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Impact of previous active dipping in *Fucus spiralis* extract on the quality enhancement of chilled lean fish

José M. Miranda [a], Mónica Carrera [b], Jorge Barros-Velázquez [a], Santiago P. Aubourg [b, *]

[a] Department of Analytical Chemistry, Nutrition and Food Science, School of Veterinary Sciences, University of Santiago de Compostela, Lugo, Spain
[b] Department of Food Science and Technology, Marine Research Institute (CSIC), Vigo, Spain

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ABSTRACT

The first goal of this work was to enhance the quality of chilled fish by applying a preliminary dipping treatment containing a bioactive extract from the alga *Fucus spiralis*. Megrim (*Lepidorhombus whiffiagonis*) specimens were dipped in aqueous solutions containing two different concentrations of the alga extract (0.50% and 0.10% alga/dipping solution, w/v; HAC and LAC batches, respectively). In addition, two control batches were considered (water dipping control and no dipping control). Microbial, chemical and sensory qualities were assessed in fish specimens after 0, 2, 6, 9 and 13 days of chilled storage. An antimicrobial effect at advanced stages of storage (9–13-day period) was observed as determined by the comparative evolution of aerobes, psychrotrophs and Enterobacteriaceae counts in megrim muscle. Likewise, a protective effect against specific lipolytic and proteolytic spoilage bacteria was also achieved at that storage period, as well as a decrease of free fatty acids formation. However, alga extract in the dipping medium had no effect ($p > 0.05$) on the increase of lipid oxidation in fish muscle throughout chilled storage. Interestingly, average scores revealed improved sensory quality in megrim corresponding to the HAC batch for the 9–13-day chilled period. Consequently, the proposed novel dipping treatment can be considered of interest for both on-board and in-land fish storage, due to the simple methodology employed and the resulting protective effects on fish quality.

1. Introduction

From the very moment wild marine species are caught till they reach the final consumer, they suffer a variety of handling and technological processes that can be decisive for the quality of the final product. To keep the original properties of the fish species, and to offer a high-quality fresh product, ice storage has been recognised as the most-employed method (Campos, Gliemmo, Aubourg, & Barros-Velázquez, 2012). However, because of the high perishability of marine species, refrigerated storage has been applied in combination with other preservative strategies such as ozone presence (Pastoriza, Bernárdez, Sampedro, Cabo, & Herrera, 2008), addition of salt (Huidobro, Montero, Tejada, Colmenero, & Borderías, 1990), high-pressure treatment (Tabilo-Munizaga, Aubourg, & Pérez-Won, 2016), and the presence of natural compounds such as organic acids (Sallam, 2007) or plant extracts (Oral, Gülmez, Vatansever, & Güven, 2008).

During on-board and in-land handling, a water dipping step has often been employed prior to chilled storage, to remove blood, digestive juices, slime and faeces, and to partially prevent microbial contamination (López-Caballero, Huidobro, Pastor, & Tejada, 2002; Erkan, 2007). To enhance such preservative effects, dips have included preservative compounds such as sodium acetate (Manju, Leema Jose, Srinivasa Gopal, Ravishankar, & Lalitha, 2007) and ellagic acid alone or in combination with ascorbic acid (Zambuchini, Fiorini, Verdenelli, Orpianesi, & Ballini, 2008). Previous research including a dipping step prior to refrigeration (at around 4 °C) of fish has also been reported to lead to enhanced quality. Previous studies have involved the employment of tea polyphenols and rosemary extract combined with chitosan (Li et al., 2012), carboxymethyl cellulose combined with *Zataria multiflora* essential oil and grape seed extract (Raeisi, Tajik, Aliakbarlu, Hamed Mirhosseini, & Mohammad Hashem Hosseini, 2015), alginate-based vitamin C solution combined with tea polyphenols (Song, Liu, Shen, You, & Luo, 2011), lactic acid (Metim, Erkan, Varlik, & Aran, 2001), and rosemary (*Rosmarinus officinalis*) combined with sage tea (*Salvia officinalis*) (Özogul, Kuley, & Kenar, 2011).

* Corresponding author.
E-mail address: saubourg@iim.csic.es (S.P. Aubourg).

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In the last decades, the use of seaweeds as food ingredients in Western countries has received increasing attention due to their excellent nutritional and functional properties (Ruperez, 2002; Peinado, Girón, Koutsidis, & Ames, 2014). Seaweeds have been shown to be a relevant source of several nutrients important for human health such as lipids, vitamins, trace minerals, dietary fibre and amino acids (Díaz-Rubio, Pérez-Jiménez, & Saura-Calixto, 2009; Paiva, Lima, Ferreira Patarra, Neto, & Baptista, 2014). Additionally, seaweeds have attracted great attention due to their profitable variety of chemical components including potential antimicrobial (Gupta & Abu-Ghannam, 2011; Sandsdalen, Haug, Stensvag, & Styrvold, 2003) and antioxidant (Halldorsdóttir, Sveinsdóttir, Gudmundsdóttir, Thorkelsson, & Kristinsson, 2014; Wang et al., 2010) compounds.

The first goal of the present work was to enhance the quality of chilled fish by applying a preliminary dipping treatment containing a seaweed extract. Thus, a brown alga, *Fucus spiralis*, was selected on the basis of its relative abundance on the European and North-American Atlantic coasts, and its relevant content of preservative compounds (Andrade et al., 2013; Tierney et al., 2013) with reported antioxidant and antimicrobial activity (García-Soto et al., 2015; Miranda, Trigo, Barros-Velázquez, & Aubourg, 2016). Specimens of megrim (*Lepidorhombus whiffiagonis*), a remarkable fish species of commercial interest in Western Europe, were dipped in aqueous solutions containing different concentrations of an ethanolic-aqueous extract of *F. spiralis*, and then stored under chilled conditions for up to 13 days. Microbial, chemical and sensory quality was monitored in fish after 0, 2, 6, 9 and 13 days of storage.

2. Materials and methods

2.1. Extraction of lyophilised F. spiralis and dipping media

The lyophilised alga *F. spiralis* was provided by a local food industry (Porto-Muiños, Cerceda, A Coruña, Spain). A combined (i.e., ethanol and water) alga extract was applied in agreement with previous research (Barros-Velázquez, Miranda, Ezquerra-Brauer, & Aubourg, 2016); 66 g of the lyophilised alga was mixed with absolute ethanol (2×528 mL), stirred for 30 s and afterwards centrifuged at $3500 \times g$ for 10 min at 4 °C. The supernatants were recovered, pooled together and then adjusted to 1 L by the addition of absolute ethanol. Then, the remaining lyophilised alga was mixed with distilled water (2×528 mL), stirred for 30 s and centrifuged at $3500 \times g$ for 10 min at 4 °C. The supernatants were recovered, pooled together and adjusted to 1 L by the addition of distilled water.

Two different concentrations of the combined alga extracts were employed, so that two kinds of dipping medium containing the alga extract were prepared. For the more concentrated batch (HAC batch, more concentrated alga condition), 834 mL of ethanolic extract and 834 mL of aqueous extract were pooled together and adjusted to 11 L by the addition of distilled water (0.50% lyophilised alga/dipping solution, w/v). To prepare the less concentrated dipping medium (LAC batch, less concentrated alga condition), 166 mL of the ethanol extract and 166 mL of the water extract were pooled together and adjusted to make an 11-L solution with distilled water (0.10% lyophilised alga/dipping solution, w/v); previously, 668 mL of absolute ethanol was also included in this solution. Thus, the same amount of ethanol was present in both dipping media.

Two kinds of control batch were considered in this study. In the first, 834 mL of absolute ethanol was diluted to an 11-L solution with distilled water, and employed as dipping control (DC batch) medium. In the other, a blank control batch with no dipping treatment (ND batch) was also considered.

2.2. Fish sample collection, dipping and chilled storage

Fresh megrim (102 specimens) were obtained at Vigo harbour, and carried to the laboratory in an ice chest. The length and weight of the individual fish employed in this work ranged from 20 to 23 cm and from 95 to 120 g, respectively.

After arrival at the laboratory, six specimens were sampled and analysed to determine the initial fish quality on day 0. These specimens were divided into three groups (two specimens per group) which were analysed independently ($n = 3$). The remaining megrim specimens were divided into four batches (24 specimens in each batch) which were subjected to different dipping treatments. One batch (ND) was kept under chilled conditions without prior treatment. The remaining three batches were immersed either in DC, LAC or HAC conditions prior to storage. Fish specimens were immersed for 5 min in the different dipping solutions, this being performed inside an isothermal room at 4 °C. After draining for 5 min, the fish specimens were placed in ice and subjected to chilled storage.

Polypropylene boxes allowing drainage of the water derived from melted ice were employed. In all cases, ice was renewed daily to maintain a constant 1: 1 fish to ice ratio. All fish batches were stored for a 13-day period, with samples being taken for subsequent analysis on days 2, 6, 9 and 13. On each sampling day, six individuals were taken from each batch and divided into three groups (two specimens per group) which were analysed independently ($n = 3$).

2.3. Analysis of lyophilised alga

The total polyphenol content of lyophilised *F. spiralis* was assessed in triplicate by means of the Folin–Ciocalteu colorimetric method (Cary 3E UV–Visible spectrophotometer, Varian, Mulgrave, Victoria, Australia), as previously described (Rodríguez-Bernaldo de Quirós, Frecha-Ferreiro, Vidal-Pérez, & López-Hernández, 2010). Gallic acid (GA) was used as standard, and the results obtained were expressed as mg GA g^{-1} lyophilised alga.

The content of different kinds of tocopherol compounds (α-, β-, γ- and δ-) in the lyophilised alga was determined according to the method of Cabrini, Landi, Stefanelli, Barzanti, and Sechi (1992). Qualitative analysis was carried out by employing the corresponding commercial standards. For quantitative analysis, content was calculated with calibration curves prepared from the corresponding commercial tocopherol compound. The results obtained were expressed as mg kg^{-1} lyophilised alga.

2.4. Microbiological evaluation of chilled megrim muscle

Samples of megrim muscle (10 g each) were aseptically dissected from chilled fish specimens, mixed with 90 mL of 0.1% peptone water (Oxoid Ltd, London, UK), and subsequently homogenised in a masticator (Seward Medical, London, UK), as previously described (Ben-Gigirey, Vieites Baptista de Sousa, Villa, & Barros-Velázquez, 1999). Serial decimal dilutions (10^{-1} to 10^{-7}) of the extracts were performed in 0.1% peptone water. Total mesophilic aerobes were investigated by deep plating in plate count agar (PCA; Oxoid), and subsequent incubation at 30 °C for 72 h. Deep plating in PCA (Oxoid) was also employed for determination of psychrotrophs, with subsequent incubation being carried out at 7–8 °C for 10 days. Enterobacteriaceae were investigated by deep plating in crystal violet neutral red bile glucose agar (VRBD agar; Merck, Darmstadt, Germany) after incubation at 37 °C for 24 h. Microorganisms exhibiting a proteolytic or lipolytic phenotype were determined on casein agar or tributyrin agar medium, respectively, after incubation at 30 °C for 48 h, as previously