Available online at www.sciencedirect.com
SCIENCE DIRECT®
Food Chemistry 88 (2004) 275–280
Food Chemistry
www.elsevier.com/locate/foodchem

Chemical, microbiological and sensory changes in thawed frozen fillets of sardine (*Sardina pilchardus*) during marination

Berna Kilinc *, Sukran Cakli

Ege University, Fisheries Faculty, Fish Processing Technology Department, Bornova-Izmir 35100, Turkey

Received 24 November 2003; received in revised form 8 January 2004; accepted 8 January 2004

Abstract

Frozen fillets of sardine (*Sardina pilchardus)* were used to make marinades. The marinating process was performed in 7% acetic acid and 14% sodium chloride in barrels. The fish : solution ratio was (1.5:1). The marination was carried out at 4 °C until the end point of marinating was assessed by sensory texture analysis. The complete marinating of sardine fillets required 22 days at 4 °C. According to statistical analysis at the beginning and at the end of the storage in barrels there were no significant differences ($p > 0.05$) in chemical analytical results. After the sardine fillets were put into the barrels, total viable count, lactic acid bacteria count, psychrotrophic bacteria count, yeast and mould counts were reduced.

Keywords: Fish; Sardine; Marination; Marinades; Quality control

1. Introduction

Sardine (*Sardina pilchardus*) is the most abundant fish species in the Ege Sea of Turkey. 16,500 t of sardine were caught in 2000 (Anonymus, 2000). Sardine is generally consumed fresh, canned or salted and the fish is also utilised as fishmeal and fish oil in Turkey (Kilinc, 1998). If sufficient amounts of sardine are available there is the possibility of developing new products with sardines. One possible alternative is the production of marinaded sardines. Sardines are very suitable for marination because of their fat contents but usage of marinaded sardine is not common in Turkey (Kilinc, 2003).

Marinated fish is preserved by the simultaneous action of organic acids, such as acetic acid, and salt. The combined preservative action prevents the growth of pathogenic bacteria and most spoilage bacteria. The products obtained have a pleasant taste without being too tough and have a reasonable shelf-life (Fuselli, Casales, Fritz, & Yeannes, 2003; Karl, 1994; McLay, 1972). The initial quality of raw materials, in terms of their freshness, microbiological loads and physical damage, is an important factor which influences the quality of the end product (Fuselli, Casales, Fritz, & Yeannes, 1994).

The aim of the present work was to study the physical, chemical and microbiological changes promoted by the marinating process in sardine fillets. The objectives of marinating are to decrease the pH value, to soften the texture until it becomes edible and to develop flavour.

2. Materials and methods

2.1. Raw materials

Frozen fillets of sardine (*Sardina pilchardus*) were used for marination. They were obtained from a fish processing plant. They had been rapidly and individually frozen at −40 °C and then stored at −18 °C for five months.

2.2. Process

Frozen sardine fillets were thawed in chill storage (4 °C) over night. Then they were washed with tap

* Corresponding author. Tel.: +90-232-388-4000/1300; fax: +90-232-3883685.
E-mail addresses: bernakilinc@mynet.com, kilinc@sufak.ege.edu.tr (B. Kilinc).

0308-8146/$ - see front matter © 2004 Elsevier Ltd. All rights reserved.
doi:10.1016/j.foodchem.2004.01.044

water. The brining process was done in a bath with 10% NaCl for 1 h at 4 °C and the fish : solution ratio was (1:1). Sardine fillets were then removed from the brine and placed in 70 l barrels with the marinating solution. The marinating process was performed in 7% acetic acid and 14% sodium chloride. Fish:solution ratio was (1.5:1). Marinating was carried out at 4 °C until the end point of marination was assessed by sensory texture analysis.

2.3. Chemical composition analysis

The chemical composition of sardine fillets was determined as crude protein ($N \times 6.25$) (AOAC, 1984), crude fat (Blig & Dyer, 1959), crude ash (AOAC, 1984) and moisture (Ludorff & Meyer, 1973). Analyses were done in triplicate.

2.4. Physical and chemical analysis

pH value was measured, as described by Lima dos Santos et al. (Lima Dos Santos, James, & Teutscher, 1981), by using a digital pH meter (HANNA). Thiobarbituric acid (TBA, mg malonaldehyde/kg) and total volatile basic nitrogen (TVB-N, mg N/100 g) values were determined, as described by Tarladgis, Watts, Younathan, and Dugan (1960) and Antonacopoulas and Vyncke (1989), respectively. The amount of formaldehyde extractable with $HClO_4$ [free formaldehyde; FA(ex), mg FA/kg] was measured by using the Nash (1953). Free and bound formaldehyde [FA(dest), mg FA/kg] values were measured by steam-distillation of acidified samples (Rehbein, 1986). Trimethylamine (TMA-N, mg/100 g) was determined as described by FAO (1986). Acetic acid and salt concentrations in fish flesh were measured as described by Karl (1994); acetic acid and salt concentrations in brine were measured, as described by (Schormüller, 1968; Ludorff & Meyer, 1973). Analyses were done in triplicate.

2.5. Microbiological analysis

For all microbiological counts, 10 g of sample were taken and transferred into 90 ml 0.1% peptone water (Difco, 0118-17-0) and homogenized. From the 10^{-1} dilution, other decimal dilutions were prepared. Total viable count was determined by using the pour plate method. Plate Count Agar (Difco, 0479-17) was used as medium. Plates were incubated at 30 °C for 24–48 h. (Harrigan & McCance, 1976). Plate Count Agar was also used for psychrotrophic bacteria count. In this case plates were incubated at 7 °C for 10 days (Ariyapitun, Mustapha, & Clarke, 1999). Oxytetracycline Yeast Extract Agar (LAB M X89) was used for moulds – yeast count. Plates were incubated at 30 °C for 3–5 days. (Harrigan & McCance, 1976). Lactic acid bacteria count was determined by using the pour plate method. MRSA (LAB M 93) was used as medium. Plates were incubated at 30 °C for 3–5 days (Dalgaard & Jorgensen, 1999).

Analyses were done in 3 parallels. The results of the all counts are recorded given as the mean values of 3 parallels.

2.6. Sensory analysis

For the determination of the sensory quality of frozen sardine fillets, a scoring test was used. The sardines without salt and spices were put into polyethylene bags which were tightly closed. The bags, including the samples of sardine fillets, were put into boiling water and cooked for 10 min. After cooking, they were cooled to 50 °C and samples were served to the panellists to evaluate the sensory attributes (appearance, odour, structure, flavour) of the samples by using the scoring test of Neuman, Molnar, and Arnold (1983). According to the scoring table, a total score of sensory attributes of 20 indicated excellent quality. Scores between 18.2 and 19.9 indicated (very good) quality, scores between 15.2 and 18.1 indicated (good) quality and scores between 11.2 and 15.1 indicated (middle) quality, scores between 7.2 and 15.1 indicated the limit of acceptibility and scores between 4.0 and 7.1 indicated spoiled samples.

During the immersion in the marinating bath, a scale that takes into account the changes in the texture because of fillet was used (Cabrer, Casales, & Yeannes, 2002). The scale for texture ranged from 1 for "inadequate texture from deficient or excess of marinating" (typical of raw fish without marinating or excessively soft, respectively) to 4 for "the best degree of marinating with a firm and consistent texture" (fillets are cut easily with a table fork). The point 2 in the sensory scale corresponds to a dry and hard texture, resistant to cut with a table fork for deficient marinating or soft and watery for excess of marinating. The point 3 corresponds to the hard, dry and fibrous texture for deficient marinating or less firm for excess of marinating.

2.7. Statistical analysis

Statistical evaluations of the physical, chemical and sensory analyses were made by using Microsoft Excel 7.0. and SPSS 9.05 programmes.

3. Results and discussion

Table 1 shows the changes of moisture, protein, lipid and ash contents of frozen fillets, brined fillets and marinated fillets. According to statistical analysis results of the Krusskal Wallis test, significant differences ($p < 0.05$) between chemical compositions of these fillets were found. The results of chemical composition anal-

Table 1
Changes in chemical composition (% on a wet weight basis)

Sample	% Moisture	% Protein	% Lipid	% Ash
Frozen fillet	79.47 ± 2.02	13.2 ± 0.10	3.60 ± 0.66	2.36 ± 0.41
Brined fillet (10% NaCl)	77.20 ± 1.77	13.6 ± 0.06	1.93 ± 1.07	4.02 ± 0.45
Marinated fillet (7% acetic acid, 14% NaCl)	73.70 ± 3.56	15.4 ± 0.31	4.44 ± 1.58	6.17 ± 0.29

$n = 3$, mean ± SD.

Table 2
Changes in chemical analysis during marination

Chemical analyses	Raw material	Marination process (day 0)	Marination process (day 22)
TBA (mg malonaldehyde/kg)	1.03 ± 0.64	2.47 ± 0.24	2.91 ± 0.37
TVB-N (mg/100 g)	10.24 ± 1.62	6.53 ± 1.62	11.2 ± 2.80
TMA-N (mg/100 g)	0.88 ± 0.13	1.07 ± 0.25	1.48 ± 0.17
FA(ex) (mg/kg)	1.03 ± 0.39	1.05 ± 0.22	1.25 ± 0.19
FA(dest) (mg/kg)	3.06 ± 0.29	3.19 ± 0.50	3.94 ± 0.19
pH (Flesh)	6.72 ± 0.01	4.23 ± 0.03	4.11 ± 0.11
pH (Brine)		3.86 ± 0.01	4.11 ± 0.01
Acetic acid % (Flesh)		1.09 ± 0.07	2.10 ± 0.07
Acetic acid % (Brine)		–	2.28 ± 0.03
Salt % (Flesh)		4.93 ± 0.04	7.12 ± 0.18
Salt % (Brine)		–	7.41 ± 0.17

$n = 3$, mean ± SD.

ysis of fillets during marination were in agreement with the marination study of Cabrer et al. (2002).

Table 2 shows the results of chemical analysis of raw material and the marination process. TBA is a good indicator of the quality of the fish, whether it was frozen, chilled or stored with ice (Tarladgis et al., 1960; Vareltzis, Zetou, & Tsiaras, 1988). It has been proposed that a maximum TBA value (indicating the good quality of the fish frozen, chilled or stored with ice) is 5 mg malonaldehyde/kg, while the fish may be consumed up to a level of 8 mg malonaldehyde/kg in TBA value (Schormüller, 1969). TBA value of raw material was found to be 1.03 mg malonaldehyde/kg. In the marination process, TBA value increased from 2.47 mg malonaldehyde/kg to 2.91 mg malonaldehyde/kg. But, according to statistical variance analysis, this increase was ($p > 0.05$) not significant. The data obtained in the present study suggest that TBA values of marinated fish are within the good quality limits after 22 days of processing in barrels at 4 °C.

TVB-N is used for determination of the spoilage level of fish during the storage period (Cobb & Venderzont, 1975; Kietzmann, Priebe, Rakov, & Reichstein, 1969; Oehlenschlager, 1981). In studies of the storage of different frozen fish species, it was suggested that TVB-N value may be at a high level (depending on the fish species) at the beginning of storage and may change, depending on the spoilage flora and analysis methods (Antonacopoulas & Vyncke, 1989; Kornop, 1976; Rehbein & Oehlenschlager, 1982). El Marrakchi, Bennour, Bouchriti, Hamama, and Togafait (1990) reported that the TVB-N value was more useful for assessing the degree of sardine deterioration than for evaluating the changes occurring during the first stages of storage. TVB-N value is affected by species, catching season and region, age and sex of the fish. A level of 35 mg/100 g has been considered the upper limit, above which fishery products are considered unfit for human consumption (Ludorff & Meyer, 1973; Schormüller, 1968). The TVB-N value of raw material was found to be 10.3 mg/100 g. After sardine fillets were put into barrels, TVB-N values decreased to 6.53 mg/100 g because of the effects of acetic acid and salt which were used in marination and which leached out the TVB-N components. According to variance analysis, no significant differences were found between TVB-N values of sardine fillets during the marination process at 4 °C ($p > 0.05$). In one report, the TVB-N value of fresh anchovy was found to be 8.7 mg/100 g. This value decreased to 7.41 mg/100 g in anchovy marinated with 6% acetic acid and 16% salt (Aksu et al., 1997). It has been reported that TVB-N value of raw shrimp was 27.5 mg/100 g. The TVB-N value in shrimp marinated with 2% citric acid and 4% salt decreased to 7.00 mg/100 g because of the citric acid and salt (Cadun, 2002). These results were very similar to our findings about the decrease of TVB-N value during marination.

TMA-N results from the reduction of TMAO by bacterial activity and partly by intrinsic enzymes. The quantity of TMA found in fish is used as an index of spoilage. In fresh fish, the TMA-N value is about 1 mg/100 g; in spoiled samples it is above 8 mg/100 g (FAO,

1986). The TMA-N value of raw material was found to be 0.88 mg/100 g. While, at the beginning of the marination process, the TMA-N was 1.07 mg/100 g, it was found to be 1.48 mg/100 g at the end. But, according to variance analysis, there were no significant differences ($p > 0.05$) in TMA-N values between the beginning and the end of the marination process. Gökoğlu, Cengiz, and Yerlikaya (2002) found that TMA-N values in sardine marinated with acetic acid solution (2%) were higher than those in sardine marinated with a solution containing acetic acid of 4%. TMA-N values, at both concentrations, significantly increased during the storage at 4 °C. In another report, TMA-N values in anchovy marinated with 2% acetic acid and 10% salt were increased from 1.11 mg/100 g to 4.07 mg/100 g after ten weeks of storage (Yapar, 1998). TMA-N values in anchovy marinated with 2% acetic acid solution and 15% salt were increased from 1.26 to 3.44 mg/100 g at the end of 10 weeks of storage. Similar results were reported with marinated fish in previous investigations (Aksu et al., 1997; Yapar, 1998).

FA values of raw material and marination stage were given as FA(ex) and FA(dest) in Table 2. In the present study, FA(ex) was 1.03 mg/kg and FA(dest) was 3.06 mg/kg in the raw material. At the start of the marination process FA(ex) began to increase from 1.05 mg/kg and rose to 1.25 mg/kg at the end of the marination process. FA(dest) value increased from 3.19 to 3.94 mg/kg at the end of the marination stage. In one study of fresh sardines, the FA(ex) value was 2.06 ± 0.59 mg/kg; FA(dest) was 1.59 ± 0.05 mg/kg. Depending on the increase of storage time at −18 °C, FA values increased. In Group A (sardine fillets), FA(ex) value changed from 1.10 ± 0.11 mg/kg to 1.94 ± 0.22 mg/kg while FA(dest) value changed from 0.56 ± 0.11 mg/kg to 4.31 ± 0.11 mg/kg. In Group B (whole sardine), while FA(ex) changed from 0.47 ± 0.11 mg/kg to 1.76 ± 0.14 mg/kg, FA(dest) value changed from 1.50 ± 0.22 mg/kg to 4.38 ± 0.17 mg/kg. Significant differences have been noted between FA values of two groups during storage at −18 °C for 60 days (Kilinc, 1998).

Formaldehyde and dimethylamine (DMA) are produced by enzymatic (TMOAase activity) degradation of trimethylamine oxide (TMAO), a natural constituent in the muscle of a large number of marine fish and shellfish (Rehbein, 1987). During freezing of the products, the formaldehyde produced is a highly reactive molecule, leading to inter- and intramolecular linkages between protein chains (Aubourg, 1998). As a result, protein denaturation and the loss of quality of the frozen fish have been associated with the formation of formaldehyde (Aubourg, 1998; Orlick, Oehlenschlager, & Schreiber, 1991).

It was reported by Meyer (1965) that there was an increase in the amount of formaldehyde in marinades. Because of this, in marinades, only small amounts of preservative usage are recommended for human consumption. An FA(dest) value of less than 10 was reported to represent very good quality and 75 the limit value for human consumption (Rehbein, 1987). In the present study, the FA(dest) value of sardine fillets in barrels was below the limit.

The pH of fresh fish is often between 6.0 and 6.5. During storage, pH always increases according to storage time. But the pH value does not offer a certain criterion of spoilage. It has to be supported by other chemical and sensory analyses (Ludorff & Meyer, 1973; Schormüller, 1968; Varlík, 1993). pH value of raw material was 6.72. After sardine fillets were put into the marinating barrels, the pH value decreased to 4.23 at the beginning of the storage. And at the end of the storage period (in barrels) the pH was decreased to 4.11. In contrast to this, the acetic acid concentration of sardine fillets increased from 1.09% to 2.10% at the end of the storage period in barrels. As soon as the marinating bath comes into contact with the fish, a diffusion of acetic acid and salt takes place into the tissue of the fish flesh until a concentration equilibrium is reached (Cabrer et al., 2002; Karl, Roepstorff, Huss, & Bloemsma, 1995; Meyer, 1965). Similar results were reported with fish marination in other investigations (Aksu et al., 1997; Cabrer et al., 2002; Karl et al., 1995; Yapar, 1998) showing decreasing pH value and increasing acetic acid concentration of fish fillets in marinades.

Results of microbiological analysis are given in Table 3. The total viable bacteria count, psychrotrophic bacteria count, lactic acid bacteria count, yeast and mould counts of raw material were 4.5×10^4 CFU/g, 7.6×10^4 CFU/g, 4.2×10^3 CFU/g, 20/g yeast and 10/g mould, respectively. After the sardine fillets were put into barrels, all these microorganisms were inhibited. After marination, the

Table 3
Changes in microbiological analysis during marination

Microbiological analyses	Raw material	Marination process (day 0)	Marination process (day 22)
Total viable count (CFU/g)	4.5×10^4	<10	<10
Lactic acid bacteria count (CFU/g)	4.2×10^3	<10	<10
Psychrophilic bacteria count (CFU/g)	7.6×10^4	<10	<10
Mould-yeast count (CFU/g)	10/g mould 20/g yeast	<10	<10

Table 4
Sensory analysis result of raw material

Raw material	Score
Appearance	4.43 ± 0.79
Texture	4.43 ± 0.53
Odour	4.57 ± 0.53
Flavour	4.43 ± 0.53
Total result	17.86 ± 2.38

$n = 5$, mean $\pm$ SD.

same negative results for microbiological counts were also found in other studies (Aksu et al., 1997; Fuselli et al., 1994).

The results of sensory analysis of raw material used in this study was 17.86 points. This score corresponds to good quality. The averages for individual attributes are shown in Table 4.

Taking into account the results of sensory analysis, the complete marinating of sardine fillets in barrels required 22 days at 4 °C in the marinating bath. At this time, the textures of sardine fillets were of the exact grade. When sardine fillets were put into the barrels, they were found to be at point 1. At storage period of 2, 3, 6 and 8 days, they were at point 2. At the storage periods of 10, 13, 15, 17 and 20 days, they were at point 3 and at the storage period of 22 days, the texture of sardine fillets reached the full marinating optimum (point 4). In the present study, we used the same sensorial analyses as Cabrer et al. (2002) to determine the end point of texture. In this study, the complete marinating of anchovy flesh required 9 days at 20 °C in the marinating bath, which was composed of 3% acetic acid and 10% sodium chloride (Cabrer et al., 2002). The bath temperature, fish species, thickness of fish flesh, fish:solution ratio and concentrations of acetic acid and sodium chloride are all very important for the marination.

4. Conclusion

In this study the score of full (complete) marinating was reached at 22 days at 4 °C, when the structural changes had concluded.

According to statistical analysis at the beginning and at the end of the storage in barrels there were no significant differences ($p > 0.05$) in chemical analysis results. After sardine fillets were put into the barrels, total viable count, lactic acid bacteria count, psychrotrophic bacteria count, yeast and mould counts were reduced.

Acknowledgements

This study was a summary of Berna Kilinc's PhD thesis. It was supported by (TUBITAK) Turkish Veterinary and Animal Research Group (Project No. 2002/VHAG 1839), Ege University Science Technology and Research Centre (EBILTEM) (Project No. 2002/BIL/024) and also it was supported by the Research Fund of Ege University Fisheries Faculty (Project No. 2001/SUF/012).

References

Aksu, H., Erkan, N., Çolak, H., Varlík, C., Gökoğlu, N., & Uğur, M. (1997). Some changes in anchovy marinades during production in different acid- salt concentrations and determination of shelf life. *Y.Y.Ü. Veteriner Hayvancílík Dergisi, 8*, 86–90.

Anonymus (2000). Fisheries Statistics. State Institute of Statistics, Prime Ministry Republic of Turkey, Ankara.

Antonacopoulos, N., & Vyncke, W. (1989). Determination of volatile basic nitrogen in fish: A third collaborative study by the West European Fish Technologists' Association (WEFTA). *Zeitschrift Fur Lebensmittel-Untersuchung Und-Forschung, 189*, 309–316.

AOAC (1984). *Official methods of analysis* (14th ed.). Washington, DC, USA: Association of Officialis Analytical Chemistry.

Ariyapitun, T., Mustapha, A., & Clarke, A. D. (1999). Microbial shelf-life determination of vacuum-packaged fresh beef treated with polylactic acid, lactic acid and nisin solutions. *Journal of Food Protection, 62*, 913–920.

Aubourg, S. P. (1998). Influence of formaldehyde in the formation of fluorescence related to fish deterioration. *Zeitschrift Fur Lebensmittel-Untersuchung Und-Forschung A, 206*, 29–32.

Blig, E. G., & Dyer, W. J. (1959). A rapid method of total lipid extraction and purification. *Canadian Journal of Biochemistry and Physiology, 37*, 911–917.

Cabrer, A. I., Casales, M. R., & Yeannes, M. I. (2002). Physical and chemical changes in anchovy (*Engraulis anchoita*) flesh during marination. *Journal of Aquatic Food Product Technology, 11*(1), 19–31.

Cadun, A. (2002). A study about shrimp marination and its quality. MSc Thesis, Fish Processing Technology Department, Fisheries Faculty, Ege University, Izmir, Turkey.

Cobb, B. F., & Venderzont, G. (1975). Development of a chemical test for shrimp quality. *Journal of Food Science, 40*, 121–124.

Dalgaard, P., & Jorgensen, L. V. (1999). Cooked and brined shrimps packed in a modified atmosphere have a shelf life of >7 months at 0 °C, but spoiled in 4–6 days at 25 °C. *International Journal of Food Science and Technology, 35*, 431–442.

El Marrakchi, A., Bennour, M., Bouchriti, N., Hamama, A., & Togafait, H. (1990). Sensory, chemical, and microbiological assessment of moroccan sardines (*Sardina pilchardus*) stored in Ice. *Journal of Food Protection, 53*(7), 600–605.

FAO (1986). *FAO food and nutrition paper manuals of food quality control food analysis: quality, adulteration, and tests of identity*. Rome: Food And Agriculture organization of the United Nations.

Fuselli, S. R., Casales, M. R., Fritz, R., & Yeannes, M. I. (1994). Microbiology of the marination process used in anchovy (*Encraulis anchoita*) production. *Lebensmittel-Wissenschaft Und-Technologie, 27*, 214–218.

Fuselli, S. R., Casales, M. R., Fritz, R., & Yeannes, M. I. (2003). Typical microorganisms in cold marinated anchovies (*Engraulis anchoita*) filled with corn oil and spices. *Journal Aquatic Food Product Technology, 12*, 55–62.

Gökoğlu, N., Cengiz, E., & Yerlikaya, E. (2002). Determination of shelflife of marinated sardine (*Sardina pilchardus*) stored at 4 °C. *Food Control, 3*, 4–7.

Harrigan, W. F., & McCance, M. E. (1976). *Laboratory methods in food and dairy microbiology*. London: Academic Press Inc.

Karl, H. (1994). Überlegungen zur Berechnung der Salz und Sauregehalte im Fischgewebewasser von marinierten Fischereierzeugnissen. *Informationen für die Fischwirtschaft, 41*, 47–59.

Karl, H., Roepstorff, A., Huss, H. H., & Bloemsma, B. (1995). Survival of Anisakis larvae in marinated herring fillets. *International Journal of Food Science and Technology, 29*, 661–670.

Kilinc, B. (1998). Physical, chemical, microbiological and sensory changes of sardine (*Sardina pilchardus*) during frozen storage. MSc Thesis, Fish Processing Technology Department, Fisheries Faculty, Ege University, Izmir, Turkey.

Kilinc, B., 2003. A study on marination of sardine (*Sardina pilchardus*) fillets and its shelflife. PhD Thesis, Institute of Fish Processing Technology, Ege University, Izmir, Turkey.

Kietzmann, U., Priebe, K., Rakov, D., & Reichstein, K. (1969). *Seefisch als lebensmittel*. Hamburg–Berlin: Paul Parey Verlag.

Kornop, G. (1976). Die lokale verteilung fluchtigen basen (TVB-N) im Gewebw von Ganzfischen Wahrend der Eisbgerug. *Archiv für Fischerei Wissenschaft, 27*, 159–169.

Lima Dos Santos, C., James, D., & Teutscher, F. (1981). Guidelines for chilled fish storage experiments. *FAO Fisheries Technical Paper*, 210.

Ludorff, W., & Meyer, V. (1973). *Fische und fischerzeugnisse*. Hamburg-Berlin: Paul Parey Verlag.

McLay, B.R. (1972). Marinades. Ministry of Agriculture Fisheries and Food. Torry Advisory Note No. 56 (14).

Meyer, V. (1965). *Marinades fish as food*. New York: Academic Press Inc., 489 p.

Nash, T. (1953). The colorimetric estimation of formaldehyde by means of the hantzsch reaction. *Biochemical Journal, 55*, 416–421.

Neuman, R., Molnar, P., & Arnold, S. (1983). *Sensorische Lebensmitteluntersuchung*. Leipzig: VEB Fachbuchverlag.

Oehlenschlager, J. (1981). Variation der gehelte an fluchtigen stickstofgehaltigen basen und 'TVB-N' in Retbersch. *Informationen für die Fischwirtschaft, 53*, 33–34.

Orlick, B., Oehlenschlager, J., & Schreiber, W. (1991). Changes in lipids and nitrogenous compounds in cod (*Gadus morhua*) and saithe (*Pollachius virens*) during frozen storage. *Archiv für Fischerei Wissenschaft, 41*(1), 89–99.

Rehbein, H. (1986). Formaldehyd in fischproducten. 1. Herkunff und gehalt. *Information Für Die Fischwirtschaft, 33*(1), 36–43.

Rehbein, H. (1987). Determination of the formaldehyde content in fishery products. *Zeitschrift Fur Lebensmittel-Untersuchung Und-Forschung, 185*, 292–298.

Rehbein, H., & Oehlenschlager, J. (1982). Zur zusammensetzung der TVB-N fraktion in sauren extrakten und alkalischen destillaten von seefishfillet. *Archiv Fur Lebensmittelhygiene, 33*, 44–48.

Schormüller, J. (1968). *Handbuch der lebensmittelchemie (Band III/2)*. Berlin-Heidelberg-New York: Springer Verlag.

Schormüller, J. (1969). *Handbuch der lebensmittelchemie (Band IV)*. Berlin-Heidelberg-New York: Springer Verlag.

Tarladgis, B. G., Watts, B. M., Younathan, M. S., & Dugan, L., Jr. (1960). A Distillation method for the quantitative determination of malonaldehyde in rancid foods. *Journal of American Oil Chemistry Society, 37*, 44–48.

Vareltzis, K., Zetou, F., & Tsiaras, I. (1988). Textural deterioration of chub mackerel (*Scomber japonicus collias*) and smooth hound (*Mustelus mustelus* L.) in frozen storage in relation to chemical parameter. *Lebensmittel-Wissenschaft Und-Technologie, 21*, 206–211.

Varlík, C., Uğur, M., Gökoğlu, N., & Gün, H. (1993). *Su ürünlerinde kalite kontrol ilke ve yöntemleri*. İstanbul: Gída Teknolojisi Derneği.

Yapar, A. (1998). Some quality changes in anchovy (*Engraulis encrasicolus*) marinades produced by using two different ripening solution. *Ege University Turkish Fisheries, 15*, 1–7.