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Postmortem changes in cazon fish muscle stored on ice

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ABSTRACT

The biochemical, chemical and physical postmortem changes and their relation to the cazon muscle eating quality were studied during an 18 day storage period at 0° C (ice). Content of ATP and breakdown products, K value, pH, trimethylamine (TMA-N), total volatile bases (TVB-N), water-holding capacity (WHC), colour and texture changes were examined. At the beginning of the study, the cazon muscle showed a low concentration of ATP and a high value of IMP. Regarding to the signs of freshness and deterioration, K value presented a linear increase (r^2 = 0.97) with an initial value of 1.05% and a final value of 58.9%. The TBV-N and TMA-N significantly increased (P < 0.05). As for the physical analysis whereas the pH and the WHC changed ($P < 0.05$); texture was not affected ($P < 0.05$). The overall results of this study indicated that the edible quality of cazon fish muscle was maintained during at least 18 days of ice storage.

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1. Introduction

The cazon is a cartilaginous fish with a minor size of one and a half metre long that belongs to the family of sharks. The large amount of fish caught and the price make it one of the most important fishing resource in Mexico; reaching in 2004, 7000 tons [\(Anu](#page-4-0)[ario Estadístico de Pesca, 2004\)](#page-4-0) and a market price of 4.5 dollars per kilogram. It also represents a precious food source due to the muscle nutritional quality and delicious flavour. Despite the importance of this fishing resource that is mainly catch in an artesanal way and commercialised fresh on ice, nowadays is little known about this species. To evaluate the impact that the artesanal practices of post-capture handling have in the quality of cazon fish fillet, is necessary first determine the species postmortem biochemical behaviour as well as freshness and muscle quality along a storage study.

It is known that fish loss of freshness and spoilage pattern markedly varies from specie to specie. Once the fish dies several postmortem changes take place. These changes are due to the breakdown of the cellular structure and biochemistry as well as to the growth of microorganisms that are either naturally associated with the fish or that become part of the flora because of the contamination during handling [\(Ehira & Uchiyama, 1987](#page-5-0)). Within this postmortem changes that directly and strongly affect its quality and shelf-life there are the protein degradation, ATP degradation, drop of pH, lipid oxidation, undesirable compounds production as trimethylamine (TMA-N) and the molecular low weight volatile bases (TVB-N), which are produced by bacterial action. Likewise, the muscle experiences changes in texture, waterholding capacity and colour.

Deterioration of fish normally follows four stages: rigor mortis, resolution of rigor, autolysis (loss of freshness) and bacterial spoilage. These stages occur faster or slower depending on the species, the physiological condition of fish, microbial contamination and temperature. The autolytic processes are carried out through endogenous enzymes present in the muscle whilst deterioration is caused by bacterial growth. It has been observed that capture, handling and processing conditions determine these changes, being temperature the factor with the strongest impact. Therefore, to preserve muscle initial freshness and quality it is important, after catching the animal, decreased temperature as quick as possible [\(Ocano-Higuera, Maeda-Martínez, Lugo-Sánchez, & Pacheco-](#page-5-0)[Aguilar, 2006](#page-5-0)).

Nowadays, methods for evaluating freshness and quality of different marine species are based on measurements of postmortem changes associated with sensory, chemical and physical changes and microbiological growth ([Gökodlu, Özden, & Erkan, 1998\)](#page-5-0). Lately, quality indices based on nucleotide degradation have received special attention for monitoring fishery products freshness during handling and processing, whilst other measurements like trimethylamine (TMA-N), total volatile bases (TVB-N) and biogenic amines such as histamine, remain as bacterial deteriorario indices ([Ryder, 1985](#page-5-0)).

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In fish muscle, adenosine-5'-triphosphate (ATP) is metabolised according to the following sequence: $ATP \rightarrow adenosine-5'-diphos$ phate (ADP) \rightarrow adenosine-5'-monophosphate (AMP) \rightarrow inosine-5'monophosphate $(IMP) \rightarrow inosine$ $(HxR) \rightarrow hypoxanthine$ $(Hx) \rightarrow x$ anthine (X) ([Ehira & Uchiyama, 1987](#page-5-0)). Regardless of the species and muscle type, ATP rapidly decreases within the first 24 h postmortem. In most postharvest fish, the initial catabolism of ATP normally results in a fast and temporary accumulation of IMP, an intermediate metabolite that contributes to the pleasant fresh flavour of the meat ([Massa, Palácios, Paredi, & Crupkin,](#page-5-0) [2005\)](#page-5-0). This compound is slowly degraded to HxR and then to Hx which is related to the progressive loss of the fish desirable fresh flavour ([Howgate, 2005\)](#page-5-0). [Massa et al. \(2005\)](#page-5-0) pointed out that changes rates and patterns in nucleotides and their related compounds considerably differ with fish species, muscle type and factors related to handling and storage conditions.

K value is calculated from the ATP concentration and its products of degradation and it is used to measure how fast these compound degrade. It shows the relation, expressed in percentage, between the sum of the HxR concentrations and Hx between the sum of the ATP concentrations and related catabolite compounds. This index is broadly used to evaluate the freshness in fish and presents a very good correlation with the storing time of fish [\(Ehira &](#page-5-0) [Uchiyama, 1987; Shahidi, Chong, & Dunajski, 1994](#page-5-0)). However, other indicators like K_i , K_0 (in which adenosine and xanthine are also included), G, and P, which are derived from the K value, can be used. The suitability of one indicator or another depends on the degradation pattern of these metabolites. [Shahidi et al.](#page-5-0) [\(1994\)](#page-5-0) evaluated in seal meat the applicability of all these indicators. They found a good correlation of K, K_i and K_0 for the first 19 days of storage whilst P was linear from 2 to 19 days. G value did not show clear trend during the early storage period (up to 13 days); however, a linear increase in the G values of meat was noticed during a prolonged storage.

Overall, whilst a K value of 20% or lower is a very good quality fish, a value of 60% is the rejection point in the Japanese market and 80% it is in the occidental countries [\(Lin & Morrissey, 1994\)](#page-5-0). On the other hand, values of 15 mg TMA-N/100 g muscle, 30 mg TVB-N/100 g muscle and 200 ppm of histamine, are considered levels that denote fish deterioration ([Huss, 1995\)](#page-5-0).

The cazon fish is, in Mexico, an important and highly appreciated fishing resource. Despite that, there are few studies that evaluate postmortem changes in its muscle. This study however, informs about the changes and effects on the quality of the cazon fish muscle when it is under the appropriate post-capture handling conditions (0 °C). Such information is necessary for a proper postcapture handling and processing. Findings suitable applications could generate higher profitable margins for producer and improve development to this fishery.

2. Materials and methods

2.1. Collecting and handling sample

During May in 2006, live cazon fish Mustelus lunulatus were collected in the Gulf of California. A 24 live specimen sample was collected with a gill net 40 km. away from Bahia de Kino in Sonora, Mexico. Immediately after the catch fish were gutted and washed with sea water to be placed on ice inside a hermetic cooler and be transported to the Laboratorio de Investigacion en Alimentos of the Universidad de Sonora. Time spent from the catch to the reception of the sample in the lab was 5 h. Already in the lab, specimens were filleted and fillets were packed in polythene bags to be later on placed inside a hermetic cooler in alterning layer of ice–fillet–ice. Fillets were subjected to 18 day storage on ice study under these conditions. During this time, the cooler was drained and refilled with ice when was necessary to do it.

ATP and related compounds, K value, trimethylamine (TMA-N), total volatile bases (TVB-N), pH, water-holding capacity (WHC), texture and colour were carried out to assess postmortem changes in the cazon fish muscle. All biochemical and chemical determinations were made on days 0, 3, 6, 9, 12, 15 and 18, whilst physical determinations were only on days 0, 6, 12 and 18. An $n = 6$ was used for each determinations. It is important emphasise that whilst samples were frozen at -86 °C to be later on analysed for biochemical postmortem, the physical changes were monitored the sampling day. In the case of day 0 samples, they correspond to 5 h post-capture sample.

2.2. Analyses

2.2.1. ATP, related compounds, and K value

Determinations of nucleotides and related compounds were carried out by a reverse phase high-performance liquid chromatography procedure ([Ryder, 1985](#page-5-0)). The identification of nucleotides, nucleosides, and bases was made by comparing their retention times with those of commercially obtained standards and by adding or spiking of standards. The K value was calculated as the percent rate of HxR and Hx to the sum of ATP and degradation products as follows:

 $\%K = \left[\frac{HxR + Hx}{4R + ADP + AMP + IMP + HxR + Hx}\right] \times 100.$

2.2.2. Chemical analyses and pH

TMA-N, TVB-N, and pH were determined by following previously described methods [\(Woyewoda, Shaw, Ke, & Burns, 1986\)](#page-5-0).

2.2.3. Texture

Shear force was used to evaluate texture in the muscle of cazon fish. Shear force was measured in the muscle using a Warner–Bratzler blade in a universal testing machine (Model 1130, Instron Corp., Canton, MA) equipped with a 50-kg cell. Speed was set at 20 cm/min and shearing force was transversally applied to the direction of the muscle fibres. Standardised cuts ($10 \times 10 \times 20$ mm) were used and necessary force (kg_f) to shear the muscle was recorded.

2.2.4. WHC

Water-holding capacity was measured using a standard methodology [\(Cheng, Hamann, Webb, & Sidwell, 1979\)](#page-5-0). WHC was expressed as ''loss of water" which was the percentage of weight lost by the sample compared to the initial weight.

2.2.5. Colour

Changes of colour in the cazon fish muscle were determined with a tri-stimulus colorimetre (Model CR-300, Minolta Co., New York, NY). Measurements were taken in the surface of the muscle.

2.2.6. Statistical analysis

Analyses were performed with the NCSS 2000 statistics software (NCSS, Kaysville, UT). Descriptive statistics (mean, standard deviation and coefficient of variation), one-way ANOVA, multiple comparison with the Tukey test and linear regression analysis were applied. Significance level was set at 5%.

3. Results and discussions

3.1. Nucleotide catabolism and K value

The ATP concentration as well as its products of degradation until hipoxantine have been widely used as fish muscle freshness indicator ([Massa et al., 2005](#page-5-0)). In Fig. 1 it is shown the degradation pattern of ATP and its products until hipoxantine in the cazon fish muscle stored at 0 °C for 18 days. The mean total molar concentration (TMC) for ATP and derivatives was $7.8 \pm 1.7 \mu$ mol/g. This value is similar to 8.0μ mol/g reported in black skipjack muscle ([Mazorra-Manzano, Pacheco-Aguilar, Diaz-Rojas, & Lugo-Sanchez,](#page-5-0) [2000](#page-5-0)), but lower than 9.3 μ mol/g in yellowtail (Seriola quinqueradiata) [\(Murata & Sakaguchi, 1986](#page-5-0)). Variations in TMC are due to species, season, physiological condition, feeding, etc. [\(Ocano-Higu](#page-5-0)[era et al., 2006](#page-5-0)). There were found ATP initial values of 0.15 ± 0.04 µmol/g, similar result to the registered by [Özogul and](#page-5-0) [Özogul \(2002\)](#page-5-0) who reported a value of 0.1 μ mol/g of ATP in rainbow trout (Oncorhychus mykiss). Literature reports that the ATP is degraded within the first 24 h postmortem [\(Haard, 1992](#page-5-0)); however, ATP low initial values found in the cazon fish's muscle could be mainly attributed to the energy consumption for the struggle in the net. Same behaviour was previously reported in Sierra (Scomberomorus Sierra) by [Castillo-Yáñez, Pacheco-Aguilar, Mar](#page-5-0)[quez-Rios, Lugo-Sánchez, and Lozano-Taylor \(2007\).](#page-5-0) On the other hand, since the net was left in the depths of sea for 18 h, fishermen caught a much fatigued and stressed cazon fishes which has as a result an exhausted ATP due to the wrangling of the animal in the net; therefore, the predominant nucleotide in the cazon fish muscle after the catch was the IMP with an initial value of 6.35 ± 0.67 µmol/g. The high concentration of IMP in this study reported a rapid degradation of ATP to IMP. Because of the rapid disappearance of ATP in the muscle, levels of adenosine-5'-triphosphate (ATP), adenosine-5'-diphosphate (ADP), adenosine-5'-monophosphate (AMP) did not show significant changes as regards the storage time ($P \ge 0.05$). Furthermore, it was found that the inosine 5'-monophosphate (IMP) concentration decreased in accordance with equation $y = -0.25x + 5.70 + 0.024x^2$ with an $r^2 = 0.8$, $(P < 0.05)$, such degradation is different to the usually showed by other species where it is linearly presented, correlating quite well with the freshness reduction and the sensorial acceptability ([Howgate, 2005\)](#page-5-0). On the other hand, HxR concentrations $[y = -0.128x + 0.4, r^2 = 0.7, P < 0.05]$ and Hx $[y = 0.056x + 0.117,$ r^2 = 0.9, P < 0.05] were significantly increased (P < 0.05) during storage time. Sometimes, IMP degradation or HxR and/or Hx accumulation prove to be a good indicator in the freshness reduction. All previously mentioned effects are due to the linearity how these compounds are degraded or formed. In this study, the Hx accumulation could be used as a freshness indicator due to the good correlation of its formation regarding the storage time.

Fig. 1. ATP and related endogenous degradation products in the muscle of cazon fish (Mustelus lunulatus) stored at 0 °C for 18 days. Data point are the mean of n = 6 for each sampling day. Bars represent the standard deviation. ATP = adenosine-5'triphosphate, ADP = adenosine-5'-diphosphate, AMP = adenosine-5'-mophosphate, $IMP = inosine-5'-monophosphate, HxR = inosine and Hx = hypoxantine.$

In Fig. 1 can be observed that this species is a moderate HxR producer, because during the sixth day of storage, this compound reached a value of 1.71 ± 0.26 umol/g. These values are consistent with the behaviour registered by [Iwamoto, Yamanaka, Watabe, and](#page-5-0) [Hashimoto \(1987\)](#page-5-0) for plaice who reported a value close to 1 μ mol/ g of this compound. Likewise, it is noted that this species is a slower producer of hypoxanthine because values of $1.09 \pm$ 0.38 μ mol/g were obtained until the 18th day of storage.

Literature reported that IMP and AMP are responsible for sweetness, fresh fish muscle characteristic [\(Howgate, 2005](#page-5-0)), whilst HxR and Hx production is related to the loss of freshness and flavour (bitterness) in some fish species. In this study, the Hx accumulation in cazon fish muscle after day 18 (1.3 \pm 0.1 μ mol/g) reflects the initial phase of autolytic deterioration as well as bacterial spoilage ([Woyewoda et al., 1986](#page-5-0)).

Quantification of the ATP concentration and its products of degradation until Hx were used as basis to calculate the K value or freshness index, which is defined as the ratio (x 100) of nonphosphorylated ATP breakdown products to the total ATP breakdown products and has been used as a freshness measure in many species ([Ehira & Uchiyama, 1987\)](#page-5-0). Fig. 2 shows a significant and linear increase $[y = 2.94x + 2.28, r^2 = 0.97, P < 0.05]$ in K value of the cazon fish muscle during the storage period, from a value (not adjusted) of $1.1 \pm 0.6\%$ (day 0) to a final value of $58.9 \pm 7.4\%$ (day 18). Using this prediction equation, the K value at time 0 was 2.3%.

[Saito, Arai, and Matsuyoshi \(1959\)](#page-5-0) described fishing products with K values lower than 20% as very fresh ones, with less than 50% as moderately fresh, and higher than 70% as not fresh. Based on these K value categories and using the prediction equation, the cazon fish muscle under the experimental conditions of this study can be considered very fresh at least the sixth day, moderately fresh up to day 16.2 and with a minimum freshness until the end of the storage. It is important to stress that to use the classification previously described, it is necessary to have in mind that this rate depends on the species; therefore, it must be calculated for each kind of fish.

In this study no spoilage-related odours were detected even at day 18; however, starting at day 18 (K value = 70.6%), a change in the characteristic initial fresh odour was detected. [Ehira and](#page-5-0) [Uchiyama \(1987\)](#page-5-0) pointed out that spoilage odour in fish kept at $0 °C$ did not appear before day 17, where the viable bacterial count was on the order of 10^5 CFU/g, the minimum of the range considered to be the threshold for spoilage.

3.2. Total volátile base and trimethylamine

Fish decomposition is a progressive proteolysis of the muscle tissue brought about primarily by the action of microorganisms

Fig. 2. K value in the muscle of cazon fish (Mustelus lunulatus) stored at 0° C for 18 days. Data point are the mean of $n = 6$ for each sampling day. Bars represent the standard deviation.

and, to a lesser extent, by autolytic enzymes. Because the changes during the decomposition are known to be very complex, a single chemical index may not be a reliable indicator for a particular sample of fish [\(Castillo-Yáñez et al., 2007\)](#page-5-0).

TMA-N and TVB-N analysis are accurate in predicting the fish spoiling process. These analyses have been traditionally used as indicators of quality in fishing products stored on ice. TVB-N is a term that includes measurement of trimethylamine, dimethylamine, ammonia and other compounds associated with seafood spoilage and increases as spoilage progresses. For several fish species, TVB-N values were reported to increase curvilinearly or linearly with the time and a level of 30 mg. Muscle TVB-N/100 g has been considered the upper limit above which some fishing products are considered spoiled and unfit for human consumption ([Gökodlu et al., 1998\)](#page-5-0). In this study the average values of TVB-N content were 40.0 ± 3.3 and 51.0 ± 5.0 mg of Nitrogen/100 g of muscle at 0 and 18 days stored in ice, respectively. This increasing may be because of the ammonia production in the muscle during the storage time, due to the fact that TVB-N levels still rise as a result of the $NH₃$ formation and other volatile amines ([Mazorra-](#page-5-0)[Manzano et al., 2000](#page-5-0)). Considering the information above, our results indicate that from the beginning of the storage cazon fish muscle did not fit human consumption; however, this one is considered fresh because it was recently caught despite the high content of TVB-N. The TVB-N high initial value may be the result of the high content of nitrogenous compounds such as urea (not measured), which degrades to ammonia, quantified compound inside the TVB-N. Urea is a characteristic compound of elasmobranchs like sharks and cazon fishes, where can be found concentrations of 2000 mg/100 g of muscle ([Huss, 1995](#page-5-0)). However, further studies are required to explain the nature of the high initial levels of TVB-N in cazon fish muscle.

In another hand, TVB-N and spoilage depend on the species and the TVB-N cut-off value of 30 mg/100 g of muscle may not coincide with organoleptic measures of spoiled fish. [Hozbor, Saiz, Yeannes,](#page-5-0) [and Fritz \(2006\)](#page-5-0) working with salmon (Pseudopercis semifasciata) studied the TVB-N in relation to bacterial deterioration. They detected 35 mg TVB-N/100 g of muscle the first day of storage on ice and 70 mg TVB after 20 days on ice. [McCarthy, Ellis, Silvia,](#page-5-0) [and Mills \(1989\)](#page-5-0) reported that cod (Gadus morhua) showed some signs of spoilage (odour) when TVB-N was at 64 mg/100 g. [Varga,](#page-5-0) [Keith, Michalik, Sims, and Reiger \(1980\)](#page-5-0), using an experienced test panel and a chemical analysis to assess freshness of cod fillets, showed a TVB-N value of 20 mg/100 g for fresh fish, 60 mg/100 g for marginal fish, and >80 mg/100 g for inedible fish. Therefore, the content of BVT-N in which a marine product is considered deteriorated must be set for each species.

TMA-N is often used as an index in assessing the shelf-life and keeping quality of fishing products because rapidly accumulates in the muscle under refrigerated conditions. The TMA-N production in fish tissue during cold storage could be used as an indicator of bacterial activity and it is an accepted deterioration measure. The pungent odour of spoiled fish has been often related to TMA tissue levels, also with the number of spoiling organisms present in many fish species and the rejection limit is usually from 5 to 10 mg TMA-N/100 g muscle [\(El Marrakchi, Bennour, Bouchriti, Hamama, &](#page-5-0) [Tagafait, 1990\)](#page-5-0). In this study, the production of TMA-N followed a similar pattern to TVB-N when it was stored in ice where a significant increase was observed $(P < 0.05)$ with time. TMA-N content day 0 was 0.62 ± 0.07 mg TMA-N/100 g of muscle whilst 2.84 ± 0.61 mg of TMA-N was detected day 18, results agreed with those in literature. [Pacheco-Aguilar, Lugo-Sánchez, and Robles-](#page-5-0)[Burgueño \(2000\)](#page-5-0) reported for the winter Monterey sardine muscle initial values of 0.818 \pm 0.33 mg/100 g at 0 °C whereas at the end of the storage (day 15) a value of 1.62 mg of TMA-N/100 g of the muscle was obtained. In another hand [Bilinski, Jonas, and Peters](#page-5-0) [\(1983\)](#page-5-0) reported for dogfish (Squalus acanthias) stored at 0 \degree C average values of 0.4 and 0.7 mg of TMA-N/100 g, days 0 and 20, respectively, whilst [Ryder, Buisson, Scott, and Fletcher \(1984\)](#page-5-0), reported 2 mg TMA-N/100 g of muscle in jack mackerel (Trachurus novaezelandie) after remaining in ice 12 days. Afterwards, they found a drastic increase in TMA-N associated with a viable bacterial count (VBC) higher than 10^6 CFU/g of muscle that confirms the TMA-N usefulness as indicator of the bacterial spoilage onset rather than indicator of freshness. Because TMA-N levels did not exceed the rejection limits of day 18 cited in the literature, our results suggested that the cazon fish muscle kept an edible quality during at least 18 days of storage. Our experimental data suggested that microbial activity was not responsible for the TVB-N initial content in accordance with the TMA-N initial value.

3.3. pH

Fig. 4 shows changes in the pH of the cazon fish muscle during the 18 days it remained in ice. Day 1 pH was 6.43 ± 0.04 . This value is similar to 6.35 ± 0.5 reported by [Barnett, Nelson, and Poysky](#page-5-0) [\(1991\)](#page-5-0) for pink salmon stored at 2 °C; nevertheless, it is lower than the reported by [Love \(1976\)](#page-5-0) for fishing products after being caught, which is found between values 6.7 and 7.0. Likewise, it is lower than 6.9 reported by [Kristoffersen et al. \(2006\)](#page-5-0) for the Atlantic cod (Gadus morhua L). Variations amongst the initial values of pH may due to the species, season, diet, level of activity or stress during the catch and type of muscle.

In the same figure it can be noticed that pH significantly increased ($P < 0.05$) with regards to the storage time, reaching a value of 6.78 ± 0.1 the day 18. This suggests bacterial growth as a result of the production of ammonia and other volatile bases. How-

Fig. 3. Postmortem changes in TMA and TVB-N in the muscle of cazon fish (Mustelus lunulatus) stored at 0 °C during 18 days. Data point are the mean of $n = 6$ for each sampling day. Bars represent the standard deviation.

Fig. 4. Postmortem changes in pH in the muscle of cazon fish (Mustelus lunulatus) stored at 0 °C during 18 days. Data point are the mean of $n = 6$ for each sampling day. Bars represent the standard deviation.

ever, the final value is even below the pH that indicates damage (pH 7). According to the information above and based on the results obtained in this study, the cazon fish muscle keeps an acceptable quality degree for at least 18 days when stored in ice.

As it was previously described, sharks and cazon fishes are distinguished by presenting high levels of TMAO in their muscle (500–1000 mg/100 g muscle). This compound can be reduced to TMA by bacterial enzymes; however, as it was observed in [Fig. 3,](#page-3-0) TMA-N values were very low, which could be the result of the pH low values observed in the cazon fish because the optimum pH of reducing enzymes of TMAO is 7.2–7.4 ([Castro, Penedo-Pa](#page-5-0)[dron, Caballero-Cansino, Sanjuan-Velazquez, & Millan De Larriva,](#page-5-0) [2006](#page-5-0)).

3.4. Texture and WHC

Lost of texture during the fishing products storage has been reported in the literature ([Sato, Ohashi, Ohtuki, & Kawabata, 1991\)](#page-5-0). Whilst several researchers have associated low muscle pH with tough texture and high drip loss ([De Vido, Paredi, & Crupkin,](#page-5-0) [2001](#page-5-0)), others suggest the involvement of several enzymes in texture deterioration during storage ([Sato et al., 1991\)](#page-5-0). In our study, no significant ($P \ge 0.05$) difference was obtained in the cazon fish muscle for shear force (SF) during the 18 day period (Fig. 5), where a initial value of 7.20 ± 1.22 kg_f was obtained.

On the other hand, a significant $(P < 0.05)$ difference was observed for WHC during the storage period, which decreased from 92.7 \pm 1.9% on day 0 to 86.2 \pm 1.5% the day 18. It is well known that fish meat properties change along decreasing freshness. In addition, magnitude of change depended on the original condition of the muscle [\(Ocano-Higuera et al., 2006](#page-5-0)). Fish muscle usually becomes tougher when accompanied by a progressive loss of fluid and reduction of water-holding capacity. Nevertheless, the WHC has been reported to be a good indicator for fish quality evaluation, its decrement has shown to result in loss of texture ([Sato et al.,](#page-5-0) [1991\)](#page-5-0). This may be possible due to a loss of the myofibrillar protein integrated during storage ([Flores & Bermell, 1984\)](#page-5-0), Our results indicated that denaturation (aggregation and/or hydrolysis) that could have occur in the myofibrillar protein of cazon fish during the period of storage affected its WHC but in accordance with the SF data, did not affect its texture. Further researchs are needed to evaluate the nature of the possible conformational changes in the protein structure of this species muscle during its chilled storage.

Fig. 5. Postmortem changes in texture (shear force) and water-holding capacity (WHC) in cazon fish muscle (*Mustelus lunulatus*) stored at 0 °C for 18 days. Data point are the mean of $n = 6$ for each sampling day. Bars represent the standard deviation.

Fig. 6. Postmortem changes in color parameters in cazon fish muscle stored at 0 \degree C for 18 days. Data point are the mean of $n = 6$ for each sampling day. Bars represent the standard deviation.

3.5. Colour

Colour is one of the most important parameters used to evaluate the quality of fishing products. [Haard \(1992\)](#page-5-0) reported that the initial colour of fishing products changed during the storage in ice affecting the quality. At the same time, determines the acceptance of the product by the consumer. Surface colour parameters for the cazon fish are shown in Fig. 6. There it may be noted that initial values of "L," "a" and "b" were 45.7 ± 1.82 , 0.69 ± 0.46 and 0.94 ± 0.43 , respectively, which placed the product within the red yellow quadrant with a hue angle of 53.71, indicating that muscle has an initial colour towards orange. Whereas "a" and "b" values were very low, the product can be considered opaque. With respect to the storage time, it was found a significant increase ($P < 0.05$) for parameters "a" and "b", which decreased the hue angle to a value of 41.55, indicating that the fillet acquired a more reddish hue at the end of storage.

4. Conclusions

The postmortem behaviour of cazon fish muscle indicated that both endogenous and microbial processes could be controlled with appropriate post-capture handling practices. The biochemical, chemical, and physical parameters used in this study proved their usefulness in assessing the quality of the cazon fish muscle. Since K value was linearly increased with respect to the storage time, this can be used as a good indicator for monitoring the loss of the muscle freshness whilst kept in ice. Overall results indicated 18 day minimum shelf-life for the cazon fish muscle handled under the experimental conditions, with no effect on texture characteristics. Based on this information, it is highly recommended that Mexican fishermen modify commercial handling operations because their current procedures are reducing the shelf-life of their products.

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References

Anuario Estadístico de Pesca (2004). Comisión Nacional de Acuicultura y Pesca. Secretaría de Agricultura, Ganadería, Desarrollo Rural, Pesca y Alimentación. México. p. 264.

- Barnett, H. J., Nelson, R. W., & Poysky, F. T. (1991). A comparative study using multiple indices to measure changes in quality of pink salmon during fresh and frozen storage. NOAA Technical Memorandum NMFS F/NWC-208. US Department of Commerce. National Oceanic and Atmospheric Administration. National Marine Fisheries Service. p. 59.
- Bilinski, E., Jonas, R. E. E., & Peters, M. D. (1983). Factors controlling the deterioration of the spiny dogfish Squalus acanthias during iced storage. Journal of Food Science, 48, 809-812.
- Castro, P., Penedo-Padron, J. A., Caballero-Cansino, M. J., Sanjuan-Velazquez, E., & Millan De Larriva, R. (2006). Total volatile base nitrogen and its use to assess freshness in European sea bass stored in ice. Food Control, 17, 245–248.
- Castillo-Yáñez, F. J., Pacheco-Aguilar, R., Marquez-Rios, E., Lugo-Sánchez, M. E., & Lozano-Taylor, J. (2007). Freshness loss in sierra fish (Scomberomorus sierra) muscle stored in ice as affected by postcapture handling practices. Journal of Food Biochemistry, 31(1), 56–67.
- Cheng, C. S., Hamann, D. D., Webb, N. B., & Sidwell, V. (1979). Effects of species and storage time on minced fish gel texture. Journal of Food Science, 44, 1087–1092.
- De Vido, D. M. N., Paredi, M. E., & Crupkin, M. (2001). Postmortem changes in the adductor muscle of scallop (Chlamys tehuelchus) in chilled and frozen storage. Journal of Aquatic food Product Technology, 10(3), 49–59.
- Ehira, S., & Uchiyama, H. (1987). Determination of fish freshness using the K value and comments on some other biochemical changes in relation to freshness. In D. E. Kramer & J. Liston (Eds.), Seafood quality determination (pp. 185–193). Amsterdam: Elsevier Science.
- El Marrakchi, A., Bennour, M., Bouchriti, N., Hamama, A., & Tagafait, H. (1990). Sensory, chemical, and microbiological assessments of Moroccan sardines (Sardina pilchardus) stored in ice. Journal of Food Protection, 53, 600–605.
- Flores, J., & Bermell, S. (1984). Propiedades funcionales de las proteínas miofibrilares: Capacidad de retención de agua. Agroquímica y Tecnología de Alimentos, 24(2), 151–158.
- Gökodlu, N., Özden, Ö., & Erkan, N. (1998). Physical, chemical and sensory analyses of freshly harvested sardines (Sardina pilchardus) stored at 4 °C. Journal of Aquatic Food Product Technology, 7(2), 5–15.
- Haard, N. (1992). Biochemistry and chemistry of color and color change in seafood. In J. Flinck, Advances in seafood biochemistry, composition and quality (pp. 305– 360). Louisiana: Technomic Publishing Co.
- Hozbor, M. C., Saiz, A. I., Yeannes, M. I., & Fritz, R. (2006). Microbiological changes and its correlation with quality indices during aerobic iced storage of sea salmon (Pseudopercis semifasciata). LWT – Food Science and Technology, 39, 99–104.
- Howgate, P. (2005). A review of the kinetics of degradation of inosine monophospate in some species of fish during chilled storage. International Journal of Food Science and Technology, 41, 341–353.
- Huss, H.H. 1995. Quality and Quality Changes in fresh fish. FAO. Fisheries Technical Paper 348. Food and Agriculture of the United Nations. (p. 202). Rome Italy.
- Iwamoto, M., Yamanaka, H., Watabe, S., & Hashimoto, K. (1987). Effect of storage temperature on rigor-mortis and ATP degradation in plaice (Paralichthys olivaceus) muscle. Journal of Food Science, 52, 1514–1517.
- Kristoffersen, S., Tobiassen, T., Esaiassen, M., Olsson, G. B., Godvik, L. A., Seppola, M. A., et al. (2006). Effects of pre-rigor filleting on quality aspects of Atlantic cod (Gadus morhua L.). Aquaculture Research, 37(15), 1556–1564.
- Lin, D., & Morrissey, M. T. (1994). Iced storage characteristics of Northern squawfish (Ptychocheilus oregonensis). Journal of Aquatic Food Product Technology, 3, 25–43.
- Love, R. (1976). Processing cod: The influence of season and fishing ground. Torry Advisory Note No. 71. Aberdeen, Scotland: Torry Research Station.
- Massa, A. E., Palácios, D. L., Paredi, M. A., & Crupkin, M. (2005). Postmortem changes in quality indices of ice stored flounder (Oaralichthys patagonicus). Journal of Food Biochemistry, 29, 570–590.
- Mazorra-Manzano, M. A., Pacheco-Aguilar, R., Diaz-Rojas, E. I., & Lugo-Sanchez, M. E. (2000). Postmortem changes in black skipjack muscle during storage in ice. Journal of Food Science, 65, 774–779.
- McCarthy, H. T., Ellis, C. P., Silvia, M. L., & Mills, B. (1989). Comparison of volatile acid number test with enzymatic acetic acid assay for assessment of seafood Quality. Journal of the Association of Official Analytical Chemists, 72, 828–834.
- Murata, M., & Sakaguchi, M. (1986). Storage of yellowtail (Seriola quinquerediata) white and dark muscles in ice: Changes in content of adenine nucleotides and related compounds. Journal of Food Science, 55, 321–326.
- Ocano-Higuera, V. M., Maeda-Martínez, A. N., Lugo-Sánchez, M. E., & Pacheco-Aguilar, R. (2006). Postmortem biochemical and textural changes in the adductor muscle of catarina scallop stored at 0° C. Journal of Food Biochemistry, 30(4), 373–389.
- Özogul, Y., & Özogul, F. (2002). Degradation products of adenine nucleotides in rainbow trout (Oncorhynchus mykiss) stored in ice in modified atmosphere packaging. Turkish Journal of Zoology, 26, 127–130.
- Pacheco-Aguilar, R., Lugo-Sánchez, M. E., & Robles-Burgueño, M. R. (2000). Postmortem biochemical and functional characteristic of Monterey sardine muscle stored at 0° C. Journal of Food Science, 65, 40–47.
- Ryder, J. M., Buisson, D. H., Scott, D. N., & Fletcher, G. C. (1984). Storage of New Zealand jack mackerel (Trachurus novaezelandiae) in ice: Chemical, microbiological and sensory assessment. Journal of Food Science, 49, 1453–1456.
- Ryder, J. M. (1985). Determination of adenosine triphosphate and its breakdown products in fish muscle by high-performance liquid chromatography. Journal of Agriculture and Food Chemistry, 33, 678–680.
- Saito, T., Arai, K., & Matsuyoshi, M. (1959). A new method for estimating the freshness of fish. Bulletin of the Japanese Society of Scientific Fisheries, 24, 749–750.
- Sato, K., Ohashi, C., Ohtuki, K., & Kawabata, M. (1991). Type V collagen in trout (Salmo gairdneri) muscle and its solubility change during chilled storage of muscle. Journal of Agriculture and Food Chemistry, 39, 1222–1225.
- Shahidi, F., Chong, X., & Dunajski, E. (1994). Freshness quality of harp seal (Phoca groenlandica) meat. Journal of Agriculture and Food Chemistry, 42, 868–872.
- Varga, S., Keith, R. A., Michalik, P., Sims, G. G., & Reiger, L. W. (1980). Stability of lean and fatty fish in hypobaric storage. Journal of Food Science, 45, 1487–1491.
- Woyewoda, A. D., Shaw, S. J., Ke, P. J., & Burns, B. G. (1986). Recommended Laboratory Methods for Assessment of Fish Quality. Canadian Technical Report of Fisheries and Aquatic Sciences, No. 1448. Halifax, N.S.