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Physicochemical and functional changes in jumbo squid (*Dosidicus gigas*) mantle muscle during ice storage

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ABSTRACT

Physicochemical and functional changes of jumbo squid (*Dosidicus gigas*) mantle muscle stored in ice for 15 days were evaluated. Proximate analysis, pH, protein solubility (PS) and SDS-PAGE profile changes were monitored for muscle proteins, while texture profile analysis (TPA) (gel strength, elasticity and cohesiveness), water holding capacity (WHC) and colour changes were monitored for gels produced from this muscle. Fresh muscle chemical composition varied according to fishing season and/or physiological status presented by specimens. pH and PS were stable ($p \geqslant 0.05$) throughout the study with a slight tendency to decrease towards the end of study. SDS-PAGE revealed no drastic changes in proteins influencing gelation (i.e., myosin). TPA and WHC of gels decreased towards the end of experiment; however, due to sampling variability, no significant differences ($p \geqslant 0.05$) were found over storage time. Changes in gel colour were observed with hue angle decreasing towards yellow hues. Improved post-capture management of mantle muscle preserved its integrity and functionality for up to two weeks.

1. Introduction

Jumbo squid (*Dosidicus gigas* Orbigny, 1835), an endemic species from the pelagic zone of the Eastern Pacific, is the largest and most abundant squid species found from Chile up to the Northwest coast of the United States (Markaida, 2005; Nigmatullin, Nesis, & Arkhipkin, 2001). In Mexico, it is quite abundant in the Gulf of California, representing 95–99% of total jumbo squid captures and approximately 5% of the total national production (CONAPESCA, 2004). Its mantle represents around half of the weight of the edible portion (60–80% of animal weight) (Slabyj, Ramsdell, & True, 1981); however, by the time it reaches market the mantle is of low value due to a rapid decrease in quality post-capture (if not managed properly). For this reason there is little interest in this fishery in the region.

Several studies have mentioned that this type of cephalopod muscle suffers a rapid quality loss, and attribute this behavior to the high proteolytic activity it possesses immediately after catch, affecting its protein functionality (Gómez-Guillén, Solas, Borderías, & Montero, 1996; Goméz-Guillén & Montero, 1997; Gómez-Guillén, Montero, Solas, & Borderías, 1998). Most studies have dealt with 1 to 6 months old frozen specimens and probable without having any post-capture control. Working with frozen muscles in such studies should not be a problem, especially if the fish has been properly handle after its catch (i.e., rapid evisceration and cooling processes). Jumbo squid caught in the Gulf of California during the last decade were frequently subjected to poor post-capture management. For example, specimens were piled up on fishing vessels without any evisceration and cooling treatment for several hours, sometimes reaching temperatures above 35 °C, especially in the hot months from May to October (Grajeda-Rodriguez, 2007). This management could have damaged the integrity of the visceral cavity and muscle fibers, thus releasing gastric and muscle enzymes. Producers and processors have now established higher requirements for this type of fishing activity (Grajeda-Rodriguez, 2007). This may explain why jumbo squid muscle presented a high proteolytic activity as mentioned by those authors. Thus, it is proposed that good squid management (i.e., rapid evisceration and icing of mantle muscle) could improve and maintain the mantle muscle quality and functionality for a lapse time.

Hence, the objective of the present study was to elucidate the physicochemical and functional changes presented in the proteins from jumbo squid (*D. gigas*) mantle muscle during ice storage.

2. Materials and methods

2.1. Raw material

Jumbo squid was harvested in the Gulf of California (Sonora, México) using the hand-jig method. Three samplings trips were conducted, two in the autumn (October and November for

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sampling I and II, respectively) of 2005 at approximately 27°46′ N and 110°59′ W off the coast of San Carlos, Nuevo Guaymas, Sonora, and the last done in the spring (March for sampling III) of 2006 at approximately 28°40′ N, 107°, 112°20′19′ W off the coast of Kino Bay, Sonora.

Specimens with mantles weighing 0.6–6 kg/each (<40–62 cm mantle length) were eviscerated immediately after capture, and mantles were stored on board in alternate layers with crushed ice for their transportation to the laboratory facilities for processing. The elapsed time from capture to start of processing never exceeded 12 h. All mantles were held in crushed ice (0 °C) prior to processing.

Once in the pilot plant at the research center, mantles (approximately 10 per sample trip) were measured, weighed, and washed with cold water; fins were cut off and 13 × 13 cm mantle pieces were cut and manually mixed to have a random sample. Nine polyethylene bags were filled with three mantle squares each (approximately total weight of 400 g per bag) which were immediately stored in ice and analysed at days 0, 2, 4, 6, 9, 13 and 15, based on a study by Ramírez-Olivas, Rouzaud-Sánchez, Haard, Pacheco-Aguilar, and Ezquerra-Brauer (2004).

2.2. Mantle muscle analyses

2.2.1. Proximate analysis and pH

Proximate analysis (water, protein and ash content of muscle and gels) was carried out following the AOAC (2000) procedures. Lipid content was obtained according to Woyewoda, Shaw, Ke, and Burns (1986). Three repetitions were conducted on fresh muscle (day 0 only). In order to obtain rapid water content readings of muscle samples, and adjust sols to 80% water content, a thermobalance (Ohaus Co. Flornam Park, NY) was used. Briefly, approximately 50 g of sample were homogenized in a blender for 2 min (at 15 s intervals), always keeping the sample at <10 °C. Ten grams of sample were used for the analysis. Results were used only for gel production and are not discussed in the results and discussion section. Muscle pH was obtained from readings done directly to the homogenized sample using a model 240 Corning digital potentiometer (Corning Science Product, New York, NY).

2.2.2. Muscle protein solubility

Protein solubility from muscle stored on ice was measured according to Hsu and Ko (2001). Briefly, samples were homogenized for 1 min in 20 mM phosphate buffer (pH 7.0) containing 0.6 M NaCl at a 1:19 (v/v) ratio (sample:buffer). Protein measurements were done before and after centrifugation at 20,000*g* for 15 min, by the biuret method (Gornall, Bardawill, & David, 1949). PS was expressed as a percentage, calculated as follows:

$$\mathrm{PS} = \{(\text{protein concentration of supernatant after centrifugation}) / (\text{protein concentration of supernatant before centrifugation})\} \times 100.$$

Analysis was done in triplicate on each sampling day.

2.2.3. SDS-PAGE

Sodium dodecyl sulfate-polyacrylamide gel electrophoresis (SDS-PAGE) was run to observe possible proteolytic changes occurring during ice storage, and to elucidate dynamic protein changes during heat-induced gel network formation. Samples were heated from 25 to 90 °C at a rate of approximately 1 °C/min. Samples were taken at 25, 40, 60 and 90 °C, and aliquots of 2 mL (pre-gel) or 2 g (gel) were blended with 18 mL of dissolving solution (5% SDS, 0.1% β-mercaptoethanol) using an UltraTurrax homogenizer (IKA Works Inc. Wilminton, NC). Fresh mantle muscle was also treated with the dissolving solution. The homogenates were incubated at 80 °C for 1 h to allow maximal protein solubilization and extraction, and subsequently centrifuged at 3000*g* for 15 min. The supernatants, after measuring protein concentration by the Biuret method, were diluted to a 0.2% protein concentration with water and then mixed (at a 1:1, v/v ratio) with SDS-PAGE sample buffer before electrophoresis (Ramirez-Suarez, Addo, & Xiong, 2005).

SDS-PAGE was performed accordingly to Laemmli (1970) with some modifications (Wang & Xiong, 1998). Ten and four percent (separating and stacking gels respectively) discontinuous gels (width × height × thickness = 80 × 60 × 1.5 mm) were prepared. Treated protein samples (0.2% protein concentration) were mixed (at a 1:1 v/v ratio) with SDS–PAGE sample buffer (4% SDS, 20% glycerol, 10% β-mercaptoethanol, 0.125 M Tris, pH 6.8) and dissolved by heating in boiling water for 3 min. Aliquots of 30 μg of protein per lane were loaded onto the acrylamide gel.

2.3. Gelation of mantle muscle and their analyses

2.3.1. Gel preparation

Samples (400 g) of squid muscle, taken each sampling day, were mixed after the addition of NaCl, sucrose (or crushed ice when necessary) to adjust the sol composition to 80% water content, 18% solids and 2% NaCl. Each sample was homogenized in a Cuisinart DCl 8 food processor for 2 min, with intervals of 15 s to prevent the temperature from increasing above 10 °C. Each sol was packed into a Petri dish (1 cm height) and vacuum sealed in moisture/vapor-proof film bags (Nylon 0.75 MIL-Adhesive-Polyethylene 2.25 MIL, Cryovac Corp., Duncan, SC) with a Super Vac Smith vacuum machine (Smith Equipment Co., Clifton, NJ). Each sol was heat-set in a water bath at 90 °C/30 min. Heat-set gels were immediately chilled to <5 °C in an ice-water bath and held overnight at 2–4 °C prior to texture evaluation.

2.3.2. Rheology

Sols produced from squid mantle muscle were analysed for changes in dynamic gelation due to the cold storage. Samples were subjected to dynamic rheological testing using a Model RFS II rheometer (Rheometric Scientific, Inc. Piscataway, NJ) equipped with two parallel plates with a 2 mm gap. Samples were loaded in the space between the parallel plates, and the exposed rim was covered with a thin layer of mineral oil to prevent dehydration. One minute was given before the start of the measurement to allow for temperature equilibration. Gels were formed by heating the protein mixtures from 10 to 80 °C at a rate of 1 °C/min. The gelling samples were continually sheared in an oscillatory mode at a fixed frequency of 1 rad/s with a maximum strain of 0.05. Changes in the storage modulus (G′, i.e., rigidity due to elastic response of the material) were monitored throughout the gelling process. Analysis was carried out twice and as both runs were very similar only one set of data is shown.

2.3.3. Gel quality characteristics

Texture profile analysis (TPA), water holding capacity (WHC) and colour characteristics of the gels were evaluated. TPA was carried out in a TAXT2 Texture Analyzer (Stable Micro Systems, Ltd, Godalming, Surrey, UK) as follows: Cylindrical shaped samples of uniform dimensions (1 cm height × 1.5 cm diam. × 3–4 g) were cut from gels and left for an hour at room temperature inside a polyethylene bag to avoid sample dryness. Running conditions were as follows: A double compression at 75% (5 s waiting-time between compressions) and normal stress at 1 mm/s crosshead velocity. Results were analysed with the Texture Expert program for Windows calculating gel hardness (kgf/g sample), elasticity and cohesiveness (both dimensionless). WHC was determined according to Cheng, Hamann, and Webb (1979). Briefly, samples (5 g) were centrifuged at 27,000*g* for 30 min at 4 °C. Results were reported as % retained water by the sample.

The colour of gelled samples was measured using a Konica-Minolta CR-400 Tristimulus Colorimeter (Konica Minolta Sensing, Inc., Japan). Colour coordinates were used to measure the degree of lightness (L^*), redness–greenness (+ or $-a^*$), and yellowness–blueness (+ or $-b^*$). Additional colour traits such as hue angle (ϕ) and whiteness index (WI) were calculated from the "L^*", "a^*", "b^*" values (Minolta Corp., 1990). For a better integration and interpretation of a^* and b^* values, hue angles (ϕ) were calculated using the formula:

$$\Phi = \text{Arctang}(b^*/a^*)$$

The WI was calculated using the formula:

$$\text{WI} = 100 - [(100 - L^*)^2 + (a^*)^2 + (b^*)^2]^{\frac{1}{2}}$$

2.4. Statistical analysis

A randomized block design using the sampling period as the blocking criteria was used. For ANOVA and Tukeýs multiple range test a significance level was established at $p < 0.05$ using the SAS statistical package (Statistical Analysis System V8). Three sampling trips were carried out and the analyses were made at least in triplicate.

3. Results and discussion

3.1. Raw material characteristics

Specimens used in the present study were obtained from the three different sampling trips. They varied in physical characteristics (weight and length of muscle) over the sampling periods. The biggest specimens were caught during the first two sampling trips, with sampling I having means of 5.4 ± 1.4 kg and 52.0 ± 8.4 cm while sampling II with means of 4.7 ± 1.2 kg and 62.8 ± 3.4 cm for weight of mantle plus fins and length of mantle, respectively. Both samplings were made in the fall season. The smallest specimens were caught in the spring season, corresponding to sampling III, with weight of mantle plus fins and length of mantle of 0.6 ± 0.2 kg and <40 cm (proximate length) respectively. Chemical composition of the samples was also affected by the sampling period (Table 1). These differences can be related to: (a) fishing season, which in turn can be correlated with the physiological stage of specimens (mature *vs.* juveniles); (b) sex, as it is well established that females are bigger in mantle length (ML) than males (Markaida & Sosa-Nishizaki, 2001); and/or (c) different fishing area. These results agree with those of Ezquerra-Brauer, Haard, Ramírez-Olivas, Olivas-Burrola, and Velázquez-Sánchez (2002), who found smaller specimens during the spring season (April) of squid capture in the Gulf of California. The feeding stage could also influence the chemical composition of the mantle muscle, since it has been found that smaller specimens feed mainly on crustaceans while bigger organisms consume fish and other mollusks, even from their own species (Abugoch, Guarda, Pérez, & Paredes, 1999; Caddy, 1983). From Table 1, it is observed that jumbo squid contains low fat and relatively high (crude) protein contents, although not as high as most teleost fish. In addition, its mantle muscle contains around 1% of non-protein nitrogen, thus reducing even less the real protein content, which could have a functional role in the gelation process (Cortés-Ruiz, 2007).

Table 1
Chemical composition of jumbo squid (*Dosidicus gigas*) mantle muscle according to sampling trip

Component (%)	Sampling I	Sampling II	Sampling III	Means
Water content	86.4 ± 0.3	86.5 ± 0.1	83.0 ± 0.1	85.3 ± 2.0
Protein	12.1 ± 0.6	12.2 ± 0.1	10.2 ± 0.1	11.5 ± 1.1
Lipid	0.2 ± 0.0	0.3 ± 0.06	0.9 ± 0.0	0.5 ± 0.4
Ash	0.7 ± 0.2	0.9 ± 0.3	1.2 ± 0.0	0.9 ± 0.3
*Carbohydrates	0.6	0.1	4.6	1.8 ± 2.5

* Obtained by difference.

3.2. Changes in mantle muscle during ice storage

3.2.1. pH

pH is considered one of the most influential parameters in muscle protein functionality (Ofstad, Kidman, Myklebust, Olsen, & Hermansson, 1995). Post mortem changes in muscle have great functional and economical implications as they can greatly interfere with its quality. An animal's nutritional stage and high stress or exercise levels before an animal's death modify the glycogen concentration stored in muscle, consequently influencing in the post mortem pH (Haard, 1992; Massa, Paredi, & Crupkin, 2003). Fig. 1 shows the changes in pH at 0 °C over the 15 days of storage. Muscle pH was stable ($p \geqslant 0.05$) during the storage time with a slight tendency to decrease towards the end of the study. This suggests that good post-capture management retards the pH changes associated with bacterial and/or endogenous enzyme action that could promote the drop in pH. However, it could also demonstrate that squid mantle muscle could present a different post mortem rigor setting than the one displayed by land animals and some fishes, since no pH change was observed during the entire storage period. This agrees with the results of Ando, Ando, Tsukamasa, Makinodan, and Miyoshi (1999) who did not find a post mortem rigor settlement in arrow squid (*Loligo bleekert*) mantle.

The results in the present study are in agreement with those of Moran-Palacio (2002) and Ramírez-Olivas (2000) who observed a slight decrease in pH during the first days of cold storage, then an increase, and finally a drop towards the end of the storage period.

3.2.2. Muscle protein solubility

Protein solubility is considered one of the most important functional properties in the preparation of gelled-emulsified type products. In the present study, a significant reduction ($p < 0.05$) in the jumbo squid mantle muscle protein solubility was observed at the fourth day of ice storage (from 93.4 ± 4.0 to 87.4 ± 4.1 at days 0 and 4, respectively). In general, the muscle protein solubility was found to slightly decrease over the storage period at 0 °C (from

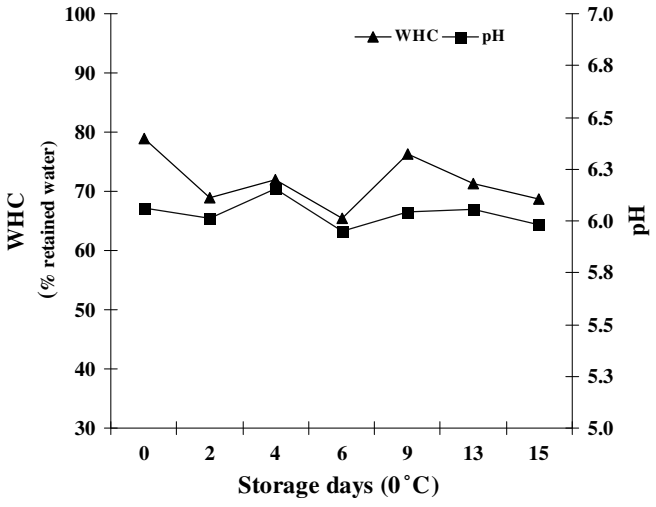

Fig. 1. Relation of jumbo squid (*Dosidicus gigas*) mantle muscle pH change with water holding capacity (WHC) of gels produced from the same muscle stored at 0 °C for 15 days. Values are the mean of three repetitions.

data mentioned previously to 89.5 ± 3.8 at day 15, $p \geqslant 0.05$) (all data not shown). The same pattern was obtained by Gómez-Guillén, Martinez-Alvarez, and Montero (2003) from squid (*Loligo vulgaris*) mantle muscle stored at 2 °C. They found that protein solubility in saline solution decreased during the first four days of storage, and increased towards the end of the study. These results indicate that mantle muscle proteins are stable under the conditions of cold temperature storage, contrary to what most manuscripts in the past decade have stated for this species.

3.2.3. SDS-PAGE

Mantle muscle protein changes promoted during cold storage (0 °C) are shown in Fig. 2a. Several observations can be made from this figure; however, it shows that only minor protein changes occurred during the 15 days of ice storage. It can be observed that a slight decrease in the myosin heavy chain (MHC) band intensity occurred over the storage time with a concomitant band appearance at 153 kDa and a slight increase in the paramyosin (PM) band intensity. These changes possibly occurred due to MHC hydrolysis; nevertheless, no new bands were detected in or below the 50 kDa region which would be the expected location of new bands after such hydrolysis. On the contrary, a disappearance of the 50–58 and 85 kDa bands was found, which was possibly due to cross-linking by endogenous transglutaminase (TGase) enzyme action, forming trimers and dimmers with an approximate molecular weight of 153 kDa, respectively. Park et al. (2003) found in *Todarodes pacificus* muscle paste both activities (hydrolytic and TGase) simultaneously. Neither Paramyosin nor actin were affected during the cold storage, in agreement with reports by Gómez-Guillén et al. (1998) and Konno, Young-Je, Yoshioka, Shinho and Seki (2003).

Several authors have suggested that jumbo squid mantle muscle possess a high proteolytic activity, which in turn affects its commercialization (Dublán-García, Cruz-Camarillo, Guerrero-Legarreta, & Ponce-Alquicira, 2006; Ezquerra-Brauer et al., 2002; Gómez-Guillén et al., 1996; Goméz-Guillén & Montero, 1997; Gómez-Guillén et al., 1998; Ramírez-Olivas et al., 2004). However, this activity is highly suppressed if jumbo squid mantle muscle is treated properly from the beginning (i.e., immediate post-catch evisceration and icing) and during its storage, as a great amount of MHC (mainly protein involved in the gelation process) can still be observed at day 15 of cold storage (Fig. 2a).

In order to elucidate the dynamic protein changes that occur during heat-induced gel network formation of squid muscle sols an experiment was carried out. Samples were taken at 25, 40, 60 and 90 °C during the gelation process and prepared for electrophoresis. Fig. 2b shows the results of samples taken at 90 °C only. Comparing both (Figs. 2a and b), it can be shown that no proteolytic activity was observed during the gelation process, as most MHC remained intact and only minor proteins are affected during this process. No TGase cross-linking (at least for MHC and actin) was observed. Results show that good post-catch handling minimizes the autolytic activity of muscle proteins in this muscle. If this type of activity would be present in the muscle, the gelation temperatures used did not activate it (comparing Fig. 2a and b). Konno, Young-Je, Yoshioka, Shinho, and Seki (2003) showed that jumbo squid, similar to the one used in the present study, presented autolysis of MHC at 25 °C; however, this activity was relatively slow (activity occurring over 0.5–4 h). This activity, if present in our samples, should not affect the gelation process since no incubation of sols occurred during this procedure.

3.3. Changes in gel quality characteristics

3.3.1. TPA and rheology

Table 2 shows the TPA of gels made of jumbo squid mantle muscle stored in ice during 15 days. A decreasing tendency in all parameters was observed towards the end of the cold storage period. Two important results were found regarding gel hardness. Firstly, the gel hardness dropped sharply at day 6 which coincided with the appearance of a dense protein band (at 153 kDa) in the electrophoresis analysis (Fig. 2a) and also a denser paramyosin band, which according to Konno et al. (2003) originates from myosin hydrolysis, a protein important in the formation of the three-dimensional gel network. Secondly, the gel hardness data were highly variable, which resulted from the variability in physical and chemical composition of the specimens (see Section 3.1 and Table 1, respectively). With respect to gel elasticity and cohesiveness, a similar behavior to that of gel hardness was found, in that

Table 2
Texture profile analysis of gels made of jumbo squid (*Dosidicus gigas*) mantle muscle stored in ice for 15 days

Storage day	Gel hardness (kgf/g)	Elasticity (%)	Cohesiveness (%)
0	0.64 ± 0.4^{ab}	74.0 ± 9.0^{a}	30.1 ± 3.7^{a}
2	0.66 ± 0.0^{ab}	76.3 ± 8.0^{a}	29.7 ± 5.0^{ab}
4	0.72 ± 0.4^{a}	67.4 ± 7.6^{a}	24.9 ± 2.5^{ab}
6	0.38 ± 0.2^{b}	68.9 ± 10.1^{a}	28.3 ± 3.8^{ab}
9	0.86 ± 0.7^{a}	70.7 ± 8.2^{a}	26.3 ± 3.4^{ab}
13	0.66 ± 0.5^{ab}	67.3 ± 12.1^{a}	25.1 ± 2.1^{ab}
15	0.51 ± 0.4^{ab}	64.2 ± 12.6^{a}	24.1 ± 5.0^{b}

Values with the same letters are statistically equal ($p \geqslant 0.05$).
Values are the mean and SD from six repetitions.

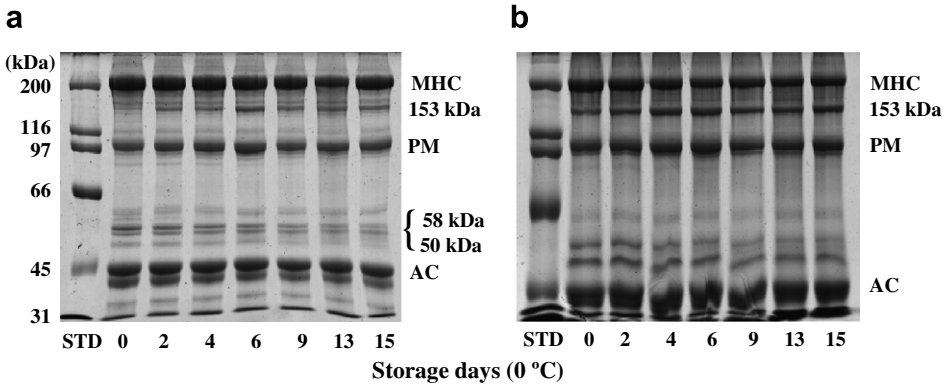

Fig. 2. Electrophoresis analysis of jumbo squid (*Dosidicus gigas*) mantle muscle. (a) Changes from muscle stored at 0 °C for 15 days, and (b) dynamic protein changes during heat-induced gel network formation from sols of the same muscle (samples taken at 90 °C). STD = molecular weight standards, MHC = myosin heavy chain, PM = paramyosin and AC = actin.

decreasing values were found towards the end of the study; nevertheless, elasticity did not show a significant difference ($p \geqslant 0.05$), while cohesiveness did so ($p < 0.05$). All these changes may be due to the effect of cold storage and gelation process exerted over the myosin molecule (see Fig. 2a and b). It has been proposed that the weakness of gels from this type of muscle is mainly due to the low protein concentration the squid pastes contain, with only around 10% of net protein (crude protein minus non-protein nitrogen, which in squid is present in high amounts) available for gelation (Cortés-Ruiz, 2007). It is theorized that if the protein concentration could be increased (whichever methodology used), squid gel hardness could be greatly improved. However, this still remains to be elucidated.

Heat-induced rheological changes in sols are displayed in Fig. 3. The storage modulus (G′) curves reported here differ from the one obtained by Pérez-Mateos, Montero, and Gómez-Guillén (2002) on fresh squid muscle (*Loligo vulgaris*); however, they are in agreement with results from the same study when proteolytic inhibitors and/or TGase were used, indicating either that *D. gigas* do not present the high proteolytic activity suggested by many authors (see Introduction and Fig. 2b) or it is species dependent. G′ of samples followed a linear behavior during the first 20°, then started to drop sharply around 31 °C to reach a minimum around 41 °C. The gel elasticity then gradually increased up to around 51 °C, the temperature at which it started to increase sharply up to an inflection point that was affected by the cold storage, i.e., day 0 showed it at 69.5 °C versus 67.5 °C at day 15. As shown in Fig. 3, days 2 and 4 showed higher ($p \geqslant 0.05$) G′ values than day 0, after which the values started to drop. These results agree with those obtained by TPA, where values also increased during the first days. Nevertheless, rheology results showed that gels were not greatly affected by the cold storage or by such proteolytic activity.

3.3.2. Water holding capacity (WHC)

Fig. 1 shows the changes in WHC from gels made from jumbo squid mantle muscle stored for 15 days at 0 °C. This parameter did not display statistical difference ($p \geqslant 0.05$) throughout the study probably due to sampling variability shown by the sampling trips (Table 1). However, a tendency to decrease towards the end of the study was observed (82.9 ± 8.6% at day 0 versus 75.3 ± 14.0% at day 15) which is in agreement with texture results. Similar WHC results obtained at day 0 were reported by Ayensa, Montero, Borderías, and Hurtado (2002) in gels made of squid muscle (*Todaropsis eblanae*) (88.5 ± 0.9%).

It is well known that pH affects the WHC of protein systems and that muscle proteins possess the least WHC at their isoelectric point. Thus, according to this fact, Fig. 1 shows the relationship between pH and WHC. Although no significant differences ($p \geqslant 0.05$) were found for either parameter during the study, the fact is corroborated as WHC followed almost parallel the pH changes.

3.3.3. Colour changes

Colour, one major food quality attribute, was evaluated for gels produced from jumbo squid muscle stored in ice for 15 days (Table 3). a^* and b^* values are not shown since they become more useful when they are integrated into the hue angle (θ) formula. Based from the hue angle results, the gels were grouped into the yellow-green quadrant of the colour sphere. Gel luminosity (L^*) was very stable ($p \geqslant 0.05$) throughout the storage period. Visual analysis supported these results as all gels (and sols) showed a very intense white tonality in agreement with whiteness index values, which were not affected ($p \geqslant 0.05$) by the cold storage. More yellow tonalities were observed at the end of the study.

Table 3
Changes in L^*, whiteness index, and hue angle (θ) of gels made of jumbo squid (*Dosidicus gigas*) mantle muscle stored in ice for 15 days

Storage day	L^*	Whiteness index (WI)	Hue angle (θ)
0	85.2 ± 1.2^a	83.6 ± 3.4^a	126.7 ± 21.9^a
2	85.3 ± 1.4^a	84.5 ± 1.5^a	120.5 ± 15.6^{bc}
4	85.7 ± 2.4^a	84.0 ± 3.7^a	113.9 ± 19.1^{bc}
6	86.5 ± 2.9^a	85.0 ± 2.9^a	110.5 ± 14.6^{bc}
9	86.4 ± 1.5^a	85.3 ± 2.0^a	112.5 ± 21.7^{bc}
13	85.5 ± 2.4^a	84.3 ± 3.5^a	108.4 ± 14.9^{bc}
15	84.3 ± 3.8^a	82.5 ± 5.5^a	102.7 ± 17.6^c

Values with the same letters are statistically equal ($p \geqslant 0.05$).
Values are the mean and SD from six repetitions.

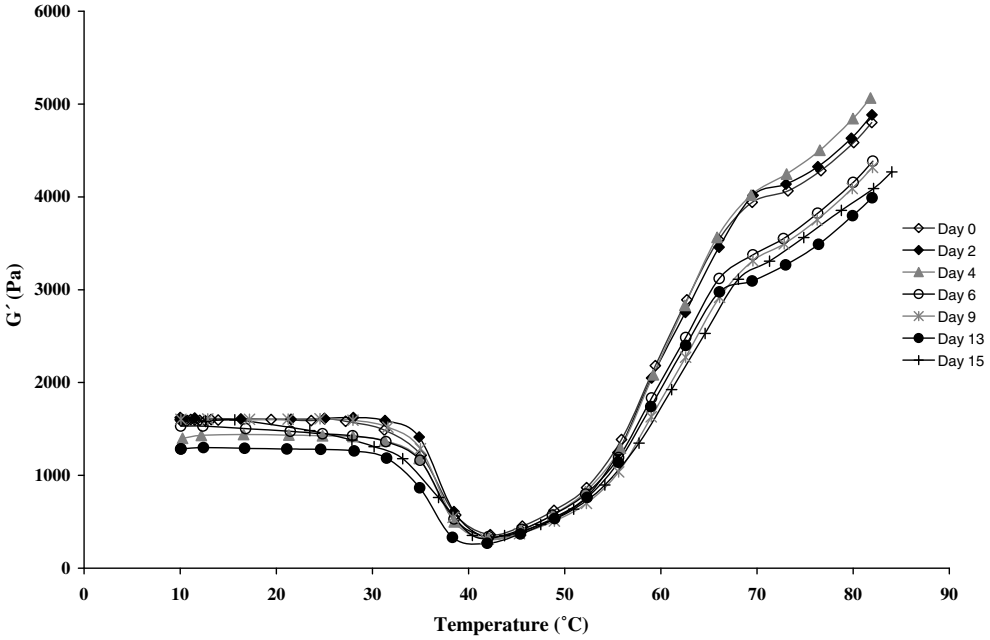

Fig. 3. Changes in elastic modulus (G′) during thermal treatment at 1 °C/min of sols made from jumbo squid (*Dosidicus gigas*) mantle muscle stored at 0 °C for 15 days.

4. Conclusions

Results from the present study show that adequate management of jumbo squid (*D. gigas*) mantle muscle during the different post-capture stages (i.e., evisceration, cold storage and transportation) help to maintain the protein muscle integrity and functionality, and almost completely reduced the proposed high proteolytic activity these specimens presented in other studies, at least for 15 days. They also support that by using this preservation method, squid muscle could be a good raw material for use in value-added products, such as gelled products. The weak texture characteristic of squid muscle gels might be due to the low protein content in the muscle *per se* since no important protein changes were found during gel formation. However, this still remains to be elucidated.

A great statistical variability was shown in the different functional properties measured. However, overall trends were identified that allowed conclusions to be drawn. If this specimen is going to be used as a raw material, it is advisable to take into account factors such as fishing season, physiological stage, cold storage and transportation of jumbo squid, as these factors have a marked effect on the results.

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