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Oxidative stability of lipid components of mullet (*Mugil cephalus*) roe and its product "bottarga"

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ABSTRACT

The lipid composition and oxidative stability of mullet (*Mugil cephalus*) raw roes and cured products (bottarga) from two different fishing areas have been studied and compared to find lipid modifications due to manufacturing procedures. Moreover, n-3 PUFA oxidation in raw roes and cured products was monitored during storage at −20 °C, and in grated sample of bottarga at room temperature. Oxidative degradation of n-3 PUFA was evaluated by conjugated dienes fatty acids hydroperoxides determination. The treatments on mullet roe did not affect the lipid level (fatty alcohols and acids, cholesterol) and the amount of hydroperoxides. No significant difference is observed between n-3 PUFA levels during processing and at different storage conditions. The order of oxidative stability, as calculated by the hydroperoxides determination was: grated bottarga < whole bottarga < raw roe.

1. Introduction

The potential health benefits related to fish and fish-derived products consumption are due to the presence of proteins, minerals, vitamins, and n-3 polyunsaturated fatty acids (n-3 PUFA) (Alamed, McClements, & Decker, 2006; Cahu, Salen, & de Lorgeril, 2004; Sidhu, 2003). Many studies have suggested that long chain n-3 PUFA, namely eicosapentaenoic acid (EPA), and docosahexaenoic acid (DHA), have an important role in human health, in the treatment of various diseases and disorders (Boran, Karaçam, & Boran, 2006; Schram et al., 2007; Sidhu, 2003; Simopoulos, 1991). Although, health benefits, n-3 PUFA are subject to rapid and/or extensive oxidation by exposure to air, light or heat during processing (Alamed et al., 2006), resulting in potential alteration in nutritional composition and quality of food (Boran et al., 2006; Drusch, Serfert, Scampicchio, Schmidt-Hansberg, & Schwarz, 2007; Moriya et al., 2007). Prolonged storage even at freezing temperatures of fish and fish oil can lead to oxidation of n-3 PUFA, which can have detrimental effects on product quality (Baron, Kjærsgård, Jessen, & Jacobsen, 2007; Boran et al., 2006). Several PUFA oxidative degradation products have been measured in fish products, like lipid hydroperoxides and volatile secondary lipid oxidation products (Baron et al., 2007; Boran et al., 2006; Drusch et al., 2007). Fish roe products contain significant amount of lipids having high levels of long chain n-3 PUFA (30–50% of total fatty acids), mainly EPA and DHA (Bledsoe, Bledsoe, & Rasco, 2003; Moriya et al., 2007; Shirai, Higuchi, & Suzuki, 2006). Amongst them, the salted and semidried mullet (*Mugil* genus) ovary product, known as "bottarga" in Italian and "karasumi" in Japanese, is produced in several countries in the world and in Sardinia (Italy) (Bledsoe et al., 2003; Scano et al., 2008). Sardinian bottarga is the final product of a number of treatments, like salting and drying in particular conditions of humidity and temperature, on the whole roe of mullets (*Mugil cephalus*) by traditional and industrial procedures; the final product can be sold as whole ovaries under vacuum packaging, or grated in jar (Murgia, Tola, Archer, Vallerga, & Hirano, 2002; Scano et al., 2008). In a recent work (Scano et al., 2008) we have studied the major lipid classes and the fatty acids and alcohols composition of commercial products of grated bottarga manufactured in Sardinia. Amongst the major lipid classes we found wax esters (50 mol%), triacylglycerols, phospholipids, and cholesterol; EPA and DHA amounted to c.a. 22% of total fatty acids. Furthermore, the samples showed a relative high content of free fatty acids (FFA) and a conjugated dienes fatty acids hydroperoxides (HP) level of 0.1 mmol/g edible portion (0.4 nmol/mg of lipid), probably due to hydrolysis and oxidation processes induced on the original lipid matrix by the manufacturing procedures and storage conditions (Scano et al., 2008). In view of the importance to evaluate the fish products oxidative stability for implications in food quality and human health, the objective of this paper was to investigate the susceptibility of mullet roe lipids to oxidative degradation induced by the procedures (salting, drying, and grating) adopted to obtained bottarga. To this goal, the lipid composition and the oxidative status of raw mullet roes and cured products from two different fishing areas were examined and the results

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compared. Furthermore, we studied the oxidative stability of unprocessed roe and bottarga lipids at different storage conditions. Lipid oxidation was evaluated by the determination of the HP level, the stable primary products of the oxidative processes that have been found to be detrimental to health (Thanh et al., 2007). The modifications of lipid components and the HP level in samples of raw roes and cured products (as whole or grated bottarga) were monitored during storage at −20 °C (60 or 90 days), and in a commercial grated bottarga sample during storage at room temperature (32 days). Taking into account that the initial step of the oxidative processes is the hydrolysis (either non-enzymatic or catalysed by enzymes) of the ester bonds of lipid molecules followed by the release of FFA (Falch, Storseth, & Aursand, 2007), the extent of this process, due to processing and storage, was also monitored in mullet roes by ^{13}C NMR spectroscopy.

2. Materials and methods

2.1. Materials

All solvents used, of the highest available purity, were purchased from Merck (Darmstadt, Germany). Cholesterol, triolein, trilinolein, oleyl oleate, cholesteryl palmitate, fatty acids, fatty alcohols, and fatty acids methyl esters standards, desferal (deferoxamine mesylate salt), and deuterated chloroform ($CDCl_3$) were purchased from Sigma–Aldrich (Milan, Italy). The reagents 20% BF_3 in MeOH and 1,1,1,3,3,3-hexamethyldisilazane (HMDS) – trimethylchlorosilane (TMCS) – pyridine (3:1:9) were purchased from Supelco (Bellefonte, USA). *cis*,*trans*-13-Hydroperoxyoctadecadienoic acid (*c*,*t*-13-HPODE) and *cis*,*trans*-9-hydroperoxyoctadecadienoic acid (*c*,*t*-9-HPODE) were purchased from Cascade (Cascade Biochem. Ltd., London). All other chemicals used in this study were of analytical grade.

2.2. Raw roes and bottarga samples

Raw roes and bottarga samples of mullets were kindly supplied by the company "Stefano Rocca s.r.l." located in Sardinia (Italy). Mullets were caught in two different fishing areas: FAO area 34 and FAO area 41 for roes 1 and 2, respectively. Ovaries were divided sagittally in two parts, one was salted, dried, and sealed (as a whole) under vacuum, with the procedures adopted in the firm to obtain bottarga; no additive was added. Both parts were immediately subjected to lipid extraction and stored at −20 °C. Also commercial grated mullet bottarga was manufactured by the same company; ingredients were reported in the label as mullet roe and salt.

2.3. Storage conditions

Samples of mullet raw roes and cured products (as whole roe) were stored at −20 °C for 90 and 60 days, respectively. A commercial sample of grated bottarga was stored for 90 days at −20 °C or 32 days at room temperature in an open jar exposed to air and light.

2.4. Lipid extraction and preparation of cholesterol, fatty alcohols and fatty acids

Total lipids were extracted from the mullet raw roes and cured products using the method described by Folch, Lees, and Sloane-Stanley (1957), by addition of 12 ml of $CHCl_3$/MeOH (2/1, v/v) solution, and quantified by the method of Chiang, Gessert, and Lowry (1957). An aliquot of total lipid extract recovered from the $CHCl_3$ phase was analysed by ^{13}C NMR to assess the free fatty acids (FFA) level. Separation of cholesterol, fatty alcohols and fatty acids was obtained by mild saponification (Rosa et al., 2005) as follows: Aliquot (2 ml) of the $CHCl_3$ fraction, containing the lipids, from each sample was dried and dissolved in 5 ml of EtOH, then 100 μl of Desferal solution (25 mg/ml of H_2O), 1 ml of a water solution of ascorbic acid (25% w/v), and 0.5 ml of 10 N KOH were added. The mixtures were left in the dark at room temperature for 14 h. After addition of 10 ml of *n*-hexane and 7 ml of H_2O, samples were centrifuged for 1 h at 900*g*. The hexane phase containing the unsaponifiable fraction (cholesterol and fatty alcohols) was collected and the solvent was evaporated. A portion of the dried residue was dissolved in 1 ml of MeOH and injected into the HPLC system. Aliquot of dried fatty alcohols was converted to trimethylsilyl ether by a mixture of TMCS, HMDS, and anhydrated pyridine (1:3:9, v/v/v) (200 μl) for 2 h at room temperature, before being applied to capillary gas chromatography. After further addition of 10 ml of *n*-hexane to the mixtures, samples were acidified with 37% HCl to pH 3–4 and then centrifuged for 1 h at 900*g*. The hexane phase with fatty acids was collected and solvent evaporated. A portion of the dried residue was dissolved in 1 ml of CH_3CN with 0.14% (v/v) CH_3COOH and aliquots of the samples were injected into the HPLC system. Aliquot of dried fatty acids was methylated with 1 ml of 20% BF_3 in MeOH (Christie, 1993) for 30 min at room temperature. After addition of 4 ml of *n*-hexane and 2 ml of H_2O, samples were centrifuged for 20 min at 900*g*. The hexane phase with fatty acids methyl esters was collected, the solvent was evaporated, the residue was dissolved in 250 μl of *n*-hexane, and aliquots of the samples were injected into the GC system. The recovery of fatty acids, fatty alcohols, and cholesterol during the saponification process was calculated by using an external standard mixture prepared by dissolving 1 mg of triolein, trilinolein, oleyl oleate, cholesterol, and cholesteryl palmitate in 5 ml of EtOH and processed as samples. All solvent evaporation was performed under vacuum.

2.5. HPLC analyses

Analyses of cholesterol and unsaturated fatty acids were carried out with an Agilent Technologies 1100 liquid chromatograph (Agilent Technologies, Palo Alto, USA) equipped with a diode array detector. Cholesterol, detected at 203 nm, was measured with use of a Chrompack column (Chrompack, Middelburg, The Netherlands), Inertsil 5 ODS-3, 150 × 3 mm, and MeOH as mobile phase, at a flow rate of 0.4 ml/min. Analyses of unsaturated fatty acids and conjugated dienes fatty acids hydroperoxides (HP), detected at 200 and 234 nm, respectively, were carried out with a Chrompack column, Inertsil 5 ODS-2, 150 × 4.6 mm with a mobile phase of $CH_3CN/H_2O/CH_3COOH$ (70/30/0.12, v/v/v) at a flow rate of 1.5 ml/min. The identification of cholesterol, fatty acids, and HP was made by using standard compounds and the second derivative as well as conventional UV spectra, generated with the Agilent Chemstation A.10.02 software (Rosa et al., 2005).

2.6. GC analyses

Fatty acids methyl esters were measured on a gas chromatograph Hewlett–Packard HP-6890 (Hewlett–Packard, Palo Alto, USA) with a flame ionisation detector and equipped with a cyanopropyl methylpolysiloxane HP-23 FAME column (30 m × 0.32 mm × 0.25 μm) (Hewlett–Packard). Nitrogen was used as carrier gas at a flow rate of 2 ml/min. The oven temperature was set at 175 °C, injector temperature 250 °C, and detector temperature 300 °C. The fatty acids methyl esters were identified by comparing the retention times with those of standard compounds. The percentage of composition of individual fatty acids was calculated

by using a calibration curve with components injected at different concentrations, using the Hewlett–Packard A.05.02 software.

2.7. NMR analyses

The extracted lipids (c.a. 100 mg) were dissolved in 0.6 ml of $CDCl_3$ and placed in 5 mm NMR tubes. The NMR analyses were performed at 25 °C on a Varian VXR-300 operating at the frequency of 75.42 MHz. NOE-suppressed, proton-decoupled ^{13}C NMR spectra were recorded with a 2 s acquisition time, a sweep width of 26 kHz, a 45° pulse angle and a 20 s relaxation delay; 10,240 scans were collected. Zero filling and a line broadening of 0.3 Hz were applied prior to Fourier transformation to minimise the noise, but not at expense of resolution.

2.8. Statistical analyses

Graph Pad INSTAT software (GraphPad software, San Diego, CA, USA) was used to calculate the means and standard deviations of two independent experiments involving triplicate analyses for each sample ($n = 6$). Evaluation of statistical significance of differences was performed using One-way ANOVA.

3. Results

Total lipids were extracted from mullet raw roes from two different fishing areas (149.4 ± 7.9 mg/g of the edible portion for roe 1 and 200.1 ± 5.2 for roe 2) and the fatty alcohols and fatty acids compositions, expressed as percentage of the total amount, are reported in Tables 1 and 2, respectively. There was no considerable variation between fatty alcohols composition of the two analysed samples, showing 70% and 73% of saturated fatty alcohols (mainly 16:0) for samples 1 and 2, respectively. Differences were observed for the fatty acids composition, with raw roe 1 showing a prevalence of polyunsaturated compared to the monounsaturated, whilst an opposite trend was observed for raw roe 2. The lipid composition of raw mullet roes was compared to that of their cured products in order to evaluate the biochemical modifications due to salting and drying procedures. An average lipid content of 229.9 ± 13.5 and 325.2 ± 21.8 mg/g edible portion was measured for bottarga 1 and bottarga 2, respectively. Fig. 1 shows the values of the total saturated (SFa) and monounsaturated (MUFa) fatty alcohols (%), cholesterol (μg/mg lipid) (Fig. 1A), the total saturated (SFA), monounsaturated (MUFA) and polyunsaturated fatty acids (PUFA) (%) (Fig. 1B) measured in mullet raw roes and cured samples (bottarga 1 and 2). The raw materials and the corresponding cured samples exhibited the same levels of lipid components. As index of the n-3 PUFA peroxidation process, the levels of EPA, DHA, and conjugated dienes fatty acids hydroperoxides (HP) were analysed. The values of EPA and DHA (expressed as μg/mg lipid), and HP (nmoles/mg lipid) measured in raw roes and cured samples are shown in Fig. 2. Both bottarga samples exhibited similar amount of oxidative products than starting materials, although raw roe 1 showed a superior level of HP than roe 2, correlated to a major level of PUFA.

The FFA levels, as an estimation of hydrolytic process due to the manufacturing procedures, were assessed in raw roes and the corresponding processes products by ^{13}C NMR analysis. Fig. 3 shows the carbonyl region (178–171 ppm) of the ^{13}C NMR spectra of the lipid fractions extracted from raw roe 2 (Fig. 3A) and its cured product bottarga 2 (Fig. 3B). Fig. 3A shows only one cluster of res-

Table 1
Fatty alcohols composition (%) of roe samples by GC.

Fatty alcohol	Roe 1	Roe 2
14:0	14.4 ± 0.9	7.3 ± 0.2
15:0	1.1 ± 0.1	3.4 ± 0.1
16:0	49.2 ± 2.7	55.9 ± 0.9
18:0	5.8 ± 0.5	7.0 ± 0.3
16:1 $n-7$	11.6 ± 0.58	7.5 ± 0.1
18:1 $n-7$	4.4 ± 0.3	2.2 ± 0.1
18:1 $n-9$	2.1 ± 0.1	3.8 ± 0.2
SFa	70.5 ± 3.8	73.5 ± 10.2
MUFa	18.1 ± 1.0	13.3 ± 0.6

SFa, saturated fatty alcohols; MUFa, monounsaturated fatty alcohols.
Mean and standard deviation of six samples.

Table 2
Fatty acids composition (%) of roe samples by GC.

Fatty acid	Roe 1	Roe 2
12:0	0.03 ± 0.01	0.02 ± 0.00
14:0	1.81 ± 0.03	1.61 ± 0.02
15:0	0.15 ± 0.01	0.55 ± 0.01
16:0	10.30 ± 0.29	11.96 ± 0.24
18:0	3.52 ± 0.28	3.21 ± 0.28
16:1 $n-7$	14.70 ± 0.18	20.59 ± 0.12
18:1 $n-7$	7.45 ± 0.07	6.72 ± 0.12
18:1 $n-9$	7.71 ± 0.06	17.94 ± 0.12
16:2	1.74 ± 0.05	1.38 ± 0.04
16:3	1.06 ± 0.09	1.68 ± 0.02
16:4	0.81 ± 0.15	0.32 ± 0.20
18:2 $n-6$	1.33 ± 0.10	0.94 ± 0.06
18:3 $n-3$	0.47 ± 0.02	0.43 ± 0.03
18:3 $n-6$	0.42 ± 0.04	0.34 ± 0.02
18:4 $n-3$	2.29 ± 0.06	0.25 ± 0.02
20:3 $n-3$	0.04 ± 0.01	0.06 ± 0.01
20:3 $n-6$	0.22 ± 0.03	0.40 ± 0.03
20:4 $n-6$	0.76 ± 0.04	3.37 ± 0.11
20:5 $n-3$	12.75 ± 0.15	4.33 ± 0.02
22:4 $n-6$	0.26 ± 0.05	0.59 ± 0.05
22:5 $n-3$	8.06 ± 0.05	4.31 ± 0.10
22:6 $n-3$	12.93 ± 0.18	8.29 ± 0.14
SFA	15.80 ± 0.48	17.35 ± 0.49
MUFA	29.87 ± 0.20	45.25 ± 0.14
PUFA	43.11 ± 0.33	26.68 ± 0.33

SFA, saturated fatty acids; MUFA, monounsaturated fatty acids; PUFA, polyunsaturated fatty acids.
Mean and standard deviation of six samples.

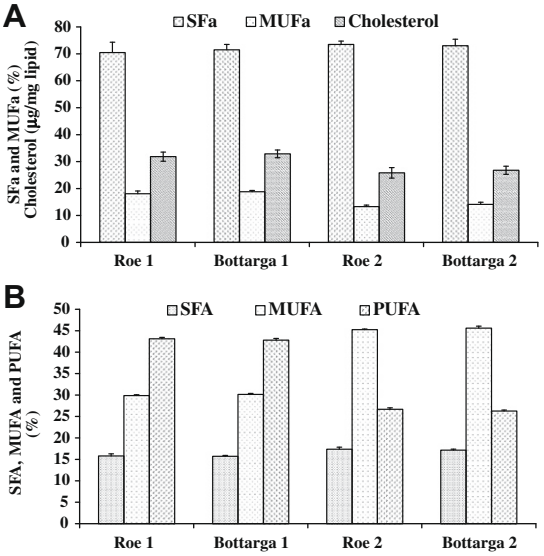

fig. 1. Values of the total saturated (SFa) and monounsaturated (MUFa) fatty alcohols, expressed as percentage of total fatty alcohols (%), cholesterol (μg/mg lipid) (A), the total saturated (SFA), monounsaturated (MUFA), and polyunsaturated (PUFA) fatty acids (% total fatty acids) (B), measured in mullet raw roes and cured samples; ($n = 6$).

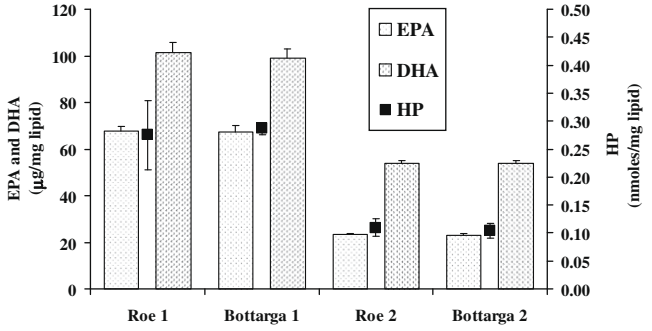

fig. 2. Values of the fatty acids 20:5 $n-3$ (EPA) and 22:6 $n-3$ (DHA) (expressed as μg/mg lipid), and conjugated dienes fatty acids hydroperoxides (HP) (nmoles/mg lipid) measured in raw roes and cured samples; ($n = 6$).

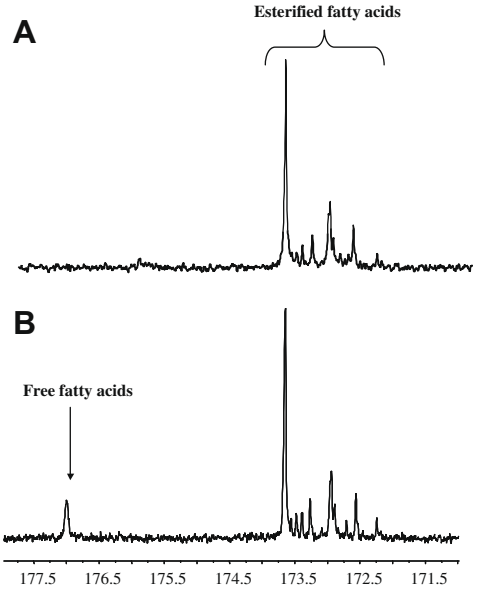

fig. 3. The carbonyl spectral region (178–171 ppm) of the ^{13}C NMR spectra of raw roe (A), and bottarga (B).

onances, arising from the –COO functional groups of the esterified fatty acids, whilst after manufacturing (Fig. 3B) a new peak at c.a. 177 ppm appears, assigned to –COO functional groups of FFA. From an integration procedure of the peaks in this spectral region, it was calculated that, after processing, c.a. 9 mol% of fatty acids were present in their free form. An initial value of 5 mol% of FFA was found for raw roe 1 (spectra not shown), and this value increased up to 15 mol% in the cured product. Moreover, the PUFA oxidative modification in samples of raw roes and cured products (as whole roe) was monitored during storage at −20 °C. Fig. 4 shows the values of total PUFA (mg/mg lipid) and HP (nmoles/mg lipid) measured in control samples and after storage at −20 °C for 30–90 days for raw samples (Fig. 4A) and 60 days for cured products (Fig. 4B). Storage did not affect the PUFA level, nevertheless an increase of the HP level for cured products was observed after 60 days, superior to that observed for raw roes after 90 days. Furthermore, only a slight increase of the FFA amount in raw roes and bottarga samples was observed during storage at −20 °C by ^{13}C NMR analysis. The modification of lipid components and the oxidative process of n-3 PUFA were also monitored in a grated commercial sample of bottarga at 30–90 days at −20 °C and 8–32 days at room temperature. The levels of total fatty alcohols, fatty acids, and cholesterol were not affected by storage for 90 days at −20 °C (data not shown) nor for 32 days at room temperature

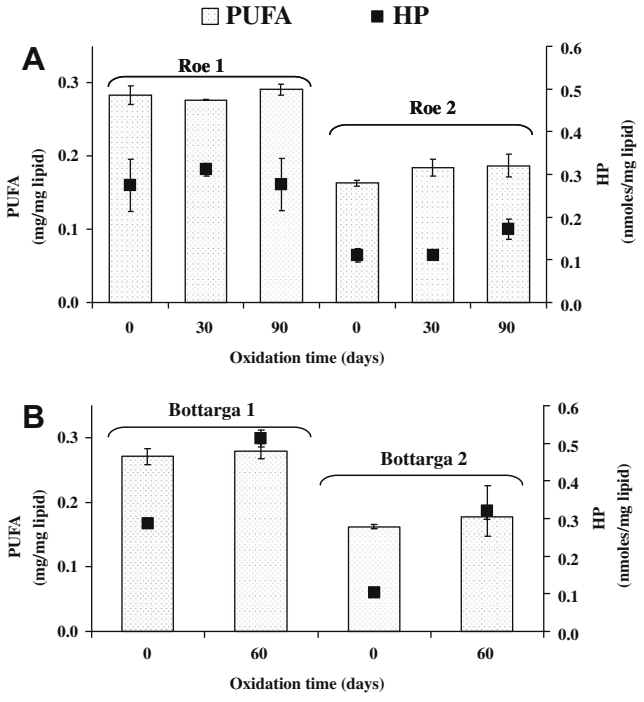

fig. 4. Values of the total polyunsaturated fatty acids (PUFA) (expressed as mg/mg lipid) and conjugated dienes fatty acids hydroperoxides (HP) (nmoles/mg lipid) measured in control raw roes and after 30 and 90 days of storage at −20 °C (A), in control cured samples (0) and after 60 days of storage at −20 °C (B); ($n = 6$).

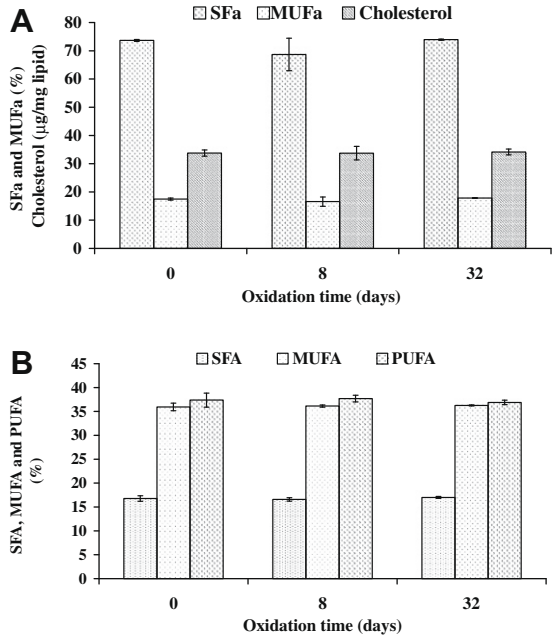

fig. 5. Values of the total saturated (SFa) and monounsaturated (MUFa) fatty alcohols, expressed as percentage of total fatty alcohols (%), cholesterol (μg/mg lipids) (A), total saturated (SFA), monounsaturated (MUFA), and polyunsaturated (PUFA) fatty acids (% total fatty acids) (B), measured in control grated bottarga samples (0) and after 8 and 32 days of storage at room temperature; ($n = 6$).

(Fig. 5). In Fig. 6 the values of EPA, DHA, and HP (as % control), measured in grated bottarga samples at 0 (control), 30, and 90 days of storage at −20 °C (Fig. 6A) or 8 and 32 days at room temperature (Fig. 6B) are shown. Stored samples did not exhibit a significant variation of n-3 PUFA level, nevertheless a significant HP increase, about six fold of control value (0.27 ± 0.03 nmol/mg lipid) was

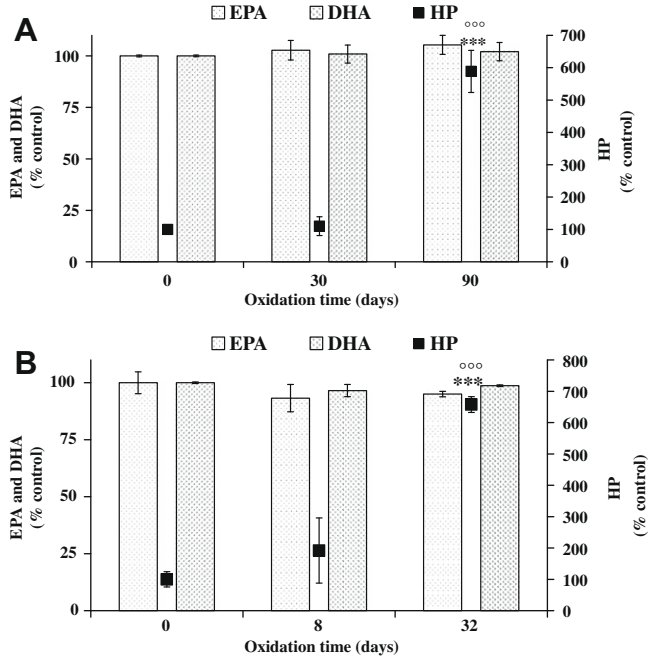

fig. 6. Values of the fatty acids 20:5 $n-3$ (EPA), 22:6 $n-3$ (DHA), and conjugated dienes fatty acids hydroperoxides (HP) (expressed as % control), measured in grated bottarga samples of control (0) and after 30 and 90 days of storage at −20 °C (A), and after 8 and 32 days of storage at room temperature (B). $^{***} = p < 0.001$ versus control; $^{\circ\circ\circ} = p < 0.001$ versus previous time; ($n = 6$).

reached after 90 and 32 days at −20 °C and room temperature, respectively.

4. Discussion

The consumption of fish or fish oil containing n-3 PUFA has been found to be clinically beneficial to human health. The health benefits of dietary n-3 fatty acids include reduced susceptibility to mental illness, protection against heart disease, improved brain and eye function in infants, and alleviation of rheumatoid arthritis symptoms (Boran et al., 2006; Cahu et al., 2004; Sidhu, 2003; Simopoulos, 1991). In spite of nutritional benefits, *n*-3 PUFA are extremely sensitive to lipid oxidation, resulting in potential alteration in nutritional composition and quality of food (Alamed et al., 2006; Baron et al., 2007; Boran et al., 2006; Drusch et al., 2007; Gorreta et al., 2002; Schram et al., 2007). Increase in n-3 PUFA oxidation has been previously reported in the development of rancidity in fish products during storage and several degradation products have been reported (Baron et al., 2007; Boran et al., 2006; Moriya et al., 2007), amongst them fatty acids hydroperoxides, important non-radical intermediates of PUFA peroxidation with many undesirable biological effects, such as cytotoxicity (Kaneko, Kaji, & Matsuo, 1994), membrane fluidity changes, and mitochondrial membrane damage (Thanh et al., 2007).

Also fish roe products are proposed as rich sources of n-3 PUFA, containing high amounts of EPA and DHA (Bernasconi et al., 2007; Bledsoe et al., 2003; Shirai et al., 2006). Mullet roe is regarded as a delicacy and Sardinian bottarga has an exceptional reputation in the market for its quality (Murgia et al., 2002; Scano et al., 2008). In a previous work we studied the lipid profile of commercial samples of grated bottarga; bottarga was found to be rich in wax esters (WE) and a relatively high concentration of FFA was also detected (Scano et al., 2008).

In this work we have studied the oxidative stability of lipids components, with particular regard to n-3 PUFA, of mullet roes from two different fishing areas during the procedures adopted to obtain their cured products, bottarga, and during different storage conditions of mullet products. The salting and drying procedures did not affect the levels of cholesterol, fatty alcohols and n-3 PUFA. The cured products showed similar oxidative status than raw materials, and HP level seemed to be correlated to the amount of PUFA in the starting samples. Mullet roe n-3 PUFA showed a high oxidative stability during processing. This might be due to the fact that a significant amount of n-3 PUFA in mullet roe are WE components (Bernasconi et al., 2007; Scano et al., 2008), and it was reported that WE enriched in n-3 fatty acids have a low degree of susceptibility to oxidation (Gorreta et al., 2002). The major biochemical change observed during processing was the increase of FFA in cured products, originated from hydrolytic processes on the lipid matrix. Bottarga samples (as whole roe) showed a minor resistance to oxidation during storage at −20 °C in respect to raw materials, as indicated by the HP level. Taking into consideration that FFA are the first target of the oxidative process, the major oxidation observed in cured product was probably due to its initial higher level of FFA during the manufacturing processes, since the hydrolytic processes are less marked during storage at freezing temperatures. The levels of EPA and DHA in grated bottarga sample were not affected by storage for 90 days at −20 °C nor for 32 days at room temperature, nevertheless a significant HP increase (six fold of control) was observed at these storage conditions. The grated bottarga showed a minor resistance to oxidation than the whole bottarga, probably because of its physical state, that results in a larger exposed surface, and its high level of FFA, as previously reported (Scano et al., 2008), that may facilitate the lipid peroxidative process. The hydroperoxides decomposition generates particularly offensive off-flavours even at low levels of oxidation (Kolanowski, Jaworska, & Weiβbrodt, 2007). Further research should include studies on flavour characterisation.

5. Conclusion

Our studies have shown that the PUFA oxidative stability in mullet roe products (in the order grated bottarga < whole bottarga < raw roe) is influenced by different parameters, such as manufacturing procedures, storage, and physical state of the matrix. The salting and drying procedures, usually adopted in Sardinia to obtain bottarga, and the different storage conditions did not induce a marked oxidative degradation of n-3 PUFA; a significant increase of HP level was observed only in grated bottarga during storage at room temperature (32 days) and at −20 °C for 90 days. Mullet roe may be therefore regarded as stable natural source of health beneficial n-3 PUFA.

Acknowledgements

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