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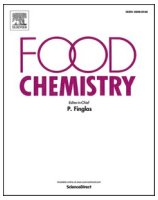

Valorisation of sea urchin (*Paracentrotus lividus*) gonads through canning

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ABSTRACT

Fresh sea urchin (*Paracentrotus lividus*) gonads are a delicacy with short seasonal availability, very often heterogeneous in size and intrinsic characteristics. This study aimed to valorise this resource through the preparation of canned products (with/without *Porphyra* spp.) and evaluate their physicochemical and sensory quality (3–12 months). Canning contributed to a decrease in protein, K and most carotenoids contents; and a concentration of lipids, ash, Na and Se levels. A simulated 12-month ageing led to decrease the protein and β-carotene contents; and the Na and lutein levels concentration. The macroalgae addition resulted in an orange, darker and less soft product, with higher carbohydrates, Na, Se and carotenoids contents. A 25 g-dose contributes to significant daily intakes of protein (8–9%), EPA+DHA (47–53%), I (35–62%) and Se (30–47%). The products were commercially stable/sterile and had good sensory acceptance. Overall, canning constitutes a strategy to provide a nutritionally balanced product available all year-round.

1. Introduction

The sea urchin *Paracentrotus lividus* is seasonally harvested in Atlantic and Mediterranean coastal areas (Boudouresque & Verlaque, 2013). Its gonads (or roe) are considered a luxury product, highly appreciated for their unique sensory characteristics. However, since the gonads are only available with high quality during a short period throughout the year (from November to March; Rocha et al., 2019), the application of preservation and processing technologies is of utmost importance to extend their shelf-life and meet demand during off-season. Several thermal (freezing and canning) and non-thermal (*e.g.*, brine-salting, fermentation and high pressure) processes have been tested and accepted to valorise such resource, especially those that come from sea urchins with smaller sizes, atypical colour or damaged shape (Tillocca, 2016).

The second most popular method of processing seafood intended for human consumption is canning which has a long traditional in some European countries (Aubourg, 2001; Hall, 2011). In this sense, canning seems to be a good solution to valorise sea urchin gonads and contribute for their consumption all year round, even in some restaurants, where chefs complain about irregular supply throughout the year (Baião, Moura, Rocha, Valente, & Cunha, 2021). Although canned sea urchin gonads are rarely sold in stores, some can be found in European retailers with a shelf-life of approximately 2 years (Monfort, 2002).

The effect of canning on physicochemical quality of sea urchin gonads has been assessed. Some works analysed such effect on the levels of

Abbreviations: K, potassium; Na, sodium; Se, selenium; I, iodine; EPA, eicosapentaenoic acid; DHA, docosahexaenoic acid..

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protein, amino acids, fatty acids (Cruz-García, López-Hernández, González-Castro, Rodríguez-Bernaldo De Quirós, & Simal-Lozano, 2000), volatile components (De Quirós, López-Hernández, González-Castro, De la Cruz-Garcia, & Simal-Lozano, 2001), carotenoids and liposoluble vitamins (De Quirós, López-Hernández, & Simal-Lozano, 2001) of sea urchin gonads from *P. lividus*. Moreover, Lee, Lee, Kim, and Kim (2012) also tested some physicochemical properties (proximate composition, fatty acids and minerals) of steamed and canned gonads of three popular sea urchin species in Korea. However, such studies did not contemplate the stability and sterility nor consider the addition of other ingredients (*e.g.* macroalgae) with the potential of enhancing product nutritional profile.

Despite the popularity of roe in many markets, to the best of authors' knowledge, there is relatively little information on the impact of canning on its quality. In this context, the aim of this study was to i) develop canned sea urchin (*P. lividus*) gonads (with and without addition of the macroalgae *Porphyra spp.*) in a light brine; ii) assess the nutritional, physical and sensory quality of the developed products, and iii) evaluate their stability and sterility (*i.e.*, over a 12-month ageing period in the case of the product without the addition of macroalgae; and after 3-month period at room temperature for the product prepared with macroalgae).

2. Materials and methods

2.1. Raw materials

Live sea urchins (*P. lividus*) of commercial size (test diameter $\geq$ 5 cm) were harvested at *Praia Norte* (Viana do Castelo, North of Portugal) during the winter, corresponding to the season with the highest gonad somatic index (Rocha et al., 2019). The specimens were transported in polystyrene boxes with cold accumulators and processed within approximately 14 h. For raw material characterisation, the gonads of twelve live sea urchins were removed and 3 independent pools were randomly constituted with 20 gonads of different specimens. The macroalgae (*Porphyra spp.*; ALGAMAR, Pontevedra, Spain) was purchased in a local supermarket (Lisbon) and used as ingredient in the preparation of some canned gonads.

2.2. Canning of gonads

Processing was performed at a seafood canning factory. Sea urchins were steamed for 10 min and after cooling, the gonads were removed with a stainless-steel spoon. Then, for batch 1 (*ca.* 30 cans), approximately 50 g of gonads were randomly distributed in easy-to-open aluminium cans (cylinder shape; mass = 10 g; volume = 80 cm^3, diameter = 7 cm; height = 3 cm) to which 5 mL of a brine (5% NaCl) was added (corresponding to 0.25 g of salt); the remaining volume was filled with water. The batch 2 (*ca.* 15 cans) was prepared similarly but with a previous addition of 500 mg of crushed *Porphyra spp.* to the cans.

Cans of both batches were hermetically sealed and properly heat sterilised (121 °C/30 min) according to the common practices of the industrial unit. After cooling and washing, cans were left during a maturation period at room temperature and then tested for stability (NP 4404–1, 2002). Then, both batches were sent to laboratories. Fifteen cans of gonads from batch 1 (Control cans = canned gonads in light brine) and the same number from batch 2 (+Macroalgae group = canned gonads with macroalgae in light brine) were maintained at room temperature (≈20 °C) for 3 months.

2.3. Accelerated ageing

The ageing of canned gonads was carried out with packages from the control batch (without macroalgae). The calculation of accelerated ageing was based on the Arrhenius equation, which indicates that an increase of 10 °C at a given temperature doubles the rate of chemical reactions and/or microbiological growth (Labuza & Fu, 1993). To this end, 15 packages were stored at 40 °C for 3 months to simulate a 12-month ageing period at 20 °C. This batch of canned gonads was entitled "Aged" group.

2.4. Analyses

All canned products (Control, +Macroalgae, Aged) were assessed after 3 months of storage, which corresponds to the time to obtain the typical palatability of most canned seafood products (Aubourg, 2001). Nine packages of each product were used in stability and sterility tests. The remaining were utilised for chemical, physical and sensory analyses.

2.4.1. Stability and sterility

The stability test was performed according to the Portuguese standard NP 4404–1 (2002). This test consists in the evaluation of the i) external examination of package, ii) appearance (colour and texture) and odour of the product, and iii) pH (pH meter 539, WTW, Weilheim, Germany) after a 7-day period at three different temperatures (≈20 °C, 37 °C and 55 °C). Canned products sterility was determined following standard NP 2309–2 (1988) for low-acid food, which involves the detection of aerobic and anaerobic mesophiles and thermophiles at 37 °C and 55 °C, respectively.

2.4.2. Chemical analyses

Homogenized samples (N = 3 pools of raw gonads or packages of canned products; and N = 3 in the case of macroalgae; 5000 rpm; Retsch Grindomix GM200, Haan, Germany) were stored at −80 °C, freeze-dried (24 h at −45 °C under ≈10,132.5 Pa; Power Dry 150 LL3000, Heto, Mukarov, Czech Republic), powdered by means of mortar and pestle and kept at −80 °C until further analysis. Proximate composition, fatty acids (FA) profile, macro (sodium (Na) and potassium (K)) and micro (iodine (I) and selenium (Se)) elements levels as well as carotenoids contents were determined in both raw materials (sea urchin gonads and macroalgae) and canned products (Control, +Macroalgae, Aged). Calculations were made to present data in drained weight of gonads.

2.4.2.1. Proximate composition. Moisture content was determined by mass difference through freeze-drying process. The ash content was quantified by incineration in a muffle furnace (TYP.MR170, Heraeus, Hanau, Germany) for 16 h at 500 ± 25 °C according to standard procedure (AOAC, 2005). The protein level was determined according to the Dumas method (Saint-Denis & Goupy, 2004) using an automatic analyser (FP-528 DSP LECO, St. Joseph, USA). The nitrogen released after combustion (850 °C) was quantified using EDTA calibration curve and converted into protein using the usual factor (6.25) for animal proteins (FAO, 2003). The total lipids were quantified following the Folch method (Folch, Lees, & Stanley, 1957). This procedure comprised the homogenization of sample in a mixture of chloroform:methanol (2:1), followed by the addition of an acid (0.1 N HCl) and a saline solution (0.5% $MgCl_2$), contributing to the protein precipitation and to promote the lipid migration to the organic phase. Afterwards, the lower phase was carefully collected; a second step of extraction was conducted and both organic phases were combined and evaporated to dryness.

2.4.2.2. Fatty acids profile. The determination of fatty acid methyl esters (FAMEs) was performed according to the Lepage and Roy (1986) procedure with modifications (Bandarra, Batista, Nunes, Empis, & Christie, 1997), through acid-catalysed transesterification. Each sample (200 mg dry weight, dw) and 50 μL of an internal standard solution (C23:0 at 10 $mg \cdot mL^{-1}$) were homogenized in 5 mL of acetyl chloride-methanol (1:19 *v*/v) and heated in a water bath for 1 h at 80 °C. After cooling, 1 mL of Milli-Q water and 2 mL of *n*-heptane (99.5%) were added. Transesterified extracts were centrifuged (2300 *g*, 5 min, 5 °C; Sigma 3 K30, Osterode, Germany), the upper phase was collected, and

moisture content was removed with anhydrous sodium sulphate (99.0%). About 2 μL was injected into a gas chromatograph (Bruker Scicon 456, Livingston, UK). The FAMEs separation was performed in a Stabilwax-MS polyethylene glycol capillary column (30 m length × 0.25 mm internal diameter, 0.25 μm thickness; Shimadzu, Kyoto, Japan) with helium as a carrier gas, programmed to start at 180 °C, held for 5 min, then raised to 220 °C at 4 °C min^{-1} and maintained at 220 °C for 25 min. Detection was done on a flame ionization detector at 250 °C and the identification was accomplished by retention times comparison using standards (Sigma-Aldrich, St. Louis, MA, USA): Supelco polyunsaturated fatty acids (PUFA) No.1 (Marine Source, 99%-Ref. 47033) and PUFA No.3 (Menhaden oil 99%-Ref. 47085-U). Quantitative data were calculated using peak areas (% of total FA).

2.4.2.3. Macro and microelements. K and Na were analysed based on the method described by Jorhem (2000) through flame atomic absorption spectrophotometry (Spectr AA 55B spectrophotometer, Agilent, Santa Clara, USA). The concentrations were calculated using linear calibration obtained from absorbance measurements of different concentrations of standard solutions (KNO_3 and $NaNO_3$, both dissolved in 0.5 M HNO_3).

I and Se were determined by inductively coupled plasma mass spectrometer (ICP-MS, Thermo X series II, Thermo Fisher Scientific, Waltham, USA), after acid digestion (Se) or alkaline extraction (I). Se content was quantified using the European standard procedure EN 15763:2009 (CEN, 2009). Briefly, about 200 mg of sample (dw) was weighed into 50 mL of polypropylene DigiTUBEs (SCP Science, Quebec, Canada) and digested overnight in 7 mL of nitric acid (60% ultrapure *w/w*). Then, digestion continued in a 48-well heating block (DigiPREP, SCP Science, Courtaboeuf, France) for 3.5 h at 85 °C after adding 1 mL of hydrogen peroxide (30% w/w). After cooling, the digests were diluted up to 25 mL with MilliQ water and kept at 5 ± 3 °C until ICP-MS analysis (Coelho et al., 2019). Quantification of I was performed separately, following the standard procedure EN 15111:2007 (CEN, 2007). About 200 mg of sample (dw) was weighed into polypropylene DigiTUBEs (SCP Science) and extracted with tetramethylammonium hydroxide (25%) in MilliQ water (1:8 ratio) using a 48-well graphite heating block (DigiPREP) for 3 h at 90 °C. After cooling, the extracts were diluted up to 50 mL with MilliQ water and centrifuged at 10000 rpm (15 min at 20 °C). The supernatant was filtered through a 0.45 μm syringe filter (Millipore) before ICP-MS analysis (Delgado et al., 2019). Quantification was attained by linear calibration using solutions from single elements high purity ICP stock standards for Se (0.5–5 $\mu g \cdot g^{-1}$; SCP Science, Marktoberdorf, Germany) and I (1–50 $\mu g \cdot g^{-1}$; Inorganic Ventures, Christiansburg, Virginia).

2.4.2.4. Carotenoids. The method to analyse carotenoids was adapted from Serrano et al. (2017). Briefly, carotenoids (two technical replicates of 200 mg each) were extracted twice with 2 mL of water-saturated *n*-butanol. The mixture was centrifuged (5 min at 10000 *g*) and the supernatant was collected after filtration through a 0.22 μm syringe filter (PET-20/25). All the procedures were performed whenever possible in the absence of light. The extracts were kept at −80 °C until further analysis. 20 μL were injected on a high-performance liquid chromatography-photo diode array (HPLC-PDA) workstation system (2795, Waters, Massachusetts, USA) equipped with a photodiode array detector (2996, Waters, Massachusetts, USA) and a degassed inline system. The detector slider and diode width were 2.4 nm and the response time was 1 s. The wavelength was monitored between 250 and 750 nm. The chromatographic separation was achieved on a Carotenoid C30 reversed phase column (4.6 mm × 100 mm, 5 μm particle size; YMC, Kyoto, Japan) at 35 °C in an oven (Waters, Massachusetts, USA), using a flow rate of 0.6 $mL \cdot min^{-1}$. The mobile phase was methanol/water (75:25, *v/v*, eluent A) and ethyl acetate (eluent B); the elution gradient was as follows: 0–25 min, 70% A and 30% B; 25–50 min, 45% A and 55% B; 50–60 min, 10% A and 90% B; 60–70 min, 70% A and 30% B. The identification of the carotenoids was performed by retention times comparison and ultraviolet spectra at 450 nm with the following reference compounds: astaxanthin, lutein, zeaxanthin, cryptoxanthin, echinenone and β-carotene. Quantification was based on external calibration using linear regression analysis.

2.4.3. Instrumental colour and texture

Instrumental colour and texture determinations were done in canned products; and colour was also determined in raw gonads.

Colour was measured using a colorimeter (Colour-eye 3000, Macbeth, New Windsor, NY, USA) at standard illuminant D65 and 10-degree observer, previously calibrated with a white standard tile. The L^*, a^* and b^* coordinates from CIELAB system were recorded. In this system, L^* values denote lightness on a scale of 0 (black) to 100 (white); the a^* values describe the intensity from green (−) to red (+); and the b^* values from blue (−) to yellow (+). The chroma (saturation; C^*) was determined according to the following equation (Schubring, 2009): $C^*_{a,b} = (a^{*2} + b^{*2})^{1/2}$. The colour difference was calculated through the Euclidean distance between two colour stimuli in CIELAB space (Schubring, 2009; Sharma & Bala, 2017): $\Delta E^*_{a,b} = [(L^* - L^*_s)^2 + (a^* - a^*_s)^2 + (b^* - b^*_s)^2]^{1/2}$. This colour distance was calculated after canning (raw gonads as standard product, L^*_s, a^*_s, b^*_s), the addition of macroalgae and ageing (control canned gonads as standard product, L^*_s, a^*_s, b^*_s).

Texture profile analysis (TPA, double compression test) was carried out on a TA.XTplus analyser and software (Stable Micro Systems, Surrey, UK) using a 5 kg load cell. The individualized gonads were compressed twice up to 50% of the original height with a cylindrical probe of 50 mm diameter (P50) and applying a constant speed of 1 $mm \cdot s^{-1}$. The time between compressions was 5 s. The primary characteristics (hardness, springiness and adhesiveness) were obtained.

2.4.4. Sensory evaluation

The sensory analysis was performed after 3 months of storage, and after confirmation of stability and sterility tests. The three canned products (Control, +Macroalgae and Aged) were assessed in a sensory laboratory, equipped according to international standard ISO (2007), through a quantitative descriptive method used by five trained panellists. The cans were open 15 min before testing and one or two gonads were presented to each panellist in white coded plates.

The panellists were asked to use a line scale (12 cm; with marks at the ends and in the middle: 0 – absent; 6 – moderate; 12 – extreme) to rate their perceived intensity of the selected attributes/descriptors (Meilgaard, Civille, & Carr, 2016): odour (typical of canned seafood, marine, algae, fresh, uncharacteristic/off odour), appearance (colour, apparent firmness, shape, graininess), flavour (typical of canned seafood, sweet, salty, acid, bitter, tropical), texture (firmness, succulence, adhesivity) and mouth sensations (astringency, sweetness, saltiness).

An acceptance test of the three products was also performed with 32 frequent consumers of canned seafood. The assessment was focused on appearance, odour, taste, texture and overall acceptance using a 5-point hedonic scale (0 – dislike very much, 1 – dislike slightly; 2 – neither like nor dislike; 3 – like slightly; 4 – like very much) (Meilgaard et al., 2016).

2.5. Nutritional contribution

The nutritional contribution (NC) of canned sea urchin gonads was calculated based on protein, eicosapentaenoic acid and docosahexaenoic acid (EPA + DHA), macro (Na and K) and micro (I and Se) elements, considering a portion of 25 g since this product is usually consumed as a side dish, appetizer or even used as an addition to give flavour and texture to several recipes (Baião et al., 2021; Sun & Chiang, 2015). The dietary reference values (DRVs) recommended by European Food Safety Authority (EFSA, 2017; EFSA Panel on NDA et al., 2016; EFSA Panel on NDA et al., 2019) were considered and the following formula was used:

$$NC\ (\%) = 100 \times (C \times M)/AI$$

where C = mean concentration of the nutrient (mg·kg^{-1}); M = weight of the defined portion (kg); and AI = adequate intake (mg·day^{-1}).

2.6. Statistical analysis

Data were tested for normality of distribution and homogeneity of variances using Kolmogorov–Smirnov and Levene's tests, respectively. The effect of canning, that is the comparison between raw and control canned gonads, on the analysed parameters was tested by the Student's *t*-test, with the exception of texture (due to insufficient amount of raw gonads). The impact of the macroalgae addition and ageing on the assessed parameters was tested by analysis of variance (one-way ANOVA), followed by the post-hoc one-sided Dunnett's test (+Macroalgae and Aged groups: lower or higher compared to Control group). Statistical analysis was performed using the STATISTICA™ software, version 12 (StatSoft Inc., Tulsa, OK, USA). Significance was considered at p-value<0.05 for all tests (Zar, 2014).

3. Results and discussion

3.1. Stability and sterility of canned gonads

External visual examination of the package did not reveal any physical damage or leaks, both in the canned products stored for 3 months at room temperature and in the aged ones (those that mimic a 12-month storage at room temperature). The appearance and odour of the sea urchin gonads were characteristic of a canned product. The differences in pH between cans maintained at the three different temperatures (≈20, 37 and 55 °C) were less than 0.5, which is line with the standard (NP 4404–1, 2002).

No microbial growth was observed under the conditions of the tests. Hence, the outcomes of the microbiological analysis were negative for thermophilic and mesophilic aerobes and anaerobes. Based on these results, it was considered that the products had commercial stability and sterility in the evaluated times *i.e.*, for at least 3 (canned gonads with macroalgae) and 12 months (canned gonads without macroalgae) after processing.

3.2. Nutritional quality

3.2.1. Proximate composition

Thermal processing techniques or culinary processes affect the composition of seafood products, leading to the loss of nutrients and breakdown of some constituents with subsequent interactions, depending on the intrinsic characteristics of the product, the methods used, as well as the exposure time and temperature (*e.g.* Aubourg, 2001; Oliveira et al., 2019).

The proximate composition of raw materials and canned products can be found in Table 1. In general, the values obtained for raw gonads are in agreement with those reported by other studies with wild *P. lividus* (Rocha et al., 2019).

The moisture content in fresh gonads was ≈73 g·(100 g)$^{-1}$, without significant differences (p = 0.334, *t*-Student test) after canning. Similarly, the moisture content of canned tuna prepared with water as filling medium was equivalent to that of the raw material, as observed by Mol, Alakavuk, and Tosun (2008). When oil is used as filling medium, the water loss can vary in the range of 9–28% (Aubourg, 2001).

On the other hand, total lipids, protein and ash contents were significantly influenced by processing (p < 0.019, *t*-Student test). The increase of total lipids may be due to the water loss stimulated by pre-cooking. Moreover, the extract presented an orange colour, suggesting that other compounds (pigments, such as carotenoids, which are fat soluble; Pither, 2003), were co-extracted and consequently the total lipids content may have been overquantified. The protein decrease can be due to solubilisation during pre-cooking, diffusion into filling medium and denaturation during thermal processing (Sampels, 2015). Regarding ash, the increase can be ascribed to the NaCl added to the light brine (Estevez et al., 2021).

Table 1
Proximate composition of raw materials (sea urchin gonads and macroalgae) and canned gonads.

Parameter (g·(100 g)$^{-1}$)	Raw materials		Canned sea urchin gonads		
	Gonads	Macroalgae[1]	Control[2]	+Macroalgae[2]	Aged[3]
Moisture	73.8 ± 0.5	9.5 ± 0.3	73.1 ± 0.9	72.8 ± 0.3	72.3 ± 0.3
Protein	17.0 ± 0.1*	36.7 ± 0.6	15.7 ± 0.6*$^{a;A}$	13.1 ± 0.1^{b}	14.7 ± 0.4^{B}
Total lipids	4.1 ± 0.2*	1.4 ± 0.2	5.1 ± 0.3*	4.7 ± 0.2	5.0 ± 0.4
Ash	2.5 ± 0.1*	10.2 ± 0.6	3.6 ± 0.1*^{a}	5.5 ± 0.0^{b}	3.7 ± 0.2
Carbohydrates[4]	2.6 ± 0.5	42.3 ± 1.0	3.1 ± 0.5^{a}	4.2 ± 0.3^{b}	3.8 ± 0.4

Results are given as mean values ± standard deviation (n = 3). For each parameter, values assigned with asterisk (effect of canning) indicate significant differences (*t*-Student test, $p \leq 0.018$); values assigned with different small (a-b) and capital (A-B) letters indicate significant lower or higher values than the control (Dunnett's one-side test, $p \leq 0.040$), in the case of +Macroalgae and aged canned gonads, respectively.
[1] Dried macroalgae;
[2] Three months of storage at ≈20 °C;
[3] Three months of storage at ≈40 °C;
[4] Calculated by difference.

Other studies have mentioned slight effects of sterilisation on chemical composition, such as higher moisture and lower levels of protein and lipids in canned roe of *P. lividus*, *Anthociadaris crassispina* and *Pseudocentrotus depressus* (Cruz-García et al., 2000; Lee et al., 2012). With steaming, the slight alterations observed are generally the opposite of canning (Lee et al., 2012).

Aged canned gonads differed significantly from the control products in terms of protein (lower content, p = 0.024, Dunnett's one-side test). This trend is certainly due to an increased solubilisation to the filling medium owed by the higher temperature during storage (Sampels, 2015).

The incorporation of macroalgae induced changes in protein, ash and carbohydrates levels (p < 0.024). The increase in ash and carbohydrates contents may be related to the addition of this ingredient, which is rich in such components (Table 1; Circuncisão, Catarino, Cardoso, & Silva, 2018). The lower protein value may be ascribed to a probable solubilisation of this macronutrient.

3.2.2. Fatty acids profile

In raw and canned gonads, 38 FA were identified (the main ones are shown in Supplementary Table 1). The PUFA were the most abundant (46–50%), followed by monounsaturated FA (MUFA, 27–29%) and saturated FA (SFA, 14–17%). Identical profiles of FA groups were found in the three canned products.

In raw gonads, EPA was the most abundant (355 mg·(100 g)$^{-1}$) within PUFA, followed by arachidonic acid (ARA, 314 mg·(100 g)$^{-1}$). The PUFA 16:3*n*-3, 18:4*n*-3, 20:3*n*-3 and 20:4*n*-3 were found at levels between 55 and 80 mg·(100 g)$^{-1}$ and the remaining below 29 mg·(100 g)$^{-1}$. The most abundant MUFA was 20:1*n*-9 (170 mg·(100 g)$^{-1}$), followed by 20:1*n*-11 (133 mg·(100 g)$^{-1}$). All other MUFA were found in levels equal to or lower than 101 mg·(100 g)$^{-1}$. Concerning SFA, 16:0 was the one with the highest concentration (177 mg·(100 g)$^{-1}$) in raw gonads. Such profile is in line with what has been reported by other authors, although in different proportions for some FA groups (Angioni & Addis, 2014; Volpe et al., 2018).

In general, the individual FA levels were not significantly influenced

by canning process and storage. However, some exceptions can be pointed out for SFA (18:0), MUFA (20:1*n*-11 and 22:1*n*-9), PUFA (22:6*n*-3, 20:2*n*-6, 20:4*n*-6) and Σ*n*-6 PUFA, with higher values being observed after processing ($p \leq 0.049$, *t*-Student test). Other study carried out with sea urchin gonads reported no significant changes or a significant decrease in some FA, specially SFA, after canning (Cruz-García et al., 2000). Other authors also did not report changes in FA concentrations due to heat processing in canned sardine, herring, mackerel (Hale & Brown, 1983) and tuna (Aubourg, Sotelo, & Gallardo, 1990).

Ageing did not induce significant changes ($p > 0.05$) in the concentrations of most FA, which is an indicator of their stabilisation up to a simulated 12-month storage period. Similarly, no significant differences ($p > 0.05$) were found between the +Macroalgae formulation and the control group. Such results can be attributed to the reduced amount of macroalgae added, which in turn contained very low levels of total lipids (Table 1), but high PUFA concentrations (Supplementary table 1). It is also noteworthy that the ratio Σ*n*-3/Σ*n*-6 (around 1.7) was not affected ($p > 0.05$) by canning, ageing and macroalgae addition.

3.2.3. Macro and microelements

Concentrations of macro and micronutrients are presented in Table 2. The mean Na and K contents of raw gonads were 270 and 430 mg·(100 g)$^{-1}$, respectively. The concentration of Na significantly increased 3.9-fold after canning ($p < 0.0001$, *t*-Student test) due to the salt (NaCl) addition in the preparation; while K significantly decreased ($p < 0.0001$, *t*-Student test).

Table 2
Concentrations of macro and microelements of raw materials (sea urchin gonads and macroalgae) and canned gonads.

Elements	Raw materials		Canned sea urchin gonads		
	Gonads	Macroalgae[1]	Control[2]	+Macroalgae[2]	Aged[3]
Macro, mg·(100 g)$^{-1}$					
K[4]	434.3 ± 25.8*	3899.4 ± 69.4	266.8 ± 21.6*	244.3 ± 34.7	292.6 ± 5.1
Na[5]	267.3 ± 22.6*	1262.0 ± 148.1	1050.7 ± 26.8*[a;A]	1427.5 ± 27.9[b]	1134.7 ± 19.70[B]
Micro, mg·kg^{-1}					
I[6]	2.1 ± 0.4	12.5 ± 0.4	2.9 ± 0.5	3.7 ± 0.6	2.1 ± 0.3
Se[7]	0.29 ± 0.03*	1.74 ± 0.25	0.84 ± 0.05*[a]	1.32 ± 0.10[b]	0.84 ± 0.18

Results are given as mean values ± standard deviation (n = 3); For each parameter, values assigned with asterisk (effect of canning) indicate significant differences (*t*-Student test, $p < 0.001$); values assigned with different small (a-b) and capital (A-B) letters indicate significant lower or higher values than the control (Dunnett's one-side test, $p \leq 0.005$), in the case of +Macroalgae and aged canned gonads, respectively.

[1] Dried macroalgae;

[2] Three months of storage at ≈20 °C;

[3] Three months of storage at ≈40 °C.

[4] Detection limit = 0.01 mg·kg^{-1}; Certified reference material = Dorm-4, fish protein (National Research Council of Canada, Canada); Certified value (average ± uncertainty) = 15500 ± 1000 mg·kg^{-1}; Present work (average ± standard deviation) = 14500 ± 495 mg·kg^{-1}; Certified reference material = LUTS-1, non-defatted lobster hepatopancreas (National Research Council of Canada, Canada); Certified value (average ± uncertainty) = 948 ± 72 mg·kg^{-1}; Present work (average ± standard deviation) = 791 ± 0 mg·kg^{-1}.

[5] Detection limit = 0.09 mg·kg^{-1}; Proficiency Test = FAPAS Test 01120, Nutritional Components in Canned Meat, January–March 2018 (Fera Science Ltd., York, UK); Certified value (average ± uncertainty) = 0.60 ± 0.03 mg·kg^{-1}; Present work (average ± standard deviation) = 0.55 ± 0.02 mg·kg^{-1}.

[6] Detection limit = 0.07 mg·g^{-1}; Certified reference material = ERM®-BB422, fish muscle (Joint Research Centre (JRC), Brussels); Certified value (average ± uncertainty) = 1.40 ± 0.40 mg·g^{-1}; Present work (average ± standard deviation) = 1.10 ± 0.04 mg·g^{-1} (79 % recovery).

[7] Detection limit = 0.03 mg·g^{-1}; Certified reference material = ERM®-BB422, Fish muscle (Joint Research Centre (JRC), Brussels); Certified value (average ± uncertainty) = 1.33 ± 0.13 mg·g^{-1}; Present work (average ± standard deviation) = 1.33 ± 0.18 mg·g^{-1} (105 % recovery).

The macroelements fluctuations in canned products are generally caused by interactions between the foodstuff and the filling medium (Pither, 2003). In the case of Na, there was an uptake from the light brine, and in the case of K, a leaching of about 38% to the filling medium. K is particularly susceptible to leaching in canned products with losses between 15 and 50% in the case of canned vegetables (Pither, 2003). Both trends, *i.e.*, increases and decreases in some macroelements (Na, K, calcium (Ca), and copper (Cu)) contents, were also reported in seasoned and canned gonads from three different sea urchin species (Lee et al., 2012). The addition of macroalgae led to a significant increase of the Na content ($p < 0.0001$, Dunnett's one-sided test), while K level remained identical to that found in the control product. Ageing induced a similar trend on the content of these elements. It is important to mention that the Na/K ratio in canned products was above the reference threshold recommended by World Health Organization (WHO; <1) for maintaining cardiovascular health (Whelton, 2014). Thus, such products should be consumed with moderation and within a balanced diet.

The concentrations of I and Se in the raw gonads were 2.1 and 0.29 mg·kg^{-1}, respectively. A significant effect of canning was registered for Se content ($p < 0.0001$, *t*-Student test); higher concentration in the control gonads compared to the raw ones. In the case of I, significant differences were not found between groups ($p > 0.05$). Previous studies suggest a primary binding of Se and I to proteins (Hou, 2009; Vicente-Zurdo, Gómez-Gómez, Pérez-Corona, & Madrid, 2019), which can make these elements less susceptible to be leached during processing (including that using heat). Additionally, higher I and Se contents after steaming and boiling were reported by other authors, who linked such trends to a moisture loss (Oliveira et al., 2019), a change not observed in the present study.

On the other hand, the contents of I and Se remained stable during ageing ($p > 0.05$), since the aged products presented concentrations in the same range of the values observed in the control (≈2.1 mg·kg^{-1} and 0.84 mg·kg^{-1}, respectively). Therefore, the present study demonstrated that canning and ageing has no detrimental effect on the I and Se naturally present in the gonads.

The addition of the macroalgae (+Macroalgae group) led to a significantly higher concentration of Se in comparison to that found in control canned gonads (about 50%) ($p = 0.0024$; Dunnett's one-sided test). The I content found in the +Macroalgae group was higher, but not significantly different ($p = 0.0829$, Dunnett's one-sided test) from that observed in the control formulation.

3.2.4. Nutritional contribution

Canning can affect the nutritional value of seafood depending on the filling medium (Sampels, 2015). In the case of canned gonads in light brine, the protein contribution can be almost 10% of the daily average requirements (AR) for adults with a 25 g serving (Table 3), which constitutes a valuable level for tissue maintenance (EFSA, 2017). In the same portion, the levels of *n*-3 FA are quite relevant in all canned products, since consumption provides between 47% (+Macroalgae group) to 53% (Aged group) of the daily adequate intake (AI) of EPA + DHA for adults (Table 3), an important contribution in the prevention of cardiovascular diseases (Sheppard & Cheatham, 2018). Thus, products prepared with sea urchin roe can be considered an additional and interesting source of PUFA.

Regarding macroelements, the contribution is ≈2% and ≈15% of the daily AI of K and Na, respectively, for adults (Table 3), corresponding to a very low amount in relation to the limit of 5 g NaCl (equivalent to a 2 g of Na) recommended by EFSA Panel on NDA et al., 2019).

A higher NC was found specifically for I (between 35 and 62% in Aged and + Macroalgae groups, respectively). The consumption of a 25 g dose of control and Aged canned gonads contributes to approximately 30% of the daily AI of Se for adults. The formulation with macroalgae

Table 3
Nutritional contribution (%) of canned sea urchin gonads, considering a portion of 25 g.

	DRVs[1] (mg·day^{-1})	Canned sea urchin gonads		
		Control[2]	+Macroalgae[2]	Aged[3]
Proximate composition				
Protein	41778[4,*] (AR)	9.4 ± 0.4^{a,A}	7.8 ± 0.1^{b}	8.8 ± 0.3^{B}
n-3 fatty acids				
EPA + DHA	250[4] (AI)	48.4 ± 5.7	47.2 ± 2.7	52.6 ± 4.9
Macroelements				
K	3500[4] (AI)	1.9 ± 0.2	1.7 ± 0.3	2.1 ± 0.1
Na	2000[4,5] (AI)	13.1 ± 0.3^{a,A}	17.8 ± 0.3^{b}	14.2 ± 0.2^{B}
Microelements				
I	0.15[4] (AI)	49.0 ± 8.4	61.6 ± 9.6	34.7 ± 4.8
Se	0.07[4] (AI)	30.1 ± 1.7^{a}	47.1 ± 3.4^{b}	30.1 ± 6.2

Values are means±standard deviation (n = 3). For each parameter, values assigned with different small (a-b) and capital (A-B) letters indicate significant lower or higher values than the control (Dunnett's one-side test, $p \leq 0.024$), in the case of +Macroalgae and aged canned gonads, respectively.
[1] The Dietary Reference Values (DRVs) given as average requirements (AR) or adequate intakes (AI) are presented for adults (women/men);
[2] Three months of storage at ≈20 °C;
[3] Three months of storage at ≈40 °C;
[4] EFSA (2017);
[5] EFSA (2019);
[*] Reference body weight for adults (63.3 kg, mean of the values for males and females; EFSA, 2017) was considered to calculate DRV.

Table 4
Carotenoids concentrations of raw materials (sea urchin gonads and macroalgae) and canned gonads.

Carotenoids (μg·g^{-1})	Raw materials		Canned sea urchin gonads		
	Gonads	Macroalgae[1]	Control[2]	+Macroalgae[2]	Aged[3]
Echinenone[4]	51.3 ± 2.9*	n.d.	27.3 ± 3.0$^{*\ a}$	41.8 ± 0.9^{b}	26.4 ± 8.0
β-carotene[5]	7.8 ± 1.1*	15.1 ± 3.4	4.5 ± 0.4$^{*a;A}$	8.8 ± 0.9^{b}	<3.3 (LQ)^{B}
Lutein[6]	1.6 ± 0.7	n.d.	0.5 ± 0.1$^{a;A}$	2.4 ± 0.2^{b}	1.2 ± 0.4^{B}
Cryptoxanthin[7]	2.3 ± 0.3*	n.d.	1.1 ± 0.1*	0.9 ± 0.1	1.0 ± 0.0
Zeaxanthin[8]	n.d.	11.6 ± 1.9	n.d.	n.d.	n.d.

Results are given as mean values±standard deviation (n = 3). For each parameter, values assigned with asterisk (effect of canning) indicate significant differences (*t*-Student test, $p \leq 0.008$); values assigned with different small (a-b) and capital (A-B) letters indicate significant lower or higher values than the control (Dunnett's one-side test, $p \leq 0.043$), in the case of +Macroalgae and aged canned gonads, respectively. Abbreviations: n.d. – not detected; LQ – quantification limit.
[1] Dried macroalgae;
[2] Three months of storage at ≈20 °C;
[3] Three months of storage at ≈40 °C.
[4] Detection and quantification limits = 1.9 and 5.9 mg·g^{-1}, respectively.
[5] Detection and quantification limits = 1.1 and 3.3 mg·g^{-1}, respectively.
[6] Detection and quantification limits = 3×10-3 and 9×10-3 mg·g^{-1}, respectively.
[7] Detection and quantification limits = 0.1 and 0.3 mg·g^{-1}, respectively.
[8] Detection and quantification limits = 0.2 and 1.1 mg·g^{-1}.

can provide up to about 47% of such intake (Table 3). The health effects associated with the consumption of foods rich in I (such as canned gonads) are related to the structural and functional tasks of the thyroid hormones. Selenium is essential in the protection against organs degeneration (EFSA, 2017).

All Se and I values obtained in a 25 g portion of canned products (Table 3) also reached >30% of the nutrient reference value (NRV) (70 and 150 μg)·day^{-1}, respectively, defined by EFSA (2017). Hence, these data support the claim that these products are "high in selenium" and "high in iodine" (European Parliament, 2006, 2011). Thus, these products can be valuable options for target groups that have special Se and I needs (*e.g.*, pregnant women for increased thyroid hormone production to cover maternal and foetal needs; EFSA, 2017). Moreover, these products had I levels (Table 2) far below the Upper Limit (600 μg·day^{-1}) defined for adults including pregnant and lactating women (EFSA, 2006).

3.2.5. Carotenoids

Carotenoids are pigments with great importance in foods since colour is one of the first quality criteria and can also play an important role as antioxidants (*e.g.* Baião et al., 2021; Rocha et al., 2019). Their levels in raw materials and canned products can be found in Table 4. Four pigments were identified in the gonads: echinenone, β-carotene, lutein and cryptoxanthin.

In the raw gonads, echinenone was the predominant carotenoid (51.3 μg·g^{-1}), followed by β-carotene (7.8 μg·g^{-1}), cryptoxanthin (2.3 μg·g^{-1}) and lutein (1.6 μg·g^{-1}). Zeaxanthin was only found in the macroalgae. The dominance of echinenone is in accordance with what was previously reported for the *P. lividus* species (Rocha et al., 2019; Symonds, Kelly, Caris-Veyrat, & Young, 2007). Other pigments such as echinochrome A, a quinone with pharmaceutical properties, can also be found in sea urchins (Rubilar, Barbieri, Gazquez, & Avaro, 2021).

Canned gonads (Control group) exhibited lower carotenoid's levels (echinenone, β-carotene and cryptoxanthin; $p < 0.008$, *t*-Student test) than the raw gonads, except in the case of lutein ($p = 0.061$, *t*-Student test). Similarly, De Quirós, López-Hernández, and Simal-Lozano (2001), registered a 20–30% decrease in the carotenoids levels (except for echinenone) after canning. This reduction was expected since these compounds are typically damaged, oxidised or even susceptible to isomerisation through heat processing and low pH, as is the case of canning (Pither, 2003).

Ageing had a significant effect on β-carotene and lutein levels ($p < 0.008$, Dunnett's one-sided test). In the case of lutein, such change (*i.e.*, increase) can be attributed to the intrinsic variability of the raw materials. The decrease in β-carotene may be due to the displacement of the equilibrium between its cis and trans forms, which can be easily isomerized into cis-isomers, the less stable form (Khoo, Prasad, Kong, Jiang, & Ismail, 2011).

The addition of macroalgae seems to contribute to the slowdown of carotenoids degradation induced by heat treatment. The carotenoids concentrations (except in the case of cryptoxanthin) were significantly higher in the +Macroalgae group in relation to control ($p < 0.044$, Dunnett's one-sided test). This result can be attributed to the great antioxidant activity of *Porphyra spp.* (Supawong, Park, & Park, 2022).

3.3. Physical quality: colour and texture

The colour and texture parameters of raw and canned products are shown in Table 5.

Colour parameters (L^*-lightness; a^*-redness; b^*-yellowness) and chroma ($C^*_{a,b}$) were not significantly affected by canning ($p > 0.085$, *t*-Student test), which was unexpected since the compounds strictly linked to colour *i.e.*, the carotenoid pigments, were deteriorated due to heat processing (section 3.6), a quality change usually associated with product discoloration (Pither, 2003). Still, the Euclidean distance showed a value of 1.9 between the raw and canned gonads, which is a small difference related to heat processing. Additionally, it is known that the colour can be also affected by variability in the shape and size of the gonads (Schubring, 2009).

The colour of the canned gonads was not influenced by ageing ($p > 0.085$ for L^*, a^*, b^* and C^*, Dunnett's one-sided test), with a small colour difference of 1.0. On the other hand, the macroalgae addition

Table 5
Colour and texture of raw and canned sea urchin gonads.

Parameter	Raw material (gonads)	Canned sea urchin gonads		
		Control[1]	+Macroalgae[1]	Aged[2]
Colour				
L^*	64.3 ± 1.5	64.5 ± 0.2[a]	62.8 ± 0.2[b]	64.6 ± 0.4
a^*	5.4 ± 0.6	6.7 ± 0.7[a]	5.1 ± 0.6[b]	7.3 ± 0.4
b^*	9.2 ± 0.7	9.6 ± 0.5[a]	8.3 ± 0.8[b]	9.6 ± 0.2
Chroma ($C^*_{a,b}$)	10.7 ± 0.9	11.7 ± 0.8[a]	9.7 ± 1.0[b]	12.1 ± 0.2
$\Delta E^*_{a,b}$ (colour difference)	–	1.9 ± 0.4[#]	3.2 ± 0.6[$]	1.0 ± 0.4[¥]
Texture[3]				
Hardness (N)	–	0.78 ± 0.19[a]	1.12 ± 0.37[b]	0.56 ± 0.18
Adhesiveness ($g \cdot cm^{-1}$)	–	−0.004 ± 0.001	−0.010 ± 0.004	−0.006 ± 0.003
Springiness (%)	–	72.6 ± 6.4	75.3 ± 3.1	69.2 ± 5.0

Results are given as mean values±standard deviation (n = 3 for colour; $n = 9$ for texture). For colour parameters, no significant differences were found between raw and control canned gonads (t-Student test, $p \geq 0.085$; effect of canning). For each parameter, values assigned with different small letters (a-b) indicate significant lower or higher values than the control (Dunnett's one-side test, $p \leq 0.023$) in the case of +Macroalgae canned gonads; no significant differences were found between control and aged canned gonads (Dunnett's one-side test, $p > 0.683$).

[1] Three months of storage at ≈20 °C;
[2] Three months of storage at ≈40 °C;
[3] It was not possible to evaluate the texture parameters in the raw material;
[#] small difference between raw and control canned gonads;
[$] noticeable difference between control and + Macroalgae groups;
[¥] small difference between control and aged groups.

significantly changed all the colour parameters analysed ($p < 0.022$, Dunnett's one-sided test). Specifically, the L^*, a^* and b^* values decreased, turning the gonads less bright, which promoted a reduction in the colour saturation. The colour difference was 3.2, which falls within the criteria of a noticeable colour difference (Sharma & Bala, 2017) between control and + Macroalgae groups.

It was not possible to evaluate the texture properties of raw gonads but indeed, the texture of seafood is susceptible to be affected by sterilisation (Pither, 2003; Sampels, 2015). For instance, Hui et al. (2006) mentioned that the oxidation of proteins that may occur after a thermal process can cause protein cross-linking and thus tissue toughening. However, only few studies reported colour and texture changes due to processing of roe products (Farag, Abib, Tawfik, Shafik, & Khattab, 2021).

The texture of canned gonads was not influenced by ageing ($p > 0.08$). In turn, the hardness was slightly affected by macroalgae addition ($p = 0.023$), which can be related to a possible interaction of some compounds presented in macroalgae with the surface of the gonads during the heat treatment. Moreover, *Porphyra spp.* has high swelling and water holding capacity when in the flakes form (Supawong et al., 2022) and consequently, it may affect the textural properties of processed foods when added as an ingredient.

3.4. Sensory quality

The main results of the descriptive test performed with the canned products are presented in Fig. 1.

The panel identified the three products as having a moderate typical odour of canned food (5.5–6.4 cm); but almost absent in what regards sea and algae odours (≈1 cm). In terms of visual evaluation, the macroalgae addition affected the appearance of gonads, making the halves distinction (slight-moderate), graininess (moderate) and firmness (moderate-strong) of product significantly lower in relation to control

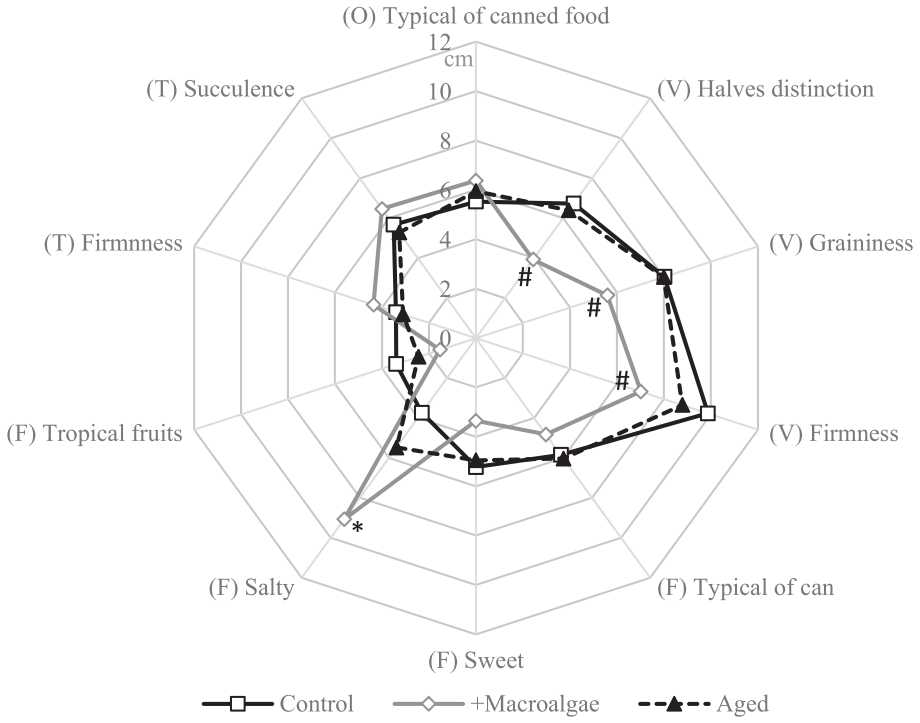

Fig. 1. Sensory profile of canned sea urchin gonads.
Results correspond to mean values ($0.1 \leq SD \leq 2.0$; $N = 5$ assessors); 12-cm scale was used: 0-absent, 6-moderate, 12-extreme; (O)-odour; (V)-visual assessment; (F)-flavour/taste; (T)-texture in the mouth; *indicates significant higher value in relation to Control ($p = 0.0002$; Dunnett's one-sided test); #indicates significant lower value in comparison to Control ($p < 0.0218$; Dunnett's one-sided test). Tests performed after 3 months of storage at ≈20 °C (Control and + Macroalgae groups) and 40 °C (Aged group).

(intensity: moderate-strong or strong-extreme) ($p < 0.0218$; Dunnett's one-sided test). The control gonads were scored as having a slight to almost moderate sweet taste (≈5 cm), which remained during ageing. The macroalgae addition did not significantly affected the sweet taste intensity. Concerning salty taste, the classifications given were 3.7 (slight-moderate) and 5.5 cm (almost moderate) in the control and aged gonads, respectively. In turn, the macroalgae addition induced a significant increase of the salty taste compared to control ($p = 0.0002$; Dunnett's one-sided test), *i.e.*, from slight-moderate (3.7) to strong (≈9 cm). Indeed, the Na content found in the formulation with macroalgae was higher than that obtained in the control canned gonads (Table 2).

Both macroalgae addition and ageing did not significantly influence the perception of other flavours (typical of can - slight-moderate or moderate; and tropical fruits - absent-slight) and texture in the mouth, perceived as slight or slight-moderate firm and slight-moderate or moderate succulent. The astringency was absent in all canned gonads. The off-odours/off-flavours were negligible.

The acceptance test evidenced a slightly lower hedonic rate to the appearance of gonads with macroalgae compared to the control formulation ($p = 0.0160$, Dunnett's one-sided test) (Table 6).

In general, the products were well accepted (Table 6), although the formulation with macroalgae can be refined in future studies to improve certain attributes, particularly the appearance and salty taste.

4. Conclusion

The results show that canning (without macroalgae addition) did not significantly affect the levels of moisture, carbohydrates, most FA, I and lutein, nor the colour of gonads. Nonetheless, there was a reduction in the contents of protein, K and most carotenoids (echinenone, β-carotene and cryptoxanthin), as well as an increase in the levels of total lipids, ash, Na and Se. This product was commercially stable and sterile for up to 12 months (evaluated by accelerated ageing), with few changes in the nutritional profile (higher Na content; lower 16:3*n*-3 and β-carotene levels). The addition of macroalgae led to a decrease in 16:3*n*-3 content and, on the other hand, an increase in carbohydrates, Na, Se and carotenoids levels (echinenone, β-carotene and lutein). This prototype still needs slight adjustment to improve the colour and texture, reduce the Na content and consequently decrease the intensity of the salty taste. Despite the reduction of some constituents, the consumption of a 25 g portion of any of the canned sea urchin gonads contributes with more than 30% of the daily requirements of EPA + DHA, I and Se that are beneficial to adult health.

In summary, canning is an approach of valorising gonads that are less uniform in terms of size and intrinsic characteristics. At the same time, it is an alternative to satisfy demand from consumers who do not appreciate raw gonads. In addition, canning also contributes to make available sea urchin gonads all year round, circumventing the disadvantage of the seasonality of fresh resource, without major losses in its nutritional value.

CRediT authorship contribution statement

Carolina Camacho: Writing – review & editing, Writing – original draft, Visualization, Validation, Investigation, Formal analysis, Data curation. **Helena Oliveira:** Writing – review & editing, Writing – original draft, Visualization, Validation, Formal analysis. **Carmo Serrano:** Writing – review & editing, Investigation. **Inês Coelho:** Writing – review & editing, Investigation. **Sónia Pedro:** Writing – review & editing, Investigation. **Helena Lourenço:** Writing – review & editing, Investigation. **Narcisa M. Bandarra:** Writing – review & editing. **António Marques:** Writing – review & editing, Funding acquisition. **M. Fernanda Pessoa:** Writing – review & editing. **Amparo Gonçalves:** Writing – review & editing, Validation, Supervision, Conceptualization. **M. Leonor Nunes:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization.

Table 6
Acceptance test of canned sea urchin gonads.

Attributes	Canned sea urchin gonads		
	Control[1]	+Macroalgae[1]	Aged[2]
Appearance	3.7 ± 0.8[a]	3.2 ± 1.0[b]	3.5 ± 0.8
Odour	3.7 ± 0.7	3.3 ± 1.0	3.6 ± 0.6
Flavour	3.7 ± 1.0	3.3 ± 1.0	3.7 ± 1.0
Texture	3.7 ± 0.9	3.6 ± 1.0	3.5 ± 0.9
Overall acceptance	3.6 ± 0.9	3.3 ± 1.0	3.7 ± 1.0

Results are given as mean values±standard deviation ($N = 32$ people, frequent consumers of canned seafood); values assigned with different small letters (a-b) indicate significant lower values than the control ($p = 0.0160$, Dunnett's one-sided test) in the case of +Macroalgae canned gonads; no significant differences were found between control and aged canned gonads (Dunnett's one-side test, $p > 0.377$). Hedonic scale (0 – dislike very much, 1 – dislike slightly; 2 – neither like nor dislike; 3 – like slightly; 4 – like very much.
[1] Three months of storage at ≈20 °C;
[2] Three months of storage at ≈40 °C.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2024.139184.

References

Angioni, A., & Addis, P. (2014). Characterization of the lipid fraction of wild sea urchin from the Sardinian Sea (western Mediterranean). *Journal of Food Science, 79*(2), C155–C162. https://doi.org/10.1111/1750-3841.12330

AOAC. (2005). *Official methods of analysis of the Association of Official Analytical Communities International* (18th ed.) Washington DC.

Aubourg, S. P. (2001). Loss of quality during the manufacture of canned fish products. *Food Science and Technology International, 7*(3), 199–215. https://doi.org/10.1106/4H8U-9GAD-VMG0-3GLR

Aubourg, S. P., Sotelo, C. G., & Gallardo, J. M. (1990). Changes in flesh lipids and fill oils of albacore (*Thunnus alalunga*) during canning and storage. *Journal of Agricultural and Food Chemistry, 38*(3), 809–812. https://doi.org/10.1021/jf00093a047

Baião, L. F., Moura, A. P., Rocha, C., Valente, L. M. P., & Cunha, L. M. (2021). Dimensions for the valorisation of sea urchin (*Paracentrotus lividus*) gonads production through the eyes of experienced chefs. *International Journal of Gastronomy and Food Science, 26*, Article 100438. https://doi.org/10.1016/j.ijgfs.2021.100438

Bandarra, N. M., Batista, I., Nunes, M. L., Empis, J. M., & Christie, W. W. (1997). Seasonal changes in lipid composition of sardine (*Sardina pilchardus*). *Journal of Food Science, 62*(1), 40–42. https://doi.org/10.1111/j.1365-2621.1997.tb04364.x

Boudouresque, C. F., & Verlaque, M. (2013). Paracentrotus lividus. In J. M. Lawrence (Ed.), *Vol. 38. Sea urchins: Biology and ecology (third)* (pp. 297–327). Tampa, Florida https://doi.org/10.1016/B978-0-12-396491-5.00021-6.

CEN. (2007). *European Standard EN 15111 - Foodstuffs - Determination of trace elements - Determination of iodine by ICP-MS (inductively coupled plasma mass spectrometry)*.

CEN. (2009). *European Standard EN 15763 - Foodstuffs - Determination of trace elements - Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion*.

Circuncisão, A. R., Catarino, M. D., Cardoso, S. M., & Silva, A. M. S. (2018). Minerals from macroalgae origin: Health benefits and risks for consumers. *Marine Drugs, 16* (11), 400. https://doi.org/10.3390/md16110400

Coelho, I., Matos, A. S., Teixeira, R., Nascimento, A., Bordado, J., Donard, O., & Castanheira, I. (2019). Combining multielement analysis and chemometrics to trace the geographical origin of Rocha pear. *Journal of Food Composition and Analysis, 77*, 1–8. https://doi.org/10.1016/j.jfca.2018.12.005

Cruz-García, C., López-Hernández, J., González-Castro, M. J., Rodríguez-Bernaldo De Quirós, A. I., & Simal-Lozano, J. (2000). Protein, amino acid and fatty acid contents in raw and canned sea urchin (*Paracentrotus lividus*) harvested in Galicia (NW Spain). *Journal of the Science of Food and Agriculture, 80*(8), 1189–1192. https://doi.org/10.1002/1097-0010(200006)80:8<1189::AID-JSFA618>3.0.CO;2-7

De Quirós, A. R.-B., López-Hernández, J., González-Castro, M., De la Cruz-Garcia, C., & Simal-Lozano, J. (2001). Comparison of volatile components in fresh and canned sea urchin (*Paracentrotus lividus*, Lamarck) gonads by GC–MS using dynamic headspace sampling and microwave desorption. *European Food Research and Technology, 212*(6), 643–647. https://doi.org/10.1007/s002170100315

De Quirós, A. R.-B., López-Hernández, J., & Simal-Lozano, J. (2001). Determination of carotenoids and liposoluble vitamins in sea urchin (*Paracentrotus lividus*) by high performance liquid chromatography. *European Food Research and Technology, 212* (6), 687–690. https://doi.org/10.1007/s002170100316

Delgado, I., Ventura, M., Gueifão, S., Coelho, I., Nascimento, A. C., Silva, J. A. L., & Castanheira, I. (2019). 12th IFDC 2017 special issue–iodine, selenium and iron contents in Portuguese key foods as consumed. *Journal of Food Composition and Analysis, 79*, 39–46. https://doi.org/10.1016/j.jfca.2019.03.004

EFSA. (2006). Tolerable upper intake levels for vitamins and minerals. Scientific Panel on Dietetic Products, Nutrition and Allergies. Parma, Italy. Retrieved from https://www.efsa.europa.eu/sites/default/files/efsa_rep/blobserver_assets/ndatolerableuil.pdf.

EFSA. (2017). *Dietary reference values for nutrients. Summary report. e15121*. EFSA Supporting Publication. https://doi.org/10.2903/sp.efsa.2017.e15121

EFSA Panel on NDA, Turck, D., Bresson, J.-L., Burlingame, B., Dean, T., Fairweather-Tait, S., … Naska, A. (2016). Dietary reference values for potassium. *EFSA Journal, 14*(10), 4592. https://doi.org/10.2903/j.efsa.2016.4592

EFSA Panel on NDA, Turck, D., Castenmiller, J., Henauw, S., Hirsch-Ernst, K.-I., Kearney, J., … Naska, A. (2019). Dietary reference values for sodium. *EFSA Journal, 17*(9), 5778. https://doi.org/10.2903/j.efsa.2019.5778

Estevez, A., Camacho, C., Correia, T., Barbosa, V., Marques, A., Lourenco, H., … Pires, C. (2021). Strategies to reduce sodium levels in European seabass sausages. *Food and Chemical Toxicology, 153*, Article 112262. https://doi.org/10.1016/j.fct.2021.112262

European Parliament. (2006). Regulation (EC) no 1924/2006 of the European Parliament and of the council of 20 December 2006 on nutrition and health claims made on foods. *Official Journal of the European Union, 404*(9), 9–25.

European Parliament. (2011). Regulation (EU) no 1169/2011 of the European Parliament and of the council of 25 October 2011 on the provision of food information to consumers, amending regulations (EC) no 1924/2006 and (EC) no 1925/2006 of the European Parliament and of the council. *An Official Journal of the European Union, 20*(L304), 168–213.

FAO. (2003). Food energy - methods of analysis and conversion factors. In *, Vol. 77. FAO food and nutrition paper*. Rome. Retrieved from http://www.fao.org/uploads/media/FAO_2003_Food_Energy_02.pdf.

Farag, M. A., Abib, B., Tawfik, S., Shafik, N., & Khattab, A. R. (2021). Caviar and fish roe substitutes: Current status of their nutritive value, bio-chemical diversity, authenticity and quality control methods with future perspectives. *Trends in Food Science & Technology, 110*, 405–417. https://doi.org/10.1016/j.tifs.2021.02.015

Folch, J., Lees, M., & Stanley, G. H. S. (1957). A simple method for the isolation and purification of total lipides from animal tissues. *Journal of Biological Chemistry, 226* (1), 497–509. https://doi.org/10.1016/S0021-9258(18)64849-5

Hale, M. B., & Brown, T. (1983). Fatty acids and lipid classes of three underutilized species and changes due to canning. *Marine Fisheries Review, 45*(4–6), 45–48.

Hall, G. M. (2011). Canning fish and fish products. In G. M. Hall (Ed.), *Fish processing - sustainability and new opportunities* (pp. 30–50). Wiley Online Library.

Hou, X. (2009). Iodine speciation in foodstuffs, tissues, and environmental samples: Iodine species and analytical method. In V. R. Preedy, G. N. Burrow, & R. Watson (Eds.), *Comprehensive handbook of iodine* (pp. 139–150). Academic Press.

Hui, Y. H., Cross, N., Kristinsson, H. G., Lim, M. H., Nip, W. K., Siow, L. F., & Stanfield, P. S. (2006). Biochemistry of seafood processing. In Y. H. Hui (Ed.), *Food biochemistry and food processing* (pp. 350–378). Blackwell Publishing.

ISO. (2007). International standard 8589. Sensory analysis - general guidance for the design of test rooms. In *International standard ISO*. Genève: International Organization for Standardization.

Jorhem, L. (2000). Determination of metals in foods by atomic absorption spectrometry after dry ashing: NMKL1 collaborative study. *Journal of AOAC International, 83*(5), 1204–1211. https://doi.org/10.1093/jaoac/83.5.1204

Khoo, H.-E., Prasad, K. N., Kong, K.-W., Jiang, Y., & Ismail, A. (2011). Carotenoids and their isomers: Color pigments in fruits and vegetables. *Molecules, 16*(2), 1710–1738. https://doi.org/10.3390/molecules16021710

Labuza, T. P., & Fu, B. (1993). Growth kinetics for shelf-life prediction: Theory and practice. *Journal of Industrial Microbiology, 12*, 309–323.

Lee, S.-U., Lee, H.-Y., Kim, S.-H., & Kim, D.-J. (2012). Changes in physicochemical composition of sea urchin roe by steaming treatment. *Journal of the Korean Society of Food Science and Nutrition, 41*(4), 550–560. https://doi.org/10.3746/jkfn.2012.41.4.550

Lepage, G., & Roy, C. C. (1986). Direct transesterification of all classes of lipids in a one-step reaction. *Journal of Lipid Research, 27*(1), 114–120. https://doi.org/10.1016/S0022-2275(20)38861-1

Meilgaard, M. C., Civille, G. V., & Carr, B. T. (2016). *Sensory evaluation techniques* (5th ed.). Boca Raton: CRC Press.

Mol, S., Alakavuk, D., & Tosun, Ş. Y. (2008). Effects of different processing technologies on the chemical composition of seafoods. *Food Science and Technology Research, 14* (5), 467. https://doi.org/10.3136/fstr.14.467

Monfort, M. C. (2002). *Fish roe in Europe: Supply and demand conditions*. Rome, FAO: Food and Agriculture Organization of the United Nations, GLOBEFISH, Fishery ….

NP 2309—2. (1988). *Microbiologia Alimentar: Conservas. Parte 2: Apreciação da esterilidade*.

NP 4404—1. (2002). *Microbiologia Alimentar: Conservas. Parte 1: Apreciação da estabilidade, provas de estufa (método corrente)*.

Oliveira, H., Muniz, J. A., Bandarra, N. M., Castanheira, I., Coelho, I. R., Delgado, I., … Duarte, M. P. (2019). Effects of industrial boiling on the nutritional profile of common octopus (*Octopus vulgaris*). *Foods, 8*(9), 411. https://doi.org/10.3390/foods8090411

Pither, R. J. (2003). *Canning| quality changes during canning* (pp. 845–851). Elsevier.

Rocha, F., Rocha, A. C., Baião, L. F., Gadelha, J., Camacho, C., Carvalho, M. L., … Valente, L. M. P. (2019). Seasonal effect in nutritional quality and safety of the wild sea urchin *Paracentrotus lividus* harvested in the European Atlantic shores. *Food Chemistry, 282*, 84–94. https://doi.org/10.1016/j.foodchem.2018.12.097

Rubilar, T., Barbieri, E. S., Gazquez, A., & Avaro, M. (2021). Sea urchin pigments: Echinochrome a and its potential implication in the cytokine storm syndrome. *Marine Drugs, 19*(5), 267. https://doi.org/10.3390/md19050267

Saint-Denis, T., & Goupy, J. (2004). Optimization of a nitrogen analyser based on the dumas method. *Analytica Chimica Acta, 515*(1), 191–198. https://doi.org/10.1016/j.aca.2003.10.090

Sampels, S. (2015). The effects of processing technologies and preparation on the final quality of fish products. *Trends in Food Science & Technology, 44*(2), 131–146. https://doi.org/10.1016/j.tifs.2015.04.003

Schubring, R. (2009). Colour measurement. In H. Rehbein, & J. Oehlenschläger (Eds.), *Fishery products: Quality, safety and authenticity* (pp. 127–172). Wiley Blackwell.

Serrano, C., Carbas, B., Castanho, A., Soares, A., Patto, M. C. V., & Brites, C. (2017). Characterisation of nutritional quality traits of a chickpea (*Cicer arietinum*) germplasm collection exploited in chickpea breeding in Europe. *Crop and Pasture Science, 68*(11), 1031–1040. https://doi.org/10.1071/CP17129

Sharma, G., & Bala, R. (2017). *Digital Color Imaging Handbook*. Webster, New York: CRC Press.

Sheppard, K. W., & Cheatham, C. L. (2018). Omega-6/omega-3 fatty acid intake of children and older adults in the US: Dietary intake in comparison to current dietary recommendations and the healthy eating index. *Lipids in Health and Disease, 17*(1), 1–12. https://doi.org/10.1186/s12944-018-0693-9

Sun, J., & Chiang, F. (2015). Use and exploitation of sea urchins. In N. Brown, & S. Eddy (Eds.), *Echinoderm Aquaculture* (pp. 25–45). New Jersey: John Wiley and Sons, Inc.

Supawong, S., Park, J. W., & Park, J. (2022). Functional and chemical properties of Gim (*Porphyra yezoensis*) as affected by the product form. *Journal of Aquatic Food Product Technology, 31*(5), 418–429. https://doi.org/10.1080/10498850.2022.2060053

Symonds, R. C., Kelly, M. S., Caris-Veyrat, C., & Young, A. J. (2007). Carotenoids in the sea urchin *Paracentrotus lividus*: Occurrence of 9′-cis-echinenone as the dominant carotenoid in gonad colour determination. *Comparative Biochemistry and Physiology Part B: Biochemistry and Molecular Biology, 148*(4), 432–444. https://doi.org/10.1016/j.cbpb.2007.07.012

Tillocca, M. G. (2016). *Tecnologia di produzione della Polpa di Riccio di mare della specie Paracentrotus lividus e controlli sanitari*. Università di Pisa. Retrieved from. https://core.ac.uk/download/pdf/79622289.pdf.

Vicente-Zurdo, D., Gómez-Gómez, B., Pérez-Corona, M. T., & Madrid, Y. (2019). Impact of fish growing conditions and cooking methods on selenium species in swordfish and salmon fillets. *Journal of Food Composition and Analysis, 83*, Article 103275. https://doi.org/10.1016/j.jfca.2019.103275

Volpe, M. G., Fabbrocini, A., Siano, F., Coccia, E., Scordella, G., Licchelli, C., … Paolucci, M. (2018). Gonad quality of sea urchin *Paracentrotus lividus* cultured in an offshore pilot-scale trial on the south-east Italian coast. *Aquaculture Nutrition, 24*(5), 1444–1455. https://doi.org/10.1111/anu.12681

Whelton, P. K. (2014). Sodium, potassium, blood pressure, and cardiovascular disease in humans. *Current Hypertension Reports, 16*(8), 1–8. https://doi.org/10.1007/s11906-014-0465-5

Zar, J. H. (2014). *Biostatistical analysis: Pearson new international edition (fifth edit)*. New Jersey: Pearson Higher Ed.