBARS values from 5.3 to 10.4 mg malondialdehyde (MDA)/kg sample (7). Numerous freeze–thaw cycles during the drying of Alaska pollack affected the formation of TBARS, but not significantly (Table 2). However, comparatively high temperatures accelerate lipid oxidation during sun drying. The levels of TBARS in CFD were much lower than in SD (Table 2). The acid and peroxide values were highest in SD, and lowest in CFD. Slight increases in acid ($p<0.05$) and peroxide values were found during the aging of CFD to complete *hwangtae* manufacturing (Table 2), but the changes were not significant. However, these results suggest that further lipid oxidation in *hwangtae* during storage and distribution should be avoided by using air-tight or vacuum packaging. According to Fitsche and Johnston (16), the TBA values of cod liver oil reached about 150 mg/kg after 3 days of storage and increased to a maximum of about 1,400 mg/kg after 5 days with aeration at 45°C in the dark. This abusive treatment is perhaps not representative of conditions likely to be encountered during the normal storage of fat and lipid products, but the oxidized lipids may rapidly induce changes that occur normally during long periods of processing, storage, and shipping.

Color characteristics The color of dried fish is a measure of its biochemical and structural degradation. The Maillard reaction is primarily responsible for the yellow-brown color of fish during and after drying. However, a small degree of browning results from the degradation of L-methyl-histidine, even in the absence of sugar (17). Furthermore, the oxidation of lipids is an important factor in the browning of the skin of dried fish by interacting with free amine groups of proteins via aldolization reactions (18). Sun drying accelerates the hydrolysis of glycerides in fish, resulting in an increase in free fatty acids and a marked decrease in some unsaturated fatty acids (20:3, 20:5; 18). The levels of free fatty acids in fish increased during drying due to the enzymatic hydrolysis of lipids (4, 19). Free fatty acids and decomposed products of free fatty acids are involved in the browning of dried fish (18). The Hunter's color values of lightness, redness, and yellowness were lowest for SD, reflective of its darker overall color among the 3 dried fish products, although there were no statistical differences among them in lightness and redness (Table 3). During the aging of CFD,

Table 1. Moisture content, water activity, and pH[1] of sun-dried Alaska pollack, cyclic freeze-thaw dried Alaska pollack, and *hwangtae*[2]

Type of fish drying	Moisture content (wt. %)	Water activity[3]	pH
Sun-dried	18.71±0.63[b]	0.66±0.07[b]	6.66±0.02[a]
Cyclic freeze-thaw dried	10.13±0.07[a]	0.49±0.02[a]	7.17±0.02[c]
Hwangtae	10.83±0.01[a]	0.48±0.01[a]	6.99±0.01[b]

[1]Mean±SD; groups with different letters are significantly different (ANOVA, $p<0.05$).
[2]Cyclic freeze-thaw dried Alaska pollack aged for 1 year under ambient conditions without exposure to direct sun rays and at the same location where cyclic freeze-thaw drying was carried out.
[3]Measured at 26.5°C.

Table 2. Acid values, peroxide values, and thiobarbituric acid-reactive substances (TBARS) levels in sun-dried Alaska pollack, cyclic freeze-thaw dried Alaska pollack, and *hwangtae*[1]

Type of fish drying	Acid value	Peroxide value (meq/kg)	TBARS
Sun-dried	47.49±1.36[c]	25.61±4.39[b]	170.43±9.96[b]
Cyclic freeze-thaw dried	9.13±0.48[a]	7.07±1.02[a]	48.51±7.86[a]
Hwangtae	11.94±0.47[b]	7.71±0.56[a]	60.2±8.66[a]

[1]Mean±SD; groups with different letters are significantly different (ANOVA, $p<0.05$).

yellowness increased gradually providing the typical yellow color of *hwangtae* with a yellowness value higher than that of the CFD and SD (Table 3). Non-enzymatic browning reactions, including carbonyl-amino-type reactions, are known to be affected by multiple variables such as water activity, moisture content, storage time, and chemical inhibitors (20). Lea and Hannan (21) reported that the rate of browning in a casein-glucose system conforms to the Arrhenius equation over a temperature range of 9-90°C, where a linear relationship is apparent between the rate of reaction and temperature. Browning progressed slowly during 1 year of storage of CFD at ambient temperatures away from direct sunlight.

Specific volume and water holding capacity Both drying and aging affected the specific volume of each type of dried fish (Table 4). The specific volume of SD was lowest (1.62 mL/g), whereas that of CFD was highest (3.37 mL/g). Specific volume can be regarded as an indicator of textural intensity and the degree of protein aggregation in dried fish. The aging of CFD for 1 year under ambient conditions seemed to affect the specific volume of *hwangtae*. Incubation under ambient conditions for 1 year may induce the rearrangement of the protein matrix in a way that reduces the specific volume of *hwangtae*. If the effects of sample variance and other conditional variances such as temperature, local intensity of sun rays, air humidity, and flow rate during the drying period are neglected, the re-equilibrium between moisture and the protein matrix could affect the specific volume during aging. However, the water holding capacity of CFD was not significantly different from that of *hwangtae*, suggesting that no serious changes in protein structure occurred during the aging period. As with specific volume, the water holding capacity of CFD was significantly higher than that of SD. Lea and Hannan (21) suggested that changes in the water holding capacity of meat are directly related to the spaces within and between the myofilaments. Numerous cycles of freezing and thawing during the drying period affect the swelling of myofibrils (6, 22) and thus the ability to bind water profoundly. Volume expansion as a result of the phase change of intercellular and intracellular water to ice may also contribute to the swelling of myofibrils.

Table 3. Color measurements of sun-dried Alaska pollack, cyclic freeze-thaw dried Alaska pollack, and *hwangtae*[1)]

Type of fish drying	L	a	b
Sun-dried	47.28±4.95[a]	-0.85±1.26[a]	12.92±2.30[a]
Cyclic freeze-thaw dried	54.38±5.37[a]	0.61±2.47[a]	17.90±1.78[b]
Hwangtae	56.39±3.82[a]	0.52±1.10[a]	27.31±1.60[c]

[1)]Mean±SD; groups with different letters are significantly different (ANOVA, $p<0.05$). L: lightness; a: redness; b: yellowness.

Table 4. Specific volume and water holding capacity of sun-dried Alaska pollack, cyclic freeze-thaw dried Alaska pollack, and *hwangtae*[1)]

Type of fish drying	Specific volume (mL/g)	Water holding capacity (g water/g solid)
Sun-dried	1.62±0.51[a]	3.30±0.05[a]
Cyclic freeze-thaw dried	3.37±0.26[b]	4.23±0.08[c]
Hwangtae	2.50±0.97[ab]	4.02±0.09[b]

[1)]Mean±SD; groups with different letters are significantly different (ANOVA, $p<0.05$).

Textural properties Sensorial qualities are highly dependent on the textural properties of various dried fishes. LeBlanc and LeBlanc (23) showed that protein aggregation in frozen stored cod is attributable to changes in reactive -SH groups. An association between the loss of solubility and the decrease in -SH reactive groups during frozen storage has been reported for myosin (24) and actomyosin solutions (25) in which the reactive -SH groups decreased to 55% of the initial value after 5 days of frozen storage (26). Most of the protein may not occur in a monomeric form, and myosin heads could be involved in the myosin aggregation in fish. The total -SH groups decreased continuously over 15 days and reached 33% of their initial value, confirming the importance of disulfide bonds in the freeze-induced aggregation of fish myosin in solution. Sikorski and Kotakowaska (27) showed the importance of this aggregation in the freeze-induced aggregation of fish myosin and actomyosin.

CFD had the lowest chewiness value (1,430.5 g_f), whereas SD had the highest value (2,102.68 g_f) when measured parallel to the fish backbone (Fig. 1). Hurling and McArthur (28) described chewiness as the effort required to convert the sample to a swallowable state when chewed with the back teeth during the breakdown process. The textural hardness of the dried fish was determined using a penetration test measuring strength and shearing force. The drying method significantly affected the textural strength of the dried samples (Fig. 2). SD showed the highest textural strength (1,229.3 g_f) and shearing force (1,414.0 g_f/cm^2). No serious differences in textural properties were found between *hwangtae* and CFD. Although the aggregation of myosin during freezing leads to muscle toughening and drip loss during thawing (29), several repetitive freeze-thaws with concomitant sublimation and vaporization increased the porosity of the

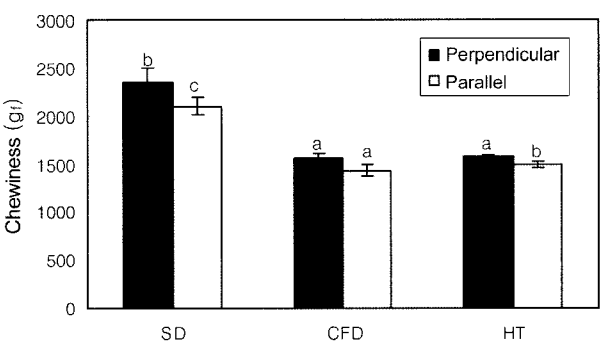

Fig. 1. Chewiness of sun-dried Alaska pollack (SD), cyclic freeze-thaw dried Alaska pollack (CFD), and *hwangtae* (HT), measured by placing a probe parallel and perpendicular to the direction of the fish backbone. Bars with different letters are significantly different (ANOVA, $p<0.05$).

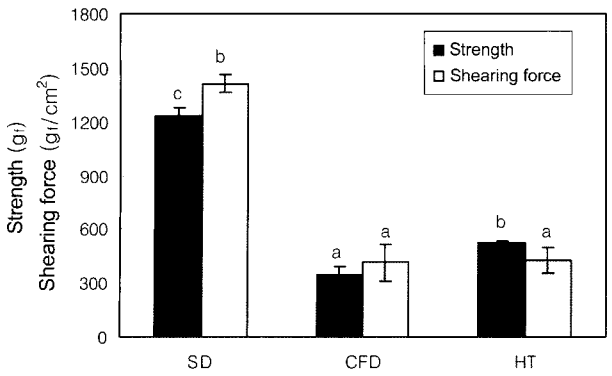

Fig. 2. Strength and shearing strength of sun-dried Alaska pollack (SD), cyclic freeze-thaw dried Alaska pollack (CFD), and *hwangtae* (HT), measured by placing a probe perpendicular to the direction of the fish backbone. Bars with different letters are significantly different (ANOVA, $p<0.05$).

Fig. 3. Scanning electron microscope images of (A) sun-dried Alaska pollack, (B) cyclic freeze-thaw dried Alaska pollack, and (C) *hwangtae*.

muscle protein matrix and the swelling of muscle fibrils, resulting in decreased toughness. The sun drying of Alaska pollack occurs mainly by vaporization, whereas both sublimation and vaporization occur during cyclic freeze-thaw drying. As drying continues, shrinkage of the muscle along with protein agglomeration toughens the dried fish products. However, intermittent snowing during cyclic freeze-thaw drying supplies moisture to the fish surface. Consequently, the fish flesh can continue to freeze and thaw for a considerably longer period, resulting in the porous structure and less rigid strength of cyclic freeze-thaw dried fish.

Surface structure The surface morphology of SD and CFD was measured using a scanning electron microscope at 2,000× magnification (Fig. 3). CFD and *hwangtae* exhibited characteristic ruptures of protein fibrils with increased gaping, whereas SD showed a coalescent protein matrix. Evidence has also been found indicating that refrozen cod samples had a slightly less regular fiber alignment and more disrupted muscle fibers (28). Gaping increased when salmon was frozen as fillets before smoking (30). Ice crystals grew in columns between approximately parallel sets of compressed muscle fibers. Holes made by ice sublimation in CFD and *hwangtae* suggest a similar ice crystal distribution in muscle and a similar compression of muscle fibers resulting from water extraction to extracellular spaces upon freezing. Rupture of the muscle protein into thread-like bundles was observed in the CFD and *hwangtae*.

Acknowledgments

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References

1. Arai K, Takashi R. Studies on muscular proteins of fish XI. Effect of freezing on denaturation of actomysin ATPase from carp muscle. B. Jpn. Soc. Sci. Fish 39: 533-535 (1973)
2. Park JW, Lanier TC, Green DP. Cryoprotective effects of sugar, polyols and/or phosphates on Alaska pollack surimi. J. Food Sci. 53: 1-3 (1988)
3. Min B, Ahn DU. Mechanism of lipid peroxidation in meat and meat products - A review. Food Sci. Biotechnol. 14: 152-163 (2005)
4. Lee EH, Han BH, Kim YG, Yang ST, Kim KS. Studies on the processing method and taste compounds of dehydrated Alaska pollack -1. Changes of nucleotides and their related compounds and free amino acid composition of Alaska pollack during hot-air dehydration. Bull. Korean Fish Soc. 12: 25-36 (1972)
5. Anderson ML, Ravesi EM. Reaction of free fatty acids with protein in cod muscle frozen and stored at -29°C after aging in ice. J. Fish. Res. Board Can. 26: 2727-2735 (1969)
6. Wilding P, Hedges N, Lillford P. Salt-induced swelling of meat. The effect of storage time, pH, ion-type, and concentration. Meat Sci. 18: 55-75 (1986)
7. Thanonkaew A, Benjakul S, Visessanguan W, Decker EA. The effect of metal ions on lipid oxidation, colour, and physicochemical properties of cuttlefish (*Sepia pharaonis*) subjected to multiple freeze–thaw cycles. Food Chem. 95: 591-599 (2006)
8. Hastings RJ, Rodger GW, Park R, Matthews AD, Anderson EM.

Differential scanning calorimetry of fish muscle: the effect of processing and species variation. J. Food Sci. 50: 503-506 (1985)
9. AOAC. Official Methods of Analysis of AOAC Intl. 17th ed. Method 934.01, 965.33. Association of Official Analytical Chemists, Arlington, VA, USA (2000)
10. Schreiber GA, Schulzki G, Spiegelberg A, Helle N, Bogl KW. Evaluation of gas chromatographic method to identify irradiated chicken, pork, and beef by detection of volatile hydrocarbons. J. AOAC Int. 77: 1202-1217 (1994)
11. Hiroko K, Takashi K, Kiyomi K. Thiobarbituronic acid-reactive substances from peroxidized lipids. Lipids 24: 873-881 (1989)
12. Chen H, Rubenthaler GL, Schanus EG. Effect of apple fiber and cellulose on the physical properties of wheat flour. J. Food Sci. 53: 304-308 (1988)
13. SAS Institute, Inc. SAS User's Guide. Statistical Analysis Systems Institute, Cary, NC, USA (1985)
14. Leaf A, Weber PC. Cardiovascular effects of *n*-3 fatty acids. New Engl. J. Med. 318: 549-557 (1988)
15. Cho SY, Miyashita K, Miyazawa T, Fujimoto K, Kaneda T. Autoxidation of ethyl eicosapentaenoate and docosahexaenoate. J. Am. Oil Chem. Soc. 64: 876-879 (1987)
16. Fitsche KL, Johnston PV. Rapid autoxidation of fish oil in diets without added antioxidants. J. Nutr. 118: 425-426 (1988)
17. Nicolas L, Frédéric J, Victurnien F, Pierre L. A novel colorimetry analysis used to compare different drying fish processes. Food Control 15: 327-334 (2004)
18. Pokorny J. Browning from lipid–protein interactions. Prog. Food. Nutr. Sci. 5: 421-428 (1981)
19. Oh KS. Changes in lipid components of pollack during sun-drying. Korean J. Food Sci. Technol. 26: 123-126 (1994)
20. Eskin NAM, Henderson HM. Browning reactions in foods. pp. 83-85. In: Biochemistry of Foods. Academic Press, Inc., New York, NY, USA (1971)
21. Lea CH, Hannan RS. Studies of the reaction between proteins and reducing sugars in the dry state. The effect of activity of water, of pH, and of temperature on the primary reaction between casein and glucose. Biochim. Biophys. Acta 3: 313-325 (1949)
22. Fennema OR. Comparative water holding properties of various muscle foods. J. Muscle Foods 1: 363-381 (1990)
23. LeBlanc EL, LeBlanc RJ. Determination of hydrophobicity and reactive groups in proteins of cod (*Gadus morhua*) muscle during frozen storage. Food Chem. 43: 3-11 (1992)
24. Chen CS, Hwang DC, Jiang SZ. Effect of storage temperatures on the formation of disulfides and denaturation of milkfish myosin (*Chanos chanos*). J. Agr. Food Chem. 37: 1228-1231 (1989)
25. Jiang ST, Hwang DC, Chen CS. Denaturation and changes in SH group of actomysin from milkfish (*Chanos chanos*). J. Food Sci. 53: 1333-1335 (1988)
26. Jose AR, Martha OM, Everett B. Fish myosin aggregation as affected by freezing and initial physical state. J. Food Chem. Toxicol. 65: 556-560 (2000)
27. Sikorski ZE, Kolakowska A. Seafood proteins, pp. 99-112. In: Changes in Proteins in Frozen Stored Fish. Sikorski ZE, Pan BS, Shahidi F (eds). Chapman & Hall, New York, NY, USA (1994)
28. Hurling R, McArthur H. Thawing, refreezing, and frozen storage effects on muscle functionality and sensory attributes of frozen cod (*Gadus morhus*). J. Food Sci. 61: 1289-1296 (1996)
29. Mackie IM. The effects of freezing on flesh proteins. Food Rev. Int. 9: 575-610 (1993)
30. RØRÅ AMB, Einen O. Effects of freezing on quality of cod-smoked salmon based on the measurements of physicochemical characteristics. J. Food Sci. 68: 2123-2128 (2003)