ORIGINAL RESEARCH

Changes on quality parameters and sensory attributes of the Patagonian red octopus (*Enteroctopus megalocyathus*) meat under different postharvest treatments

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ABSTRACT. *Enteroctopus megalocyathus* is an important commercial resource harvested by small-scale fisheries from Argentina and Chile, with limited access to cooling preservation methods. In this sense, the use of alternative postharvest conservation methods could be useful to preserve the good quality of the raw product. The effect of postharvest treatments using seawater immersion, flaked ice, 0.1% acetic acid, and a control was investigated on raw octopus during storage at 4 °C for seven days. Under these treatments, changes in physical, chemical, microbiological parameters and sensory attributes were evaluated. Results showed that for control and seawater treatments, octopus became unacceptable at the third day. Ice and 0.1% acetic acid treatments exhibited better physical, chemical and microbiological quality parameters along the storage days. Based on sensory attributes, octopus meat immersed in 0.1% acetic acid remained within the limits of acceptability until the fifth day, while the ice treatment extended the initial quality at least for seven days. Thus, 0.1% acetic acid would become an economical and easily applicable method during postharvest handling of *E. megalocyathus* fishery.

Key words: Post-capture quality, octopus, artisanal fishing, acetic acid, quality parameters, sensory attributes.

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Cambios en los parámetros de calidad y atributos sensoriales de la carne del pulpo colorado patagónico (*Enteroctopus megalocyathus*) bajo diferentes tratamientos poscosecha

RESUMEN. *Enteroctopus megalocyathus* es un importante recurso comercial capturado por pesquerías de pequeña escala de Argentina y Chile, con acceso limitado a métodos de preservación con frío. En este sentido, el uso de métodos alternativos de conservación poscosecha podría ser útil para preservar la buena calidad del producto crudo. Se investigó el efecto de los tratamientos poscosecha mediante inmersión en agua de mar, hielo en escamas, ácido acético al 0,1% y un control en pulpo crudo durante el almacenamiento a 4 °C durante siete días. Bajo estos tratamientos, se evaluaron cambios en parámetros físicos, químicos, microbiológicos y atributos sensoriales. Los resultados mostraron que para el tratamiento control y agua de mar, el pulpo se volvió inaceptable al tercer día. Los tratamientos con hielo y ácido acético al 0,1% presentaron mejores parámetros de calidad física, química y microbiológica a lo largo de los días de almacenamiento. Con base en los atributos sensoriales, la carne de pulpo sumergida en ácido acético al 0,1% se mantuvo dentro de los límites de aceptabilidad hasta el quinto día, mientras que el tratamiento con hielo extendió la calidad inicial al menos durante siete días. Por tanto, el ácido acético al 0,1% podría ser un método económico y fácilmente aplicable durante el manejo poscosecha en la pesquería de *E. megalocyathus*.

Palabras clave: Calidad poscaptura, pulpo, pesca artesanal, ácido acético, parámetros de calidad, atributos sensoriales.

INTRODUCTION

On a global scale, official statistics on cephalopod landings are dominated by the large-scale squid fisheries; notwithstanding, a wide range of small-scale fisheries for coastal octopuses, cuttlefish and squid exist throughout the world (Boyle and Rodhouse 2005; FAO 2018). Small-scale octopus fisheries are poorly documented, but they make a vast contribution to the well-being of coastal communities (Boyle and Rodhouse 2005; Pierce and Portela 2014; Emery et al. 2016). In addition, the growing worldwide popularity of some traditional cuisines in recent years, coupled with a good nutritional quality, have helped boosting the octopus demand (Arvanitoyannis and Varzakas 2009; FAO 2018; Zamuz et al. 2023).

The Patagonian red octopus, *Enteroctopus megalocyathus* (Gould, 1852), is a species of subantarctic origin inhabiting the coasts of the southern tip of South America, from ~ 42° S southward, in the Argentine (Atlantic Ocean) and Chilean (Pacific Ocean) Patagonia. It is a relatively large benthic species, reaching 1.20 m of total length and more than 5 kg of total weight (Ré 1998; IFOP 2010), representing an important commercial resource in both Argentine and Chilean coasts. This species is harvested by small-scale fisheries operating by diving. In Argentina, red octopus is also extracted from rocky intertidal shores during low tides (Ortiz et al. 2011; Ortiz and Ré 2019). In this country, *E. megalocyathus* was historically sold into the informal markets for local consumption. However, due to the high acceptance of the meat and the high commercial value attained in the market, the fishing industry started getting interested in this species.

To obtain a final competitive product, the industrialization process of marine products requires various industrial operations starting with the good quality of the raw material (Samples 2014). Due to its high autolytic activity, octopuses are usually highly perishable, and their sensory quality attributes decrease quickly after capture, tree times faster than squids (Hurtado et al. 2001; Atrea et al. 2009; Shalini et al. 2015). Enzymatic and chemical reactions are usually responsible for the initial loss of freshness, whereas microbial activity is responsible for the obvious spoilage and thereby defines the product's shelf life (Gram and Huss 1996).

Because octopus post-mortem deterioration has high kinetics, the application of postharvest handling treatments is crucial to maintain the good quality and safety of the final product (Ashie et al. 1996; Mouritsen and Styrbæk 2018). Cooling is the most common and effective treatment used to extend the shelf life of seafood (Samples 2014). However, most of the *E. megalocyathus* intertidal fishing sites are located in rural areas, far from the cities. In fishing areas, fishermen do not have access to ice and they do not apply any other post capture preservation treatment until the octopuses are frozen. The lack of post-harvest preservation treatment in the *E. megalocyathus* catches was also observed in Chile on board of the artisanal boats and at loading ports (Chong et al. 2001; IFOP 2010). The lack of cooling methods was also seen in octopus artisanal fisheries from India. Before being sold, octopus catch is cleaned-up and then sun-dried (Aditi and Deepak 2013), an old traditional practice done to obtain dried fish (Jain and Pathare 2007). These conditions would promote decomposition of the food, affect the quality and safety of the final product, the retail prices and, in consequence, the whole fishery.

In the artisanal fisheries, packing seafood in ice on board of small fishing vessels is a labor-intensive task, while onboard mechanical refrigeration requires more specialized installations and is a relatively costly system (Shawyer and Pizzali 2003). Considerable research has been focused on the use of various methods to preserve or extend the shelf life, while ensuring the safety of fresh seafood products (Márquez-Ríos et al. 2007; Pacheco-Aguilar et al. 2008; Atrea et al. 2009; Ozogul et al. 2010; Zamuz et al. 2023). The use of sodium salts or organic acids, such as acetic acid have been used to control microbial growth, improve sensory attributes and extend the shelf life of various food systems, representing an easy and economical alternative with low toxicity for humans (Mitsuda et al. 1980; Kurita and Koike 1982; Sallam 2007; Ozogul et al. 2010; Manimaran et al. 2016, Zamuz et al. 2023).

Quality parameters are important features when assessing seafood shelf life. Seafood quality decreases due to a complex process in which physical, chemical and microbiological forms of deterioration are implicated (Gram and Huss 1996). Commercial decisions as whether to accept or reject a product depend on these quality parameters, and their change in the sensorial attributes. In addition, sensory attributes are one of the most important characteristics when evaluating the freshness of a product because this attribute is directly related to the consumers preferences (Arvanitoyannis et al. 2005).

There is a lack of information on quality changes and storage preservation occurring during post-harvest handling in *E. megalocyathus*. Moreover, other than ice, there are not studies that evaluate post-capture conservation techniques in octopuses. In this sense, an evaluation of alternative post-harvest conservation methods could be useful for octopus fisheries with limited access to cooling preservation methods. Thus, the objective of the present work was to evaluate the effect of different post-harvest treatments (control, seawater, ice and 0.1% acetic acid) on the changes in physical and chemical properties, total microbial counts and sensory attributes of the Patagonian red octopus meat.

MATERIALS AND METHODS

Sample preparation

Specimens were collected during sampling trips carried out during spring 2017 in Golfo Nuevo, Patagonia Argentina. Octopuses were caught at depths ranging from 10 to 15 m by scuba diving, using a hook especially designed to capture red octopus. In a time interval of two hours after harvest, octopuses were taken to the laboratory and their total weight was registered to the nearest 1g using a digital scale (Ohaus Scout Pro). Nine octopuses between 850 to 1,500 g of total weight were used. Arm samples of 500-700 g from the different octopuses were used for each treatment. Samples were individually placed in covered plastic containers of 9 l and stored at 4 °C using four conditioning methods: i) control, ii) immersion in filtered seawater (35 mg salt g^{-1}), iii) flake ice (2-3 mm size), and iv) immersion in 0.1% acetic acid (C_2H_4O acetic acid Merck, Buenos Aires, Argentina). The ratio between octopus meat and seawater, acetic acid or ice was 40-60 g l^{-1} depending on the arm weight. Seawater, flaked ice, and acetic acid solution in each container were replaced daily.

Determination of physical-chemical and microbiological parameters

For each treatment, changes in physical, chemical and microbiological parameters were investigated immediately after capture and every 48 h along seven days of storage time.

The total volatile base nitrogen (TVB-N) content present in raw meat samples was determined according to the method developed by Antonacopoulos and Vyncke (1989) and was expressed as TVB-N mg 100 g^{-1} octopus meat.

The water holding capacity (WCH) was determined as 'centrifuge drip'. About 10 g of each arm sample was weighed and centrifuged at 10,000 rpm for 15 min at 4 °C (Sorvall Instruments, Model RC5C; Thermo Scientific, Waltham, MA, USA). Values were calculated on a wet weight basis as $100 \times (A/B)$, where A is the final sample weight after water expulsion, and B is the initial sample weight (Gullian-Klanian et al. 2017).

Drip loss (%) was measured gravimetrically in octopus arm samples weighting 10-15 g each, using the following equation: Drip loss (%) = [(Wi-W)/Wi] × 100, where Wi is weight of the first day and W is weight at each day. Moisture content was determined by drying the sample in an oven at 100 °C up to reach constant weight (AOAC 1990).

The value of pH was determined by homogenizing each muscle sample in distilled water in the ratio 1:5 (w/v). The pH of the homogenate was measured using a pH meter (Hanna Instrument, Smithfield, RI, USA) previously calibrated.

Changes in color were determined by using a Minolta CR14 Instrument (Osaka, Japan) on the skin, in the dorsal side of the arm samples. Data were transformed into L^* (lightness), a^* (redness) and b^* (yellowness) values according to Hunter Lab Scale.

The aerobic plate count was performed according to the AOAC (1990). Samples of 10 g of octopus arm were aseptically homogenized with 90 ml of peptonized solution 0.1%. After homogenization, decimal dilutions of the sample were prepared using the same diluent and were plated onto Plate Count Agar. Each dilution was plated and incubated at 37 °C for 48 h. Results were expressed as number of colony forming units per gram of sample (CFU g^{-1}). All analyses were done in triplicate.

Sensory analysis

For each treatment, fifteen seafood consumers daily evaluated arm octopus samples. They were asked individually to assess samples for color, odor, and overall acceptability by using a 9-point hedonic scale for each sensory attribute: 1, dislike extremely; 2, dislike very much; 3, dislike moderately; 4, dislike slightly; 5, neither like nor dislike; 6, like slightly; 7, like moderately; 8, like very much; 9, like extremely.

Statistical analysis

Results were expressed as mean value ± standard deviation (SD). One-way ANOVA was used to compare each physicochemical parameter among treatments and storage days. Having demonstrated significant differences between the groups, the Tukey test for *post hoc* multiple comparisons was applied. For attributes of the sensory analysis, a Repeated Measures ANOVA was applied. Statistical analysis was performed using software R (R Core Team 2019). Significant differences were defined at $p < 0.05$.

RESULTS AND DISCUSSION

Physicochemical and microbiological parameters

There were significant differences among treatments and during the storage time. A significant increase of TVB-N was observed in control and seawater treatments (Figure 1). Although some reports indicate that TVB-N is not a reliable as index of cephalopod quality (Paarup et al. 2002; Ohashi et al. 1991), for most markets TVB-N is a quality determinant of fresh fish because of its close relationship with sensory scores and bacterial counts (Huss 1998; Jinadasa 2014). In this sense, after three days of storage, TVB-N values for control and seawater treatments exceeded the acceptability limits allowed by the European Commission Regulations (2005) for fishery products (25-35 mg 100 g^{-1}) and by the Argentine Food Code (2019) (35 mg 100 g^{-1}) (Figure 1 A). Likewise, for control and seawater treatments, increments in TVB-N

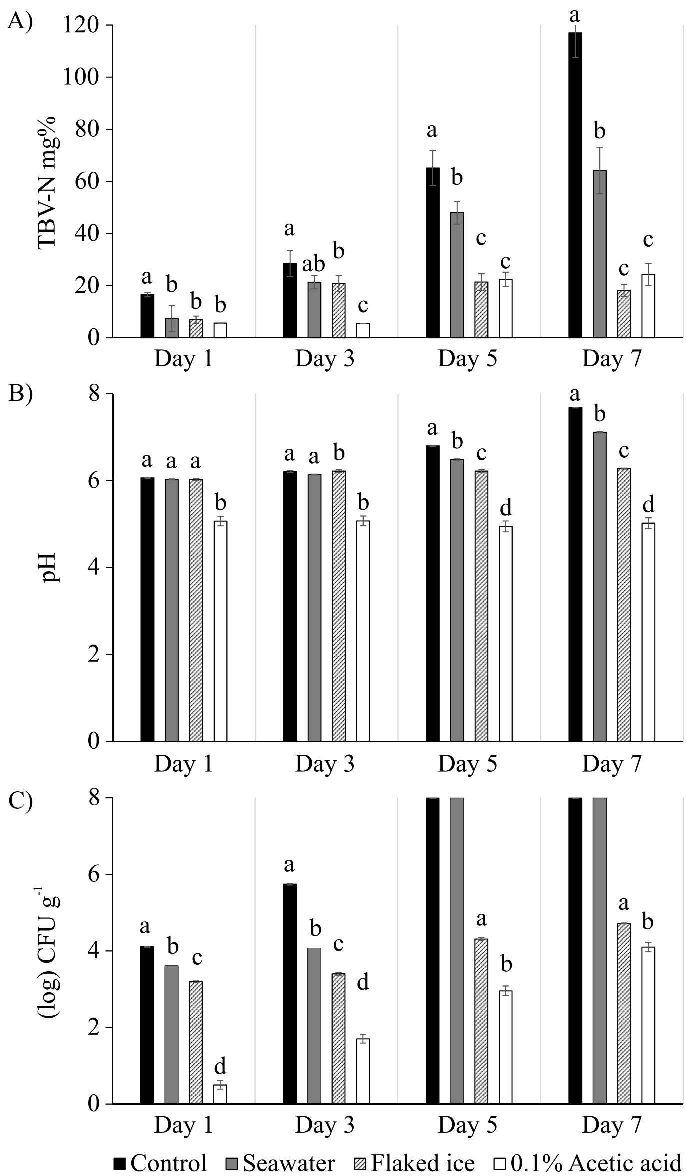

Figure 1. Quality parameters of *Enteroctopus megalocyathus* meat stored at 4 °C as a function of storage time. A) Total volatile base nitrogen (TVB-N) content. B) pH value. C) Agar plate count values. Colony forming unit per gram (CFU g^{-1}). Different letters represent significant differences among treatments for each day.

values were reflected in the pH increase (Figure 1 A and 1 B).

The nitrogen of total volatile bases (TVB-N) quantified in marine products had also been utilized as indicator of bacterial spoilage. High TVB-N values observed for control and seawater treatments were probably because of the high autolytic activity, a common feature in cephalopods (Hurtado et al. 1999; Atrea et al. 2009), and the high proliferation of degenerative flora with the subsequent and rapid decomposition (Márquez-Ríos et al. 2007). For these treatments, the agar plate count exceeding the value of 7 log CFU g^{-1} after the third day of storage (Figure 1 C) is considered by different authors as the upper acceptability limit for marine species (Hurtado et al. 2001; Lougovois et al. 2008; Vaz-Pires et al. 2008; Atrea et al. 2009). In addition, the Argentine Food Code (2019) requires a limit of 7 log CFU g^{-1} for marine food.

For ice and 0.1% acetic acid treatments, TVB-N values showed a significant increment on the third day, and then remained low and stable. Low TVB-N values would be the result of a decrease in bacterial growth due to low temperatures (ice treatment) and low pH (acetic acid treatment). For the 0.1% acetic acid the pH was 5, lower than the others treatments, and remained stable during the storage time (Figure 1 B). Lastly, for ice and 0.1% acetic acid a slight increase in bacterial growth was observed (Figure 1 C). In the case of ice, it is known that bacterial growth is constrained by low temperatures (Graham et al. 1993; Márquez-Ríos et al. 2007; Lougovois et al. 2008), while for acetic acid treatment, the growth of microorganism was restrained probably by the low pH values observed in this treatment (Figure 1 B and 1 C).

There were significant differences among treatments and throughout storage time (Figure 2). Drip loss is an important physical quality parameter, which reflects a decrease in weight in the final product as a consequence of water loss, along with some water-soluble vitamins from the muscle (Mohan et al. 2012). For the control treatment, drip loss of the muscle decreased in average 18.8% from the first to the last day of storage. For seawater and ice treatments, a significant increase in the water content (i.e. an increase in drip loss) was observed (Figure 2 A). Myofibrils proteins are the primary site for water holding in meat (Yu et al. 2022), this would be the case for cephalopods which have a high level of protein content (Mouritsen and Styrbæk 2018). Likewise, Graham et al. (1993) and Zeng et al. (2005) reported an uptake of water for marine species rich in protein when they were treated with ice and refrigerated seawater. For 0.1% acetic acid treatment the drip loss of muscle remained stable. There is no information about the effect of acetic acid on drip loss in shellfish. According to Mitsuda et al. (1980) and Mouritsen and Styrbæk (2018), acid tends to alter muscular proteins producing change in texture of seafood meat. This phenomenon could be attributed to the capacity of acetic acid for partial or total denaturation of myofibril proteins present in octopus (Kariya et al. 1986) affecting the ability to retain water (Baxter and Skonberg 2008).

In addition, WHC has been reported to be a good indicator for the evaluation of seafood quality since its decrease caused texture loss (Pacheco-Aguilar et al. 2008). In the control treatment, WHC decreased the first day and it remained stable onwards. For seawater treatment, a steadily WHC decrease from 95 to 73% during storage was noted (Figure 2 B). This pattern could be related to the increase in growth of microorganisms (Figure 1 C), which probably produced a deterioration of textural proteins leading to a lower WHC (Graham et al. 1993; Baxter and Skonberg 2008). On the other hand, according to Zeng et al. (2005), in marine foods treated with refrigerated seawater, the descent in WHC could be explained by the increase of loosely bound water in the muscle due to the brine uptake (i.e. drip loss increase) (Figure 2 A and 2 B). For the ice treatment, a significantly WHC decrease occurred as from the fifth day of storage, coinciding with the same pattern observed in *Octopus vulgaris* (Hurtado et al. 2001). For 0.1% acetic acid treatment, the WHC showed a slightly difference between the first and the last day of storage. The moisture percentage remained stable in the control treatment, while in seawater, ice, and 0.1% acetic acid, the moisture percentage increased during the last day of storage, probably due to the increase in water content (i.e. drip loss) (Figure 2 C).

Color is one of the most important parameters used to evaluate the quality of seafood (Ocaño-Higuera et al. 2011). Particularly in octopus, the quality sensory attributes rely on skin color and brightness (= lightness) (Barbosa and Vaz-Pires 2004; Gullian-Klanian et al. 2016). Color was af-

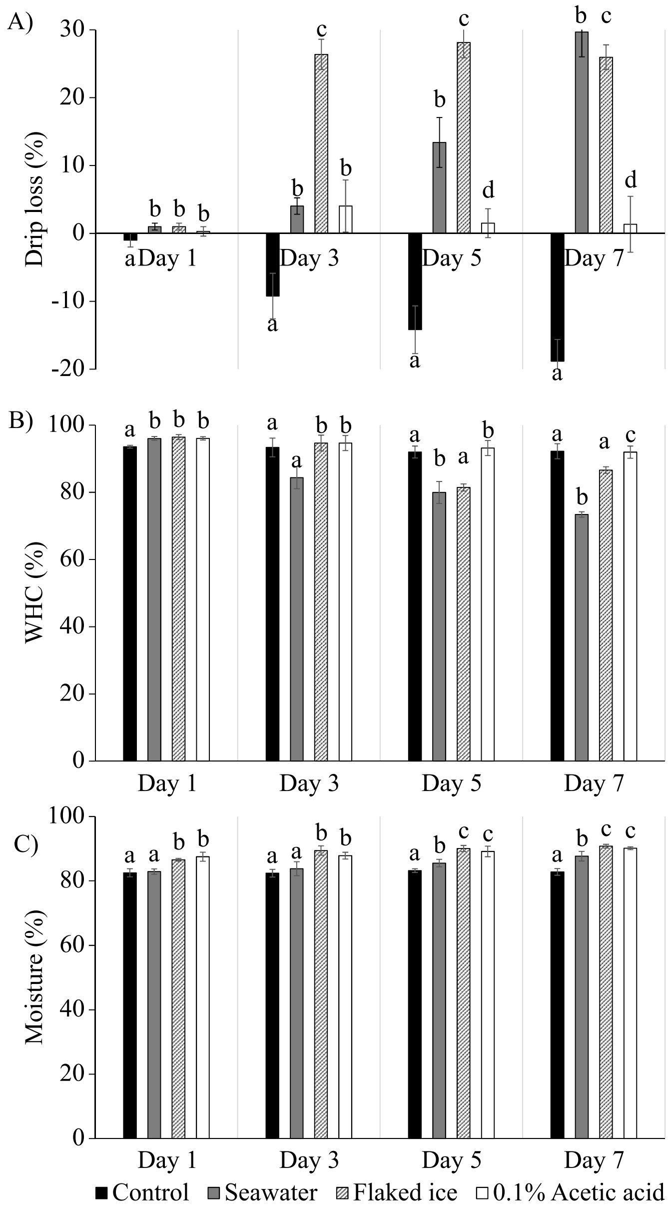

Figure 2. Quality parameters of *Enteroctopus megalocyathus* meat stored at 4 °C as a function of storage time. A) Drip loss percentage. B) Water Holding Capacity (WHC) percentage. C) Moisture percentage. Different letters represent significant differences among treatments for each day.

fected during storage time and according to the treatment applied (Figure 3). For the control, the L^* (lightness) parameter remained stable, while in the seawater treatment it significantly decreased as from the third day. For the ice treatment, a slight increase was observed during the storage time. This treatment showed the higher value of lightness at the end of the storage period. For 0.1% acid acetic, samples L^* remained stable during the storage time.

The reddish-brown color is a morphological at-

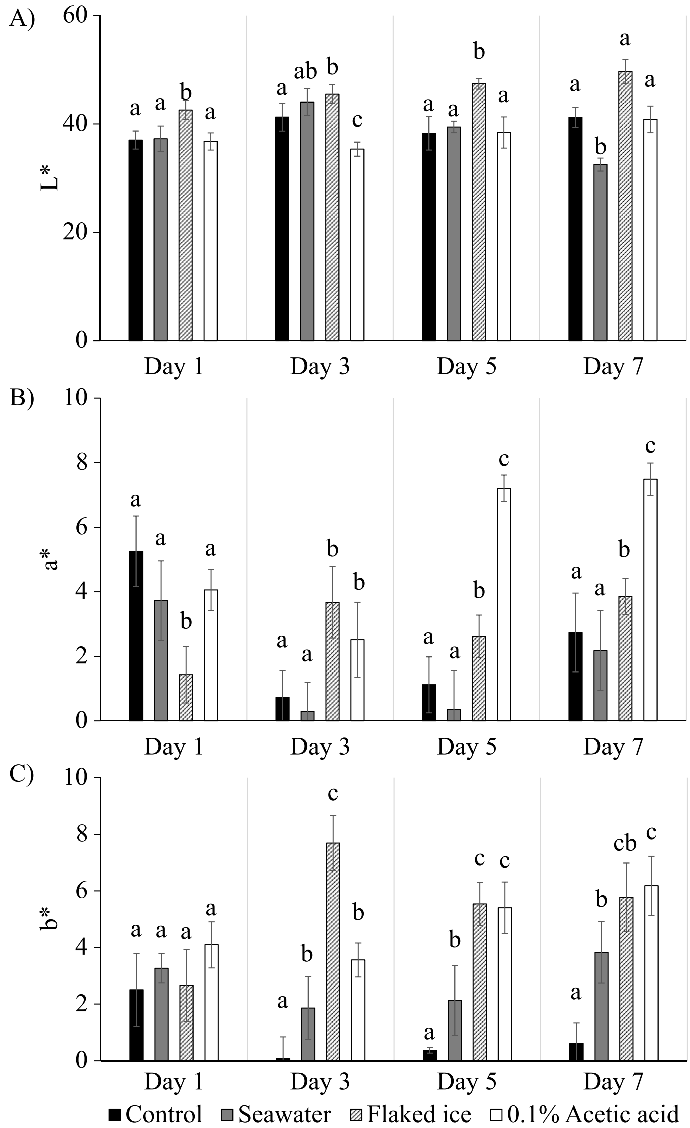

Figure 3. Changes in color parameters L^* (A), a^* (B) and b^* (C) of *Enteroctopus megalocyathus* meat stored at 4 °C. Different letters represent significant differences among treatments for each day.

tribute of *E. megalocyathus* skin (Ré 1998). The a^* parameter (reddish) significantly changed among treatments. For the control and seawater treatments, a significant decrease was observed on the third day, and then the values of this parameter remained stable. For the ice treatment, a^* parameter ranged from 2.5 to 3.5 with a significant increase on the third day. The 0.1% acetic acid treatment duplicated the initial value of the redness index on the seventh day with a significant increase in reddish on the fifth day. These increases in reddish parameter were probably because of alterations in chromatophore organs on the skin. Chromatophore organs consist in a pigment-filled bag with radial muscles and nerves that surround the bag. Combined radial muscles contractions (i.e. chromatophore expanded) or relaxation (i.e. chromatophore contracted) give octopuses their ability to change color (Messenger 2001). Thus, denaturation of muscle proteins produced by low pH would generate the expansion of chromatophore organs, turning the skin more reddish. For yellowness parameter (b^*) in the case of control and seawater treatments, a decrease at third day was observed. For the ice treatment, an increase was noted on the third day while for acetic acid on the fifth day. In these last treatments, yellowness mean values remained high up to the end of storage.

Sensory attributes

Significant differences among treatments and during storage time were observed for sensory attributes (Figure 4). The ice showed the highest values throughout storage. For control and seawater treatments, sensory values ranged from neither like nor dislike (5) to dislike very much (2), and stayed up 5 points only until the second day. These values agree with the highest bacterial counts observed for these treatments (Figure 1 C). Microbial contamination is one of the major cause of fish quality deterioration that occurs from nature as well as during post-harvest processing and marketing (Huss 1998; Majumdar et al. 2023). Octopus samples with ice treatment ranged from like moderately (7) to neither like or dislike (5), and showed values above 5 during all the storage time. These sensory values are in agreement with results from Hurtado et al. (1999), Barbosa and Vaz-Pires (2004), Shalini et al. (2015) and Gullian-Klanian et al. (2016), reporting that the acceptability of whole raw octopus (*O. vulgaris* and *O. maya*) preserved in ice or refrigerated at 0 °C, based on the external sensory attributes, lasted until 6-7 days of storage. In the case of 0.1% acetic acid, sensory values were higher than control and seawater but lower than flaked ice treatment. The average ranged from like slightly (6) to dislike slightly (3), and stayed up 4 points only until the fifth day.

In summary, Patagonian red octopus meat loses quality after capture depending on the post-capture treatment applied. Without post-capture treatment or immersion in seawater, octopus meat attains high values of TBV-N and microbial counts and low score of sensory attributes, giving rise a shelf life of three days. Furthermore, without post-capture treatment octopus meat loses the ability to retain water, which implies a weight loss of the raw product. Samples kept in flaked ice or in 0.1%

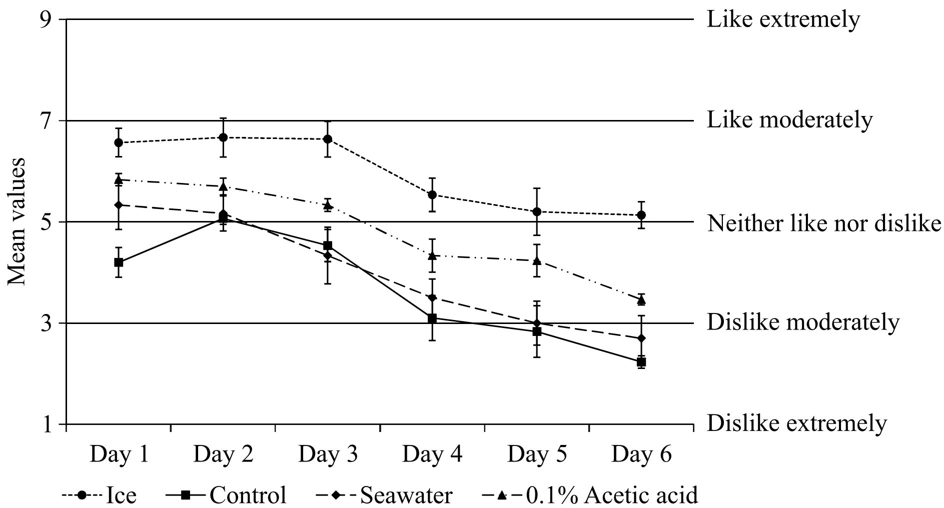

Figure 4. Mean value and standard deviations of sensorial attributes for different post-capture treatments on *Enteroctopus megalocyathus* meat stored at 4 °C.

acetic acid exhibited a restrain of the deterioration process during the storage time, showing similar physical, chemical and microbiological parameters. Based on sensory attributes, octopus meat immersed in 0.1% acetic acid remained within the limits of acceptability until the fifth day, while *E. megalocyathus* meat preserved in flaked ice proved to be the best treatment to extend the initial quality up to at least 7 days. However, availability of ice is a limiting factor in the Patagonian red octopus fishing areas. Thus, 0.1% acetic acid solution would become an easy and economic method to maintain the quality and safety of raw *E. megalocyathus*, and it could be tested in other octopus species in places where fishermen have limited postharvest handling resources.

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Author contributions

Jimena B. Dima: conceptualization, investigation, formal analysis, funding acquisition, methodology, resources, validation, visualization, writing-original draft, writing-review and editing. Martina V. Fiedorowicz Kowal: data curation, investigation. Jorge Castañeda: data curation, investigation. Nicolás Ortiz: conceptualization, investigation, funding acquisition, methodology project administration, resources, supervision, validation, writing-original draft, writing-review and editing.

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