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Effect of brining and frozen storage on physicochemical properties of well-fed Atlantic mackerel (*Scomber combrus*) intended for hot smoking and canning

Paulina E. Romotowska
María Guðjónsdóttir
Magnea G. Karlsdóttir
Sigurjón Arason
Ásbjörn Jónsson
Hordur G. Kristinsson
Telma B. Kristinsdóttir

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Report summary

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Titill / Title	**Effect of brining and frozen storage on physicochemical properties of well-fed Atlantic mackerel (*Scomber scombrus*) intended for hot smoking and canning**		
Höfundar / Authors	Paulina E. Romotowska[1, 2], María Gudjónsdóttir[2,] Magnea G. Karlsdóttir[1], Sigurjón Arason[1, 2], Ásbjörn Jónsson[1], Hordur G. Kristinsson[1, 3], Telma B. Kristinsdóttir[1, 2] [1]*Matís ohf. / Icelandic Food and Biotech R&D. Vínlandsleið 12, IS-113 Reykjavík. Iceland* [2]*University of Iceland, Faculty of Food Science and Nutrition.Vinlandsleid 14. IS-113 Reykjavík. Iceland* [3]*University of Florida, Department of Food Science and Human Nutrition, 359 FSHN Building, Newell Drive, Gainesville, FL 32611, USA.*		
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Ágrip á íslensku:	Makríll (*Scomber scombrus*) er tiltölulega ný nytjategund við strendur Íslands. Þar sem makríll er feitur fiskur með stutt geymsluþol, krefst hann því hámörkunar á geymsluaðstæðum og vinnsluferlum. Í þessu verkefni voru breytingar á efna- og eðliseiginleikum við hitameðhöndlun á söltuðum og ósöltuðum makríl rannsakaðar. Fyrir vinnslu var fiskurinn geymdur í 6, 9 og 12 mánuði við -18 °C og -25 °C með það fyrir augum að kanna hversu vel íslenskur frosinn makríll hentar sem hráefni í niðursoðnar og heitreyktar vörur. Til þess að athuga þau áhrif sem hitameðhöndlun hefur á vinnslueiginleika makríls voru sýnin hituð upp í 75 °C (til að herma eftir reykingu) og 90 °C (til að herma eftir niðursuðu). Langvarandi geymsla í frosti hafði neikvæð áhrif á hráefnið vegna aukinnar þránunar og var fiskurinn sem geymdur var við -18 °C með marktækt lakari gæði samanborið við fisk sem geymdur var við -25 °C fyrir vinnslu. Niðurstöðurnar sýndu að afurð hituð að 75 °C hafði hærra vatnsinnihald, hærri vatnsheldni og hærri nýtingu og var auk þess meyrari samanborið við afurð hitaða að 90 °C. Á heildina litið þá gefa niðurstöðurnar til kynna að feitur sumarmarkíll gæti hentað vel til vinnslu á niðursoðnum og heitreyktum afurðum.		
Lykilorð á íslensku:	*Uppsjávarfiskur; frostgeymsla; hitastig; niðursuða; heitreyking; þráun; eðliseiginleikar*		

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Summary in English:	Atlantic Mackerel (*Scomber scombrus*) is a novel species in Iceland and as a fatty fish with a short shelf-life it requires optimization of storage and processing conditions. Physicochemical changes of brined and un-brined mackerel were analysed during frozen storage (6, 9, 12 months) at -18 °C vs. -25 °C with the aim of investigating the suitability of using well-fed frozen mackerel as raw material for canned and hot-smoked products. Heat treatments to a core temperature of 90 °C (representing canning) and 75°C (representing hot-smoking) were applied. Prolonged frozen storage showed negative effects on the raw material prior to heat processing due to an increased level of lipid oxidation, where fish stored at -18 °C was of significantly poorer quality than fish stored at -25 °C. Moreover, the results indicated that heat treatment resulting in a core temperature of 75 °C showed higher water content, liquid holding capacity, heating yield as well as lower maximum shear force of texture compared to mackerel heated to a core temperature of 90 °C. Overall, analyses indicated that the fatty summer mackerel was well suitable for production of canned and hot-smoked products.
English keywords:	*Pelagic fish; frozen storage temperature; canning, hot-smoking, lipid oxidation; physicochemical properties*

Contents

1 Introduction

Atlantic mackerel is a streamlined fast-swimming pelagic fish that resides in shoals and migrates vast distances in its lifespan (Godø, Hjellvik, Iversen, Slotte, Tenningen & Torkelsen, 2004). Since 2007, mackerel has been caught in Icelandic waters and its abundance has increased steadily along with its importance. For instance, exportation values of frozen mackerel from Iceland reached 21.3 million ISK in 2013 and 23.7 million ISK in 2014 (Statistics Iceland, 2015). The mackerel migrates into Icelandic waters early summer in order to search for feed and restore the energy reserves. However, limited information exists on the physicochemical properties and stability of the Atlantic mackerel caught during the heavily feeding period (Icelandic fishing time), which may affect the initial raw material characteristics and therefore in-depth analysis is required. Previous studies have shown that the muscle lipid content of the mackerel increases during the heavy feeding period, which may lead to great seasonal and individual variation in the fatty acid composition and quality of the Icelandic mackerel (Overholtz, Hare & Keith, 2011; Jansen, Campbell, Kelly, Hatun & Payne, 2012; Astthorsson, Valdimarsson, Gudmundsdottir & Oskarsson, 2012; Valdimarsson, Astthorsson & Palsson, 2012).

Mackerel is also a great source of omega-3 polyunsaturated fatty acids (PUFAs) which makes it an excellent choice of food for human consumption. Several studies have shown beneficial roles of marine omega-3 PUFAs in regards to coronary artery disease, reducing effects for cardiovascular dealings, as well as affecting psychological health such as depression (Delagado-Lista, Perez-Martinez, Lopez-Miranda, & Perez-Jimenez, 2012; Perica, 2011).

However, high amounts of PUFAs result in instability of the raw material during processing and storing due to lipid degradation and oxidation (Saeed & Howell, 2002; Zotos, Hole, & Smith, 1995). In addition, lipid oxidation is the main cause for quality loss of fatty fish, by causing off-flavour, loss of taste as well as it may create health hazards such as the formation of highly reactive radicals (Jónsdóttir, Bragadóttir, & Ólafsdóttir, 2008). It is therefore important to minimize lipid oxidation during storage prior to any secondary processing.

Freezing is a commonly used preservation method to prolong the shelf-life of fish. During the freezing process, the conversion of unbound water to ice occurs in the fish muscle and the portion of frozen water depends on the food material as well as the freezing temperature (Karlsdottir,

Sveinsdottir, Kristinsson, Villot, Craft, & Arason, 2014a). In frozen fish, the unfrozen liquid phase may lead to deterioration processes which continue throughout the frozen storage duration. Furthermore, it should be noted that concentrations of enzymes, salts and other dissolved substances increase in the unfrozen solution during the freezing process (Hall, 1997).

During heat treatment of fish, myofibrils undergo specific structural changes that are associated with the liquid holding capacity (Ofstad, Kidman, Myklebust, Olsen, & Hermansson, 1995). Tender and juicy products are more likeable to consumers while dryness or toughness in texture are less appealing (Esaiassen, Ostli, Elvevoll, Joensen, Prytz, & Richardsen, 2004). Therefore, it is important to optimize proper heat processing in order to obtain the highest quality product.

The aim of present project was to explore the processing properties of Atlantic mackerel caught in Icelandic waters after its heavy feeding period in September and to evaluate the potential of using the well fed mackerel as raw material for the production of canned and hot-smoked products for human consumption after frozen storage. Firstly, the purpose of this study was to conclude the impact of frozen storage temperature (-18 °C, -25 °C) and duration (6, 9 and 12 months) on various physical and chemical properties of Atlantic mackerel prior to two types of heat treatment, representing pre-cooking prior to canning (core temperature of 90 °C) and hot-smoking (core temperature of 75 °C). Secondly, the effect of brining before heat treatment on the canned mackerel was investigated.

2 Materials & Methods

2.1 Material, processing and sampling

Atlantic mackerel (*Scomber scombrus*) was caught during the summer of 2012 (beginning of September) in the South-East of Iceland (North-East Atlantic Ocean - FAO no 27) by trawler (Börkur NK-122). The whole mackerel (300g – 500g) was frozen in blocks in a vertical plate freezer and stored at either -18 °C or -25 °C prior to further processing. Six fishes (in blocks) from each storage temperature (-18 °C and -25 °C) were sampled at each sampling occasion at 6, 9 and 12 months of storage (n=24). The fish blocks were allowed to thaw in air at room temperature for 17 hours, thereafter the sampled fishes were filleted by hand.

The fillets from each frozen storage temperature were then divided into 4 groups (8 groups in total) with three fillets in each (n=3). Two groups from each storage temperature were brined in 10% NaCl (100g/L), followed by draining for 35 min at 4°C, while the other groups were left unsalted. All brined fillets were then placed in heat-resistant polyamide bags (PMT, Reykjavik, Iceland), two fillets (one fish) in each. The samples were then cooked in a steam oven (Convotherm, Elektrogeräte, GmbH, Eglfing, Germany) at either 160 °C for 15 min to reach a core temperature of at least 90 °C, representing heat treatment during canning, or at 100 °C for 10 min to reach a core temperature of 75 °C, representing heat treatment during hot-smoking. The non-brined fillets were either placed in plastic bags and cooked the same way as the canned group (core temperature of 90 °C) or kept raw as a control group. Samples of raw and heat treated fillets were then minced with skin and used for analysis. All physicochemical analyses were performed in triplicates.

All chemicals used during analysis of samples were of analytical grade from Fluka (Buchs, Switzerland) or Sigma-Aldrich (Steinheim, Germany / St. Louis, MO, USA).

2.2 Chemical composition

Water content of the mackerel samples was determined by the weight difference during drying of 5 g minced samples at 104 °C ± 1 °C for 4 h (ISO, 1999). Results were calculated as g water/100 g sample.

Total lipids (TL) of the fish samples were extracted according to the method of Bligh and Dyer (1959) and the lipid content was determined gravimetrically. The results were expressed as g lipid/100 g of the sample.

2.3 Heating yield

The weight of the fillets before and after heating treatment was recorded. The heating yield was calculated as the ratio of the fillet weight after cooking to the fillet weight before cooking (raw and/or brined fillets) as presented by equation 1:

Heating yield of fillets (%) $= \frac{g\ cooked\ fillets}{g\ raw\ fillets} * 100$ **(Equation 1)**

2.4 Texture measurements

Texture analysis was performed on all heated samples. After allowing the samples to cool down to room temperature, pieces were cut from the fillet loins, approx. 4x1.5x2 cm in size. The shear force was measured with a Warner-Bratzler angled blade using a TA.XT2® Texture Analyser (Stable Micro Systems, Haslemere, Surrey, UK). The deformation rate was 2 mm s^{-1}. This angled blade was 3.2 mm in thickness, 123 mm in height and 70 mm in width and the angle was at 60 °C. The shear force (N) of the sample was recorded as the maximum peak force required to cut through the sample. Results represent the toughness of the fish muscle, incorporating to shearing of the fibres and their compression underneath the blades (Bouton, Harris, & Shorthouse, 1975).

2.5 Liquid holding capacity (LHC)

The liquid holding capacity (LHC) of mackerel samples was measured with the centrifugal method described by Eide, Børresen, and Strøm (1982). Approximately 2.0 ± 0.1g of the samples were weighed precisely into a vial and centrifuged (Biofuge Stratos, Thermo Electron Corporation,

Germany) at 1350 rpm for 5 minutes at 4 °C. LHC (%) was calculated as the ratio of liquid in the sample after centrifugation to liquid in the sample before centrifugation. This method is commonly used to analyse water holding capacity (WHC) of fish muscle. However, the term liquid holding capacity was chosen for this measurement whereas it was noted that the weight loss during centrifugation did not consist only of water, but of fat as well. Therefore, the LHC of the samples in this project was an indicator to the fish muscles ability to withhold any liquids, whether it was fat or water.

2.6 Lipid oxidation products

2.6.1 Lipid hydroperoxide values

A modified ferric thiocyanate method was used to determine the lipid hydroperoxide content of the fillets (Shantha & Decker, 1994). Five grams of sample were mixed with 10 mL of ice-cold chloroform:methanol (1:1) solution (with addition of 500 ppm butylated hydroxytoluene (BHT), which was used to prevent peroxidation during measurements). Five mL of sodium chloride (0.5 M) were added to the mixture, which was then homogenized at 2400 rpm for 10-20 sec. (Ultra-Turrax T25 basic, IKA Labortechnik, Germany). Phase separation was facilitated by centrifugation at 5000 rpm for 5 min at 4 °C (TJ-25 Centrifuge, Rotor TS-5.1-500, Beckman Coulter, California, USA). The lower chloroform layer containing the lipids was collected (100 µL) and mixed with 900 µL of chloroform: methanol (1:1) solution. For samples that had endured the longest storage time, the results only became readable (within the parameters of the prepared standard curve) after changing the ratio of chloroform to solvent to 50:950 µL. Finally, a 5 µL mixture (1:1) of ammonium thiocyanate (4 M) and ferrous chloride (80 mM) was added, before vortexing. After 10 min of incubation at room temperature, the absorbance was measured at 500 nm (Tecan Sunrice, Austria) on a polypropylene microplate (Eppendorf, microplate 96/F-PP). The concentration of lipid hydroperoxide was determined using a standard curve prepared from cumene hydroperoxide (60 µM). Results were expressed as µmol lipid hydroperoxide per g of sample.

2.6.2 Thiobarbituric acid reactive substances (TBARS)

TBARS was determined with a modified method of Lemon (1975). The sample (5 g) was homogenized (Ultra-Turrax T25 basic, IKA Labortechnik, Germany) with 10 mL of 7.5% trichloroacetic acid (TCA) solution, 0.1% propyl gallate and 0.1% etylenediaminetetraacetic acid (EDTA). After centrifugation at 5000 rpm for 20 min at 4 °C (Beckman Coulter TJ-25, Rotor TS-5.1-500, USA) the collected supernatant was filtered with a Whatman qualitative filter paper no 4. Thiobarbituric acid (0.02 M) in an amount of 900 µL was mixed with the collected supernatant (100 µl) before heating the mixture in a water bath at 95 °C for 40 min. After heating, the mixture was immediately placed on ice for cooling and the absorbance was measured at 530 nm (Tecan Sunrice, Austria). TBARS were determined using a standard curve prepared from 1.1.3.3-tetraethoxypropane (TEP). The results were expressed as µmol malomaldehyde diethyl acetal per kg of sample.

2.7 Enzymatic lipid hydrolysis

The free fatty acid (FFA) content was determined using the method of Lowery and Tinesley (1976) with a modification as described by Bernardez, Pastoriza, Sampedro, Herrera and Cabo (2005). The absorbance of the solution was read at 710 nm (UV-1800 spectrophotometer, Shimadzu, Japan) and the amount of free fatty acids was determined, using a standard curve prepared from oleic acid in a concentration range of 2 - 22 µmol. Results were expressed as grams FFA per 100 g of total lipids.

2.8 Statistical analysis

Statistical analysis of data was performed using Microsoft Office Excel 2010 (Microsoft Inc. Redmond, Wash, USA), NCSS (NCSS 2000, Utah, USA) and SigmaStat 3.5 (Dundas Software Ltd., GmbH, Germany). One-way ANOVA, General Linear Models (GLM), Duncan´s comparison test and Pearson correlation were applied on means (n = 3) for each group. The significance level was set at $p \leq 0.05$.

3 Results & Discussion

3.1 Water and lipid content

Water content of the mackerel before heat treatment ranged from 56.3% to 59.4% and was neither affected by the frozen storage time nor temperature ($p > 0.05$). The water content of the mackerel after heat treatment was ranging from 48.0% up to 62.5% (Table 1). The fish heated up to a core temperature of 75 °C maintained significantly higher water content (57.1 ± 3.8%) than the pre-cooked fish up to core temperature of 90 °C (53.3 ± 0.9%).

The total lipid content of the raw material after frozen storage varied from 12.9 to 25.7% (Table 1). The high standard deviation in the total lipid content may be explained by the high variation between individual fishes due to variation in availability of food and type of the food (Overholtz et al., 2011; Jansen et al., 2012; Astthorsson et al., 2012).

3.2 Heating yield determination

The heating yield of mackerel varied from 84.1% to 98.1% (Figure 1) but a significant change was observed between the different heat treatments. The heating yield showed high positive correlation with the analysed water content of the fish ($r = 0.7$, $p = 0.003$). Samples heated up to a core temperature of 75 °C resulted in significantly higher yield than samples heated up to core temperature of 90 °C. The heating yield determination was conducted in order to measure the ability of muscle to maintain water and thereby muscle weight. It can be assumed that increased temperatures caused greater protein denaturation and therefore higher levels of broken protein-protein bonds, shrinkage of the cells and water loss (Bertola, Bevilacqua, & Zaritzky, 1994; Ofstad, Kidman, & Hermansson, 1996; Karlsdottir, Sveinsdottir, Kristinsson, Villot, Craft, & Arason, 2014a). However, the frozen storage temperature and duration prior to heat processing had no effect on the heating yield of the mackerel.

3.3 Liquid holding capacity (LHC)

Neither frozen storage temperature, nor storage time had a significant effect on the liquid holding capacity (LHC) of mackerel prior to heat treatment (Figure 2), in correlation with the insignificant changes in water content during the frozen storage. However, the LHC of the mackerel was significantly affected by heat treatment. The highest LHC value was obtained for samples heated up to core temperature of 75 °C after 9 months of frozen storage, and the lowest LHC was observed for samples heated up to core temperature of 90 °C without brining (89.4% ± 3.6 and 48.7% ± 3.03, respectively) (Figure 2). It can therefore be assumed that changes in LHC were correlated to the protein deterioration which occurred greatly at higher temperature, leading to dehydration of the muscle through disruption of cell structures (Skipnes, Ostby, & Hendrickx, 2007).

In addition, higher LHC was obtained when the fish was brined before heat processing ($p < 0.05$). It has been previously reported that brine at low concentration may lead to anions binding to the filaments and consequently increase repulsive force of the filaments leading to swelling of the muscle (Thorarinsdottir, Arason, Bogason, & Kristbergsson, 2001; Gudjónsdóttir, Arason, & Rustad, 2011).

3.4 Texture

The maximum shear force of the heat threated mackerel ranged from 11.8 N to 176.4 N, with significantly highest shear force values for the pre-cooked fish up to a core temperature of 90 °C compared to samples heated up to core temperature of 75 °C (Figure 3). These results are in agreement with research conducted by Skipnes, Ostby, and Hendrickx (2007), where the hardness of farmed cod increased after cooking. This is probably due to heat induced toughening of the fish muscle, after denaturation of myofibrillar and sarcoplasmic proteins, as actin and other sarcoplasmic proteins denaturation occurs at 60 – 90 °C (Kong, Tang, Rasco, Crapo, & Smiley, 2007). This also explains the difference between samples heated up to 75°C and 90 °C, since more proteins denatured as the temperature rises, thus leading to increasing water loss and muscle toughening and consequently a higher shear force of the muscle.

Furthermore, the heated mackerel samples were significantly higher in shear force after pre-cooking up to core temperature of 90 °C without brine addition, compared to brined samples, indicating that the salt-induced swelling of the muscle during brining had a preventive effect on the hardness of the fillets

Muscle softening was observed with prolonged storage at -18°C in brined samples heated up to 90 °C, while no such effect was seen at -25°C. However, the muscle in the brined fillets heated up to 75 °C got a tougher texture with prolonged frozen storage, independent on frozen storage temperature. Additionally, the shear force showed significant negative correlation with water content ($r = -0.6$, $p = 0.01$), lipid holding capacity ($r = -0.5$, $p = 0.05$), as well as heating yield ($r = -0.6$, $p = 0.01$). This is in compliance with previous findings, where the shear force of fish muscle has been associated with its water and lipid content, where higher lipid or water contents the softer the texture. Takahashi, Inoue, and Shinano (1993) suggested that less water in the fish muscle and a higher level of protein results in firmer product after cooking. Moreover, Foegeding, Lanier, and Hultin (1996) findings showed that firmness of cooked fish was related with both muscle pH and moisture content.

3.5 Lipids oxidation products

Lipid oxidation development was affected by prolonged frozen storage prior to heat processing (Figure 4). Lipid hydroperoxide (PV) formation, a primary oxidation product, increased significantly in the raw material between 6 months and 12 months of storage at -18 °C. Hydroperoxide production appeared to be much higher and more rapid in the fish stored at -18 °C ($p < 0.05$), compared to the fish stored at -25 °C, which was relatively stable during the storage duration (Figure 4a).

A similar pattern was obtained for the formation of secondary oxidation products, where TBARS increased significantly with extended storage time (Figure 4b). Moreover, the results showed higher level of TBARS for the fish stored at -18 °C, indicating that the lipid oxidation development is partly inhibited at lower frozen storage temperature. These findings are in general agreement with other studies regarding the effects of storage temperature on lipid oxidation (Karlsdottir, Sveinsdottir, Kristinsson, Villot, Craft, & Arason, 2014b; Aubourg, Lago, Sayar, & González, 2007).

Regarding the effect of the different heat treatments no significant difference was seen in the oxidation products. . Although, these changes were not statistically significant, earlier studies have suggested that heat processing may lead to decomposition of oxidation products (Rodríguez, Trigo, Pérez, Cruz, Paseiro, & Aubourg, 2009). A Pearson correlation analysis indicated a positive relationship between heating yield and PV ($r = 0.6$, $p = 0.01$), as well as negative correlation between fillet weight and PV and TBARS ($r = -0.7$, $p = 0.003$; $r = -0.6$, $p = 0.01$, respectively). In addition, present of brine resulted in slightly higher lipid oxidation development which may suggest that salt addition may accelerate oxidation rate (Aubourg & Ugliano, 2002). These results are in general agreement to precious the previous studies (Kanner, Harel, & Jaffe, 1991; Osinchak, Hultin, Zajicek, Kelleher, & Huang, 1992).

3.6 Enzymatic lipid hydrolysis

Formation of free fatty acids (FFA) is generally associated with enzymatic activity inside the fish muscle leading to lipid hydrolysis. FFA in the frozen mackerel samples ranged from 1.0 to 3.2 FFA/100 g lipids (Figure 5). The greatest changes in FFA content were observed during frozen storage temperature prior to heat treatment. Fish stored for 12 months at -18 °C before processing reached the highest FFA level. A significant difference in FFA formation was observed between storage temperatures, where fish stored at -18 °C prior the processing showed significantly higher level of FFA than fish stored at -25 °C. Consequently, it can be assumed that lower temperature during frozen storage decrease level of hydrolysis by inhibition of enzyme activity, mainly lipase, occurred in the non-frozen phase and responsible for food spoilage (Aubourg, Rey-Mansilla, & Sotelo, 1999).

Heat treated samples showed lower values of FFA than the fillets before heat processing. These results indicated the possibility of FFA losses through liquids lost during the heat treatments (Rodriguez-Estrada, Penazzi, Caboni, Bertacco, & Lercker, 1997). For instance, a positive correlation was found between heating yield and FFA ($r = 0.5$, $p = 0.05$). Furthermore, heat processing may also inhibit lipases which accelerate FFA formation (Wang, Miller, & Addis, 1991; Karlsdottir, Sveinsdottir, Kristinsson, Villot, Craft, & Arason, 2014a).

Increased accumulation of the FFA during frozen storage is a consequence of enzyme activity (Aubourg & Medina, 1999; Shewfelt, 1981). Decrease of FFA with extended storage time may have been associated with loss of liquid during the heat treatments. This is in agreement with the negative correlation between FFA levels and the amount of liquid loss after heat treatment ($r = - 0.44$; data not shown).

In addition, neither different temperature of heat treatment nor brining had any significant effect on the FFA level.

4 Conclusions

The present study demonstrated physical and chemical properties of mackerel as caught in Icelandic waters during the heavy feeding period and analysed its potential as raw material for high value canned and hot-smoked products as well as its stability during prolonged frozen storage.

Different heat treatment of the frozen mackerel samples resulted in changes of physicochemical properties. Accordingly, heat treatment representing pre-cooking prior to canning (core temperature of 90 °C), in contrary to hot-smoking (core temperature of 75 °C), resulted in lower water content and liquid holding capacity and hence lower heating yield and increased toughness of texture.

However, it should be noted that different properties of mackerel are required with the intention to obtain specific product. It is important to apply proper heat treatment in order to meet consumers and market demands, as well as proper time of exposure to heat treatment.

Correspondingly, addition of a relatively small amount of brine solution before heat treatment may increase quality of the mackerel products. Salting of the fish had a beneficial effect on heating yield and texture.

Furthermore, prolonged storage of the raw material prior to heat processing had negative effect on its quality. However, present study indicated inhibited lipid oxidation rate by decreased frozen storage temperature of the raw material. Therefore, it can be recommended to store frozen mackerel before further processing at -25 °C rather than at -18 °C.

In conclusion, mackerel caught in Icelandic water has potential to be utilized for the production of value-added products, such as canned and hot-smoked products.

5 Acknowledgements

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6 Figure Captions

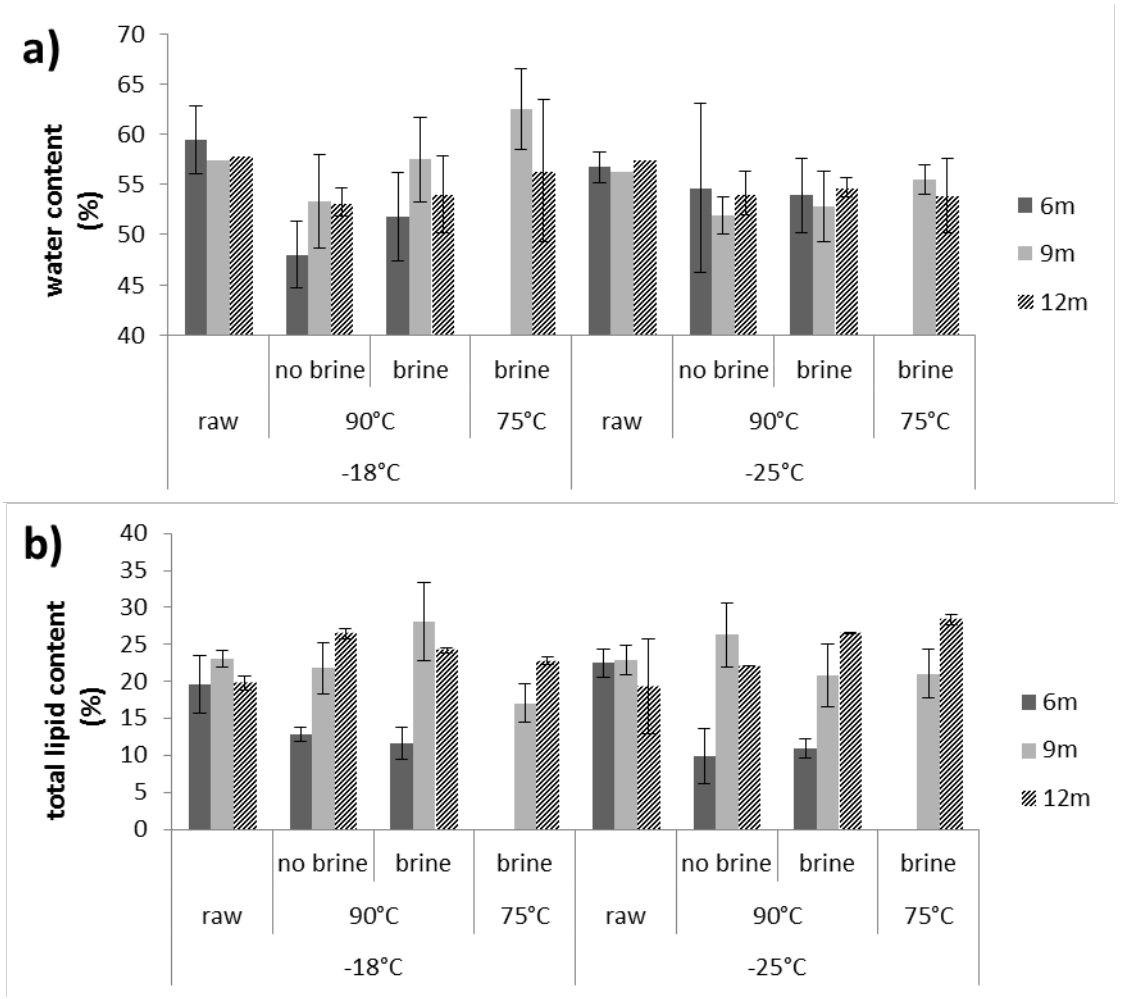

Figure 1 Water and total lipid content (%) of mackerel fillets as affected by frozen storage time (6, 9 and 12 months) and temperature (-18 °C and -25 °C) prior to processing, and different heat treatments (core temperature of 75 °C and 90 °C). Samples were either brined or non brined before heat treatment (n = 3; mean ± stdv.). Unbrined and unheated samples were used as control for each frozen storage condition (duration and temperature).

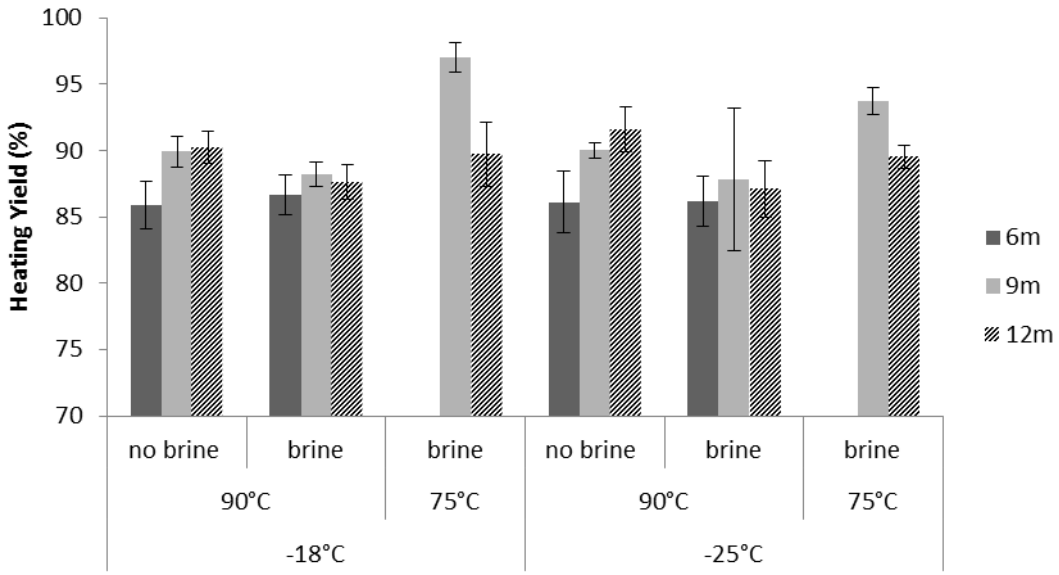

Figure 2 Changes in heating yield (%) during different heat treatments of frozen mackerel stored at -18 °C and -25 °C for 6, 9 and 12 months prior to processing. Heated fillets up to core temperature of 90 °C and 75 °C were either brined or non-brined. Bars represents standard deviation (n = 3).

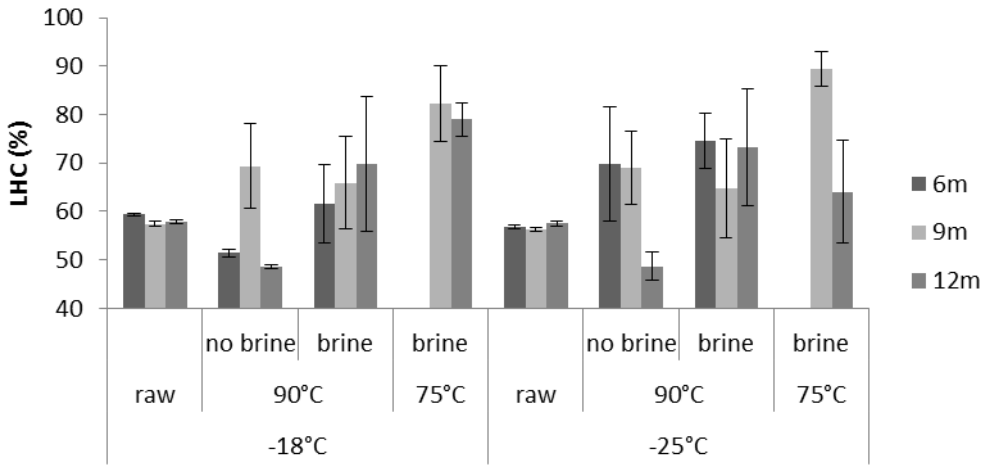

Figure 3 Changes in liquid holding capaciy (LHC%) during different heat treatments of frozen mackerel stored at -18 °C and -25 °C for 6, 9 and 12 months prior to processing. Heated fillets up to core temperature of 90 °C and 75 °C were brined and non-brined. Bars represents standard deviation (n = 3).

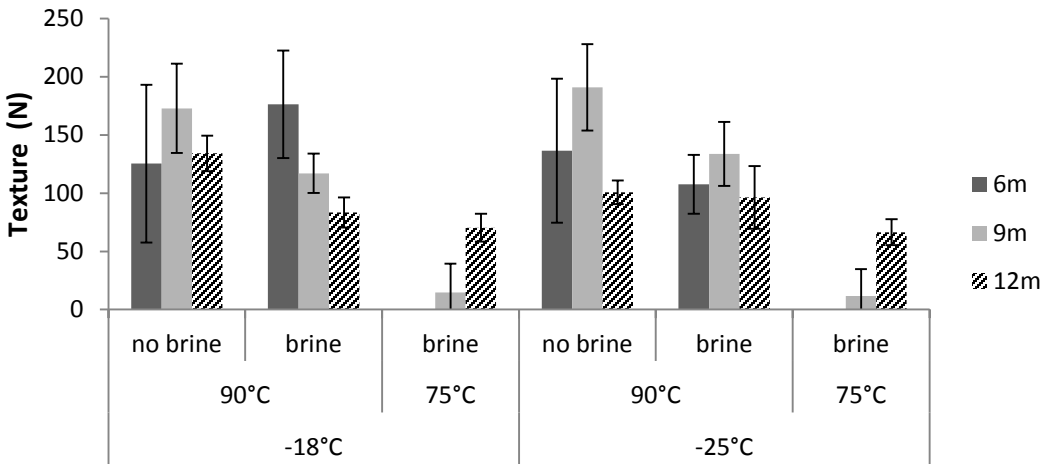

Figure 4 Changes in texture (N) during different heat treatments of frozen mackerel stored at -18 °C and -25 °C for 6, 9 and 12 months prior to processing. Heated fillets up to core temperature of 90 °C and 75 °C were brined and non-brined. Bars represents standard deviation (n = 3).

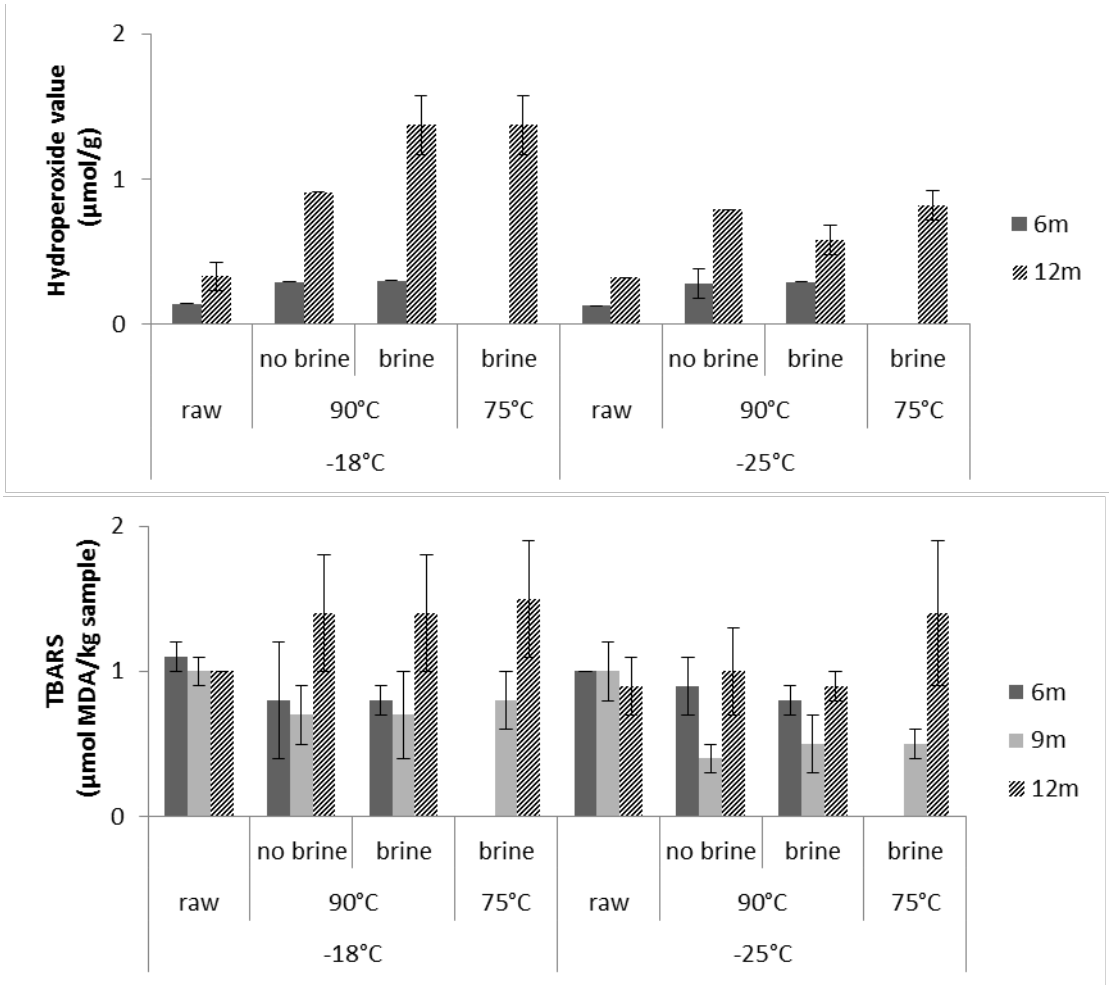

Figure 5 Lipid hydroperoxide formation (µmol/kg sample) and thiobarbituric acid reactive substances formation (TBARS; µmol MDA/ kg sample) during different heat treatments of frozen mackerel stored at -18 °C and -25 °C for 6, 9 and 12 months prior to processing. Heated fillets up to core temperature of 90 °C and 75 °C were brined and non-brined. Bars represents standard deviation (n = 3).

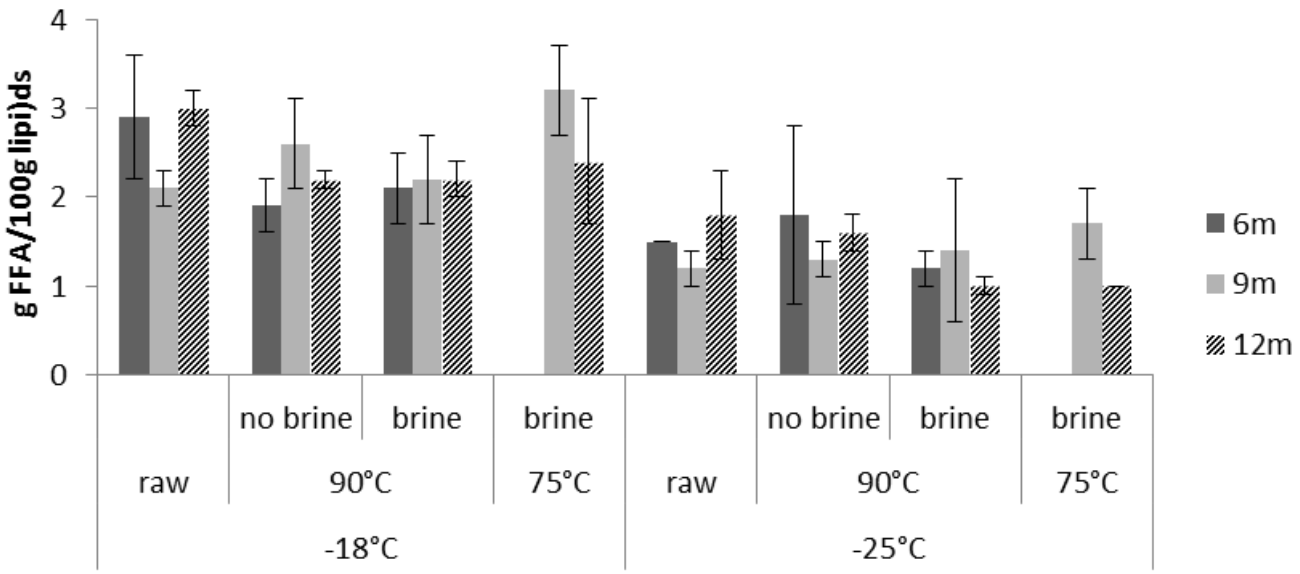

Figure 6 Evaluation of free fatty acid level (g FFA/100g lipids) during different heat treatments of frozen mackerel stored at -18 °C and -25 °C for 6, 9 and 12 months prior to processing. Heated fillets up to core temperature of 90 °C and 75 °C were brined and non-brined. Bars represents standard deviation (n = 3).

7 References

Aubourg, S., Lago, H., Sayar, N., & González, R. (2007). Lipid damage during frozen storage of Gadiform species captured in different seasons. *European Journal of Lipid Science and Technology, 109*, 608-616.

Aubourg, S., & Medina, I. (1999). Influence of storage time and temperature on lipid deterioration during cod (*Gadus morhua*) and haddock (*Melanogramus aeglefinus*) frozen storage. *Journal of Agricultural and Food Chemistry, 79*, 1943-1948.

Aubourg, S., Rey-Mansilla, M., & Sotelo, C. G. (1999). Differential lipid damage in various muscle zones of frozen hake (*Merluccius merluccius*). *Z Lebensm Unters Forsch A, 208*, 189-193.

Aubourg, S., & Ugliano, M. (2002). Effect of bine pre-treatment on lipid stability of frozen horse mackerel (*Trachurus trachurus*). *European Food Research and Technology, 215*, 91-95.

Bernardez, M., Pastoriza, L., Sampedro, G., Herrera, J. J. R., & Cabo, M. L. (2005). Modified method for the analysis of free fatty acids in fish. *Journal of Agricultural and Food Chemistry, 53*, 1903-1906.

Bertola, N. C., Bevilacqua, A. E., & Zaritzky, N. E. (1994). Heat-Treatment Effect on Texture Changes and Thermal-Denaturation of Proteins in Beef Muscle. *Journal of Food Processing and Preservation, 18*(1), 31-46.

Bligh, E. G., & Dyer, W. J. (1959). A rapid method of total lipid extraction and purification. *Canadian Journal of Biochemistry and Physiology, 37*(8), 911-917.

Bouton, P. E., Harris, P. V., & Shorthouse, W. R. (1975). Changes in shear parameters of meat associated with structural changes produced by aging, cooking and myofibrillar contraction. *Journal of Food Science, 40*, 1122.

Delagado-Lista, J., Perez-Martinez, P., Lopez-Miranda, J., & Perez-Jimenez, F. (2012). Long chain omega-3 fatty acids and cardiovascular disease: a systematic review. *British Journal of Nutrition, 107*(2), 201-213.

Eide, O., Borresen, T., & Strom, T. (1982). Minced fish production from capelin (Mallotus villosus) A new method for gutting, skinning and removal of fat from small fatty fish species. *Journal of Food Science, 47*, 347-349.

Esaiassen, M., Ostli, J., Elvevoll, E. O. J., Joensen, S., Prytz, K., & Richardsen, R. (2004). Brining of cod fillets: influence on sensory properties and consumer liking. *Food Quality and Preference, 15*, 421-428.

Foegeding, E., Lanier, T., & Hultin, H. (1996). Chapter 15. Characteristics of edible muscle tissues. In O. Fennema (Ed.), *Food Chemistry* 3rd ed., (pp. 879-942). New York: Marcel Dekker, Inc.

Hall, G. M. (1997). *Fish processing technology.* Cornwall.: TJ Press Ltd.

ISO. (1993). Determination of moisture and other volatile matter content (6496). *Geneva, Switzerland: The International Organization for Standardization*.

Jittinandana, S., Kenney, P. B., Slider, S. D., & Kiser, R. A. (2002). Effect of Brine Concentration and Brining Time on Quality of Smoked Rainbow Trout Fillets. *Journal of Food Science, 67*(6).

Jónsdóttir, R., Bragadóttir, M., & Ólafsdóttir, G. (2008). Oxun fiskvöðva - Hlutverk fosfólípíða, prótein, andoxunarefna og áhrif suðu á oxun. In). Reykjavík: Matís Report 08-08.

Kanner, J., Harel, S., & Jaffe, R. (1991). Lipid peroxidation of muscle food as affected by sodium chloride. *Journal of Agricultural and Food Chemistry, 39*(6), 1017-1021.

Karlsdottir, M. G., Sveinsdottir, K., Kristinsson, H. G., Villot, D., Craft, B. D., & Arason, S. (2014a). Effect of thermal treatment and frozen storage on lipid decomposition of light and dark muscles of saithe (Pollachius virens). *Food Chemistry, 164*(0), 476-484.

Karlsdottir, M. G., Sveinsdottir, K., Kristinsson, H. G., Villot, D., Craft, B. D., & Arason, S. (2014b). Effects of temperature during frozen storage on lipid deterioration of saithe (Pollachius virens) and hoki (Macruronus novaezelandiae) muscles. *Food Chemistry, 156*(0), 234-242.

Kong, Tang, T., Rasco, B., Crapo, C., & Smiley, S. (2007). Quality changes of Salmon (Onchorhynchus gorbuscha) muscle during thermal processing. *Journal of Food Science, 72*(2), 103-111.

Lemon, D. W. (1975). *An improved TBA test for rancidity.* . In *New Series Circular, No. 51. Fisheries and Marine Services Canada*). Halifax, Nova Scotia.

Lowery, & Tinesley. (1976). Rapid colorimetric determination of free fatty acid. *Journal of the American Oil Chemists Society, 53*, 470-472.

Moini, S., Khoshkhoo, Z., & Hemati Matin, R. (2012). The Fatty Acids Profile in Mackerel (Scomberomorus guttatus) and its Shelf Life in Cold Storage at -18°C. *Global Veterinaria, 8*(6), 665-669.

Ofstad, R., Kidman, S., & Hermansson, A. (1996). Ultramicroscopical structure and liquid loss in heated cod (Gadus morhua L) and salmon (Salmo salar) muscle. *Journal of the Science of Food and Agriculture, 72*, 337-347.

Ofstad, R., Kidman, S., Myklebust, R., Olsen, R. L., & Hermansson, A. (1995). Liquid-holding capacity and structural changes in comminuted salmon (Salmo salar) muscle as influenced by pH, salt and temperature. *LWT – Food Science and Technology, 28*(3), 329-339.

Osinchak, J. E., Hultin, H. O., Zajicek, O. T., Kelleher, S. D., & Huang, C. H. (1992). Effect of NaCl on catalysis of lipid oxidation by the soluble fraction of fish muscle. *Free Radical Biology and Medicine, 12*(1), 35-41.

Perica, M., Delas, I (2011). Essential fatty acids and psychiatric disorders. *American Society for Parenteral and Enteral Nutrition, 26*(4), 409-425.

Rodriguez-Estrada, M. T., Penazzi, G., Caboni, M. F., Bertacco, G., & Lercker, G. (1997). Effect of different cooking methods on some lipid and protein components of hamburgers. *Meat Science, 45*(3), 365-375.

Rodríguez, A., Trigo, M., Pérez, R., Cruz, J. M., Paseiro, P., & Aubourg, S. (2009). Lipid oxidation inhibition in frozen farmed salmon (*Oncorhynchus kisutch*): Effect of packaging. *Czech Journal of Food Sciences, 27*(Special Issue), S182-S184.

Saeed, S., & Howell, N. K. (2002). Effect of lipid oxidation and frozen storage on muscle protein of Atlantic mackerel (Scomber scombrus). *Journal of the Science of Food and Agriculture, 82*, 579-586.

Shantha, & Decker. (1994). Rapid, sensitive, iron-based spectrophotometric methods for determination of peroxide values of food lipids. *Association of Official Analytical Chemists International*(77), 421-424.

Shewfelt, R. (1981). Fish muscle lipolysis - a review. *Journal of Food Biochemistry, 5*(2), 79-100.

Skipnes, D., Ostby, M. L., & Hendrickx, M. E. (2007). A method for characterizing cook loss and water holding capacity in heat treated cod (Gadus morhua) muscle. *Journal of Food Engineering, 80*, 1078–1085.

Statistics Iceland. (2015). Hagtíðindi: Útflutningur og útflutningsframleiðsla sjávarafurða 2014/ Statistical series: Export and export production of marine products 2014. In *Hagtíðindi Sjávarútvegur*, vol. 21). Reykjavik: Hagstofa Íslands.

Takahashi, K., Inoue, N., & Shinano, H. (1993). Effect of storage temperature on freeze denaturation of carp myofibrils with KCl or NaCl. *Bulletin of the Japanese Society of Scientific Fisheries [Nihon Suisan Gakkai shi] 25 ref., 59*(3), 519-527.

Thorarinsdottir, K. A., Arason, S., Bogason, S. G., & Kristbergsson, K. (2001). Effects of phosphate on yield, quality, and water holding capacity in the processing of salted cod (Gadus morhua). *Journal of Food Science*, *66*(6), 821-826.

Torres, J. A., Saraiva, J. A., Guerra-Rodríguez, E., Aubourg, S. P., & Vázquez, M. (2014). Effect of combining high-pressure processing and frozen storage on the functional and sensory properties of horse mackerel (Trachurus trachurus). *Innovative Food Science & Emerging Technologies, 21*, 2-11.

Vázquez, M., Torres, A., Gallardo, J. M., Saraiva, J., & Aubourg, S. P. (2013). Lipid hydrolysis and oxidation development in frozen mackerel (Scomber scombrus): Effect of a high hydrostatic pressure pre-treatment. *Innovative Food Science & Emerging Technologies, 18*, 24-30.

Wang, Y.-J., Miller, L., & Addis, P. (1991). Effect of heat inactivation of lipoxygenase on lipid oxidation in lake herring (coregonus artedii). *Journal Of The American Oil Chemists Society*, *68*(10), 752-757.

Zotos, A., Hole, M., & Smith, G. (1995). The effect of frozen storage of mackerel (Scomber scombrus) on its quality when hot-smoked. *Journal of the Science of Food and Agriculture, 67*(1), 43-48.