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POLVO CONGELADO COMERCIAL: AVALIAÇÃO DE INDICADORES DE QUALIDADE FÍSICO-QUÍMICA, SENSORIAL E REOLÓGICA

COMMERCIAL FROZEN OCTOPUS: EVALUATION OF PHYSICOCHEMICAL, SENSORY, AND RHEOLOGICAL QUALITY INDICATORS

PULPO CONGELADO COMERCIAL: EVALUACIÓN DE INDICADORES DE CALIDAD FISICOQUÍMICA, SENSORIAL Y REOLÓGICA

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RESUMO

Introdução: O congelamento é essencial para a preservação do polvo, mas pode afetar as suas propriedades sensoriais e a textura. Uma empresa de produtos do mar que comercializa polvo do Nordeste do Pacífico, congelado a bordo dos navios, e polvo fresco do Nordeste do Atlântico, proveniente de uma zona mais próxima das suas operações, questionou a diferença de qualidade e os eventuais benefícios associados.
Objetivo: Este estudo comparou o polvo fresco e congelado do Nordeste do Atlântico com o polvo congelado do Nordeste do Pacífico, que passa por um processo de temperagem antes de ser novamente congelado. O objetivo foi avaliar a qualidade físico-química, sensorial e reológica do polvo, fornecendo informações sobre os efeitos do congelamento e as suas implicações nas decisões comerciais no setor dos produtos do mar.
Métodos: O processo de congelamento envolveu congelamento por ar forçado a -18 °C, glaciagem e armazenamento entre -18 e -22 °C durante uma semana.
Resultados: Foram observadas alterações na textura (menor dureza e fracturabilidade) do polvo congelado do Atlântico em comparação com o fresco. A nível sensorial, o polvo congelado também apresentou menor dureza e elasticidade. Ao longo do período de congelação, os valores de consistência das dispersões proteicas diminuíram, associando os efeitos do congelamento a alterações na qualidade. Por outro lado, o polvo congelado do Nordeste do Pacífico apresentou maior teor de humidade, pH, e os valores mais elevados de dureza e fracturabilidade entre as amostras.
Conclusão: O congelamento impacta negativamente a qualidade do polvo, reduzindo características sensoriais e texturais importantes. Os resultados reforçam a necessidade de otimizar processos e critérios de abastecimento na indústria de produtos de pescado.

Palavras-chave: *octopus* spp.; congelação; qualidade; sensorial; reologia

ABSTRACT

Introduction: Freezing is essential for preserving octopus, but it can affect its sensory properties and texture. A seafood company that markets Northeast Pacific octopus, frozen on board, and fresh octopus from the Northeast Atlantic, sourced from an area closer to its operations, questioned the differences in quality and potential associated benefits.
Objective: This study aimed to address this challenge by comparing fresh and frozen octopus from the Atlantic with frozen octopus from the Pacific, which undergoes a tempering process before being refrozen. The objective was to evaluate the physicochemical, sensory, and rheological quality of the octopus, providing insights into the effects of freezing and its implications for commercial decision-making in the seafood sector.
Methods: The freezing process involved forced-air freezing at -18 °C, glazing, and storage at -18 °C between 22 °C for one week.
Results: Changes in texture (lower hardness and fracturability) were observed in frozen Atlantic octopus compared to the fresh sample. At the sensory level, frozen octopus also showed reduced hardness and elasticity. Over the freezing period, consistency values of protein dispersions decreased, linking freezing effects to changes in quality. On the other hand, the frozen Pacific octopus showed higher moisture content, pH, and the highest hardness and fracturability among the samples.
Conclusion: Freezing negatively impacts the quality of octopus, reducing key sensory and textural characteristics. These findings highlight the need to optimize freezing processes and sourcing strategies in the seafood industry.

Keywords: *octopus* spp.; freezing; quality; sensory; rheology

RESUMEN

Introducción: La congelación es esencial para la conservación del pulpo, pero puede afectar sus propiedades sensoriales y su textura. Una empresa de productos del mar que comercializa pulpo del noreste del Pacífico, congelado a bordo de los buques, y pulpo fresco del noreste del Atlántico, procedente de una zona más cercana a sus operaciones, cuestionó la diferencia de calidad y los posibles beneficios asociados.
Objetivo: Este estudio comparó pulpo fresco y congelado del noreste del Atlántico con pulpo congelado del noreste del Pacífico, que se somete a un proceso de templado antes de volver a ser congelado. El objetivo fue evaluar la calidad fisicoquímica, sensorial y reológica del pulpo, proporcionando información sobre los efectos de la congelación y sus implicaciones en la toma de decisiones comerciales en el sector pesquero.
Métodos: El proceso de congelación incluyó congelación por aire forzado a -18 °C, glaseado y almacenamiento a temperaturas entre -18 y -22 °C durante una semana.
Resultados: Se observaron alteraciones en la textura (menor dureza y fracturabilidad) del pulpo del Atlántico congelado en comparación con el fresco. A nivel sensorial, el pulpo congelado también presentó menor dureza y elasticidad. A lo largo del periodo de congelación, los valores de consistencia de las dispersiones proteicas disminuyeron, relacionando los efectos del congelado con cambios en la calidad. Por otro lado, el pulpo congelado del noreste del Pacífico presentó mayor contenido de humedad, pH y los valores más altos de dureza y fracturabilidad entre las muestras.
Conclusión: La congelación impacta negativamente en la calidad del pulpo, reduciendo características sensoriales y texturales importantes. Los resultados refuerzan la necesidad de optimizar los procesos y los criterios de abastecimiento en la industria de productos pesqueros.

Palabras Clave: *octopus* spp.; congelación; calidad; sensorial; reologia

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INTRODUCTION

Octopus is a valued seafood for its high protein content and unique texture. With increasing global demand, frozen octopus has become essential for year-round availability, but the freezing process can affect its sensory and textural properties. This study, prompted by a seafood company's concern, compares fresh Northeast Atlantic octopus with frozen Northeast Pacific octopus, which undergoes tempering before refreezing. By evaluating physicochemical, sensory, and rheological properties, the research explores how freezing impacts octopus' quality, providing insights for the company's sourcing strategy and the broader seafood industry.

1. THEORETICAL FRAMEWORK

The significance and scale of octopus fisheries are expected to increase, given that numerous finfish stocks are currently either fully exploited or overexploited (Sauer et al., 2021). Octopus is an increasingly important source of food and protein, with global fisheries doubling over the past four decades (Almeida et al., 2022; Martino et al., 2021).

Cephalopods of the genus *Octopus* spp., which comprises more than 100 species, are among the most important commercially harvested octopus worldwide, with these cephalopods typically being marketed fresh, dried, salted, and frozen (Zamuz et al., 2023). This species is highly appreciated and product demand commands high prices throughout all distribution chain, thus, sustaining traditional methods as well as industrial fishing methods. The supply is achieved at the expense of domestic production and a strong contribution of imported products (Ainsworth et al., 2023).

Most of the cephalopods that are caught are stored and marketed in their frozen state, because this product deteriorates rapidly when fresh, since they contain a large amount of endogenous proteolytic enzymes that promote this degradation (Altissimi et al., 2018). The frequent and systematic monitoring of temperature serves as the primary tool for the freezing industry to ensure preservation, although it does not guarantee absolute quality. Freezing and frozen storage can modify the sensory properties of seafood, especially during prolonged storage times or at high temperatures (Erikson et al., 2021). These freeze-induced alterations, in aspects related to the quality of fish muscle, are accompanied by modifications of fiber morphology, as well as denaturation and aggregation of muscle proteins, which produce a hard, dry, and fibrous product (Tan et al., 2021). On the other hand, the freezing process can disrupt membranes by the formation and aggregation of ice crystals. The process of freezing and thawing disrupts muscle cells, leading to the release of enzymes from lysosomes and mitochondria (Tan et al., 2021). This enzymatic activity induces proteolysis, promoting partial softening, which is crucial for enhancing the texture quality of certain species, such as octopus. Even when processed at low temperatures, the rate of proteolysis remains notably high (Lv & Xie, 2021). The extent of this change depends on many factors, including the rate of freezing and thawing, storage temperature, temperature oscillations, freezing-thawing abuse during storage, transportation, retail display, and consumption (Çalışkan Koç et al., 2025).

As a rule, seafood contains 17-20% crude protein corresponding to 2-3% protein nitrogen (Cropotova et al., 2023). The changes that occur in muscle proteins during frozen storage can be evaluated by studying their functional properties: protein solubility, emulsifying capacity, and viscosity. Among the rheological techniques, viscosity is one of the most sensitive functional properties for measuring changes occurring during frozen storage of fish muscle proteins (Mehta et al., 2023). Likewise, viscosity is the second most important commercial property of gelatine, following gel strength (Derkach et al., 2020). The non-Newtonian rheological behavior most observed in food products is shear-thinning, characterized by a decrease in viscosity as shear rate increases. This viscosity, whether dependent on the shear rate or time-dependent, is called the apparent viscosity, η_{app}. During frozen storage of fish, apparent viscosity decreases at different speeds depending on the number of factors, such as species, season, mince or whole muscle, and frozen storage temperature (Xie et al., 2024).

potential quality differences between fresh/frozen Northeast Atlantic and frozen Northeast Pacific octopus, both marketed by a local company.

2. METHODS

This study aimed to evaluate the quality (physical, chemical, and sensory) of octopus subjected to freezing. Fresh Northeast Atlantic octopus was frozen using air-blast freezing at -18°C, glazed, and stored for one week at -18 to -22°C. Additionally, frozen Northeast Pacific octopus, which underwent a tempering process before refreezing, was analyzed. The study compared these two samples to assess.

2.1 Octopus' Samples

Octopus samples were provided by a local company, which commercializes specimens from two distinct areas: the Northeast Atlantic and the Northeast Pacific.

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Ten specimens of *Octopus vulgaris* were provided by artisanal fleets operating in shallow waters in the Northeast Atlantic (NA), Portugal (fishing area 27). Octopuses were caught using traditional fishing equipment, known as "alcatruz" and "covo", the first are clay pots and the second is a half-cylindrical structure trap made of iron. Once on board, octopuses were immediately refrigerated in coolers containing ice and water and transported to the seafood processing plant and subsequently to the company in Portugal.

Five octopuses of genus *Octopus* spp. from the Northeast Pacific (NP), Alaska (fishing area 67), were caught with pot fisheries, eviscerated, and, in the same way, were kept refrigerated and transported to the seafood processing plant. Regrettably, due to the geographical distance, these samples had to be exported to the company (Portugal) in frozen blocks to ensure both microbiological and physicochemical quality.

At the company, NA octopus individuals were weighted, eviscerated, and five individuals were frozen by air-blast freezing in a batch tunnel (-18 °C). On the other hand, NP octopus individuals were weighed and submitted to a tempering process (a maximum of 16 hours) before being eviscerated and frozen using the same method. The tempering process involves the controlled thawing of frozen octopus, ensuring a gradual and uniform increase in temperature to preserve its texture, flavor, and nutritional attributes. Finally, frozen octopus samples were glazed and stored for one week between -18 and -22 ºC. Samples of fresh and frozen NA octopus, as well as frozen NP octopus, were sent to the laboratory of the Polytechnic Institute of Beja. Fresh and frozen octopus were evaluated according to their physicochemical, sensory, and textural/rheological properties.

Figure 1 provides an overview of the workflow used to evaluate the quality of fresh and frozen octopus samples.

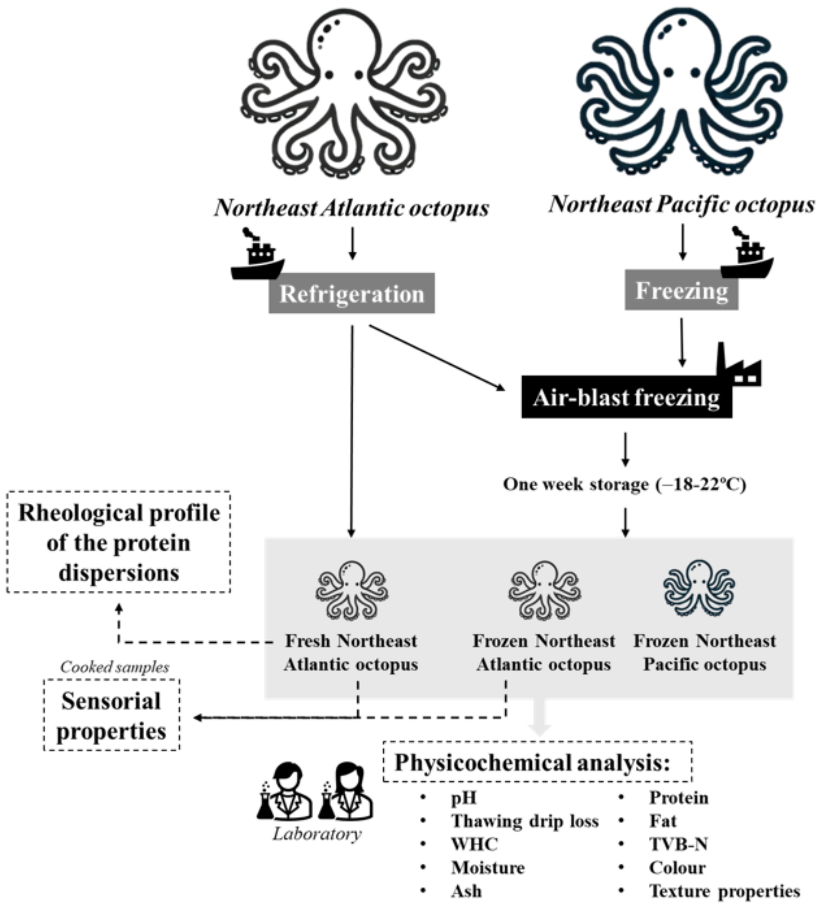

Figure 1 - Flowchart illustrating the study of fresh and frozen octopus quality from the Northeast Atlantic and Northeast Pacific regions

2.2 Quality evaluation of fresh and frozen octopus

Fresh and frozen octopus, in duplicate, were evaluated in the laboratory following overnight thawing. The assessment included direct measurements of pH, thawing drip loss, and water holding capacity (WHC). For moisture, ash, protein, fat, and total volatile basic nitrogen (TVB-N), octopus samples were previously crushed and homogenized after removing the skin, mantle, and suckers.

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The pH values were determined at 20 °C using a potentiometer (Metrohm-model 691, Switzerland), fitted with a penetration electrode. Thawing drip loss was measured using Equation 1 (Zhang et al., 2024):

$$\% \, Drip = \frac{A-B}{A} \times 100 \qquad (1)$$

Where A and B correspond to the sample initial weight (g) and sample final weight (g), respectively. WHC was determined by the methodology by Zhang et al. (2024).
Moisture, ash, protein, and fat contents were determined according to Association of Official Analytical Chemists (AOAC) methodologies. Finally, TVB-N was determined by the Conway method (Zhang et al., 2024), and the results were expressed in mg N/100 g.

2.3 Sensorial and textural properties of cooked octopus

A heat treatment for 22 min, using pressure cooking, was applied to five replicates of both fresh and frozen NA octopus samples, and the evaluation of instrumental color, texture, and sensorial properties was performed.
Color analysis was determined using a colorimeter, Minolta CR-300R (Minolta, Osaka, Japan), with ten measurements taken per replicate. Results were expressed by the coordinate system C.I.E.Lab. Chroma, Hue, and Total Color Variation (ΔE) were calculated by the following equations 2, 3, and 4 (Zhang et al., 2024), respectively:

$$Chroma = (a*^2 + b*^2)^{0.5} \qquad (2)$$

$$Hue = \arctan\left(\frac{b}{a}\right) \qquad (3)$$

$$\Delta E = [(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2]^{0.5} \qquad (4)$$

Texture analysis was made in a texturometer Texture Analyser Model TAHDi® (Stable Micro Systems, Godalming, UK), with a load cell capacity of 25 kg (Interchangeable Low Force Load Cells Model LC/25). The puncture test was analyzed five times in each replicate. The thickness of each arm was 16 mm, the probe was a cylinder with a 2 mm diameter, and the temperature of the assay was 30 ºC. The test speed was set at 1 mm/s, and the distance run was 12 mm. In the graphs, Force versus Time was determined for hardness (N), fracturability (N), adhesivity (N.S), and distance until fracture (mm).
Quantitative Descriptive Analysis (QDA) was performed on the octopus samples in a sensory room with all required specifications (ISO 8586-2, 2008), with a sensory panel of 20 people. The sensory analysis was carried out with a 10 cm quantitative unstructured scale assessment with the descriptors: visual appearance (color, brightness), smell (intensity, seaweed and ink-like), mouth feels (adhesivity, hardness, elasticity and fracturability), taste (sweet, salty, bitter and ink-like), flavor (intensity, sourness, seaweed), persistence and aftertaste. The results from the unstructured scale were converted to a 1–9 quantitative scale for each attribute. Panelists were provided with sliced samples (1 cm thickness) served at 30 ± 1 °C on Petri plates, along with water to cleanse the palate between samples.

2.4 Frozen storage study - rheological profile of the protein dispersions

After skin and suckers were removed, and the fresh NA octopus was crushed and homogenized, the samples were placed in a storage test at -20 °C to assess the rheological profile (consistency) of the protein dispersions. Muscle dispersions were prepared according to the method of Borderías et al. (1985), with slight modifications. These were then used to determine the apparent viscosity, flow behavior, and consistency determination (Equation 5). The assay was carried out with a rotational viscometer (HAAKE VT550, United Kingdom) using a cone and plate geometry (PK5, angle 1º) and equipped with a temperature-control unit. The viscometer was connected to a computer to control the acquisition of data. The apparent viscosity measurements were determined over a shear rate range from 100 to 1000 s^{-1}. During shearing, a total of 15 data points was recorded, with each measurement performed in triplicate.

$$Log \, \eta_{app} = Log \, k + (n-1) \log \gamma \qquad (5)$$

Where η_{app} is the apparent viscosity (Pa · s), k is the consistency coefficient (Pa · s^n), γ is the shear rate (1/s), and n is the flow behavior index (adimensional). The parameters k and n were determined by fitting the experimental shear stress versus shear rate data to the power-law model using linear regression on the logarithmic form of the equation.

2.5 Statistical analysis

Statistical analysis of the results was performed using Statistica 6.0 Software (TIBCO Software Inc., Palo Alto, USA). To evaluate significant differences between the means, analysis of variance (ANOVA) with one-factor and Tukey test ($p < 0.05$) was used.

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3. RESULTS

3.1 Quality evaluation of fresh and frozen octopus

Table 1 shows the results of chemical analysis, thawing drip, and WHC of fresh and frozen octopus captured in NA and frozen octopus captured in NP.

Table 1 - Chemical composition of fresh and frozen octopuses. Results are shown as mean ± standard deviation. The letters (a-b) in the same row mean that values are significantly different, according to the Tukey test at the 5% significance level (n < 0.05)

Parameter	Northeast Atlantic octopus		Northeast Pacific octopus
	Fresh	Frozen	Frozen
Proteins [1]	n.d. [3]	21.2 ± 1.15 a	17.48 ± 1.28 a
Lipids [1]	n.d. [3]	0.64 ± 0.02 a	0.56 ± 0.03 b
Moisture [1]	80.40 ± 0.54 b	80.79 ± 0.54 b	82.99 ± 0.60 a
Ash [1]	2.13 ± 0.11 a	2.36 ± 0.11 a	2.46 ± 0.12 a
pH	6.04 ± 0.13 b	6.41 ± 0.13 b	7.15 ± 0.15 a
TVB-N [2]	6.73 ± 0.66 b	9.90 ± 0.66 a	11.22 ± 0.74 a
Thawing Drip [4]	n.d. [3]	9.25 ± 1.42 a	12.50 ± 1.59 a
WHC [4]	12.30 ± 0.62 b	11.74 ± 0.62 b	14.81 ± 0.69 a

Note: [1] g/100g edible part; [2] mg NH_3/100 g; [3] not determined; [4] %.

Fresh NA octopus presented 80.40 ± 0.54 g/100 g edible part of moisture content (Table 1), showing no significant differences ($p > 0.05$) for frozen octopus from the same fishing area, but this parameter increases ($n < 0.05$) for NP octopus (82.99 ± 0.60 g/100 g edible part). A similar pattern was observed for pH, TVB-N, and WHC (Table 1), indicating an increase ($p < 0.05$), as observed in the case of pH, from 6.04 ± 0.13 (fresh) and 6.41 ± 0.13 (frozen) to 7.15 ± 0.15 in frozen NP octopus.

3.2 Sensorial and textural properties of cooked octopus

Color and texture results of fresh and frozen octopuses after the cooking process are presented in Table 2. Significant differences ($n < 0.05$) in color were more pronounced in frozen NA samples, revealing an ΔE of 18.62 ± 0.31, compared to 16.50 ± 0.31 and 17.22 ± 0.34 for fresh NA and frozen NP samples, respectively. These differences result from lower luminosity (L*), lower greenness (a*), and bigger blueness (b*). In the texture analysis (Table 2), it is observable that frozen octopus samples from the NA exhibited lower fracturability and hardness (2.81 ± 0.27 and 3.33 ± 0.18 N, respectively), displaying statistically significant differences ($n < 0.05$) when compared to the fresh ones (3.97 ± 0.28 and 4.60 ± 0.19 N, respectively). Conversely, NP samples showed significantly higher values were observed to 5.40 ± 0.31 and 5.53 ± 0.21 N, respectively.

Table 2 – Color and texture properties of fresh and frozen octopuses. Results are shown as mean ± standard deviation. The letters (a-c) in the same row mean that values are significantly different, according to the Tukey test at the 5% significance level (n < 0.05)

Parameter	Northeast Atlantic octopus		Northeast Pacific octopus
	Fresh	Frozen	Frozen
Color:			
L*	81.29 ± 0.38 a	78.94 ± 0.38 b	81.99 ± 0.42 a
a*	-2.23 ± 0.11 b	-0.97 ± 0.11 a	-1.24 ± 0.12 a
b*	2.99 ± 0.22 b	7.91 ± 0.22 a	0.59 ± 0.24 c
Chroma	3.80 ± 0.19 b	8.09 ± 0.19 a	1.62 ± 0.22 c
Hue	129.18º ± 6.48º b	97.06º ± 6.48º c	181.54º ± 7.25º a
ΔE	16.50 ± 0.31 b	18.62 ± 0.31 a	17.22 ± 0.34 b
Texture properties:			
Fracturability [1]	3.97 ± 0.28 b	2.81 ± 0.27 c	5.40 ± 0.31 a
Distance to fracturability [2]	9.93 ± 0.35 b	7.65 ± 0.34 c	12.92 ± 0.39 a
Hardness [1]	4.60 ± 0.19 b	3.33 ± 0.18 c	5.53 ± 0.21 a
Adhesivity [3]	26. 84 ± 1.25 a	27.53 ± 1.22 a	19.75 ± 1.40 b

Note: [1] N; [2] mm; [3] -N.S.

Sensory evaluation was limited to the fresh and frozen NA octopus samples due to constraints in the laboratory. Figure 2 represents the sensory parameters (brightness, hardness, elasticity, and seaweed flavor), which presented significant differences

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between fresh and frozen NA octopuses. All sensory evaluation results are presented in Table 3. It is evident that fresh NA octopus exhibited higher scores in most attributes, except for smell intensity, where frozen samples showed higher values.

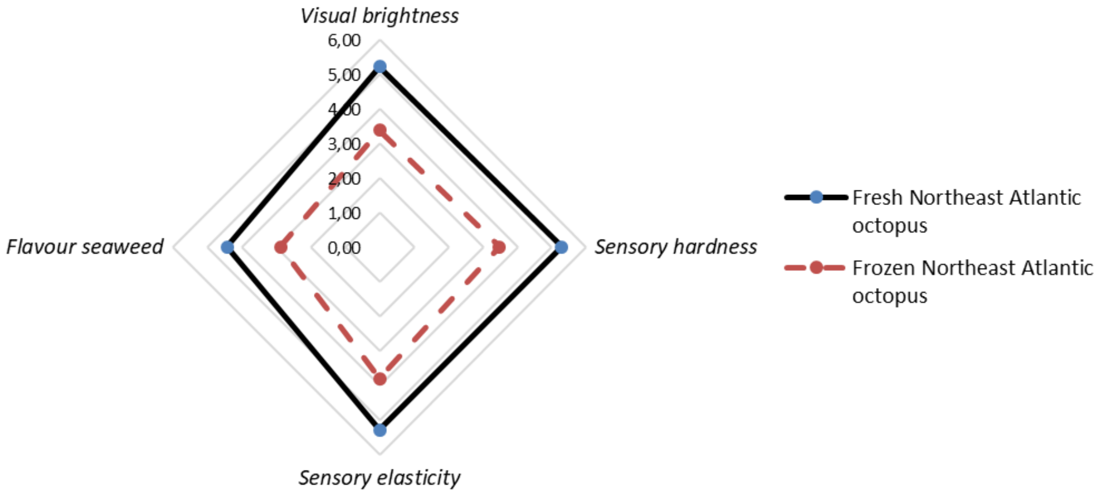

Figure 2 - Sensory parameters of cooked samples of fresh and frozen Northeast Atlantic octopuses

Table 3 – Sensory parameters of fresh and frozen Northeast Atlantic octopuses. Results are shown as mean ± standard deviation. The letters (a-b) in the same row mean that values are significantly different, according to the Tukey test at the 5% significance level ($p < 0.05$)

Sensory attribute	**Northeast Atlantic octopus** **Fresh**	**Frozen**
Visual:		
Brightness	5.21 ± 0.53 a	3.38 ± 0.50 b
Color	3.15 ± 0.63 a	3.38 ± 0.57 a
Smell:		
Smell intensity	4.50 ± 0.49 a	5.50 ± 0.46 a
Seaweed	4.86 ± 0.60 a	4.44 ± 0.56 a
Ink-like	2.45 ± 0.50 a	2.43 ± 0.45 a
Oral Tactile Sensations:		
Adhesivity	3.64 ± 0.47 a	3.12 ± 0.44 a
Hardness	5.29 ± 0.45 a	3.47 ± 0.43 b
Elasticity	5.29 ± 0.51 a	3.81 ± 0.48 b
Fracturability	3.93 ± 0.50 a	4.12 ± 0.46 a
Taste:		
Sweet	2.00 ± 0.38 a	3.08 ± 0.38 a
Salty	2.92 ± 0.54 a	2.79 ± 0.52 a
Bitter	1.64 ± 0.25 a	1.50 ± 0.26 a
Flavour:		
Intensity	5.29 ± 0.55 a	4.56 ± 0.51 a
Sour	1.56 ± 0.29 a	1.83 ± 0.34 a
Seaweed	4.42 ± 0.45 a	2.87 ± 0.44 b
Ink-like	1.82 ± 0.38 a	1.92 ± 0.36 a

3.3 Frozen storage study - rheological profile of the protein dispersions

The stability of the octopus against frozen storage was evaluated by monitoring the rheological viscosity behavior. Table 4 presents the consistency of protein dispersions from NA octopus over 14 weeks of frozen storage. A clear decrease in consistency values was observed over the storage time.

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Table 4 – Evolution of the behavior of consistency along the frozen storage. Results are shown as mean ± standard deviation

Protein dispersions	Frozen storage (weeks)	Consistency coefficient, K ($Pa.s^n$)
Northeast Atlantic octopus	0 (fresh)	0,54 ± 0,15
	7	0,48 ± 0,14
	9	0,34 ± 0,02
	13	0,24 ± 0,03
	14	0,20 ± 0,02

4. DISCUSSION

During freezing, significant nutritional or chemical changes are not expected, but thawing effects depend on the freezing method and storage time (Muniz, 2020). Diet, habitat, season, sex, and species affect fatty acid composition, while water temperature and nutrient availability influence octopus muscle composition (Lougovois et al., 2008; Muniz, 2020).

In this study, the pH data showed no significant differences between frozen (pH 6.41) and fresh (pH 6.04) octopus from the NA. However, both exhibited significant differences when compared to the frozen NP octopus (pH 7.15). Variations in pH levels can serve as indicators of glycogen conversion into lactic acid and the degradation of muscle components such as proteins and nucleotides during the storage period (Vaz-Pires & Barbosa, 2004). The pH increase in fresh fish is mainly caused by the enzymatic breakdown of nitrogenous compounds and ATP, leading to the release of dimethylamine, phosphate, and ammonia (Vaz-Pires & Barbosa, 2004). The elevated pH observed in NP octopus is likely related to the complex freezing process used during manufacturing, which includes defrosting large blocks, tempering, soaking in water, and refreezing. These repeated temperature fluctuations can influence the balance of hydrogen and hydroxyl ions through changes in redox reactions and endogenous enzyme activity (Zavadlav et al., 2019). While postmortem pH decline typically depends on glycogen reserves, cephalopods have low glycogen levels and rely primarily on protein metabolism. In these species, phosphoarginine acts as the main phosphagen, converting to octopine, a basic compound, during pre-rigor, which limits the pH drop in muscle tissue after death (Zavadlav et al., 2019).

Frozen octopuses are a good source of protein (17-21%) and exhibit low fat content, as corroborated by the results obtained in our study, where the fat content was less than 1%, being considered a lean fish (Vaz-Pires & Barbosa, 2004). While the protein content did not differ significantly between frozen NA octopus (21.20%) and NP octopus (17.48%), Lougovois et al. (2008) asserted that the freezing process did not adversely impact the protein content of octopuses, even during defrosting.These authors highlighted that, during defrosting, some intra- and extracellular fluids, primarily consisting of water-soluble proteins, might be drained due to a reduced water-holding capacity. Regarding moisture content, no significant differences were observed between fresh and frozen NA octopuses, which is likely attributed to the pre-treatment with saline solutions and the glazing freezing method, both of which helped minimize moisture losses after defrosting. However, significant differences were observed in frozen NP octopus, displaying the highest moisture content (82.99%). This difference may be linked to the soaking process following tempering, where the operation possibly facilitated the incorporation of more water into its proteins. Indeed, Altissimi et al. (2018) reported significant changes in moisture content and protein levels in defrosted samples compared to fresh ones, likely due to the effects of freezing and thawing processes.

Data for WHC after defrosting (Table 1) presented a significant difference between the NP and NA octopuses, with the higher value in the NP octopus (14.81%), compared to the lower values in the fresh and frozen NA octopuses (12.30 and 11.74%, respectively). Changes in WHC from postmortem to processing are a sensitive indicator of myofibrillar protein structure and overall fish quality. WHC is also affected by ice crystal size, which can damage muscle fibers and cellular structure (Zhang et al., 2024). Tempering might have contributed to the NP octopus WHC when compared with the NA octopus, since recrystallization and subsequent increase in crystal size, mainly due to temperature fluctuations, resulted in structural alteration of the proteins (denaturation). For instance, Hu & Xie (2022) found that repeated freezing and thawing cycles reduce fish muscle WHC due to ice crystal-induced structural damage, protein denaturation, and changes in protein-water interactions and conformation.

The TVB-N method is commonly used to assess fish product quality by measuring trimethylamine (from microbial deterioration), dimethylamine (from autolytic enzymes during freezing), ammonium (from amino acid deamination and nucleotide catabolism), and other basic volatile nitrogen compounds linked to decomposition (Bekhit et al., 2021). According to Bekhit et al. (2021), fresh, chilled, or frozen fish and fish products should present values equal to or less than 35 mg TVB-N/100g sample. In our study, fresh and frozen NA and NP octopuses showed results below this acceptability value (Table 1). Nonetheless, the NP sample presented the highest TVB-N value (11.22 mg TVB-N/100g), as well as the highest pH value (pH 7.15). Obviously, the freezing process to which the NP octopus was subjected resulted in a higher TVB-N value, which may have been related to temperature gradients that influenced and accelerated autolysis and microbial activity. However, the results obtained in our study agree with those

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obtained in other studies, for instance, values of 10 mg TVB-N/100 g in octopus's arms storage, in refrigeration (Lougovois et al., 2008).

Based on the color results (Table 2) for octopus samples after the cooking process, frozen NA octopus showed lower lightness and higher redness and yellowness values. A plausible explanation for these findings can be linked to the packaging process, or lack thereof, during refrigerated transportation to the laboratory. The absence of packaging may have influenced the color parameters, especially the yellow color (b* parameter), attributed to surface dehydration. As the outer layer of ice sublimes, this superficial dehydration facilitates protein denaturation and lipid oxidation (Vaz-Pires & Barbosa, 2004). Comparing chromaticity, the frozen NA octopus displayed higher values compared to the frozen NP octopus, indicating a less saturated and dull appearance. The fresh NA octopus exhibited brighter colors, greater color purity or intensity, and notably, the a* value was closest to the red color. This evolution may be attributed to the lesser denaturation of proteins associated with pigments. The frozen NA octopus also had the lowest hue value, placing it in the second quadrant near yellow. Additionally, the difference in color, ΔE, was more pronounced in the frozen NA octopus.

All rheological parameters exhibit significant differences among all samples (Table 2). However, in terms of adhesiveness, no significant differences were observed between the fresh and frozen NA octopus. In contrast, the frozen NP octopus displayed a significant decrease in adhesivity values. Additionally, the NP octopus exhibited higher hardness, fracturability, and a greater distance to fracture compared to the frozen NA octopus. Regarding the sensory analyses of cooked NA octopuses, the brightness was significant different between fresh and frozen samples, showing scores of 5.21 and 3.38, respectively (Figure 2). The loss of water after heat treatment, which was possibly higher in frozen octopus, may have an influence on the brightness. The smell intensity was more pronounced in the frozen octopus, whereas the seaweed smell attribute (algae) was higher in the fresh octopus. Regarding sensory hardness, significant differences were observed between fresh and frozen octopuses, with the fresh one being rated as harder. These findings align with the instrumental results presented in Table 2. In terms of sensory elasticity, the fresh octopus exhibits significantly higher elasticity compared to the frozen octopus. According to Reyes et al. (2014), freezing octopus results in a softer texture after cooking when compared to fresh samples. Other sensory attributes were evaluated, but with no significant differences between the octopus samples.

Regarding the evaluation of rheological viscosity behavior of protein dispersions throughout frozen storage, NA octopuses demonstrated a decreasing pattern of consistency. Frozen temperatures inevitably lead to the formation of ice crystals, causing mechanical damage to both the membrane and the protein network. This promotes the release of endogenous proteolytic enzymes and contributes to protein denaturation/aggregation (Tan et al., 2021). In octopus, myofibrillar proteins like paramyosin are essential for stabilizing the myosystem by balancing protein aggregation and proteolysis caused by high levels of endogenous enzymes, whose activity remains high in both fresh and frozen states (Ruiz-Capillas et al., 2003). Reyes et al. (2014) studied the effect of freezing on the electrophoretic protein pattern in the common Octopus vulgaris, and it was found that myosin and paramyosin bands were most affected by freezing prior to cooking. Additionally, this decrease in consistency can be accompanied by the consumer's possible desirable texture. These results explained and distinguished fresh and frozen octopus samples by assessing the consistency of protein dispersions. The observed differences in consistency values suggest alterations in the protein structure, potentially caused by freezing and storage processes. Understanding structural changes is key to comparing fresh and frozen octopus, revealing how freezing and storage affect protein quality and functionality. This information is valuable for the seafood industry, helping assess frozen octopus products and guide decisions on their usage and preferences.

CONCLUSION

This study assessed the quality of frozen octopus from fishing areas in the NA and NP. Freezing processes did not negatively impact the quality of the octopus, as most chemical, physical, and sensory characteristics remained within quality indicators when compared to fresh octopus. TVB-N values, an indicator of freshness, complied with EU standards. However, tempering and refreezing negatively affected the quality of frozen Pacific octopus. This process led to the highest pH and TVB-N values, indicating a decline in freshness. Sensory analysis showed that frozen octopus had reduced hardness and elasticity, while fresh octopus had a stronger seaweed flavor and greater brightness. In terms of viscosity, the power-law model accurately described the rheological properties of octopus protein dispersions influenced by frozen storage. Initial viscosity was high due to intense proteolytic activity, but it decreased rapidly over time due to protein denaturation and aggregation.

This study provides valuable insights into the quality of frozen octopus and the implications of freezing methods, including the negative effects of tempering and refreezing, on its physical attributes and overall consumer experience.

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AUTHORS' CONTRIBUTION

Conceptualization, S.P and M.J.C.; data curation, S.P., A.R. and M.J.C.; formal analysis, S.P. and M.J.C.; investigation, S.P., A.R., L.F. and M.J.C.; methodology, A.R. and M.J.C.; project administration, S.P. and M.J.C.; resources, S.P. and M.J.C.; supervision, S.P. and M.J.C.; validation, S.P. and M.J.C.; visualization, S.P., L.F. and M.J.C.; writing- original draft, S.P., A.R., L.F. and M.J.C.; writing- review & editing, S.P. and L.F.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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