Influence of lipid content and packaging methods on the quality of dried capelin (*Mallotus villosus*) during storage

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Abstract Capelin annual catch exceeds half a million tons in Iceland, with only a small quantity (<20%) of female with roe used for human food. There is a potential to use dried male capelin as a new product for human consumption, but its lipid content varies considerably (4–20% body weight). Earlier studies were more concentrated on the influence of drying conditions than the influence of storage conditions on the quality of dried fish, as dried fish are usually considered to be stable and safe during storage. Three batches of dried male capelin differing in lipid content were packaged and studied during 5 months storage at 22 ± 2 °C to establish appropriate lipid content at harvesting and product packaging method. Lipid composition, lipid hydrolysis and oxidation, sensory attributes and microbial activity were evaluated. Batches differed in composition and stability, with low lipid capelin constituting higher proportion of polyunsaturated fatty acids

(22% lipid) than high lipid (18% lipid) capelin. Lipid oxidation was influenced by lipid content and packaging method, as accelerated oxidation occurred in high lipid and open packed capelin. Lipid hydrolysis was less influenced by packaging and was greater in low lipid capelin. High lipid capelin in open bags scored the highest for rancid odor. All batches were micro-biologically stable with colony-forming unit counts increasing less than log 1 (log 5–6) during 5 months storage.

Keywords Dried capelin · Lipid content · Packaging · Stability

Introduction

Capelin (*Mallotus villosus*) is a small pelagic schooling fish that is economically and ecologically important in the northern hemisphere (ICES 2009). Large populations occur in the Atlantic and Pacific Oceans but important commercial fisheries have developed in the Atlantic Ocean (Carscadden et al. 2013). Capelin catch in Iceland exceeds half a million tons in recent years (Statistics Iceland 2015). A small quantity of it specifically female capelin with roe is used for human food, with about 80% used for fishmeal and oil production (Statistics Iceland 2015). So there is a potential to increase the use of capelin for human consumption.

In Iceland, capelin is caught during spawning migration in winter, from December to March. Its lipid content depends on maturity and season, and varies from about 4 to 20% body weight (Bragadóttir et al. 2002). There are even variations in lipid content of capelin caught within the same month of the year (Cyprian et al. 2015). Due to relatively high amounts of PUFA in fish (Stołyhwo et al. 2006; Bragadóttir et al. 2002), the lipid in dried capelin

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may adversely affects its nutritional quality, wholesomeness and sensory value during distribution and marketing. In Eastern Africa particularly Kenya, Tanzania and Uganda the consumption of dried small pelagic species is widespread (IOC 2012; Oduor-odote et al. 2010). The increased consumption of small dried pelagic species has been ascribed to the increase in human population, decline in catches of table fish and improved processing (IOC 2012). Consumer preferences for the products are not only because of the taste and flavor, but also because of low price and good stability during storage (IOC 2012). This provides a possible avenue for utilizing capelin for human consumption by marketing dried capelin in East Africa and elsewhere. A similar small temperate fish species, blue whiting (*Micromesistius poutassou*), is dried and marketed in Nigeria for human consumption.

Storage is an important part in the distribution and marketing of processed food. Storage life is determined by processing method, product composition, packaging and storage conditions. Air drying is advantageous over other preservation methods because it is affordable and efficient (Jain 2006; Bellagha et al. 2002). Quality deteriorations such as lipid hydrolysis that influence the formation of oxidation products occur during storage of dried fish (Doe 2002). Air dried products are also easily contaminated by microbes mainly air borne molds as they are often processed and sold unpackaged in an open-air environment (Park et al. 2014).

Packaging is an important part of the food industry. Food packaging has a preservation function and delivers safe, wholesome and attractive foods to the market (Kilcast and Subramaniam 2000). The methods of packaging can influence the preservation of quality of dried fish. Open air packaging is generally used for dried small pelagic species (mostly lean fish) in the intended markets and can expose dried capelin saturated with oil to oxygen. Vacuum packaging eliminates the air and is known to improve the shelf life and overall quality of muscle foods (Etemadian et al. 2012). Earlier studies were more concentrated on the influence of drying conditions than the influence of storage conditions on the quality of dried fish, as dried fish are usually considered to be stable and safe during storage (Cyprian et al. 2016; Hwang et al. 2012; Dewi et al. 2011; Oduor-odote et al. 2010; Kilic 2009; Wu and Mao 2008). The present study was aimed at assessing lipid deterioration, sensory and microbial quality during storage of dried capelin as influenced by lipid content in the fish muscle and packaging method. Samples were stored at 22 ± 2 °C in order to simulate warm climate as expected in the distribution and marketing of this kind of product.

Materials and methods

Raw material, processing and sampling

Three batches of frozen male capelin (*Mallotus villosus*) caught on 1st (C1), 13th (C2) and 28th (C3) February, 2013 in the North Atlantic were obtained from HB Grandi fishing company, Reykjavik, Iceland. The fish had been stored in chilled seawater (0 °C) for two days post-catch before freezing at -25 °C in blocks weighing 25 kg each and kept frozen for 3 months until the study time. Batches differed significantly ($p < 0.05$) in lipid content with values of 10% (10.32 ± 0.65), 9% (8.83 ± 0.46) and 7.5% (7.54 ± 0.48) for C1, C2 and C3, respectively. Before drying, the frozen fish was thawed overnight at 18 – 20 °C. Ten fish from each group were tagged for weighing during drying to determine when the samples attained hygroscopic equilibrium moisture. Fish were arranged separately by groups in a single layer on plastic meshed trays and dried in a tunnel drier under controlled temperature, relative humidity and air speed (Cyprian et al. 2016).

After drying the fish (DC1, DC2 and DC3) was taken to Matis laboratory and each group divided into three equal portions which were further divided into 30 equal size samples, of these 10 were put into open polyethylene bags, 10 in sealed polyethylene bags and 10 were vacuum packaged in 10 high-barrier film bags (40PA/70LDPE, 250 mm $\times$ 400 mm $\times$ 0.120 mm, Plastprent Iceland). Packed fish were stored at room temperature (22 ± 2 °C) and sampled every 30 days for five months when two bags were picked randomly for analyses from each packaging treatment for each batch, 18 bags in all.

Chemicals

Chemicals used in this study were of analytical grade purchased from Sigma-Aldrich (Steinheim, Germany), Sigma-Aldrich (St. Louis, MO, USA) and Fluka (Buchs, Switzerland).

Water content and water activity

Water content of raw and dried capelin was determined as the difference in weight of minced fish ($n = 4$) before and after drying at 103 ± 1 °C for 4 h (ISO 1993). Results were expressed as g water/100 g mince. Water activity (a_w) was measured ($n = 4$) using an Aqua Lab instrument (Novasina AW-Center, AWC503 RS-C, Axlar AG, Switzerland) as described by Cyprian et al. (2016).

Total lipid

Lipid was extracted from 25 g of minced fish (with $80 \pm 1\%$ water content) ($n = 4$) with methanol/chloroform/0.88% $\text{KCl}_{(\text{aq})}$ (at 1/1/0.5, v/v/v) according to the Bligh and Dyer (1959) method. The lipid content was determined gravimetrically and the results were expressed as g lipid per 100 g mince.

Fatty acid composition

The fatty acid composition of the total lipid extracts ($n = 4$) was determined by gas chromatography of fatty acid methyl esters (FAME). Methylation of fatty acids was carried out according to AOAC (2000). The lipid extract was vaporized to constant weight at 55°C under nitrogen. About 70 mg of the pure lipid extract was dissolved in 1.5 mL 0.5 N NaOH (in methanol) and incubated at 100°C for 7 min. After cooling, 2 mL BCl_3 , 12% in methanol were added and incubated again at 100°C for 30 min. The sample was cooled and 1 mL of standard solution (1 mg/mL 23:0 methyl ester in isooctane) and 5 mL saturated NaCl solution were added and mixed. After phase separation, the isooctane layer was transferred into a test tube containing 1 mm bed of anhydrous Na_2SO_4 . This was repeated with 1 mL of clean isooctane. The combined isooctane layers, containing the FAMEs were then transferred to GC vials ($2 \times 750 \mu\text{L}$). The FEME s were separated by GC (Varian 3900 GC, Varian, Inc., Walnut Creek, CA, USA) equipped with a fused silica capillary column (HP-88, 100 m $\times$ 0.25 mm $\times$ 0.20 μm film), split injector and flame ionization detector fitted with Galaxie Chromatography Data System (Version 1.9.3.2 software). The result of each fatty acid was expressed as g fatty acid per 100 g lipid.

Phospholipid and free fatty acid

Phospholipid (PL) content was determined on the lipid extract ($n = 4$) by a colorimetric method, based on a complex formation of phospholipid and ammonium ferrothiocyanate (Stewart 1980), before reading the resultant solutions absorbance at 488 nm (UV-1800 spectrophotometer, Shimadzu, Kyoto, Japan). The reported PL was based on a standard curve prepared with phosphatidylcholine in chloroform (5–50 $\mu\text{g/mL}$) and results expressed as g phosphatidylcholine equivalent/100 g lipid. Free fatty acid (FFA) content was determined on 3 mL of the lipid extract based on a complex formation with cupric acetate-pyrimidine (Bernardez et al. 2005), followed by absorbance reading of the upper layer at 710 nm. The FFA concentration was calculated as μM quantities of oleic acid, based on a standard curve spanning a

2–22 μmol range. Results were expressed as g FFA/100 g lipid.

Lipid oxidation

Lipid hydroperoxides (PV) were determined using the ferric thiocyanate method described by Santha and Decker (1994) with modifications according to Karlsdottir et al. (2014) and Cyprian et al. (2016). The results were expressed as μmol lipid hydroperoxides per kg mince ($n = 4$). Thiobarbituric acid-reactive substances (TBARS) were measured as described by Lemon (1975) with modifications according to Cyprian et al. (2015). The results were expressed as μmol of malomaldehyde diethylacetal/kg mince ($n = 4$).

Fluorescent compounds were determined with a Perkin-Elmer LS 50B fluorescent spectrometer (Perkin-Elmer, Massachusetts, USA). The absorbance of the organic phase resulting from the lipid extraction described earlier (Bligh and Dyer 1959) was measured at 393/463 and 327/415 nm excitation/emission maxima according to previous studies (Nguyen et al. 2012; Karlsdottir et al. 2014). The fluorescence shift (OFR) was calculated as the ratio between the two relative fluorescence intensity (RF) values at excitation/emission maxima 393/463 and 327/415 nm, i.e. $\text{OFR} = \text{RF}_{393/463\text{nm}}/\text{RF}_{327/415\text{nm}}$.

Microbial analyses

To determine microbial quality of dried capelin, aerobic plate count (TPC), molds and yeast counts were carried out according to general microbiological principles. Samples were analyzed in duplicate per package. Twenty five grams of minced fish ($n = 4$) were mixed with 225 ml of cooled Maximum Recovery Diluent (MRD, Oxoid) in a stomacher bag to obtain a tenfold dilution. Blending was done in stomacher for 1 min. Successive tenfold dilutions were done as required. TPC were determined by plating aliquots on plate count agar (PCA) using pour plate technique. Enumeration of TPC was performed after 3 days incubation under aerobic conditions at $30.0 \pm 1.0^\circ\text{C}$. Molds and yeast were determined from the aliquots by spread plating onto Dichloran Rose-Bengal Chloramphenicol agar (DRCB-Agar) in Petri dishes. Molds and yeast were counted separately after 5 days incubation at $22 \pm 1^\circ\text{C}$. The results were expressed as log CFU/g minced sample.

Sensory evaluation with generic descriptive analysis (GDA)

A sensory panel of 10 members participated in the GDA of the dried capelin. All participants were trained according to international standards (ISO 1993b) including detection

and recognition of tastes and odors, and in the use of scales. The members of the panel were familiar with the GDA method and experienced in sensory evaluation. The panel used an unstructured scale from 0 to 100 (Stone and Sidel 1985) to evaluate rancid odor and stock-fish odor. About 5 cm portions were cut from individual capelin after removing the head and tail. The samples were placed in aluminum boxes coded with 3-digit numbers that did not indicate sample group nor storage time or packaging method. Each panelist evaluated duplicates of samples in a random order in two sessions. A computerized system (FIZZ, Version 2.0, 1994–2000, Biosystemes, France) was used for data recording.

Data analysis

Data analyses were done using Microsoft Excel 2010 (Microsoft Inc. Redmond, Wash., USA). One way analysis of variance (ANOVA), Duncan's test (Post-hoc) and Pearson correlation analysis were performed on means of the variable values in the statistical program NCSS 2000 (NCSS, Utah, USA). Multivariate comparison of different variables and samples was performed using Principal Component Analysis (PCA) on mean level corrected variable values using full cross-validation in the Unscrambler[®] statistical program (Version 8.0 CAMO, Trondheim, Norway).

Results and discussion

Lipid, water content and water activity

Lipid content of raw fish decreased from early to late February and was significantly ($p < 0.05$) different between the batches with values of 10, 9.0 and 7.5% in C1, C2 and C3, respectively. The corresponding water content increased and was 73, 75 and 77%. This was in agreement with the variation in lipid content of capelin that was the highest in late fall and gradually decreased during migration in early winter to spawning in March/April (Bragadóttir et al. 2002) and with the general inverse relationship between water and lipid content in fish (Cyprian et al. 2015; Henderson et al. 1984).

The difference in lipid content of dried batches was also significant ($p < 0.05$) with values of 31, 28 and 25%, and corresponding water content of 23, 19 and 18% in DC1, DC2 and DC3, respectively and water activity 0.758, 0.683 and 0.676. High lipid content reduced drying rate with the less fatty capelin ending drier than the fattest capelin. The lipid was thought to play a role in limiting the drying process (Cyprian et al. 2016) by replacing the aqueous phase that served as vector for transfers or acting as a

physical barrier to heat transfer. Dried capelin batches had a moisture content $< 25\%$ and water activity < 0.80 that was enough to inhibit the growth of harmful bacteria and assure product safety (Kilic 2009). During storage, lipid and water content in dried packed capelin did not change significantly ($p > 0.05$) in any of the groups (data not shown).

Fatty acids (FA) composition

Monoenic (MUFAs) fatty acids constituted 57–60% of the total lipid in dried capelin, followed by saturated (SFAs) 20–21% and polyunsaturated (PUFAs) 19–22% of the total lipid (Table 1). Raw and smoked capelin muscle lipid has been reported to have a high concentration of MUFAs (46–57%) (Cyprian et al. 2015; Bragadóttir et al. 2002). The predominant acids among the MUFAs were long-chain C22:1 (n-11 and n-9) and eicosenoic acid (C20:1n9). Proportions of PUFAs were significantly ($p < 0.05$) higher in DC3 and DC2 compared to DC1. This may be explained by relatively high phospholipid content in DC3 and DC2 (Cyprian et al. 2015; Parmentier et al. 2007). Eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) constituted about 13% of the total lipid.

Packaging methods influenced changes in the proportion of fatty acid groups in dried capelin during storage (Fig. 1).

Table 1 Fatty acid composition (g fatty acids/100 g lipid) present in lipid extracted from dried capelin mince (n = 4)

Fatty acid	DC1	DC2	DC3
C14:0	6 ± 0 ^a	6 ± 0 ^a	6 ± 0 ^a
C16:0	12 ± 0 ^a	12 ± 0 ^a	12 ± 0 ^a
C18:0	1.2 ± 0.11 ^{a*}	1.2 ± 0.15 ^a	0.6 ± 0.31 ^b
Other SFA	2.2 ± 0.12 ^a	1.5 ± 0.06 ^b	2.5 ± 0.33 ^a
Total SFA	21.2 ± 0.56 ^a	20.6 ± 0.34 ^b	21.1 ± 0.11
C16:1n7	8 ± 0 ^a	8 ± 0 ^a	8 ± 1 ^a
C18:1n7	3 ± 0 ^a	3 ± 0 ^a	3 ± 0 ^a
C18:1n9	9 ± 0 ^a	9 ± 0 ^a	10 ± 01 ^a
C20:1n9	15.2 ± 0.00 ^a	14.6 ± 0.25 ^b	14.6 ± 0.37 ^b
C22:1(n-11 and n-9)	20.2 ± 0.11 ^a	19.3 ± 0.28 ^b	19.4 ± 0.36 ^b
Other MUFA	3.8 ± 0.55 ^a	3.3 ± 0.41 ^a	2.2 ± 0.25 ^b
Total MUFA	60.1 ± 1.22 ^a	57.7 ± 0.92 ^b	57.4 ± 1.06 ^b
C18:2n6	1 ± 0 ^a	1 ± 0 ^a	1 ± 0 ^a
C18:4n3	2 ± 0 ^a	2 ± 0 ^a	2 ± 0 ^a
C20:5n3 (EPA)	6.7 ± 0.00 ^a	6.9 ± 0.26	7.2 ± 0.33 ^b
C22:6n3 (DHA)	6.1 ± 0.06 ^a	7.2 ± 0.61 ^b	6.8 ± 0.33 ^b
Other PUFA	2.7 ± 0.43 ^a	3.9 ± 0.61 ^b	4.1 ± 0.29 ^b
Total PUFA	18.7 ± 1.02 ^a	21.5 ± 1.11 ^b	21.6 ± 0.97 ^b

DC1 dried capelin of high lipid, DC2 dried capelin of moderate lipid, DC3 dried capelin of low lipid, SFA saturated fatty acids, MUFA monounsaturated fatty acids, PUFA polyunsaturated fatty acids

* Different letters (superscript^{a–b}) within a row indicate significantly different values between samples ($p < 0.05$)

Fig. 1 Changes in major fatty acid classes (g fatty acid/100 g total lipid) in packed dried capelin (a–c) during 5 months of storage at ambient temperature (n = 4)

Proportion of PUFAs became lower in all batches during storage with a corresponding increase in both SFAs and MUFAs. This is because PUFAs are highly susceptible to oxidation (Karlisdottir et al. 2014; Nguyen et al. 2012). Oxidation of PUFAs appeared to be reduced more by vacuum packaging of the fattest capelin (DC1) than in less fat capelin (DC3 and DC2). This as discussed in Sect. 3.3 could be explained by differences in phospholipids proportion (Shah et al. 2009).

Lipid hydrolysis

Proportion of phospholipids (PL) expressed as g phosphatidyl-choline equivalent/100 g lipid was significantly ($p < 0.05$) lower with increased lipid content of dried capelin being 25, 15 and 8% of total lipid in DC3, DC2 and DC1, respectively (Fig. 2a–c). The main lipid components in fish are triacylglycerols and PL. Diet of the fish affects the proportion of PL by either increasing or decreasing triacylglycerols (Standal et al. 2010; Burri et al. 2012), leading to increase or decrease in total lipids (Mørkøre et al. 2007). Phospholipids are membrane lipids with relatively constant absolute content, explaining why the proportion of PL is higher in less fatty than in fatty capelin.

PL hydrolysis in dried capelin was rapid in all batches during early storage and most rapid in DC3 with the highest proportion of PL (Fig. 2a–c). The relatively higher PL content at the beginning of storage declining as storage progressed may explain the reduced rate of hydrolysis with time. Rapid PL hydrolysis in DC3 may be attributed to the occurrence of substrate specific enzymes such as phospholipases that catalyze cleavage of hydrophilic phosphodiester bonds but not of lipophilic triacylglycerols. Also, a higher proportion of the highly susceptible PUFAs especially n-3 PUFAs in PL than triacylglycerols (Parmentier et al. 2007) can explain the difference in rate of hydrolysis between batches of dried capelin, why rapid hydrolytic activities occurred with high PL content (DC3 > DC2 > DC1). Packaging methods (open, sealed and vacuum) did not have significant effects on PL hydrolysis (Fig. 2a–c), supporting the explanation that PL hydrolytic processes

were largely driven by enzymatic activities (Chaijan et al. 2006; Shah et al. 2009).

Free fatty acids (FFA) content was the highest in dried capelin with the lowest fat content and the highest proportion of PL (Fig. 2d–f). FFA increased in all groups and most rapidly during early storage and with significantly ($p < 0.05$) higher values for DC3 than the other groups. FFA content increased while PL content declined (significant correlation $r = -0.54$) indicating that FFA evolution was mainly due to PL degradation. Hydrolysis of glycerol-fatty acid esters occurs in fish muscle lipid with the liberation of free fatty acids (Chaijan et al. 2006; Lopez-Amaya and Marangoni 2000). The development trends of FFA in different groups can be explained by the corresponding PL hydrolysis. Packaging methods did not affect the FFA formation which is in agreement with the PL hydrolytic results (Fig. 2d–f). Generally, the results indicate that lipid hydrolysis was mainly influenced by PL content with DC3 demonstrating a greater hydrolysis and suggesting that DC1 was more stable than less fatty capelin (i.e. DC2 and DC3 group).

Lipid oxidation

Lipid content and packaging methods significantly ($p < 0.05$) affected the hydroperoxides formation, where PV developed fastest in fattier capelin and open bags (Fig. 3a–c). The difference between groups and packaging method depends on fat content and access to oxygen necessary for lipid oxidation. Peroxide value increased during storage in open and sealed bags except towards the end of storage when it decreased in the fattier DC1 and DC2 open bags (Fig. 3a, b). The increase was due to the formation of hydroperoxides which are primary lipid oxidation products. Hydroperoxides accumulate during the initial oxidation process but decrease later, as the rate of cleavage and reactions exceed their formation (Cyprian et al. 2015; Underland et al. 1999). Therefore, the progressive increase in PV during storage in open and sealed bags and a decline observed in DC1 and DC2 open bags towards the end of storage indicate the vulnerability of dried capelin to lipid

Fig. 2 Changes in phospholipid (PL) (a–c) and free fatty acid (d–f) in packed dried capelin during 5 months of storage at ambient temperature ($n = 4$). DC1 dried capelin of high lipid, DC2 dried capelin of moderate lipid, DC3 dried capelin of low lipid

Fig. 3 Lipid hydroperoxides formation (a–c) and thiobarbituric acid reactive substances formation (d–f) in packed dried capelin during 5 months of storage at ambient temperature ($n = 4$). DC1 dried capelin

of high lipid, DC2 dried capelin of moderate lipid, DC3 dried capelin of low lipid

oxidation, but more so in DC1 and DC2 open bags. Similar results were obtained with color changes (L^* and b^* values on CIELAB color scale) (data not shown), associated with lipid oxidation products (Cyprian et al. 2016). Peroxide value of vacuum packed capelin was stable during storage (Fig. 3c). It is known that vacuum packaging can delay lipid oxidation by limiting access to oxygen thereby preserving the quality of muscle foods (Etemadian et al. 2012).

Thiobarbituric acid-reactive substances (TBARS) were the highest in groups with higher lipid content, and showed a tendency of decreasing with storage time except in DC1 and DC2 open bags increasing again towards the end of

storage (Fig. 3d–f). The decrease in TBARS during storage may be explained by the low decomposition of hydroperoxides into secondary products of lipid oxidation (TBARS) as PV progressively increased. The results indicate a negative correlation between PV and TBARS ($r = -0.27$). The interactions of protein components (peptides and acid amines) with malonaldehyde to give tertiary lipid oxidation products might have contributed to decrease in TBARS (Underland et al. 1999). Increase in TBARS in DC1 and DC2 open bags towards the end of storage may be attributed to faster decomposition of existing hydroperoxides into TBARS since the corresponding PV were decreasing. Packaging methods had some impacts on TBARS

Table 2 Mean sensory scores (scale 0–100) of rancid odor (A) and stock-fish odor (B) of dried capelin (n = 2)

Group	Packaging (bags)	A	Storage time (months)						B	Storage time (months)					
			0	1	2	3	4	5		0	1	2	3	4	5
DC1	Open	***	4.5 ^a	8.4 ^b	10.9 ^b	15.3 ^c	14.8 ^c	16.5 ^c		45.7 ^a	44.6 ^a	47.5 ^a	48 ^a	46 ^a	45 ^a
	Sealed	***	4.5 ^a	6.6 ^a	7.5 ^a	7.4 ^a	10.3 ^b	13.6 ^c		45.7 ^a	45.2 ^a	47.4 ^a	41.9 ^a	40.5 ^a	42.7 ^a
	Vacuum	**	4.5 ^a	4.9 ^{ab}	4.6 ^a	6 ^c	5.7 ^{bc}	7.3 ^c	*	45.7 ^a	45.2 ^a	43.2 ^{ab}	36.7 ^b	38.1 ^{ab}	37.1 ^{ab}
DC2	Open	***	3.6 ^a	5.8 ^b	3.4 ^a	5.4 ^b	9.1 ^c	10.8 ^c		50 ^b	48.7 ^b	45.7 ^b	45.9 ^b	45.9 ^b	48.1 ^b
	Sealed	**	3.6 ^a	4.7 ^a	4.5 ^a	4.6 ^a	7.2 ^b	7.3 ^b	*	50 ^a	48.9 ^a	47.7 ^{ab}	49.7 ^a	42.3 ^b	47.5 ^{ab}
	Vacuum	**	3.6 ^a	3.8 ^a	2.8 ^a	3 ^a	3.6 ^a	6.5 ^b	**	50 ^a	42.2 ^b	51.3 ^a	48.2 ^{ac}	44.9 ^{bc}	38.8 ^b
DC3	Open	**	3.2 ^a	4.1 ^a	6.2 ^b	5.5 ^b	6.8 ^b	6.3 ^b	**	57.8 ^a	46.6 ^{ab}	45.6 ^{ab}	49.7 ^a	45.8 ^b	46.6 ^{ab}
	Sealed		3.2 ^a	4 ^a	5.1 ^a	4.4 ^a	4.4 ^a	4.3 ^a		57.8 ^a	49.1 ^a	48.2 ^a	46.6 ^a	48.6 ^a	46.9 ^a
	Vacuum	**	3.2 ^a	3.2 ^b	3.5 ^b	3.3 ^b	2.8 ^b	5.9 ^c	*	57.8 ^a	53.3 ^{ab}	49.5 ^b	51.1 ^{ab}	52.6 ^{ab}	51.6 ^{ab}

DC1 dried capelin of high lipid, DC2 dried capelin of moderate lipid, DC3 dried capelin of low lipid

* $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$ (significant difference in a sensory attribute with storage time, different letters (superscript^{a–c}) within a row under the same sensory attribute indicate significant different values)

decomposition with vacuum packed capelin being more stable than open and sealed bags. The results are in conformity with PV data in explaining the importance of limiting access to oxygen to minimize lipid oxidation.

At the beginning of storage fluorescence shift ratio (OFR) were not significantly ($p > 0.05$) different between the batches, with OFR values of 1.05, 1.29 and 1.16 in DC1, DC2 and DC3, respectively. Increase in OFR values during storage were dependent on the lipid content and packaging methods (data not shown). The increase was higher in fattier dried capelin (DC1 and DC2) in both open (9.9 and 5.0) and sealed (5.3 and 3.6) bags until towards the end of storage when the fattiest fish (DC1) scored much higher (9.9) than in the other groups. Increases in OFR values occurred in parallel to decreases in TBARS [even though no significant ($p > 0.05$) inverse correlation $r = -0.10$ was obtained]. OFR, tertiary products of lipid oxidation are derived from complex interactions of protein components with secondary lipid oxidation products such as malonaldehyde (Underland et al. 1999). Contrarily, when OFR in open bags DC1 increased towards the end of storage, the corresponding TBARS content increased whereas PV decreased. This may be due to interactions of hydroperoxides with protein components that has also been reported to form fluorescence compounds (Nguyen et al. 2012). Packaging methods had a considerable influence on OFR development with samples in open bags obtaining higher values than sealed and vacuum packed.

Sensory evaluation with generic descriptive analysis (GDA)

The generic descriptive analysis (GDA) attributes used in this study to describe dried fish degradation were rancid odor and stock-fish odor (a characteristic of dried fish). The

rancid odor increased whereas stock-fish odor decreased during storage (Table 2A, B). Both lipid content and packaging methods affected rancid odor (Table 2A). A stronger rancid odor ($p < 0.05$) was obtained in high fat DC3 as well as open packed samples. These results are in agreement with lipid oxidation indicators discussed earlier, showing positive correlations ($r = 0.67, 0.05, 0.13$ with PV, TBARS & OFR, respectively). Rancid odor was below 20 on the scale of 0–100, a limit that has been used to indicate the samples are becoming rancid (Magnússon et al. 2006). The stock-fish odor was slightly reduced as storage time progressed, with higher reduction in fattier and open packed capelin (Table 2B). The changes were however, not significantly different ($p > 0.05$) by groups during storage. This can be explained by slow accumulation of TBARS with storage since TBARS have a profound impact on food sensory properties (Stapelfeldt et al. 1997). Stock-fish odor was inversely correlated to PV and TBARS ($r = -0.22$ and -0.21 , respectively).

Microbial quality

At the beginning of storage, total plate count (TPC) was not significantly ($p > 0.05$) different between the groups with values of log 5.48, 5.24, 5.38 colony-forming units in DC1, DC2 and DC3, respectively. Slow microbial growth was obtained during storage of dried capelin with TPC increments of less than log 1 colony-forming units by the end of storage. This may be explained by the low moisture content (<25%) and water activity (<0.80) in dried capelin batches, slowing down the growth of spoilage bacteria (Kilic 2009). Lipid content appeared to have no effect on TPC development. The lowest TPC was found in vacuum packed capelin a fact attributed to the air elimination retarding aerobic microorganisms' growth. Yeast and

molds were generally not detected in the dried capelin except after extended storage in DC1 and DC2 open bags. This may in addition to the low moisture content be explained by the drying conditions and packaging methods used. Indoor drying, sealing and vacuum packaging minimized contamination by microbes mainly air borne yeast and molds (Park et al. 2014). The results indicate that dried capelin was microbiologically stable during storage.

Multivariate data analysis

Principal component analysis (PCA) was carried out to gain an overview of the similarities and differences between the variables (Fig. 4). The variables not analyzed at all sampling points (microbial and fatty acid composition; analyzed at the beginning, middle and the end of storage) were not included in the model. The samples varied mainly in phospholipid, total lipid, water content and rancidity along the first principal component (PC-1), explaining 49% of the variations in the samples, mainly due to the difference in sample groups (i.e. lipid content). Low lipid (DC3) samples were located to the left of the scores plot along PC-1 described by phospholipid and stock-fish odor variables whereas high lipid (DC1) samples were located on the right side described mainly by the total lipid, water content and rancid odor. The variables describing DC3 were negatively correlated to those that described DC1 capelin. DC2 capelin that had moderate lipid content were centrally located implying they were marginally described by the variables that described both DC3 and DC1.

Samples also varied with regard FFA, PV and TBARS along the second principal component (PC-2) accounting for 26% of the total variation, mainly due to the difference in packaging methods storage time. As seen in Fig. 4, samples are located on the lower side of PC-2 during early

storage described mainly by TBARS but also total lipid and on the upper side later during storage described mainly by FFA, PV, and fluoresce. TBARS were negatively correlated with the variables that described samples during later storage. The influence of packaging methods was not clearly portrayed in the model.

Conclusion

The study proved that a stable and safe product at room temperature with moisture content less than 25% and water activity of less than 0.80 could be produced by the raw material and drying method used. But the capelin used must be chosen carefully due to its seasonal variation in lipid content. The influence of lipid content on hydrolysis and oxidation as well as sensory properties during storage at room temperature could be significantly demonstrated even though all the batches used in the study were of medium lipid content (7.5–10%). Lipid hydrolysis analyzed by changes in the proportion of PL and FFA was higher in less fatty capelin, explained by the group's higher proportion of PUFAs notably the long chain fatty acids EPA and DHA.

The lipid content and packaging methods significantly affected the lipid oxidation progress during storage as higher PV and TBARS contents were observed in higher lipid content capelin. Vacuum packaging significantly retarded the lipid oxidation with the lowest PV values were obtained in all vacuum packaged samples throughout the storage period. Primary hydroperoxides (PV) increased first and then decreased during later storage. Secondary oxidation products (TBARS) were higher with increased lipid content and tended to decrease during storage except in higher lipid groups with access to oxygen towards the end of the 5 months at 22 °C. OFR

Fig. 4 Principal component analysis (a) scores and (b) correlation loadings describing packed dried capelin based on variables evaluated at all sampling points. PC1 49% versus PC2 26%. DC1 dried capelin

of high lipid, DC2 dried capelin of moderate lipid, DC3 dried capelin of low lipid, Op open bags, Ap sealed bags, Vp vacuum bags, M month of storage

values measuring tertiary oxidation products were highest at the end of storage and highest in the fattiest product in open bags. These conclusions were confirmed by the principal component analysis where the variation in the data (49%) was explained by low lipid with high phospholipid content, and stockfish odor, and highest lipid capelin with total lipids, water content and rancid odor. Oxidation products explained 26% of the variation with early storage, TBARS and total lipids on one side and PV, FFA, OFR and long term storage on the other side. The dried high lipid capelin was more stable during storage if oxygen in the atmosphere was excluded by vacuum packaging with less PV, odor and amount of microbes. The spoilage of lipids was both due to oxidation (PUFAs and oxygen) and hydrolysis. It is recommended to test consumer acceptability of industrially dried and vacuum packed high lipid capelin for markets accustomed to dried small fish.

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