

SENSORY, CHEMICAL and MICROBIOLOGICAL CHARACTERISTICS of CANNED-SMOKED WHITING ROE PATE

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Received / Geliş tarihi: 13.01.2013

Accepted / Kabul tarihi: 23.07.2013

Abstract

This study was carried out to determine sensory, chemical and microbiological quality of raw whiting (*Gadus merlangus euxinus* Nordman, 1840) roe obtained from Middle Black Sea, smoked roes and the roe which were canned after smoked. Analyses of TVB-N, TBA, pH, total mesophilic aerobic bacteria, total psychrophilic aerobic bacteria, total yeast-mold, total coliform bacteria and also the sensory analyses were done on the roe samples. Additionally, proximate composition values were obtained in both raw material and canned-smoked product. Moisture, protein, fat, ash, carbohydrate contents and calorie value of raw roe were 80.88%, 11.70%, 4.50%, 1.09%, 1.82% and 94.62 kcal, respectively. These values were determined as 61.34%, 8.64%, 12.52%, 1.12%, 16.37% and 212.74 kcal for the canned-smoked roe pate. In the chemical analysis findings, TVB-N and TBA values stayed within the acceptability limit values. It was observed in the microbiological analysis results that, microbial load of the raw material decreased with smoking process. No microbial load was found after the canning process. When all of the quality properties were taken into account, it was seen that the canned-smoked whiting roe pate might be offered for consumption as an alternative product and it might provide added value to food sector.

Keywords: Whiting, roe, smoking, canned, quality

KONSERVE FÜME MEZGİT HAVYARI EZMESİNİN DUYUSAL, KİMYASAL ve MİKROBİYOLOJİK ÖZELLİKLERİ

Özet

Bu çalışma Orta Karadeniz'de avlanan mezgıt balığından elde edilen çiğ havyar, füme havyar ve tütsülendikten sonra konservesi yapılan havyarın duyuşal, kimyasal ve mikrobiyolojik kalitelerini belirlemek amacıyla yürütülmüştür. Havyar örneklerine TVB-N, TBA, pH, toplam mezofilik aerobik ve psikrofilik aerobik bakteri, toplam maya-küf, toplam koliform bakteri analizleri ile duyuşal analizler uygulanmıştır. Ayrıca ham materyal ile son üründe besin kompozisyonu değerleri elde edilmiştir. Çiğ havyarın nem, protein, yağ, kül, karbonhidrat içeriđi ve kalori değeri sırası ile %80.88, %11.70, %4.50, %1.09, %1.82 ve 94.62 kcal'dir. Bu değerler konserve füme havyar ezmesi için sırasıyla %61.34, %8.64, %12.52, %1.12, %16.37 ve 212.74 kcal olarak belirlenmiştir. Kimyasal analiz bulgularında TVB-N ve TBA değerleri tüketilebilirlik sınır değerleri arasında kalmıştır. Mikrobiyolojik analiz sonuçlarında çiğ örnekteki mikrobiyal yükün dumanlama işlemleri ile azaldığı gözlenmiştir. Konservasyon işlemlerinden sonra ise mikrobiyal yüke rastlanmamıştır. Tüm kalite değışimleri dikkate alındığında konserve füme mezgıt havyarı ezmesinin alternatif bir ürün olarak tüketime sunulabileceđi ve gıda sektörüne katma değeri sağlayabileceđi görülmüştür.

Anahtar kelimeler: Mezgit balığı, havyar, tütsüleme, konserve, kalite

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INTRODUCTION

According to the statistics of 2010, caught amount of whiting is 13558 tons in the Black Sea Region where provides approximately 77% of the saltwater fish production of Turkey. Whiting is an economically valuable fish because it makes main catch of the bottom trawling (1, 2). After anchovy, horse mackerel and trout, whiting is the 4th mostly consumed fish by Turkish families (3). Besides the white and delicious flesh of whiting, its roe is also relished by consumers. It was reported that spawning of whiting continued during the year irregularly and it was the highest in November- January and March- April (4). After the fish were caught, they may be served for consumption directly or marketed as diversely processed products. One of the important products obtained from fish is roe (5). Roe or caviar is among the most valuable fish products. As long as they are big enough, almost all fish roes may be used. Among them, the most important ones are sturgeon, trout, blue fin tuna, grey mullet, herring, river herring, codfish and whiting. The ingredient of granulose roe is 46% moisture, 26.9% protein, 15% fat and 8.8% ash. Generally roe is graded when it is raw and different factors such as type, size, freshness, color and taste of the roe are considered (6).

Roe which is known as caviar is very rich in protein. These proteins are globulin, albumin, glycoprotein and phosphoprotein. Its phosphoric acid content is also very high. Some roes of the fish such as sturgeon, atlantic salmon, whiting and codfish whose economic value is quite high are important human food (7).

Salted roes are called as 'caviar' in English and French, 'caviale' in Italian, 'ikra' in Russian and 'havyar' in Turkish and they are served as delicate products. In Turkey, serving of processed roes is quite limited. This is because the roe that is prepared with traditional methods lacks desirable taste, and it is in the quite expensive products category (8). Roe production depends on salting of the roes. In addition to salting, fermented sausage production technologies, freezing, smoking and canning processes are also applied in the roe production (9).

In this study, it was aimed to determine the effect of processing on the sensory, chemical and microbiological properties of the canned-smoked whiting roe pate.

MATERIALS AND METHODS

Raw Material

Whiting (mean weight 40.50 ± 1.5 g, mean length 17.39 ± 0.20 cm) were purchased from a fisherman in Sinop. They were transferred to the laboratory in a polystyrene box with crushed ice within 30 min. Then, the roes were taken out from the fish. The mean weight and length of the roes were 4.99 ± 0.36 g and 5.37 ± 0.14 cm, respectively.

Brine Preparation

The whiting roes were washed gently in solution at 5% salt (50 g salt-1000 mL water). Later, the roes were put in boiling water and boiled them for 4-5 min. During this process, the roes became tougher by absorbing water. They were taken out from boiling water with a slotted spoon and left for cooling for a while. 5 g pimento and 2.5 g curcuma were added to solution at 25% salt (142.25 g salt- 569 mL water). The rate of roe: solution was 1:2 (w/w). The roes were immersed into the solution for 1 h.

Smoking

Smoking process was applied on the roes in 5 kg capacity semi mechanical smoking oven (Apparatebau Günther Kronawitter). Wire grids were placed into smoking oven to prevent the roes from falling and roes were collocated on these grids. They were smoked for 10-15 min at 50 °C. It had been seen in previous trials that higher smoking temperature and time caused to intense smoky smell and a bitterish taste resulted from exposing of the roes to very intense smoke.

Preparation of Pate and Canning

The smoked roes were mashed in a mortar. This mash was mixed with 15% bread crumbs to adjust consistency. The mixture was filled in 4 x 4 cm small jars and 5 mL of sunflower oil was added onto the mixture. Covers of the jars were closed and they were sterilized in an autoclave at 121 °C for 15 min. After sterilization, the jars waited at room temperature for 20 min and the

Taking out of the whiting roe and washing kindly in 5% salty water ⇒ Boiling of the roes for 4 minutes ⇒ Holding them in 25% salt solution with spices for 1 hour ⇒ Smoking process at 50 °C for 10-15 minutes ⇒ Mashing smoked roes ⇒ Adding bread crumbs to the mashed roes ⇒ Filling of the mash into glass jars ⇒ Filling the top of the glass jars with sunflower ⇒ Sterilization process with pressured vapor (at 121 °C / for 15 min.) ⇒ Sensory evaluation

Fig.1. Flow chart of canned-smoked whiting roe patties production

analyses were started. The flow chart that is used in production of the canned- smoked whiting roe patties is shown in Figure 1.

Analyses

Proximate Composition

The moisture and ash contents of roe were determined as described by AOAC (10) methods. The crude protein content was determined according to Kjeldahl method (10). It was calculated by multiplying the nitrogen content by the conversion of 6.25. The lipid content was analysed according to the method of the Bligh and Dyer (11). The carbohydrate value was calculated by [Carbohydrate value = 100-(Moisture + Protein + Lipid + Ash)] formula and then, the energy value was calculated according to Atwater method, that was formulated as

$$[\text{Energy (kcal/100 g)} = (\text{Lipid} * 9) + (\text{Protein} * 4) + (\text{Carbohydrate} * 4)] \quad (12).$$

Total volatile basic nitrogen (TVB-N) value

Analysis of total volatile basic nitrogen (TVB-N) was modified by Antonacopoulos and was done according to Lucke- Geidel method and the results were given as mg/100 g (13, 14).

Thiobarbituric Acid (TBA) Value

Analysis of thiobarbituric acid (TBA) was done according to Erkan and Ozden (15).

pH Value

pH measurements were made with a pH meter (Werkstätten 82362 Weilheim, Germany) according to Vural and Oztan (16). Sample of 2 g from the roe was weighed and 20 mL distilled water was added on it. The pH value was measured after homogenization of 1 min.

Microbiological Analysis

For all microbiological counts, dehydrated ready mediums of Merck were used. 10 g of roe sample was taken, transferred into 90 mL sterile

Physiological Saline Solution (0.85%) and then homogenized in a homogenizer (IKA Yellow Line DI 25 Basic). From the 10-1 dilution, other decimal dilutions were prepared and inoculated (17). Total mesophilic aerobic bacteria, psychrophilic bacteria, yeast-mold and coliform bacteria counts were determined by using the pour plate method. Plate Count Agar was used as medium for total mesophilic aerobic bacteria and psychrophilic bacteria counts, plates were incubated at 28 °C for 3 days and 4 ± 1 °C for 10 days, respectively. For total yeast-mold count, Potato Dextrose Agar was used as medium and plates were incubated at 28 °C for 3 days. To count coliform bacteria, Violet Red Bile Agar (Merck) was used as medium and plates were incubated at 35 °C for 24 h.

Sensory Evaluation

Sensory evaluation was carried out in 3 phases: in the raw roes, smoked roes and canned- smoked roe patties. All of the roe samples were evaluated by 6 experienced panelists (four men and two women) by giving scores from 1 to 9 in terms of appearance, odor, color, texture and flavor. Sensory evaluation of the roes were made by using the hedonic scala modified by Kurtcan and Gonul (18). In this scala; 9- like extremely, 8-like very much, 7-like moderately, 6- like slightly, 5- neither like nor dislike, 4- dislike slightly, 3- dislike moderately, 2- dislike very much, 1-dislike extremely.

Statistical Analysis

The Minitab 15 (Minitab Inc. USA) program was used to search for significant differences between mean values of different results. Differences between means were analyzed by one-way analysis of variance (ANOVA). The results are presented as mean ± SE.

RESULTS AND DISCUSSION

Proximate Composition

The moisture, protein, fat, ash and carbohydrate contents and calorie values of the raw whiting roes and the canned-smoked whiting roe pate are given in Table 1.

Table 1. Proximate analysis of raw and canned-smoked whiting roe n=3.

Sample	Moisture (%)	Protein (%)	Lipid (%)	Ash (%)	Carbohydrate (%)	Energy (kcal/100 g)
Raw	80.88±0.12 ^a	11.70±0.11 ^a	4.50±0.00 ^a	1.09±0.00 ^a	1.82±0.00 ^a	94.62±0.48 ^a
Canned-smoked	61.34±1.07 ^b	8.64±0.29 ^b	12.52±0.39 ^b	1.12±0.02 ^a	16.37±0.99 ^b	212.74±6.33 ^b

Values are shown as mean ± standard error of triplicates.

a, b.... (↓): The difference between the groups with different letters is important ($P<0.05$).

Roe has high nutritional value, and its chemical composition varies depending on fish species and processing techniques (19). According to the findings; the moisture, protein, fat, ash, carbohydrate contents and calorie value of the raw roe were 80.88±0.12%, 11.70±0.11%, 4.50±0.00%, 1.09±0.00%, 1.82±0.00% and 94.62±0.48 kcal, respectively. For the canned-smoked roe pate, these values were 61.34±1.07%, 8.64±0.29%, 12.52±0.39%, 1.12±0.02%, 16.37±0.99% and 212.74±6.33 kcal, respectively.

The moisture amount of the roes varies according to fish species and processing techniques (20). In this study, the moisture content of the raw roe was determined as 80.88%. It decreased resulted from vaporization that was occurred during the smoking and sterilization processes. Additionally, salt concentration (25%) in the solution was effective on the decrease in the moisture content. As a result, the mean moisture content of the canned-smoked roe pate was found as 61.34%. The moisture contents in the raw roe and canned-smoked roe pate were statistically significant ($P<0.05$). Sengor et al. (21) reported that moisture values of raw and smoked sturgeon roe were 75.42% and 68.40%, respectively. In another study, Inanli et al. (19) stated that moisture content of raw rainbow trout roe was 61.16%. Moisture values of various fish roes in results of previous studies (8, 22, 23) are in accordance with our study.

In the present study, the protein content of raw roe decreased after sterilization process ($P<0.05$). It was thought that, this decrease resulted from high lipid content in the roe pate because of added oil to the canned-smoked roe pate before sterilization. The protein content in the roe in this study was similar to the protein content of sea urchin roe (12.03%) reported by Mol et al. (24). A number of studies on proximate composition of roe from different fish species were made. In comparison to literature data, it was observed that protein value of the raw whiting roe was lower than trout (23), salmon (23), flathead mullet (25) and some sturgeon species (21, 23, 26).

Fish roes have high concentrations of fat and protein (27). In our study, the fat content of the raw roe was 4.50%. There was an increase in the canned-smoked roe pate and it was observed as 12.52% ($P<0.05$). It was supposed that increase in the fat content resulted from filling of the glass jars with sunflower oil. Bledsoe et al. (27) reported that the fat contents of whitefish, hake and whiting roes were 6.6-9.8%, 5-8% and 9%, respectively. The fat amount in rainbow trout roes was determined as 11.70% by Inanli et al. (19). In another study, the fat content of salmon roe was stated as 10.9-19.4%, averagely (26). The fat value of the raw whiting roe was found lower than results of several studies such as on flathead mullet (25), sturgeon (21) and codfish (8). Similarly, our protein values were lower than the results of other studies. Whiting is one of lean fish species, so low fat contents in our study may be expressed with low fat content of the whiting.

In our study, the mean ash content of the raw roe and canned-smoked roe pate was detected as 1.09% and 1.12%, respectively. No significant difference was observed between the ash values ($P<0.05$). Generally, ash values of different roe species were stated between 2.01% and 2.21% in the other studies (19, 20, 28). It was thought that boiling of the roes before immersing them into salty solution limited salt absorption.

The carbohydrate value (1.82%) of the raw roe increased to 16.37% in the canned-smoked roe. It was seen that addition of bread crumbs into the roe pate increased the carbohydrate value. Similarly it was observed that the calorie value was affected from this increase and so the calorie value of the raw roe (94.62 kcal) increased to 212.74 kcal in the canned-smoked roe. There was statistically significant difference between the carbohydrate and calorie values of the raw and canned-smoked roes ($P<0.05$).

Chemical Analysis

The TVB-N values of the raw roes, smoked roes and the canned- smoked roe pate are presented

in Table 2. A significant difference was found among groups in terms of the TVB-N values in our study ($P<0.05$). Long (29) reported that acceptability limit value of freshwater fish was 32-34 mg/100 g. Alperden et al. (30) stated that the limit value of roe was 32-34 mg/100 g, too.

In the present study, the TVB-N value of the raw roe was 9.84 mg/100 g. This value decreased to 6.13 mg/100 g after the roe was smoked and increased to 8.89 mg/100 g in the end product. All TVB-N values stayed within the acceptability limit values. It was thought that, increase in the TVB-N value did not result from the spoilage, but it was related with released nitrogen compound after denaturation (catabolism) of protein molecules with sterilization process. The TVB-N value of the raw whiting roe is similar to results of Inanli et al. (19) for salmon roe. Inanli et al. (20) reported that TVB-N value of raw trout roe was 6.90 mg/100 g. Ozpolat (28) stated that the TVB-N value of raw trout roe was 6.19 mg/100 g and it was 6.54 mg/100 g at day 0. Sengor et al. (21) reported that TVB-N values of raw and smoked sturgeon roes were 21.27 mg/100 g and 22.89 mg/100 g, respectively. These values were quite higher than the TVB-N values obtained in our study. The differences between the findings of researchers could be attributed to different fish species and application of different processes.

According to researchers, the TBA count must be less than 3 mg MA/kg in very good material and should not be more than 5 mg MA/kg in good material. The limit for consumption is between 7-8 mg MA/kg (31). The TBA values of the raw roes, smoked roes and the canned- smoked roe pate are presented in Table 2. The TBA value of the raw roe was determined as 0.36 mg MA/kg. The TBA value increased in the smoked roe (1.28 mg MA/kg) and decreased in the end product (0.58 mg MA/kg). As a reason for this situation, it might be said that sterilization process (conservation) decreased the microbial load,

therefore the TBA amount showed a decrease in the end product and the reason is the fact that microorganisms take part in oxidation of the lipids (32). In the present study, there was a significant difference among groups in terms of the TBA values ($P<0.05$). In previous studies made on flathead mullet (33) and trout (9) roes, TBA values were found low similar to our study. Gunlu (34) stated that increase in TBA after smoking process resulted from application of salting. Most probably, the increase in our TBA value that was observed for the smoked roe was related with the same reason. The decrease in our TBA value of the end product might be resulted from used additives or application of sterilization process.

The pH value should be between 6.00–6.50 for fresh fish and the upper acceptability limit is 6.80–7.00 (35). pH values of the raw roes, smoked roes and the canned- smoked roe pate are presented in Table 2. The pH value decreased in all phases of process. In a study, it was stated that pH value of smoked sturgeon roe decreased in comparison to raw samples (21). In our study, a significant difference was found among groups in terms of the pH values ($P<0.05$). pH values of fresh flathead grey mullet roe and dried roe were determined as 5.79 and 5.86 by Celik et al. (33), respectively. pH value of trout roe was reported as 7.77 (26). In another study made by Inanli et al. (9), pH values of trout roes salted at 4 and 8% were stated as 6.24 and 6.2, respectively (at day 0). These results are similar to our findings. Effects of smoking and sterilization processes might be shown as a reason for the decrease in the pH value.

Microbiological Analysis

Microbiological analysis results of the raw roes, smoked roes and the canned- smoked roe pate are given in Table 3. All microbiological analyses were carried out on the raw, smoked and canned-smoked samples. There was a significant difference among the groups ($P<0.05$).

Table 2. TVB-N, TBA, pH values of raw, smoked and canned-smoked whiting roes n=3.

Value	Raw	Smoked	Canned-smoked
TVB-N (mg/100g)	9.84±0.35 ^b	6.13±0.57 ^a	8.89±0.69 ^b
TBA (mg MDA/kg)	0.36±0.03 ^a	1.28±0.27 ^b	0.58±0.00 ^a
pH	6.55±0.03 ^c	5.16±0.00 ^a	4.98±0.00 ^a

Values are shown as mean ± standard error of triplicates.

a, b.... (→): The difference between the groups with different letters is important ($P<0.05$).

Table 3. Microbiological analysis results of raw, smoked and canned-smoked whiting roes (log cfu/g) n=3.

Sample	TMAB	TPAB	TYM	TCB
Raw	4.61±0.00 ^c	3.50±0.01 ^c	4.10±0.09 ^b	3.33±0.19 ^c
Smoked	4.28±0.02 ^b	2.85±0.1 ^b	3.79±0.16 ^b	1.14±0.41 ^b
Canned-smoked	N.D	N.D	N.D	N.D

Values are shown as mean ± standard error of triplicates.

a, b,... (↓): The difference between the groups with different letters is important ($P<0.05$).

When the roe is obtained in aseptic conditions, it is sterilized microbiologically. But commercially prepared roes are contaminated with different microorganisms in a short time. So, their sterility is affected (36). In our study, total mesophilic aerobic bacteria and psychrophilic aerobic bacteria counts of the raw roe were found as 4.61 and 3.50 log cfu/g, respectively. They decreased to 4.28 and 2.85 log cfu/g in the smoked roe, respectively. In the raw and smoked roes, respectively; total yeast-mold and coliform bacteria counts were determined as 4.10-3.33 log cfu/g and 3.79-1.14 log cfu/g. No microbial growth was observed in any of the end products.

Inanli et al. (20) determined that total mesophilic aerobic bacteria, psychrophilic aerobic bacteria and yeast-mold counts of raw rainbow trout roes were 4.74, 4.23 and 4.20 log cfu/g, respectively. These values are very similar to our findings. Counts of total mesophilic aerobic bacteria, yeast-mold and coliform in raw salmon trout roe was stated as 2.05, 0.97 and <0.48 log cfu/g by Patir et al. (37), respectively. The result in the study and the findings in our research are not similar. This difference may depend on different raw material and application of different processes. Karakas et al. (8) determined that total mesophilic aerobic bacteria counts of raw roe and salted roe were 7.88 and 3.18 log cfu/g, respectively. It was explained that, total mesophilic aerobic bacteria should be 7×10^4 - 7×10^6 cfu/g according to the APHA (38).

Sensory Evaluation

Sensory evaluation of the raw roes, smoked roes and the canned- smoked roe pate are presented in Table 4. When appearance, odor and color

points of the raw roe determined as 10 ± 0.00 by the panelists, texture points stayed as 9.6 ± 0.50 , averagely. There was no flavor evaluation for the raw roe. According to sensory results, it was observed that the smoked roe was evaluated with higher points in terms of appearance, odor and color than the canned-smoked roe pate.

It was tested that, there was no crumbling in texture after smoking and it had stiff structure which did not crumble when pressed with fork and it could also be ripped. As a result of the sensory evaluation of canned-smoked roe pate which was prepared by adding bread crumbs, it was seen that the color of it was light brown and it had an attractive smell and taste. Statistically, the smoked roe was significantly different ($P<0.05$) than the canned-smoked roe pate in terms of appearance, odor and color.

CONCLUSIONS

Besides the white and delicious flesh of whiting, its roe is also relished by consumers. Generally, it is consumed in fried form. In the present study, chemical, microbiological and sensory quality characteristics in the raw roes, smoked roes and the canned- smoked roe pate were evaluated. The TVB-N and TBA values stayed within the acceptability limit values. It was determined that the pH value increased with the smoking and sterilization processes. Besides, the total mesophilic aerobic bacteria, psychrophilic bacteria, yeast-mold and coliform counts of the raw whiting roe decreased with the effect of smoke. No growth was seen in microbiological analysis that was made at the end of the sterilization process.

Table 4. Sensory analysis results of raw, smoked and canned-smoked whiting roes n=3.

	Appearance	Odor	Color	Texture	Flavor
Raw	10±0.00 ^b	10.00±0.00 ^b	10.00±0.00 ^b	9.6±0.5a	*
Smoked	9.6±0.33 ^b	9.3±0.00 ^b	10.00±0.00 ^b	9±0.08a	9.7±0.25 ^a
Canned-smoked	8.6±0.5 ^a	8.1±0.6 ^a	8±0.5 ^a	9.5±0.08 ^a	9.7±0.25 ^a

Values are shown as mean ± standard error of triplicates.

a, b,... (↓): The difference between the groups with different letters is important ($P<0.05$).

The product is relished by the panelists in sensory analysis. As a conclusion; it may be said that immersing into the salty solution, boiling, smoking, canning and using additives contributed to the development of the product. The next step of this study can be doing extra research on related issues such as determination of its shelf life. These types of products may be varied by seafood processing sector.

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