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General and biochemical composition of caviar from Sturgeon *(Acipenser ruthenus)* farmed in Korea

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Abstract

Our study was undertaken to gain basic and detailed information about the composition of caviar from sterlet *(Acipenser ruthenus)* aqua-cultured in Korea. The moisture, protein, and lipid content of caviar samples was 51.32 ± 2.36%, 25.43 % ± 0.32, and 13.21% ± 0.44, respectively. Hunter color L, a, and b value of unprocessed caviars was 14.83, 0.58, and 1.87, respectively. In accordance with time distribution, a and b values were not significantly changed during the period of observation, but L values indicative of brightness began to decline from 5 weeks. The fatty acids of the caviars consisted of saturated (27.87%), monounsaturated (43.14%), and polyunsaturated fatty acids (28.99%), especially, omega-3 fatty acids such as docosahexaenoic acid (11.39%) and eicosapentanoic acid (4.69%). Among the total amino acids, glutamic acid of 33.5 mg/g (13.9%) was the largest portion of caviars, followed by lysine (27.1 mg/g, 11.3%), leucine (23.1 mg/g, 9.6%), and phenylalanine (22.4 mg/g, 9.3%).

Introduction

Caviar, the eggs produced by sturgeons, is a desirable food item. The most famous caviars are derived from wild harvested sturgeons in the area of the Caspian Sea: Beluga *(Huso huso)*, Osetra *(Acipenser gueldenstaedtii)* and Sevruga *(Acipenser stellatus)* caviar (Caprino *et al.*, 2008). Over the past few decades, pushed by an ever-increasing demand for caviar, wild sturgeon stocks have been over-exploited with the consequent decrease of product availability and supply. In 1997, the Convention on International Trade in Endangered Species of Wild Fauna and Flora limited caviar trade by listing all sturgeon species on the Annex II of the convention (Pikitch *et al.*, 2005). Since then, the global status of wild sturgeons has been examined (Williot *et al.*, 2001).

The ensuing rapid growth of this new branch of the aquaculture industry was possible only after a long lead-in time involving research on cultivation of these species (Williot and Rouault 1982; Doroshov *et al.*, 1983; Arlati *et al.*, 1988; Williot *et al.*, 2001, 2007). Early in the new millennium, China commenced the large-scale rearing of sturgeons, targeting the national consumer market (Bronzi *et al.*, 2011). In 2008, the global production of farmed caviar was estimated to be 110-120 tons, mostly originating from about 80 fish-farms in 16 countries. The most commonly used species is the Siberian sturgeon *(Acipenser baerii)*, which is presently reared in 22 countries, reaching a total annual production of about 8800 tons, followed by the Russian sturgeon *(Acipenser gueldensteadtii)* cultured in about 16 countries, sterlet *(Acipenser ruthenus)* produced in 15 countries, and the stellate sturgeon *(Acipenser stellatus)* cultured in 12 countries (Williot *et al.*, 2009; Bronzi *et al.*, 2011). In Korea, aqua-culture of mainly A. ruthenus began in the early 2000's.

Little is known of the chemical and biochemical composition of caviar. The proximate composition (crude protein, total lipids, moisture, and salt content) of some caviar samples have been examined (Wirth *et al.*, 2000) and compared with the fatty acid composition of total lipids from cultured and wild Gulf of Mexico sturgeon (*A. oxyrhinchus desotoi*) including the ova (Chen *et al.*, 1995; Wirth *et al.*, 2000).

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The objective of our study is to gain basic and detailed information about the composition of caviar from *A. ruthenus* aqua-cultured in Korea compared with caviar of Capelin roe, Pacific herring roe, and Japanese flying fish roe, which are caviars preferentially consumed in Korea (Lee *et al*., 2011).

Materials and Methods

Caviars

General and biochemical compositions of caviar samples collected from *A. ruthenus* aqua-cultured in Chungchongnam-do Fishery Institute Freshwater Fishery Development Center in Chungnam, Korea were determined. Caviar was collected from seven sturgeons averaging 12 kg. The weight of the produced caviar was 1.3 kg. Caviar samples were immediately stored at 1°C in a refrigerated storage room until analyses.

Proximate analyses

Caviar samples were analyzed for moisture, protein, and lipid using Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC, 2000). Briefly, the moisture content of the caviars was determined by drying at 105°C to constant weight and the Kjeldhal method using a KjeltecTM 2300 device (Foss, Höganäs, Sweden) was used to determine crude protein content (Nx6.25). Crude lipid was estimated by extraction with petroleum ether at 60-80°C with a soxhlet apparatus.

Color

Color values of samples were measured by using a Colorimeter SP-80/45 (Tokyo Denshoku, Tokyo, Japan) calibrated with a white tile (X: 84.48, Y: 86.19, Z: 99.46). Color values of the samples were determined by the Hunter L, a, b values. In the Hunter system L indicates brightness or whiteness, positive a value indicates redness, negative a value indicates greenness, positive b value indicates yellowness, negative b value indicates blueness, and ΔE indicates the color difference. Color was measured 3 times for each sample and then averaged.

Fatty acid

After the extraction of total lipids, the preparation of fatty acid methyl esters for fatty acid analysis was performed as previously described (Christie, 2003). Twenty milligrams of the recovered lipids were reconstituted in 1.5 mL of 0.5 N methanolic NaOH. A solution of triundecanoic acid (C11:0) methyl ester was added as internal standard to a final concentration of 500 ug/mL and hydrolyzed at 100°C for 5 min. After cooling, 2 mL of 14% boron trifluoride in methanol was added. Transesterification was performed at 100°C for 30 min. After transesterification, 1 mL iso-octane was added to the sample and vortexed intensely for 30 seconds. Five milliliters of saturated NaCl solution was added and cooled at room temperature. The fatty acid methyl esters (FAMEs) were extracted with 1mL×1mL of iso-octan and the extractions were injected in split mode (split ratio 1:50) into a model 6890 Series GC) gas-chromatograph (Agilent, Santa Clara, CA, USA) fitted with a FID detector. iThe fatty acid analysis used a SPTM-2560 fused silica capillary column (100 m × 0.25 mm internal diameter; 0.20 um film thickness; SUPELCO, St. Louis, MO, USA), with a programmed temperature from 140°C (5 min) to 240°C in an increment of at 4°C/min, held for 15 min, then increased from 240 to 250°C at 10°C/min, and finally held for 1 min. Carrier gas was nitrogen that was supplied at 0.8 mL/min and an inlet pressure of 30.77 psi. The resulting peak areas were corrected by theoretical relative FID response factors (Ackman, 2002) and quantified relative to the internal standard. All analyses were done in duplicate. The double bond index was obtained by calculating the sum of the percentage of each unsaturated fatty acid multiplied by the number of double bonds and divided by the percentage of saturated fatty acids.

Amino acid analysis

A modification of the AOAC method was used for amino acid analysis (AOAC, 2000). Dry samples were hydrolyzed with 25 mL 6N HCl at 110°C for 24 h. Amino acid analysis was carried out by ion-exchange chromatography using a L-8800 automatic amino acid analyzer (Hitachi, Tokyo, Japan).

Results and Discussion

In this study, we analyzed the composition of caviar from A. ruthenus aqua-cultured in Korea and compared the ingredients with Capelin roe, Pacific herring roe, and Japanese flying fish roe. The proximate composition of caviar is summarized in Table 1.

Table 1. Composition (%) of caviar from *Acipenser ruthenus* and other fish roe

Component	Caviar (this study)	Capelin roe[a]	Pacific herring roe[a]	Japanese flying fish roe[a]
Moisture	51.32	80.40	65.40	70.30
Protein	25.43	10.80	10.65	11.30
Lipid	13.21	4.30	2.30	2.90

[a]The results were reported by Lee *et al*

The moisture content of caviar samples was 51.32 ± 2.36% (g/100 g wet weight). The protein and lipid contents were 25.43%±0.32, 13.21%±0.44, respectively, which were lower than those from other sturgeon species (Wirth *et al.*, 2000). Caviar from farmed sturgeon reportedly showed lower protein and lipid contents; a possible explanation might be the peptide and fat content in the fish feed (Wirth *et al*., 2000).

The Hunter L, a, b values of the examined caviars are summarized in Table 2.

Table 2. Comparison (%) of Hunter color value of caviar from *Acipenser ruthenus* and other fish roe

Hunter color	Caviar (this study)	Capelin roe[a]	Pacific herring roe[a]	Japanese flying fish roe[a]
L	14.83	64.64	54.97	33.45
a	0.58	0.24	-1.56	8.66
b	1.87	23.44	16.37	17.11
△E	79.79	39.49	44.79	66.08

[a]The results were reported by Lee *et al*

Color is one of important quality attributes of caviar. L values indicate whiteness to blackness; the present value of 14.83 was indicative of some dark color. The a value (redness to greenness) and b value (yellowness to blueness) was 0.58 and 1.87, respectively, which were close to gray. The L, a, and b value of caviars after 8 weeks was 10.10, 0.24, and 2.35, respectively. L values were significantly lower, but a and b values were similar with the baseline values. It was confirmed that L values had no significant changes until 5 weeks after the measurements and then decreased sharply (data not shown).

The fatty acid composition of the caviars was saturated fatty acids (SFA) 27.87%, monounsaturated fatty acids (MUFAs) 43.14%, and polyunsaturated fatty acids (PUFAs) 28.99%. Among the SFAs, palmitic acid (C16:0) was dominant (22.46%), followed by tricosanoic acid (C23:0; 1.82%) and myristic acid (C14:0; 1.59%). Unsaturated fatty acid contents of MUFAs and PUFAs were about 3-times higher than SFA. The main MUFA was oleic acid (C18:1n9), accounting for 33.67% of the total fatty acids, followed by palmitoleic acid (C16:1, 7.51%). The dominant PUFA was docosahexaenoic acid (DHA; C22:6v-3) accounting for 11.39% of the total fatty acids followed by linoleic acid (C18:2n6) accounting for 10.19%. The composition of omega-3 fatty acids of caviars in the present study was 16.08% of the total fatty acids, which was higher than those of other fishes (Dhaneesh et al., 2012). Especially, the composition of DHA among omega-3 fatty acids was higher than those of other fishes. DHA and eicosapentanoic acid (EPA) prevent human coronary artery diseases (Leaf and Webber., 1988). Although many omega-3 fatty acids occur in nature, DHA and EPA are not synthesized by humans at a rate sufficient to meet metabolic needs, making a dietary source necessary (NAS/NRC, 2005). The results of fatty acid compositions of caviars in this study were similar with those of caviars from farmed white sturgeon *(A. transmontanus)* (Caprino *et al*., 2008). Although the fatty acid composition was influenced by the food of the individual (Steffens *et al*., 1995), MUFAs were high in two cases. Comparison with previously reported data of Capelin roe, Pacific herring roe, and Japanese flying fish roe that are popular in Korea confirmed that the percentage of MUFAs in caviar was relatively higher than other roes, whereas that of total SFA in caviar was lower.

Table 3. Fatty acid composition of caviar from *Acipenser ruthenus*

Fatty acid	Capelin roe[a]	Pacific herring roe[a]	Japanese flying fish roe[a]	*A. ruthenus* caviar (this study)								
					C11:0	C14:0	C15:0	C16:0	C17:0	C18:0	C20:0	C22:0	C23:0
SFA†	30.0	33.0	70.3	27.87	0.45	1.59	0.29	22.46	0.44	0.19	0.22	0.41	1.82
					C16:1	C17:1	C18:1n 9	C20:1					
MUFA††	28.5	25.9	11.3	43.14	7.51	0.81	33.67	1.15					
					C18:2n6	C18:3n 6	C18:3n 3	C20:2	C20:3n 6	C20:5n3 1)	C22:2	C22:6n3 2)	
PUFA‡	41.7	41.4	2.9	28.99	10.19	0.77	0.85	0.26	0.40	4.69	0.45	11.39	

SFA† Saturated Fatty Acid; MUFA†† Monounsaturated Fatty Acid; PUFA‡ Polyunsaturated Fatty Acid
C20:5n3[1]) EPA (Eicosapentaenoic acid); C22:6n3[2]) DHA(Docosahexaenoic acid)
[a]The results were reported by Lee *et al*.

Table 4. Amino acid contents (mg/g, %)of caviar and other fish roe

Amino acid	Caviar (this study)	Capelin roe[a]	Pacific herring roe[a]	Japanese flying fish roe[a]
Thr	9.4 (3.9)	5.0 (5.1)	5.8 (5.8)	6.8 (6.6)
Val	13.7 (5.7)	5.8 (5.9)	6.2 (6.2)	5.9 (5.7)
Met	11.4 (4.7)	3.2 (3.3)	3.0 (3.0)	3.2 (3.1)
Ile	13.4 (5.5)	4.7 (4.8)	4.9 (4.9)	5.3 (5.2)
Leu	23.1 (9.6)	8.5 (8.6)	10.0 (10.0)	10.0 (9.7)
Phe	22.4 (9.3)	5.3 (5.4)	5.3 (5.3)	5.4 (5.3)
Lys	27.1 (11.3)	7.8 (7.9)	7.1 (7.1)	7.5 (7.3)
EAA[1)]	120.5 (50.0)	40.3 (41.0)	42.3 (42.3)	44.1 (42.9)
Asp	11.7 (4.8)	8.0 (8.1)	8.3 (8.3)	7.2 (7.0)
Ser	14.6 (6.0)	6.5 (6.6)	4.2 (4.2)	6.4 (6.2)
Glu	33.5 (13.9)	12.9 (13.1)	13.6 (13.6)	12.5 (12.2)
Gly	5.5 (2.3)	4.3 (4.4)	5.1 (5.1)	4.4 (4.3)
Ala	16.8 (7.0)	6.0 (6.0)	6.4 (6.4)	5.4 (5.3)
Cys	3.1 (1.3)	1.6 (1.6)	1.4 (1.4)	1.1 (1.1)
Tyr	18.1 (7.5)	3.1 (3.2)	3.7 (3.7)	4.4 (4.3)
His	9.0 (3.7)	4.2 (4.3)	3.3 (3.3)	3.9 (3.8)
Arg	0.8 (0.3)	6.4 (6.5)	5.5 (5.5)	7.0 (6.8)
Pro	7.6 (3.2)	5.1 (5.2)	6..2 (6.2)	6.3 (6.1)
NEA[2)]	120.7 (50.0)	58.1 (59.0)	57.7 (57.7)	58.6 (57.1)
Total	241.2 (100.0)	98.4 (100.0)	100.0 (100.0)	102.7 (100.0)

EAA[1)] Essential amino acid; NEA[2)] Nonessential amino acid
[a]The results were reported by Lee *et al*

Percentages of fatty acids differ among species and organs. In red muscle and liver, lipids undergo more enzymatic activities than in smooth muscle, producing large amounts of free fatty acids in oils (Hooper et al., 1973). Detection of higher amounts of v-3 and v-6 PUFA in the present caviar samples agreed with previous findings (Huynh and Kitts, 2009). SFAs and MUFAs are generally abundant in fish from warm or temperate regions, whereas PUFAs show higher levels in fish from cold regions (Dey *et al*., 1993). Compared with freshwater fish, marine fish have higher levels of PUFAs, especially DHA and EPA. Differences in fatty acids of marine and freshwater fishes should be considered with respect to species habitat and based on their natural diet, especially whether a species is herbivorous, omnivorous, or carnivorous (Sargent *et al*., 1995). Total protein contents of caviar were relatively higher (25.43%) than for Capelin roe (10.8%), Pacific herring roe (10.65%), and Japanese flying fish roe (11.3%).

Detailed amino acid composition of caviars in this study is shown in Table 4. Among the essential amino acids, Lys (11.2%), Leu (9.6%), and Phe (9.3%) were determined. Non-essential amino acid content included Glu (13.9%), Tyr (7.5%), and Ala (7.0%). Especially, the caviars contained higher contents of the savory tasting Glu (3.01 g in 100 g), compared to reported roes (Lee *et al*., 2011).

Conclusions

Caviar has high nutritional value and is a valued haute cuisine ingredient. Sturgeon aquaculture is increasing as the demand for caviar continues to increase in Korea. In this study, we extracted caviar from *A. ruthenus*, a species of Sturgeon cultured in Korea and analyzed the composition of that. Also, it was compared with the composition of previously reported fish roes (Capelin roe, Pacific herring roe and Japanese flying fish roe) that are the major fish roes consumed in Korea. As the result, caviar contained higher levels of unsaturated fatty acids and essential amino acids than those of reported roes. So, it was suggested that the nutritional value of caviar from A. ruthenus would be high. Furthermore, it is to evaluate its nutritional values by analyzing the general and biochemical composition of caviar aqua-cultured and consumed in Korea.

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