A New Method to Evaluate Gel Properties of Fish Meat Gel Products*¹

Takeshi Sano,*² Satoshi F. Noguchi,*³ Takahide Tsuchiya,*²
and Juichiro J. Matsumoto*²

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We designed a new method to evaluate the gel properties of fish meat gel products, by a stretching test, and a film-shaped gel sample, *ca.* 1 mm thick, is used as a test piece. This method had the following advantages: The method can be applied to a small amount of sample and less than 500 mg of sample was needed for one measurement. It had high accuracy and reproducibility. It showed high correlations with the pushing-in method using cylindrical test pieces, and values could be converted to those from each method. The fracture test and measurement of Young's modulus (non-fracture test) can be performed at the same time, while for the pushing-in method using cylindrical gels, the fracture test and measurement of Young's modulus must be done separately. Thus the present stretching method was effective particularly when a large amount of sample could not be obtained, and also possessed several other advantages as above. Therefore, the present method may contribute for evaluation of the gel properties of fish meat gel products.

Elastic and tender texture (*ashi*) of kamaboko and other fish meat gel products is the most important factor in considering the quality of the products. The evaluation of the gel properties of fish meat gel products, particularly the strength of *ashi*, requires mechanical measurements to objectively define the gel properties and a sensory evaluation.

In 1935, Simidu and Takebayashi¹⁾ designed a gelometer which measured the strength of *ashi* of various fish meat gel products. Matsumoto and Arai,²⁾ in 1952, improved the Simidu's instrument³⁾ and was used to measure the firmness of fish meat paste. A spherical plunger (5 mm in diameter) was pushed into a cylindrical test piece (3 cm in diameter and 2 cm in height). The load on the plunger when it sank 5 mm into the test piece was defined as the "depression strength" and the load at the breaking point was defined as the "breaking strength". Thus Matsumoto and Arai laid the foundations for the present mechanical measurements of gel properties of fish meat gel products. Later, Fujikawa and Miyake⁴⁾ measured the jelly strength of fish meat gel products by a method similar to that of Matsumoto and Arai.²⁾ In 1958, Okada and Yamazaki⁵⁾ modified the

instrument of Matsumoto and Arai.²⁾ A spherical plunger (5 mm in diameter) was pushed into a cylindrical test piece (3 cm in diameter and 3 cm in height) and then the stress-strain curve up to the breaking point was recorded. The jelly strength, breaking strain, and softness were obtained from the profile of the stress-strain curve. Today the instruments and methods used to measure the jelly strength and gel properties of fish meat gel products are the modified instruments and methods of Okada and Yamazaki.⁵⁾ Although the measuring conditions, such as the size of the test piece and the plunger, the speed of pushing-in, etc., may be modified by each researcher, the instruments and methods are based on those of Okada and Yamazaki.⁵⁾

On the other hand, Shimizu and Simidu⁶⁾ were carrying out evaluations of fish meat gel products using the stretching test. One end of the dumbbell-shaped test piece was fixed and the other end was stretched. The stress-strain curve was recorded up to the breaking point. Breaking stress, breaking elongation, and Young's modulus were obtained from the profile of the stress-strain curve and adopted as indices of gel properties. Subsequently, the shape of the test piece was changed

*¹ Studies on Fish Meat Gels—V.

*² Department of Chemistry, Sophia University, 7-1 Kioi, Chiyoda, Tokyo 102, Japan (佐野 猛, 土屋隆英, 松本重一郎: 上智大学理工学部).

*³ Taiyo Research Laboratory, Taiyo Fishery Co., Ltd., Tsukishima 3, Chuo, Tokyo 104, Japan (野口 敏: 大洋漁業 (株) 大洋研究所).

to ring-shaped⁷⁾ or sliced,⁸⁾ and the strength of ashi was then measured by similar stretching tests.

These methods to evaluate the gel properties of fish meat gel products needed a great deal of sample because large test pieces were used. When a large amount of sample could be obtained easily such as fish meat and washed fish meat, it was possible to evaluate the gel properties easily, even by these methods. Therefore, there was little effort to make the test pieces smaller. However, in studies using the isolated constituent proteins of fish meat to investigate the formation of fish meat gel products, large quantities of the constituent muscle proteins, such as myosin, actin, tropomyosin, paramyosin, *etc.*, were needed, and it is difficult to satisfy this requirement using pure proteins. Consequently, a method to evaluate gel properties using a small test piece was needed. Such a method must also be accurate and the results must be reproducible. In the present paper, we describe a new method to evaluate the gel properties of fish meat gel products, using a small test piece, by a stretching test similar to that of Shimizu and Simidu,⁶⁾ Shimizu *et al.*,⁷⁾ and Shimizu and Nishioka.⁸⁾ The accuracy and reproducibility of this method were examined. In addition, its usefulness was investigated by comparing it with the pushing-in method⁵⁾ using a cylindrical test piece.

Materials and Methods

Materials

Frozen mince of Alaska pollack *Theragra chalcogramma*, (SA grade) processed by Taiyo Fishery Co., Ltd., Tokyo, was used.

Gel Preparation

Blocks (2–3 cm cube) of Alaska pollack frozen mince were thawed at 4°C, the water content was adjusted by adding cold water and ground with an Ishikawa grinder (type 18, Ishikawa Kojo Co., Ltd., Tokyo, 20 cm in diameter) for about 30 min. After it became homogeneous, it was reground for 30 min with 2.5% (w/w) NaCl and then de-aerated by centrifugation at 2,900×g for 5 min. The residue was used for the preparation of the film-shaped or cylindrical gel samples.

When film-shaped gels were prepared, the residue was put into a polyvinylidene chloride casing (2.0 cm in width and 1.3 cm in diameter) and set between two glass plates (7.5×15×0.3 cm) with spacers of about 1 mm thick as indicated in Fig. 1 (A), and both ends of the casing were sealed

Fig. 1. Preparation of film-shaped gels (A) and measurement of the gel properties of a film-shaped gel test piece (B). The detailed procedures were described in Materials and Methods.

with an adhesive tape.

When cylindrical gels were prepared, the residue was packed in the casing and both ends of the casing were sealed by tying with strings.

The above procedures were performed at 4°C.

The film-shaped and cylindrical samples were heated in two different ways: a) heated at 80°C for 40 min immediately after preparation (one-step heating), b) held at 4°C for 24 h and then heated at 80°C for 40 min (two-step heating). The heated samples were immediately cooled in ice-water for 30 min and then held at 25°C for 24 h.

Measurement of Gel Properties

All measurements of gel properties were performed at 25°C.

Measurements of the gel properties of film-shaped gels were carried out by the present stretching test using a rheometer (NRM-2010J-CW, Fudoh Kogyo Co., Ltd., Tokyo).

A film-shaped gel sample was cut to 5.0 mm in width and both ends were fixed at binders of the rheometer with an extending length of 2.6 cm as shown in Fig. 1 (B). We employed an extending length of 2.6 cm because we used a slide glass (2.6×7.6×0.1 cm) when fixing a test piece at binders. The test piece was stretched at a speed

Table 1. Summary of number of measurements (N), mean, standard deviation (S.D.), coefficient of variation (C.V.), and standard error (S.E.) of each batch

		Batch			
		A	B	C	Total
Breaking stress	N	10	17	12	39
	Mean/g·cm ⁻²	643	661	635	648
	S.D./g·cm ⁻²	47	36	25	38
	C.V./%	7.3	5.4	3.9	5.9
	S.E.	16	9.0	7.7	6.2
Elongation	N	10	17	12	39
	Mean	0.445	0.462	0.454	0.455
	S.D.	0.040	0.051	0.028	0.042
	C.V./%	9.0	11	6.2	9.2
	S.E.	0.013	0.013	0.0086	0.0069
Stretching strength	N	10	17	12	39
	Mean/g·cm ⁻²	287	307	289	297
	S.D./g·cm ⁻²	41	49	23	40
	C.V./%	14	16	8.0	13
	S.E.	14	12	7.0	6.6
Young's modulus	N	13	17	16	46
	Mean/g·cm ⁻²	2180	2290	2290	2260
	S.D./g·cm ⁻²	160	160	210	180
	C.V./%	7.3	7.0	9.2	8.0
	S.E.	47	40	53	27

The gel sample (film-shaped gel) was prepared from Alaska pollack frozen mince whose water content was 76.1% (w/w). The gel properties were measured by the present stretching method.

of 6 cm·min⁻¹ and the stress-strain curve up to the breaking point was recorded. The thickness of the test piece was measured with a micrometer (Mitsutoyo Manufacturing Co., Ltd., Tokyo)

The following four indices were adopted to express gel properties:

- ⟨1⟩ breaking stress
- ⟨2⟩ elongation
- ⟨3⟩ stretching strength
- ⟨4⟩ Young's modulus

"Breaking stress" was defined as the stress per cm² of the sectional area of the test piece at the breaking point and "elongation" was the ratio $\Delta l/l$, where the elongation length at the breaking point, i.e., Δl , compared to the initial length of the test piece (i.e., 2.6 cm=1). "Stretching strength" was referred to the product of "breaking stress" and "elongation". "Young's modulus" was obtained from the initial slope of the stress-strain curve.

Measurements of the gel properties of cylindrical gels were performed by the pushing-in test⁵⁾ using the rheometer.

A cylindrical gel was cut to 13 mm in length, and a spherical plunger (3 mm in diameter) was pushed into the center of the section of the test piece at a

speed of 6 cm·min⁻¹. The stress-strain curve up to the breaking point was recorded and "breaking stress" and "breaking strain" were obtained from the profile. "Jelly strength" was defined as the product of "breaking stress" and "breaking strain". When Young's modulus was measured, a cylindrical test piece (13 mm in length) was compressed with a flat disc plunger (2.0 cm in diameter) at a speed of 6 cm·min⁻¹, and Young's modulus was obtained from the initial slope of the stress-strain curve.

Water Content

Water content of Alaska pollack frozen mince was determined from the weight loss after heating at 120–130°C for 24 h.

Results and Discussion

The present method was designed for use with small test pieces. A film-shaped gel of about 1 mm thick was subjected to the measurement as shown in Fig. 1 (B). In the present method, less than 500 mg of sample was needed for one measurement.

In order to ensure accuracy and reproducibility of this method the following points are im-

portant.

The gel sample must be homogeneous. Removal of air by centrifugation was indispensable to the preparation of gel samples, because bubbles have various negative effects when evaluating gel properties, especially in the fracture test⁹⁾ to measure breaking stress, elongation, and stretching strength.

An exact sectional area of a test piece was needed in order to calculate the indices. Shimizu and Simidu⁶⁾ measured the gel properties using the sectional area of the precooked test piece. However it is necessary to measure the exact sectional area of the test piece in order to reduce errors in the values of gel properties. This is particularly important when a small test piece is subjected to measurements, because shrinkage or swelling of the gel samples sometimes occurs on heating. Therefore, the thickness of the film-shaped gel test piece was measured with a micrometer and an exact sectional area of the test piece was calculated.

First, the accuracy and reproducibility of the present method were examined.

Film-shaped gels were prepared from Alaska pollack frozen mince (water content, 76.1% (w/w)) and their gel properties were evaluated by the present method. The cycle of "preparation and measurement" was repeated 3 times and each batch was referred to A, B, and C. The number of measurements (N), mean, standard deviation (S.D.), coefficient of variation (C.V.), and standard error (S.E.) of each batch are summarized in Table 1.

For breaking stress and elongation the C.V.s (*i.e.*, the ratio of S.D. to mean) were 4.02–7.33% and 6.26–11.1%, respectively, and it is demonstrated that both indices can be measured with high accuracy. The C.V. of the stretching strength (*i.e.*, the product of breaking stress and elongation) was 7.99–15.8% and it is apparent that this index can be also measured with high accuracy, however the C.V. value is a little higher than those of breaking stress and elongation. The C.V. of Young's modulus was 6.90–9.00%, and this index also can be measured with high accuracy. Thus it is clear that all of the indices of gel properties obtained using the present method such as breaking stress, elongation, stretching strength, and Young's modulus are measurable with high accuracy.

Next the reproducibility of the method was investigated. Difference of the means among

Fig. 2. Correlation between breaking stress (F) and breaking stress (C). Data points are the average of 4–6 measurements. (F) and (C) indicate film-shaped and cylindrical gels, respectively.

batches were examined by Duncan's multiple range test¹⁰⁾ using the means and S.E.s of each index for each batch. No significant differences were observed among batches for any of the indices ($p < 0.01$). Therefore, it is demonstrated that each index of gel properties obtained by this method can be measured with high reproducibility.

The relationships between the indices of the present method and the popular pushing-in method (containing the compression test) using a cylindrical test piece also were examined.

Alaska pollack frozen mince was adjusted to water contents of 85.0%, 82.9%, 80.7%, and 78.6% (w/w) and cooked gels were prepared using both the one-step and the two-step heating methods (see Materials and Methods). Then, both film-shaped and cylindrical gels were prepared and the gel properties of each were evaluated by the present stretching or the pushing-in methods. The relationships between the indices obtained by these two methods are indicated in Figs. 2–5.

As shown in Figs. 2 and 3, the correlation coefficient between the breaking stress of film-shaped gels and the breaking stress of cylindrical gels, and the elongation of film-shaped gels and the breaking strain of cylindrical gels were 0.989 and 0.969, respectively. It is clear that each of these two pairs are highly correlated to each other. Therefore, the correlation coefficient between the stretching strength of film-shaped gels, the product of breaking stress and elongation, and the jelly strength of cylindrical gels, the products of breaking stress and breaking strain, was 0.996 and

Fig. 3. Correlation between elongation (F) and breaking strain (C). Data points are the average of 4-6 measurements. (F) and (C) are the same as in Fig. 2.

Fig. 4. Correlation between stretching strength (F) and jelly strength (C). Data points are the average of 4-6 measurements. (F) and (C) are the same as in Fig. 2.

these are also highly correlated to each other.

The relationship of Young's moduli between the film-shaped and cylindrical gels is shown in Fig. 5. The correlation coefficient for the Young's moduli was 0.926 and it is revealed that Young's moduli also are highly correlated, although the value was a little higher than those obtained for Figs. 2-4.

The conversion formulae between the indices obtained by the present stretching method using film-shaped gels and by the pushing-in method using cylindrical gels are shown as follows, where (F) and (C) indicate film-shaped gels and cylindrical gels, respectively.

$$\begin{aligned} \text{Breaking stress (C)/g} &= \\ &0.271 \times \text{breaking stress (F)/g} \cdot \text{cm}^{-2} - 29.1 \\ \text{Breaking strain (C)/cm} &= \\ &0.555 \times \text{elongation (F)} + 0.294 \\ \text{Jelly strength (C)/g} \cdot \text{cm} &= \\ &4.66 \times 10^{-2} \times \text{stretching strength (F)/g} \cdot \text{cm}^{-2} \\ &- 19.6 \\ \text{Young's modulus (C)/g} \cdot \text{cm}^{-2} &= \\ &1.07 \times \text{Young's modulus (F)/g} \cdot \text{cm}^{-2} - 188 \end{aligned}$$

Thus, high correlations were observed between the indices obtained by the present stretching method using film-shaped gels and by the pushing-in method using cylindrical gels, namely breaking stress (F)-breaking stress (C), elongation (F)-breaking strain (C), stretching strength (F)-jelly strength (C), and Young's modulus (F)-Young's modulus (C). These results suggest that many similarities exist in the properties of the two systems, despite the differences in the shape of the test pieces, the direction of the deformation, etc. There is a difference between the Young's moduli

Fig. 5. Correlation between Young's modulus (F) and Young's modulus (C). Data points are the average of 4-6 measurements. (F) and (C) are the same as in Fig. 2.

obtained by the two methods. However, in the range of Young's moduli of fish meat gel products, namely 10^2 - 10^4 g·cm⁻², the difference is small, and it was considered to be possible to convert one to the other.

As described above, it was demonstrated that the present stretching method to evaluate the gel properties of fish meat gel products had the following advantages:

- 〈1〉 The method can be applied to a small amount of sample. Less than 500 mg of sample was needed for one measurement.
- 〈2〉 It had high accuracy and reproducibility.
- 〈3〉 It showed high correlations with the

pushing-in method which is widely applied, and values could be converted to each method.

<4> The fracture test and measurement of Young's modulus (non-fracture test) can be performed at the same time, while for the pushing-in method using cylindrical gels, the fracture test and measurement of Young's modulus must be carried out separately.

Thus the present method was effective particularly when a large amount of sample could not be obtained, and also possessed several other advantages. When evaluating the gel properties of commercial fish meat gel products, we must care to avoid bubbles in a test piece. Because in the present method the test piece is small, it is easy to make test pieces containing no bubbles from commercial gel products.

Non-fracture tests to evaluate the gel properties of fish meat gel products are being used more frequently.¹¹⁻¹⁸⁾ One of the advantages of non-fracture tests is the ability to analyze the rheological properties more easily than in the case with fracture tests. However, considering that foods are fractured by chewing and this dominates the texture of foods, fracture tests can not be ignored when evaluating textural properties of foods. In this sense, the present method is very useful for evaluating the gel properties of fish meat gel products as foods.

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