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ORIGINAL ARTICLE

Chemical composition of European squid and effects of different frozen storage temperatures on oxidative stability and fatty acid composition

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Abstract The chemical composition of European squid (*Loligo vulgaris*) mantles and tentacles and the lipid oxidation during frozen storage at three different temperatures (−20º, −40º and −80 °C) were investigated. The moisture, fat, protein and ash contents of tentacles were 80.72%, 1.44%, 16.16% and 1.63% while the same contents for mantle were 78.54%, 1.37%, 18.52% and 1.45% respectively. The initial free fatty acidity (FFA), peroxide (PV) and thiobarbituric acid reactive substances (TBARS) values of tentacles were 1.17%, 1.80 meq O_2/kg fat and 0.80 mg malonaldehyde/kg respectively. The same results for mantles were 1.38%, 2.20 meq O_2/kg fat and 0.73 mg malonaldehyde/kg respectively. PV and TBARS values increased with the storage time for all samples and higher storage temperature resulted with higher PV and TBARS values. The initial fatty acid compositions of *L. vulgaris* mantles were 29.95% saturated (SFAs), 9.95% monounsaturated (MUFAs) and 59.31% polyunsaturated fatty acids (PUFAs) and tentacles were 34.16% SFAs, 10.69% MUFAs and 55.15 PUFAs. SFAs content were increased but MUFAs and PUFAs contents were decreased during frozen storage of mantles and tentacles.

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Introduction

Loligo. vulgaris is one of the most common squids along the northeastern Atlantic and the Mediterranean coasts. *L. vulgaris* (Lamarck 1798) have become of increasing importance as food and one of the most commonly consumed cephalopods around the world. Because the muscle contains little fat, little saturated fat, and is a good source of minerals. Squid consumption is limited in large parts of the world, especially, where this commodity is mainly commercialized as frozen. Until recently considerable amounts of squid are consumed in east and southeast Asia, and in the Mediterranean countries. Nowadays, in many countries that are not traditionally cephalopod consumers, the consumption is increasing mainly as chilled and frozen ready meals (Guerra and Rocha 1994; Jeyasekaran et al. 2010).

Among various conservation methods currently used, the most important are those based on the action of low temperatures which preserves taste and nutritional value with optimal quality. The purpose of frozen storage of seafood is to extend its shelf life and to limit microbial and enzymatic activity which causes deterioration. Quality and shelf life of frozen seafood depend on mainly storage temperature (Heen and Karsti 1965, Haard 1992).

The aim of this study is to research the effect of frozen storage on shelf life and quality characteristics of squid mantle and tentacle in different temperatures was investigated in highly commercial squid *L. vulgaris* (Lamarck 1798). Moreover, very little knowledge is available on the effect

of processing and storing on the shelf life of these seafood delicacies.

Materials and methods

Sample preparation

European squid (*L. vulgaris*), caught in the North-East Mediterranean in June 2006 and offloaded 24 h later, were taken from a dock in Güllük, Bodrum, Turkey. The edible portions of the squid were separated into mantle and tentacle portions. The mantle was cleaned, deskinned and eviscerated. For the tentacles, the eyes were removed but skin was left on. The tentacle portion is generally consumed with the skin attached. All squid samples were vacuum packed with polyethylene bags (0.02 g/m^2 day atm vapour permeability) and divided to three groups and stored in −20±0.1 °C, −40±0.1 °C and −80±0.1 °C. All of the analyses were carried out in mantles and tentacles separately. The proximate composition analyses were carried out in fresh samples and pH value, free fatty acidity, peroxide and thiobarbituric acid reactive substances contents and fatty acids distributions were carried out at 0, 4, 8, and 12th months.

Proximate and chemical analyses

Squid mantles and tentacles were determined for moisture, fat, protein and ash contents according to the AOAC Method (1999). pH was measured in a homogenate prepared by blending 10 g of muscle with 90 ml of distilled water for 30 s. Readings were taken with a Cole Parmer, Model 5996-50 pH meter. Squid lipids were extracted as described by Bligh and Dyer (1959). Free fatty acids were determined according to the alkaline titration method and calculated as mg KOH/g fat (AOAC 1999). Peroxide value was determined by a titrimetric method (AOAC 1999). Results were expressed as millimole of O_2 per kilogram of lipid. Thiobarbituric acid reactive substances (TBARS) were determined as described by Tarladgis et al. (1960). The fatty acid compositions were determined as fatty acid methyl esters (FAME) using a gas chromatography, Thermofinnigan TraceGC/Trace DSQ/A1300 gas chromatograph with a splitless injection, equipped with a mass spectrophotometer and a fused capillary column (SGE BPX5, 30 m, 0.32 mm inner diameter 0.25 μm film thickness). The working temperature of the injector, column and ms detector was 240 °C, 190 °C and 240 °C respectively. Helium was used as a carrier gas. Samples were injected into the column inlet using an automatic injector. Fatty acid methyl esters (FAMEs) were identified by comparison of their retention time and equivalent chain length with respect to standard FAMEs (47885-U, Supelco, Bellefonte, Penn., USA). Samples FAMEs were quantified according to their percentage area.

Statistical evaluation

All results were analyzed by analysis of variance (ANOVA) according to a two factorial design, using a split plot design with two trials. In this design, the factors were the storage temperature (−20, −40, −80±1 °C) and the storage time (months) for a specific parameter with repeated measurements in time. If required, Duncan multiple comparison test was performed to investigate which means significantly differed from each other. Minitab (Minitab, State College, PA) software (ver. 13.0 for Windows) was used for statistical analyses.

Results and discussion

The chemical composition of mantles and tentacles of *L. vulgaris* are shown in Table 1. The tentacles contained more moisture, more fat, less protein and more ash contents than the mantles. The results of proximate analyses proved that the composition of mantles and tentacles of *L. vulgaris* is significantly different. The tissue structure and the functions of mantles and tentacles are different therefore; the moisture, lipid, protein and ash contents of mantles and tentacles are in different statistical groups. The chemical compositions of cephalopods are dependent on species, growth stage, habitat, season and anatomical region of the cephalopod (Kreuzer 1984; Sinanoglou and Miniadis-Meimaroglou 1998; Özogul et al. 2008).

Table 2 shows the pH variations of mantles and tentacles during frozen storage. Statistically important increases seen in all storage temperatures of mantles and tentacles during storage however, the increases in lower temperatures were greater than higher temperatures. Yamanaka (1987) and Ohashie et al. (1991) pointed out the pH increase of squid in fresh storage and the increase in storage temperature also increase the pH levels of squid mantles.

Table 1 Chemical composition of mantles and tentacles of *L. vulgaris* (%)

	Moisture	Fat	Protein	Ash
Tentacle	80.7±0.25[a]	1.4±0.12[a]	16.1±0.62[a]	1.6±0.01[a]
Mantle	78.5±0.29[b]	1.4±0.09[b]	18.5±0.55[b]	1.5±0.01[b]

Values are given as mean±S.D. from triplicate determinations.

Different superscripts in the same column indicate significant differences ($P<0.05$).

The free fatty acidity values of −20 °C and −40 °C stored samples were increased as to form a peak and decreased during storage (Table 2). The peak formation was seen in 4th month in −20 °C and 8th month in −40 °C stored mantles and tentacles. The free fatty acidity values of −80 °C stored samples were increased in 12 months of storage. It is well known that free fatty acids are a result of enzymatic hydrolysis of esterified lipids. These results may be explained with non-enzymatic auto-hydrolysis. However, a relation between phospholipids hydrolysis during frozen storage is reported in lean fish (Han and Liston 1988). Apgar and Hultin (1982) reported that the microsomal lipid hydrolysis enzyme system is active at temperatures below freezing point. Olley and Lovern (1960) suggested that phospholipases may be activated by freezing and it would be possible that the free fatty acids formation activated by phospholipases.

Primary lipid oxidation was followed by the peroxide value. The peroxide values of all tested samples were increased during storage in all storage temperatures (Tables 3 and 4) ($P<0.05$). It was determined that the peroxide values of both mantles and tentacles were lower in lower temperatures ($P<0.05$). These results could be explained by auto-oxidation but a link between microsomal lipid peroxidation enzyme systems and enzymatic oxidation during frozen storage is reported in lean fish (Olley and Lovern 1960; Apgar and Hultin 1982; Han and Liston 1988) Peroxides are the initial lipid oxidation products which are subject to form advanced oxidation reactions. Lipid peroxides are very unstable and therefore fluctuations can be observed in peroxide value (Han and Liston 1988).

Thiobarbituric acid reactive substances analysis is a widely used indicator for the assessment of degree of secondary lipid oxidation. TBARS analysis quantifies the malondialdehyde (MA) that is released as an end product of lipid oxidation. TBARS values showed significant differences in different temperatures and storage times (Table 5) ($P<0.05$). At the beginning of the storage, TBARS values were determined as 0.73 mg MA kg^{-1} in mantle and 0,80 mg MA kg^{-1} in tentacles. The TBARS values were 6.20 and 4.33 and 3.21 mg MA kg^{-1} in −20, −40 and −80 °C stored samples while same values were 6.74, 4.74 and 3.50 mg MA kg^{-1} in tentacles respectively. TBARS values increased with the storage time for all samples ($P<0.05$). Higher storage temperature resulted with higher TBARS values ($P<0.05$). Nevertheless, since cephalopod mantle has a very small percentage of lipids in its mantle and tentacle compositions, lipid oxidation may affect sensory quality of the product. It is thought that since malonaldehyde could interact with other components of fish such as nucleosides, nucleic acid, proteins and other aldehydes, therefore TBARS values might not give actual rates of lipid oxidation (Auburg 1993). The results of FFA, peroxide4 and TBARS point that the development of lipid oxidation of mantles and tentacles of *L. vulgaris* during frozen storage at different temperatures seemed to depend on the storage temperature and storage time. The results suggest that the highest temperature was linked to the highest rate of lipid oxidation as well as storage time.

The fatty acid compositions of *L. vulgaris* mantles and tentacles during frozen storage are shown in Tables 6 and 7. The initial fatty acid compositions of *L. vulgaris* mantles were 29.95% saturated (SFAs), 9.95% monounsaturated (MUFAs) and 59.31% polyunsaturated fatty acids (PUFAs) and tentacles were 34.16% SFAs, 10.69% MUFAs and 55.15 PUFAs. The results of the fatty acid analysis reveal that *L. vulgaris* is quite rich in n-3 fatty acids. Navarro and Villanueva (2000) found that cephalopods in their early stages of growth show high requirement for PUFA. The contents of n-3 PUFA were 53.62% in mantles and 48.42% in tentacles. C22:6 n-3 (DHA) were the dominant PUFAs in lipid from both portions. DHA and 20:5 n-3(EPA) were found at the level of 38.97% and 0.77% in the lipid from mantle and 34.95 and 0.70% in the lipid from tentacles. The most abundant fatty acid in squid mantle and tentacle was DHA followed by palmitic acid and eicosapentaenoic acid (EPA). DHA and EPA are the most characteristic acid for cephalopods (Navarro and Villanueva 2000). Ozogul et al.

Table 2 The pH values of mantles and tentacles of *L. vulgaris* during frozen storage

Time (Month)	Mantle			Tentacle		
	−20 °C	−40 °C	−80 °C	−20 °C	−40 °C	−80 °C
0	6.6 ± 0.03^{Aa}	6.6 ± 0.03^{Aa}	6.6 ± 0.03^{Aa}	6.7 ± 0.03^{A}	6.7 ± 0.02^{A}	6.7 ± 0.02^{A}
4	6.7 ± 0.05^{Ba}	6.6 ± 0.05^{Ba}	6.6 ± 0.02^{Ba}	6.8 ± 0.11^{B}	6.7 ± 0.07^{B}	6.7 ± 0.07^{B}
8	6.7 ± 0.06^{Ca}	6.7 ± 0.06^{Ca}	6.7 ± 0.01^{Cb}	6.9 ± 0.13^{BC}	6.8 ± 0.08^{BC}	6.8 ± 0.07^{BC}
12	6.8 ± 0.04^{Da}	6.8 ± 0.04^{Da}	6.7 ± 0.01^{Db}	6.9 ± 0.09^{C}	6.9 ± 0.03^{C}	6.8 ± 0.07^{C}

Values are given as mean±S.D. from triplicate determinations.

Different capital superscripts in the same column indicate significant differences ($P<0.05$).

Different lowercase superscripts in the same row indicate significant differences ($P<0.05$).

Table 3 The free fatty acidity values of mantles and tentacles of *L. vulgaris* during frozen storage

Time (Month)	Mantle			Tentacule		
	−20 °C	−40 °C	−80 °C	−20 °C	−40 °C	−80 °C
0	1.4 ± 0.06^{Aa}	1.4 ± 0.06^{Aa}	1.4 ± 0.06^{Aa}	1.2 ± 0.03^{Aa}	1.2 ± 0.03^{Aa}	1.2 ± 0.03^{Aa}
4	2.9 ± 0.09^{Ba}	2.6 ± 0.11^{Bb}	1.4 ± 0.04^{Bc}	2.5 ± 0.04^{Bb}	2.3 ± 0.14^{Ba}	1.2 ± 0.12^{Bc}
8	2.0 ± 0.09^{Ca}	2.9 ± 0.09^{Cb}	1.7 ± 0.07^{Cc}	1.7 ± 0.15^{Cc}	2.5 ± 0.05^{Ca}	1.5 ± 0.04^{Cb}
12	1.7 ± 0.05^{Da}	2.3 ± 0.10^{Db}	2.1 ± 0.07^{Dc}	1.4 ± 0.07^{Dc}	2.0 ± 0.07^{Da}	1.8 ± 0.09^{Db}

Values are given as mean ± S.D. from triplicate determinations.

Different capital superscripts in the same column indicate significant differences ($P<0.05$).

Different lowercase superscripts in the same row indicate significant differences ($P<0.05$).

Table 4 The peroxide values of mantles and tentacles of *L. vulgaris* during frozen storage

Time (Month)	Mantle			Tentacule		
	−20 °C	−40 °C	−80 °C	−20 °C	−40 °C	−80 °C
0	2.2 ± 0.04^{Aa}	2.2 ± 0.04^{Aa}	2.2 ± 0.04^{Aa}	1.5 ± 0.02^{Aa}	1.5 ± 0.02^{Aa}	1.5 ± 0.02^{Aa}
4	2.6 ± 0.10^{Ba}	2.4 ± 0.07^{Bb}	2.2 ± 0.07^{Ac}	1.8 ± 0.06^{Ba}	1.7 ± 0.08^{Bb}	1.5 ± 0.04^{Ac}
8	3.0 ± 0.09^{Ca}	3.0 ± 0.11^{Ca}	2.7 ± 0.08^{Bb}	2.1 ± 0.02^{Ca}	2.1 ± 0.05^{Ca}	1.9 ± 0.02^{Bb}
12	3.4 ± 0.13^{Da}	2.8 ± 0.09^{Db}	2.6 ± 0.11^{Bc}	2.4 ± 0.14^{Da}	2.0 ± 0.03^{Cb}	1.8 ± 0.11^{Bc}

Values are given as mean ± S.D. from triplicate determinations.

Different capital superscripts in the same column indicate significant differences ($P<0.05$).

Different lowercase superscripts in the same row indicate significant differences ($P<0.05$).

Table 5 The thiobarbituric acid reactive substances values of mantles and tentacles of *L. vulgaris* during frozen storage (mg MA kg^{-1})

Time (Month)	Mantle			Tentacule		
	−20 °C	−40 °C	−80 °C	−20 °C	−40 °C	−80°C
0	0.73 ± 0.02^{Aa}	0.73 ± 0.02^{Aa}	0.73 ± 0.02^{Aa}	0.80 ± 0.01^{Aa}	0.80 ± 0.01^{Aa}	0.80 ± 0.01^{Aa}
4	3.0 ± 0.01^{Ba}	2.3 ± 0.01^{Bb}	2.1 ± 0.01^{Bc}	3.3 ± 0.01^{Ba}	2.5 ± 0.02^{Bb}	2.2 ± 0.01^{Bc}
8	4.6 ± 0.01^{Ca}	3.5 ± 0.01^{Cb}	3.0 ± 0.02^{Cc}	5.0 ± 0.02^{Ca}	3.8 ± 0.01^{Cb}	3.3 ± 0.01^{Cc}
12	6.2 ± 0.02^{Da}	4.3 ± 0.01^{Db}	3.2 ± 0.02^{Dc}	6.7 ± 0.02^{Da}	4.7 ± 0.02^{Db}	3.5 ± 0.01^{Dc}

Values are given as mean±S.D. from triplicate determinations.

Different capital superscripts in the same column indicate significant differences ($P<0.05$).

Different lowercase superscripts in the same row indicate significant differences ($P<0.05$).

Table 6 Fatty acid distribution of *L. vulgaris* mantles during frozen storage

	−20 °C				−40 °C				−80 °C			
	0	4	8	12	0	4	8	12	0	4	8	12
C14:0	1.41^{Aa}	2.05^{Ba}	2.70^{Ca}	3.45^{Da}	1.41^{Aa}	1.72^{Bb}	2.06^{Cb}	2.32^{Db}	1.41^{Aa}	1.72^{Bb}	2.05^{Cb}	2.30^{Db}
C15:0	0.76	0.77	0.76	0.79	0.76	0.77	0.77	0.76	0.76	0.76	0.76	0.76
C16:0	22.28^{Aa}	24.37^{Ba}	26.20^{Ca}	28.62^{Da}	22.28^{Aa}	23.61^{Bb}	24.82^{Cb}	25.73^{Db}	22.28^{Aa}	23.61^{Bb}	24.84^{Cb}	25.75^{Db}
C17:0	1.14^{Aa}	1.29^{Ba}	1.47^{Ca}	1.68^{Da}	1.14^{Aa}	1.25^{Bb}	1.31^{Cb}	1.41^{Db}	1.14Aa	1.25^{Bb}	1.30^{Cb}	1.41^{Db}
C18:0	4.31^{Aa}	5.07^{Ba}	5.79^{Ca}	6.72^{Da}	4.31^{Aa}	4.66^{Bb}	5.14^{Cb}	5.49^{Db}	4.31Aa	4.65^{Bb}	5.13^{Cb}	5.48^{Db}
C20:0	0.04^{Aa}	0.05^{Ba}	0.07^{Ca}	0.07^{Da}	0.04^{Aa}	0.04^{Ab}	0.05^{Bb}	0.05^{Bb}	0.04Aa	0.04^{Ab}	0.05^{Bb}	0.05^{Bb}
C22:0	0.00^{Aa}	0.05^{Ba}	0.08^{Ca}	0.16^{Da}	0.00^{Aa}	0.02^{Bb}	0.04^{Cb}	0.06^{Db}	0.00Aa	0.02^{Bb}	0.04^{Cb}	0.06^{Db}
C14:1	0.19^{Aa}	0.13^{Ba}	0.08^{Ca}	0.00^{Da}	0.19^{Aa}	0.28^{Bb}	0.39^{Cb}	0.48^{Db}	0.19^{Aa}	0.28^{Bb}	0.38^{Cb}	0.47^{Db}
C15:1	0.07^{Aa}	0.05^{Ba}	0.04^{Ca}	0.03^{Da}	0.07^{Aa}	0.05^{Bb}	0.05^{Cb}	0.04^{Db}	0.07^{Aa}	0.05^{Bb}	0.05^{Cb}	0.04^{Db}
C16:1	0.85^{Aa}	0.66^{Ba}	0.47^{Ca}	0.21^{Da}	0.85^{Aa}	0.76^{Bb}	0.66^{Cb}	0.56^{Db}	0.85^{Aa}	0.75^{Bb}	0.66^{Cb}	0.56^{Db}
C17:1	0.14	0.14	0.13	0.13	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14
C18:1 n9	4.28^{Aa}	4.18^{Ba}	4.03^{Ca}	3.94^{Da}	4.28^{Aa}	4.19^{Bb}	4.19^{Cb}	4.12^{Db}	4.28^{Aa}	4.19^{Bb}	4.18^{Cb}	4.10^{Db}
C20:1	4.25^{Aa}	3.81^{Ba}	3.30^{Ca}	2.83^{Da}	4.25^{Aa}	4.04^{Bb}	3.79^{Cb}	3.54^{Db}	4.25^{Aa}	4.05^{Bb}	3.78^{Cb}	3.52^{Db}
C22:1n9	0.08^{Aa}	0.06^{Ba}	0.04^{Ca}	0.00^{Da}	0.08^{Aa}	0.06^{Ba}	0.05^{Cb}	0.04^{Db}	0.08^{Aa}	0.06^{Ba}	0.05^{Cb}	0.05^{C}
C24:1	0.08^{Aa}	0.07^{Ba}	0.03^{Ca}	0.00^{Da}	0.08^{Aa}	0.07^{Ba}	0.06^{C}	0.04^{D}	0.08^{A}	0.07^{B}	0.05^{C}	0.04^{D}
C18:2n6	1.32^{Aa}	0.96^{Ba}	0.58^{Ca}	0.19^{Da}	1.32^{Aa}	1.16^{Bb}	0.96^{Cb}	0.79^{Db}	1.32^{Aa}	1.16^{Bb}	0.96^{Cb}	0.78^{Db}
C18:3n6	0.08^{Aa}	0.09^{Aa}	0.08^{Ba}	0.07^{Ca}	0.08^{Aa}	0.08^{Bb}	0.08^{Ba}	0.08^{Bb}	0.08^{Aa}	0.08^{Bb}	0.08^{Ba}	0.08^{Bb}
C18:3n3	0.06^{Aa}	0.04^{Ba}	0.03^{Ca}	0.02^{Da}	0.06^{Aa}	0.04^{Bb}	0.04^{Cb}	0.03^{Db}	0.06^{Aa}	0.04^{Bb}	0.04^{Cb}	0.03^{Db}
C20:2	0.24^{Aa}	0.21^{Ba}	0.18^{Ca}	0.16^{Da}	0.24^{Aa}	0.22^{Bb}	0.21^{Cb}	0.19^{Db}	0.24^{Aa}	0.22^{Bb}	0.21^{Cb}	0.19^{Db}
C20:3n3	0.30^{Aa}	0.24^{Ba}	0.18^{Ca}	0.15^{Da}	0.30^{Aa}	0.27^{Bb}	0.24^{Cb}	0.21^{Db}	0.30^{Aa}	0.27^{Bb}	0.24^{Cb}	0.20^{Db}
C20:4n6	2.65^{Aa}	2.33^{Ba}	1.99^{Ca}	1.69^{Da}	2.65^{Aa}	2.56^{Bb}	2.31^{Cb}	2.19^{Db}	2.65^{Aa}	2.56^{Bb}	2.31^{Cb}	2.19^{Db}
C20:5n3	14.30^{A}	14.19^{A}	13.87^{B}	13.79^{B}	14.30^{A}	14.50^{A}	14.00^{B}	14.42^{A}	14.30^{A}	14.52^{A}	14.04^{B}	14.47^{A}
C22:2	0.12^{Aa}	0.09^{Ba}	0.05^{Ca}	0.00^{Da}	0.12^{Aa}	0.10^{Bb}	0.09^{Cb}	0.07^{Db}	0.12^{Aa}	0.10^{Bb}	0.08^{Cb}	0.07^{Db}
C22:4n6	0.49^{Aa}	0.37^{Ba}	0.21^{Ca}	0.17^{Ca}	0.49^{Aa}	0.35^{Bb}	0.30^{Bb}	0.25^{Cb}	0.49^{Aa}	0.35^{Bb}	0.29^{Cb}	0.25^{Cb}
C22:5n6	0.77^{Aa}	0.58^{Ba}	0.32^{Ca}	0.25^{Da}	0.77^{Aa}	0.55^{Ba}	0.47^{Cb}	0.38^{Db}	0.77^{Aa}	0.55^{Ba}	0.46^{Cb}	0.38^{Db}
C22:6n3	38.97^{Aa}	37.19^{Ba}	34.78^{Ca}	32.96^{Da}	38.97^{Aa}	38.51^{ABb}	37.80^{Bb}	36.62^{Cb}	39.18^{Aa}	38.51^{ABb}	37.82^{Bb}	36.64^{Cb}
sfa	29.95	33.66	37.07	41.49	29.95	32.06	34.18	35.82	29.95	32.05	34.19	35.81
ufa	69.26	65.38	60.39	56.60	69.26	67.94	65.82	64.18	69.47	67.95	65.81	64.19
mufa	9.95	9.10	8.11	7.15	9.95	9.60	9.32	8.95	9.95	9.60	9.29	8.91
pufa	59.31	56.28	52.27	49.45	59.31	58.34	56.50	55.22	59.52	58.35	56.53	55.28
w3	53.62	51.65	48.86	46.92	53.62	53.32	52.07	51.27	53.83	53.34	52.14	51.34
w6	5.33	4.33	3.18	2.37	5.33	4.69	4.12	3.68	5.33	4.69	4.09	3.68

Different capital superscripts in the same temperature indicate significant differences ($P<0.05$).

Different lowercase superscripts in the time indicate significant differences ($P<0.05$).

Table 7 Fatty acid distribution of tentacles of *L. vulgaris* during frozen storage

	−20 °C				−40 °C				−80 °C			
	0	4	8	12	0	4	8	12	0	4	8	12
C14:0	1.53^{Aa}	2.21^{Ba}	2.90^{Ca}	3.69^{D}	1.53^{Aa}	1.86^{Bb}	2.23^{Cb}	2.50^{D}	1.53^{Aa}	1.86^{Bb}	2.21^{Cb}	2.48^{Db}
C15:0	0.89^{Aa}	0.90^{Aa}	0.89^{Aa}	0.92^{Ba}	0.89^{Aa}	0.89^{Aa}	0.89^{Aa}	0.88^{Ab}	0.89^{Aa}	0.88^{AB}	0.88^{ABa}	0.87^{Bb}
C16:0	25.46^{Aa}	27.82^{Ba}	30.04^{Ca}	32.73^{Da}	25.46^{Aa}	26.71^{Bb}	28.02^{Cb}	29.01^{Db}	25.46^{Aa}	26.72^{Bb}	28.05^{Cb}	29.03^{Db}
C17:0	1.29^{Aa}	1.46^{Ba}	1.68^{Ca}	1.90^{Da}	1.29^{Aa}	1.41^{Bb}	1.46^{Cb}	1.58^{Db}	1.29^{Aa}	1.41^{Bb}	1.46^{Cb}	1.58^{Db}
C18:0	4.94^{Aa}	5.81^{Ba}	6.67^{Ca}	7.72^{Da}	4.94^{Aa}	5.29^{Bb}	5.82^{Cb}	6.21^{Db}	4.94^{Aa}	5.28^{Bb}	5.82^{Cb}	6.20^{Db}
C20:0	0.04^{Aa}	0.05^{Aa}	0.07^{Ba}	0.08^{Ba}	0.04^{Aa}	0.04^{Ab}	0.05^{Bb}	0.05^{Bb}	0.04^{Aa}	0.04^{Ab}	0.05^{Bb}	0.05^{Bb}
C22:0	0.00^{Aa}	0.05^{Ba}	0.08^{Ca}	0.16^{Da}	0.00^{Aa}	0.02^{Bb}	0.04^{Cb}	0.06^{Db}	0.00^{Aa}	0.02^{Bb}	0.04^{Cb}	0.06^{Db}
C14:1	0.19^{Aa}	0.13^{Ba}	0.82^{Ca}	0.00^{Da}	0.19^{Aa}	0.28^{Bb}	0.38^{Cb}	0.47^{Db}	0.19^{Aa}	0.28^{Bb}	0.38^{Cb}	0.47^{Db}
C15:1	0.07^{Aa}	0.05^{Ba}	0.04^{Ca}	0.03^{Da}	0.07^{Aa}	0.05^{Bb}	0.05^{Cc}	0.04^{Dd}	0.07^{Aa}	0.05^{Bb}	0.05^{Cc}	0.04^{Dd}
C16:1	0.85^{Aa}	0.66^{Ba}	0.47^{Ca}	0.21^{Da}	0.85^{Aa}	0.75^{Bb}	0.65^{Cb}	0.55^{Db}	0.85^{Aa}	0.75^{Bb}	0.65^{Cb}	0.55^{Db}
C17:1	0.14^{Aa}	0.14^{Aa}	0.13^{Ba}	0.13^{Ba}	0.14^{Aa}	0.14^{Aa}	0.13^{Ba}	0.14^{Aa}	0.14^{Aa}	0.14^{Aa}	0.13^{Ba}	0.13^{Ba}
C18:1 n9	4.94^{Aa}	4.82^{Ba}	4.66^{Ca}	4.56^{Da}	4.94^{Aa}	4.80^{Bb}	4.79^{Cb}	4.69^{Db}	4.94^{Aa}	4.79^{Bb}	4.78^{Cb}	4.67^{Db}
C20:1	4.37^{Aa}	3.91^{Ba}	3.41^{Ca}	2.92^{Da}	4.37^{Aa}	4.11^{Bb}	3.85^{Cb}	3.59^{Db}	4.37^{Aa}	4.13^{Bb}	3.84^{Cb}	3.57^{Db}
C22:1n9	0.08^{Aa}	0.06^{Ba}	0.04^{Ca}	0.00^{Da}	0.08^{Aa}	0.06^{Ba}	0.05^{Cb}	0.04^{Db}	0.08^{Aa}	0.06^{Ba}	0.05^{Cb}	0.05^{Db}
C24:1	0.04^{Aa}	0.03^{Ba}	0.01^{Ca}	0.00^{Da}	0.04^{Aa}	0.03^{Ba}	0.03^{Bb}	0.02^{Cb}	0.04^{Aa}	0.03^{Ba}	0.02^{Cc}	0.02^{Cb}
C18:2n6	2.65^{Aa}	1.92^{Ba}	1.17^{Ca}	0.39^{Da}	2.65^{Aa}	2.30^{Bb}	1.89^{Cb}	1.55^{Db}	2.65^{Aa}	2.30^{Bb}	1.89^{Cb}	1.55^{Db}
C18:3n6	0.09^{Aa}	0.09^{Aa}	0.08^{Ba}	0.07^{Ca}	0.09^{Aa}	0.08^{Bb}	0.08^{Ba}	0.08^{Bb}	0.09^{Aa}	0.08^{Bb}	0.08^{Ba}	0.08^{Bb}
C18:3n3	0.06^{Aa}	0.04^{Ba}	0.03^{Ca}	0.02^{Da}	0.06^{Aa}	0.04^{Ba}	0.04^{Bb}	0.03^{Ca}	0.06^{Aa}	0.04^{Ba}	0.04^{Bb}	0.03^{Ca}
C20:2	0.24^{Aa}	0.21^{Ba}	0.18^{Ca}	0.16^{Da}	0.24^{Aa}	0.22^{Ba}	0.21^{Bb}	0.19^{Cb}	0.24^{Aa}	0.22^{Ba}	0.21^{Bb}	0.19^{Cb}
C20:3n3	0.30^{Aa}	0.24^{Ba}	0.18^{Ca}	0.15^{Da}	0.30^{Aa}	0.27^{Bb}	0.23^{Cb}	0.21^{Db}	0.30^{Aa}	0.27^{Bb}	0.23^{Cb}	0.20^{Db}
C20:4n6	2.48^{Aa}	2.18^{Ba}	1.87^{Ca}	1.57^{Da}	2.48^{Aa}	2.37^{Bb}	2.14^{Cb}	2.02^{Db}	2.48^{Aa}	2.37^{Bb}	2.14^{Cb}	2.02^{Db}
C20:5n3	13.11^{Aa}	12.99^{Aa}	12.76^{Ba}	12.65^{Ba}	13.11^{Aa}	13.16^{Ab}	12.68^{Bb}	13.04^{Cb}	13.11^{Aa}	13.18^{Ab}	12.72^{Bb}	13.09^{Cb}
C22:2	0.12^{Aa}	0.09^{Ba}	0.05^{Ca}	0.00^{Da}	0.12^{Aa}	0.10^{Bb}	0.08^{Cb}	0.07^{Db}	0.12^{Aa}	0.10^{Bb}	0.08^{Cc}	0.07^{Db}
C22:4n6	0.46^{Aa}	0.34^{Ba}	0.19^{Ca}	0.15^{Da}	0.46^{Aa}	0.32^{Ba}	0.28^{Cb}	0.23^{Db}	0.46^{Aa}	0.32^{Ba}	0.27^{Cb}	0.23^{Db}
C22:5n6	0.70^{Aa}	0.52^{Ba}	0.30^{Ca}	0.23^{Da}	0.70^{Aa}	0.50^{Ba}	0.43^{Cb}	0.35^{Db}	0.70^{Aa}	0.50^{Ba}	0.41^{Cb}	0.35^{Cb}
C22:6n3	34.95^{Aa}	33.30^{Ba}	31.29^{Ca}	29.57^{Da}	34.95^{Aa}	34.19^{Bb}	33.49^{Cb}	32.39^{Db}	34.95^{Aa}	34.19^{Bb}	33.51^{Cb}	32.42^{Db}
sfa	34.16	38.29	42.31	47.20	34.16	36.22	38.51	40.29	34.16	36.21	38.52	40.28
ufa	65.84	61.71	57.69	52.80	65.84	63.78	61.49	59.71	65.84	63.79	61.48	59.72
mufa	10.69	9.80	9.59	7.84	10.69	10.23	9.94	9.55	10.69	10.23	9.90	9.50
pufa	55.15	51.91	48.10	44.96	55.15	53.55	51.55	50.16	55.15	53.56	51.58	50.22
w3	48.42	46.56	44.26	42.39	48.42	47.66	46.44	45.67	48.42	47.68	46.50	45.74
w6	6.37	5.05	3.61	2.41	6.37	5.57	4.82	4.23	6.37	5.57	4.79	4.22

Different capital superscripts in the same temperature indicate significant differences ($P<0.05$).

Different lowercase superscripts in the time indicate significant differences ($P<0.05$).

(2008) reported similar results for *L. vulgaris*. Despite the fact that cephalopods contain very small amounts of fat, this organism is good sources of EPA and DHA content.

SFAs content were increased but MUFAs and PUFAs contents were decreased during frozen storage of mantles and tentacles. The increase ratio of SFAs contents were decreased while the decrease ratios of MUFAs and PUFAs contents were increased with decreasing storage temperature.

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