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## Quality enhancement of canned sardine (*Sardina pilchardus*) by a preliminary slurry ice chilling treatment

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The slurry ice technology has shown wide advantages when employed as a chilling method for marine species instead of the traditional flake ice storage. In the present work, the use of slurry ice was evaluated for the first time as a technological treatment prior to the canning processing of fish. Thus, sardine (*Sardina pilchardus*) specimens were stored in slurry ice for 2 and 5 days and then taken and subjected to canning. Quality assessment of the canned product was performed on the fish muscle and on the coating oil, in comparison with that of a parallel control batch previously stored in flake ice. Analyses included composition (water, lipid and NaCl contents), physical properties (firmness, cohesivity), volatile amine formation (total and trimethylamine), lipid oxidation development (anisidine value, polyene index,  $\alpha$ -tocopherol content and Rancimat oxidative stability) and interaction compounds formation (fluorescence assessment). An inhibition of lipid oxidation development ( $p < 0.05$ ) was obtained in canned sardine when applying slurry ice as a preliminary chilling storage system. The present work opens the way to the use of slurry ice instead of flake ice as a preliminary treatment of fish material prior to the canning process.

**Keywords:** Sardine, chilling, slurry ice, canning, lipid oxidation, quality.

### 1 Introduction

Canning belongs to the most important means of fish preservation [1–3]. Many marine species produce excellent canned products, supporting an important role in the field of human nutrition [4]. During the canning process, both enzymes and bacteria should be permanently inactivated by heat and, provided re-infection does not occur and no negative interaction with the container is produced, heat-processed fish keeps for a very long time. However, several detrimental effects have been encountered during canning (loss of essential nutrients, formation of undesirable compounds, browning development and lipid and protein damage) that can strongly influence the shelf life of canned fish products [5–8].

Since most species used for canning occur in glut quantities, canneries often store the raw material before it is canned. In this sense, two main strategies have been employed, namely chilled and frozen storage. Many of the problems with canned fish can be related to the quality of the raw material, which continuously changes during storage prior to processing, so that the

quality of canned fish will depend to a large extent on the adequacy of the preliminary holding methods used [9, 10].

The introduction of chilling systems based on slurry ice – also known as fluid ice, slush ice, flow ice or liquid ice – has afforded several advantages as compared to traditional flake ice. Among others, these are (i) sub-zero storage temperature, (ii) faster chilling rates due to the higher heat exchange power of slurry ice, (iii) less physical damage caused to the fish surface due to its microscopic spherical crystals, and (iv) the prevention of dehydration events due to full coverage of the fish surface [11–13]. Along this line, recent studies have reported significant inhibitory effects of slurry ice on microbiological and biochemical mechanisms responsible for fish spoilage, as compared to traditional flake ice. This has led to relevant increases in the shelf life of a broad variety of chilled aquatic food products such as lean fish [14], medium-fat fish [15], fatty fish [16, 17] and crustaceans [18, 19].

The present study was based on the advantages reported for aquatic food products subjected to refrigerated storage in slurry ice, the main goal being to explore the potential advantages of such storage as preliminary treatment prior to the canning of the fish material. Sardine (*Sardina pilchardus*) was selected for this study owing to its relevance as a thermally processed product of the fish

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industry [20]. Thus, the chilling storage of sardine in slurry ice was compared with that employing traditional flake icing as a preliminary treatment prior to the canning process. The effects of the preliminary storage systems considered on the quality of the canned fish are discussed.

## 2 Materials and methods

### 2.1 Slurry ice and traditional flake ice

A slurry ice prototype (FLO-ICE; Kinarca S.A.U., Vigo, Spain) was used. The composition of the slurry ice binary mixture was 40% ice/60% water, prepared from filtered seawater (salinity: 3.3%). The temperature of the slurry ice mixture was  $-1.5^{\circ}\text{C}$ . The average temperature of the specimens processed in slurry ice was in the range of  $-1.0$  to  $-1.5^{\circ}\text{C}$ .

Flake ice was prepared with an Icematic F100 Compact device (Castelmac Spa, Castelfranco, Italy). The temperature of the flake ice was  $+0.5^{\circ}\text{C}$ . The average temperature of the specimens stored in flake ice was in the range of  $+0.5$  to  $+1.0^{\circ}\text{C}$ .

The fish specimens were surrounded by slurry or flake ice at a 1 : 1 fish-to-ice ratio and stored in a refrigerated room at  $+2^{\circ}\text{C}$ . When required, the flake ice and the slurry ice mixture were renewed.

### 2.2 Raw fish, sampling and chilling storage

Sardine (*Sardina pilchardus*) specimens were caught near the Galician Atlantic coast and transported on ice to the laboratory 10 h from the capture. The length of the specimens was in the 16–21-cm range and the average weight was 150 g.

Upon arrival in the laboratory, the fish specimens were neither headed nor gutted, but were directly placed in slurry ice or flake ice in an isothermal room at  $2^{\circ}\text{C}$ . Four different groups ( $n = 4$ ) were used for each icing treatment and studied separately throughout the whole experimental period to achieve the statistical study. Fish specimens were taken for the canning process on the starting day (day 0, no previous chilling) and on days 2 and 5 of chilled storage under both icing conditions.

### 2.3 Canning process

Chilled sardine specimens were steam-cooked in our pilot plant ( $102$ – $103^{\circ}\text{C}$ ) to a final backbone temperature of  $65^{\circ}\text{C}$  [21]; the fish were then cooled at room temperature ( $15$ – $18^{\circ}\text{C}$ ) for about 2 h, headed and eviscerated.

Muscle portions (90 g) from two individual sardine specimens were placed in each small flat rectangular can ( $105 \times 60 \times 25$  mm; 150 mL). Of NaCl, 2 g was weighed and added to each can, which were then filled with sunflower oil as coating medium. The cans were vacuum sealed and sterilised in a retort ( $115^{\circ}\text{C}$ , 45 min;  $F_0 = 7$  min). After 3 months of storage at room temperature, the cans were opened and the liquid part was carefully drained off gravimetrically, filtered by means of a filter paper and collected. Then, the resulting liquid phase was centrifuged, the oil phase separated and dried with anhydrous  $\text{Na}_2\text{SO}_4$ . Sardine muscle was wrapped in filter paper. Fish white muscle and the coating oil medium were used for physical and biochemical analyses.

### 2.4 Composition analysis

Water content was determined by weight difference between the homogenised fish muscle (1–2 g) before and after 24 h at  $105^{\circ}\text{C}$ . Results are expressed as g water/100 g muscle.

The lipid fraction was extracted from the fish muscle by the Bligh and Dyer [22] method. Quantification results are expressed as g lipid/100 g muscle.

The NaCl content in fish muscle was calculated from the amount of chlorine by boiling in  $\text{HNO}_3$  with an excess of  $\text{AgNO}_3$ , followed by titration with  $\text{NH}_4\text{SCN}$  [23]. Results are expressed as g NaCl/100 g muscle.

### 2.5 Textural analysis

A shear test was used to evaluate texture. Firmness and cohesivity were determined from a stress-distance curve obtained from a Universal Testing Machine (LR-5K; Lloyd Instruments Limited, Hampshire, UK) including a load cell of 500 N [24]. The shear force or firmness was measured as the maximum peak force (N) required to shear/cut through the samples; cohesivity was measured during the upward movement of the blade and was calculated as the deformation (mm) at maximum peak force [25]. The average value of three replicates was considered in each analysis.

### 2.6 Volatile amine formation

Total volatile base-nitrogen (TVB-N) values were measured by the Antonacopoulos [26] method, with some modifications. Briefly, fish muscle (10 g) was extracted with 6% (wt/vol) perchloric acid and brought up to 50 mL, determining the TVB-N content – after steam-distillation of the acid extracts rendered alkaline to pH 13

with 2% (wt/vol) NaOH – by titration of the distillate with 10 mM HCl. The results are expressed as mg TVB-N/100 g muscle.

Trimethylamine-nitrogen (TMA-N) values were obtained by means of the picrate method, as previously described [27]. This involves the preparation of a 5% (wt/vol) trichloroacetic acid extract of fish muscle. The results are expressed as mg TMA-N/100 g muscle.

## 2.7 Lipid oxidation assessment

The anisidine value was determined in fish muscle according to the AOCS [28] method, based on the reaction between  $\alpha$ - and  $\beta$ -aldehydes (primarily 2-alkenals) with *p*-anisidine reagent. The anisidine value is expressed as 100 times the absorbance measured at 350 nm in a 1-cm path length cuvette from a solution containing 1 g lipid/100 mL reaction medium.

Lipid extracts from the fish muscle were converted into fatty acid methyl esters (FAME) by employing acetyl chloride [29]. FAME were analysed [30] by GC (Perkin-Elmer 8700 chromatograph) employing a fused-silica capillary column SP-2330 (0.25 mm i.d.  $\times$  30 m; Supelco, Bellefonte, PA, USA). The carrier gas used was N<sub>2</sub> flowing with a linear velocity of 18 cm/s. A flame ionisation detector set at 250°C was used. Peaks were identified by comparison of their retention times with standard FAME mixtures (Qualmix Fish, Larodan; FAME Mix, Supelco). Peaks were automatically integrated, with 19:0 fatty acid being used as internal standard for quantitative analysis. The polyene index (PI) was calculated as the following fatty acid ratio: C20:5 + C22:6/C16:0 [31].

Tocopherol analysis [32] was carried out by HPLC employing a Superspher Si LichroCART 60 (25 cm  $\times$  4  $\mu$ m i.d.; Merck, Germany) column. The mobile phase employed was propan-2-ol/hexane (0.5 : 99.5, vol/vol) with a 1 mL/min flow rate. Fluorescence detection (Merck Hitachi F-1050 and Merck-Hitachi D-2500 integrator) was employed at 290 nm/330 nm excitation/emission wavelengths. The presence of different tocopherol isomers was checked in sardine muscle by employing different tocopherol and tocotrienol compounds (Merck, Germany). Only the  $\alpha$ -tocopherol isomer was detected in the subject sardine samples, and its content expressed as mg/kg lipids.

The oxidative stability of coating oils from canned sardine was determined automatically by employing a Metrohm Rancimat model 679 (Metrohm, Herisau, Switzerland) operating at an air flow of 20 L/h and at 110°C temperature following the AOCS [33] procedure. The formation

rate of volatile compounds was monitored by electro-conductivity and results are expressed as the induction time (h).

## 2.8 Interaction compound formation

The formation of interaction compounds [34, 35] – also called tertiary oxidation compounds – is the result of the interaction between lipid oxidation compounds and protein-like nucleophilic molecules present in fish muscle. In the present work, such interaction events were studied in the canned fish muscle and in the coating oil by means of fluorescence analysis.

Formation of fluorescent compounds was determined with a Perkin Elmer LS 3B fluorimeter by measurements at 393/463 nm and 327/415 nm as previously described [10]. The relative fluorescence (RF) was calculated as follows:  $RF = F/F_{st}$ , where  $F$  is the fluorescence measured at each excitation/emission maximum, and  $F_{st}$  is the fluorescence intensity of a quinine sulphate solution (1  $\mu$ g/mL in 0.05 M H<sub>2</sub>SO<sub>4</sub>) at the corresponding wavelength. The fluorescence ratio (FR) was calculated as the ratio between the two RF values:  $FR = RF_{393/463\text{ nm}}/RF_{327/415\text{ nm}}$ . The FR value was determined in the lipid extract of the fish muscle and in the coating oil.

## 2.9 Statistical analyses

SPSS software (SPSS Inc., Chicago, IL, USA) was used to explore the statistical significance of the differences between slurry ice and flake ice conditions, this including multivariate contrasts and multiple comparisons by the Scheffé and Tukey tests. Data from the different measurements were subjected to one-way analysis of variance; comparison of means was performed using a least-square differences (LSD) method [36]. A confidence interval at the 95% level ( $p < 0.05$ ) was considered in all cases.

## 3 Results and discussion

### 3.1 Composition analyses

The water and lipid contents of canned sardine ranged from 63–65% and 1.1–1.9%, respectively (Tab. 1). Values for both constituents did not provide significant differences ( $p > 0.05$ ) as a result of the preliminary icing conditions. Compared to the raw fish starting value (72.03  $\pm$  0.60 and 3.01  $\pm$  0.06 for water and lipids, respectively), all kinds of canned samples showed lower ( $p < 0.05$ ) values, according to previous research [37].

**Tab. 1.** Comparative evolution of compositional parameters in canned sardine that was preliminarily chilled under slurry ice and flake ice conditions<sup>§</sup>.

Parameter	Preliminary chilling time [days]	Preliminary icing conditions	
		Slurry ice	Flake ice
Water content [g/100 g muscle]	0	63.77 (0.58)	63.77 (0.58)
	2	63.53 (0.56)	64.66 (0.81)
	5	64.64 (0.62)	64.67 (0.55)
Lipid content [g/100 g muscle]	0	1.14 <sup>a</sup> (0.14)	1.14 <sup>a</sup> (0.14)
	2	1.24 <sup>a</sup> (0.10)	1.33 <sup>a</sup> (0.29)
	5	1.89 <sup>b</sup> (0.35)	1.68 <sup>b</sup> (0.15)
NaCl content [g/100 g muscle]	0	0.68 (0.16)	0.68 (0.16)
	2	0.76 (0.10)	0.74 (0.24)
	5	0.91 (0.15)	0.83 (0.03)

§ Mean values of four independent determinations ( $n = 4$ ). Standard deviations are indicated in brackets. For each parameter and for each chilling condition, mean values followed by different letters (a, b) denote significant differences ( $p < 0.05$ ) as a result of chilling time. All parameters showed no significant differences ( $p > 0.05$ ) between both preliminary icing conditions.

Water loss can be explained in terms of heat treatment and protein degradation in canned sardine muscle, this leading to a decreasing water holding capacity of proteins [6, 38, 39]. Lipid loss in canned fish muscle has been explained as a result of lipid extraction by the coating oil [37, 40]. No significant differences were observed for the water content in canned fish as a result of the preliminary chilling time under both icing conditions. However, a chilling time increase (day 5) led to a higher lipid content under both icing conditions.

Previous research [15, 17] has shown an NaCl content increase in chilled fish muscle treated under slurry ice conditions when compared to the flake ice conditions. However, in the present case (Tab. 1) no significant differences ( $p > 0.05$ ) were observed for the NaCl presence in canned sardine when comparing both kinds of preliminary chilling treatment employed. In addition, no significant differences could be assessed as a result of the previous chilling time under both conditions. Both lacks of differences could be explained by the fact that a substantial quantity of NaCl was added during the manufacturing of the canned product (see Materials and methods) before the sterilisation step was achieved. However, compared to the raw fish starting value ( $0.07 \pm 0.02$ ), all kinds of canned samples showed a higher ( $p < 0.05$ ) NaCl content.

### 3.2 Textural properties

Firmness and cohesivity results are indicated in Tab. 2. Values obtained for both parameters in canned sardine did not show significant differences ( $p > 0.05$ ) between samples stored under slurry ice and flake ice treatments, so that a differential effect of the preliminary chilling conditions could not be obtained. However, a 2-day previous chilling storage led to an increase in firmness value and a decrease for the cohesivity score for both kinds of chilling systems; thus, a more breakable fish structure was obtained as a result of the preliminary chilling storage. Lengthening the storage time under both icing conditions for up to 5 days was not accompanied by any additional change in both textural properties.

The texture of the fish muscle depends on numerous intrinsic biological factors related to the density of the muscle fibres, as fat and collagen content of the fish [25, 41]. Previous research has shown a firmness decrease in chilled fish by increasing the chilling storage time [42]. However, heating has been shown to convert the translucent, jelly-like cellular fish mass into an opaque and firmer

**Tab. 2.** Comparative evolution of different quality parameters in canned sardine that was preliminarily chilled under slurry ice and flake ice conditions<sup>§</sup>.

Parameter	Preliminary chilling time [days]	Preliminary icing conditions	
		Slurry ice	Flake ice
Firmness [N]	0	17.6 <sup>a</sup> (2.7)	17.6 <sup>a</sup> (2.7)
	2	22.7 <sup>b</sup> (1.6)	19.9 <sup>b</sup> (2.9)
	5	23.9 <sup>b</sup> (1.6)	22.5 <sup>b</sup> (3.1)
Cohesivity [mm]	0	47.9 <sup>b</sup> (1.0)	47.9 <sup>b</sup> (1.0)
	2	36.1 <sup>a</sup> (3.7)	34.7 <sup>a</sup> (2.3)
	5	33.7 <sup>a</sup> (3.2)	33.4 <sup>a</sup> (1.9)
Total volatile base-nitrogen [mg TVB-N/100 g muscle]	0	52.7 (2.4)	52.7 (2.4)
	2	54.6 (4.3)	55.7 (3.4)
	5	55.1 (2.7)	52.7 (6.2)
Trimethylamine-nitrogen [mg TMA-N/100 g muscle]	0	6.9 (0.4)	6.9 <sup>a</sup> (0.4)
	2	6.8 (0.9)	7.9 <sup>a, b</sup> (0.7)
	5	7.5 (0.7)	8.2 <sup>b</sup> (0.6)

§ Mean values of four independent determinations ( $n = 4$ ). Standard deviations are indicated in brackets. For each parameter and for each chilling condition, mean values followed by different letters (a, b) denote significant differences ( $p < 0.05$ ) as a result of chilling time. All parameters showed no significant differences ( $p > 0.05$ ) between both preliminary icing conditions.

material, where the connective tissue holding the cells together has been degraded and blocks of cells become readily separated from one another [5]. According to the starting raw fish values ( $9.05 \pm 0.73$  and  $68.32 \pm 1.15$  for firmness and cohesivity, respectively) obtained in the actual experiment, the present results show the influence of both the preliminary chilling storage and the heat treatment on the textural parameters.

### 3.3 Volatile amine formation

The results of the TVB-N analysis are shown in Tab. 2. No significant differences ( $p > 0.05$ ) deriving from the preliminary storage system were found in the muscles of canned sardine specimens. Lengthening the storage time under either of the icing conditions was not accompanied by any significant ( $p > 0.05$ ) additional formation of TVB-N. However, a marked increase ( $p < 0.05$ ) was observed for all kinds of samples when compared to the starting raw material employed ( $31.4 \pm 2.6$ ), according to the results of Gallardo *et al.* [43].

The TMA-N content did not provide significant differences ( $p > 0.05$ ) between both kinds of icing treatment. Canned samples that were previously stored under flake ice conditions showed an increasing tendency as a result of the chilling time; such a difference was not detected for their corresponding canned samples previously stored under slurry ice conditions. When the raw sardine TMA-N value is considered ( $0.06 \pm 0.01$ ), a great increase is concluded as a result of the canning process [43].

Trimethylamine formation in the present experiment can be explained by means of two different pathways: (i) as a result of trimethylamine oxide (TMAO) bacterial catalysis breakdown during the chilled storage, and (ii) trimethylamine can be produced from TMAO by thermal breakdown during the cooking and sterilisation steps. The great difference between the starting fish TMA-N value ( $0.1 \pm 0.0$ ) and the TMA-N value reported for canned fish without previous chilling storage ( $6.9 \pm 0.4$ , day 0) leads to the conclusion that, in the actual experiment, both thermal steps (cooking and sterilisation) exert a higher effect on the trimethylamine formation than the chilled storage. Previous research [44] has shown an inhibitory effect on TVB-N and TMA-N formation for slurry ice treatment during sardine chilled storage. However, the actual results show that after the heat treatment was applied, this difference is not maintained.

### 3.4 Lipid oxidation assessment

Lipid oxidation development was analysed in the canned fish muscle and in the coating oil.

The anisidine value was studied in the canned fish muscle to assess secondary lipid oxidation development (Tab. 3). Higher ( $p < 0.05$ ) values were obtained for canned samples that had been stored under flake ice conditions, so that an inhibitory effect of slurry ice treatment could be concluded. Both preliminary icing conditions led to higher values after 2 days of chilled storage, but a lengthened storage time under both icing conditions for up to 5 days was not accompanied by any significant additional increase in this parameter.

The fatty acid composition was analysed in the canned fish muscle. The PI assessment (Tab. 3) provided a different behaviour in canned samples according to the kind of preliminary icing condition. Thus, for fish previously stored under flake ice conditions, the PI value was shown to decrease with the chilling time [10], while the opposite result was obtained for the canned fish previously stored under slurry ice conditions. As a result, a higher ( $p < 0.05$ ) PI was obtained for the canned fish muscle that had been stored preliminarily under slurry ice conditions, so that an inhibitory effect on polyunsaturated fatty acid damage is inferred.

**Tab. 3.** Comparative evolution of different lipid damage parameters in canned sardine that was preliminarily chilled under slurry ice and flake ice conditions<sup>§</sup>.

Parameter	Preliminary chilling time [days]	Preliminary icing conditions	
		Slurry ice	Flake ice
Anisidine value	0	0.80 <sup>a</sup> (0.30)	0.80 <sup>a</sup> (0.30)
	2	<sup>z</sup> 1.68 <sup>b</sup> (0.41)	<sup>y</sup> 1.91 <sup>b</sup> (0.31)
	5	<sup>z</sup> 1.53 <sup>b</sup> (0.26)	<sup>y</sup> 2.08 <sup>b</sup> (0.49)
Polyene index	0	1.06 <sup>a</sup> (0.03)	1.06 <sup>b</sup> (0.03)
	2	<sup>y</sup> 1.32 <sup>b</sup> (0.20)	<sup>z</sup> 0.86 <sup>a</sup> (0.04)
	5	<sup>y</sup> 1.41 <sup>b</sup> (0.07)	<sup>z</sup> 0.90 <sup>a</sup> (0.03)
$\alpha$ -Tocopherol [mg/kg lipids]	0	313.54 <sup>b</sup> (1.46)	313.54 <sup>b</sup> (1.46)
	2	<sup>y</sup> 298.99 <sup>a</sup> (3.69)	<sup>z</sup> 266.03 <sup>a</sup> (34.50)
	5	<sup>y</sup> 291.22 <sup>a</sup> (8.40)	<sup>z</sup> 235.76 <sup>a</sup> (4.36)
Rancimat induction time [h]	0	4.68 (0.07)	4.68 <sup>b</sup> (0.07)
	2	4.27 (0.47)	4.09 <sup>a, b</sup> (1.25)
	5	<sup>y</sup> 4.76 (0.84)	<sup>z</sup> 3.96 <sup>a</sup> (0.42)

<sup>§</sup> Mean values of four independent determinations ( $n = 4$ ). Standard deviations are indicated in brackets. For each parameter and for each chilling time (2 and 5 days), mean values preceded by different superscripts (z, y) denote significant differences ( $p < 0.05$ ) between both preliminary chilling conditions. For each parameter and for each chilling condition, mean values followed by different letters (a, b) denote significant differences ( $p < 0.05$ ) as a result of chilling time.

## Canned sardine quality and preliminary slurry ice chilling

$\alpha$ -Tocopherol is a known endogenous antioxidant widely distributed in marine species. Its content in fish foods has been shown to decrease as a result of quality loss during processing and storage [45, 46]. In the actual experiment,  $\alpha$ -tocopherol contents obtained in the canned fish muscle are expressed in Tab. 3. A higher ( $p < 0.05$ ) content was obtained for canned fish that was preliminarily stored under slurry ice conditions, so that a lower oxidation development is concluded for such kinds of samples throughout the experiment. Both icing conditions showed an  $\alpha$ -tocopherol loss increase in canned fish with the preliminary chilling time, according to an increasing product damage [45, 46].

Oxidative stability was studied in the coating oils. Thus, induction times obtained by the Rancimat method are indicated in Tab. 3. After 5 days of preliminary chilled storage, canned fish specimens that were stored under slurry ice conditions led to a higher ( $p < 0.05$ ) induction time than their counterparts stored under flake ice conditions, so that a higher oxidative stability could be concluded as a result of the slurry ice conditions [47]. The preliminary chilling time did not lead to significant differences in canned fish under slurry ice conditions; however, a decrease in the induction time was observed when lengthening the storage time under flake ice conditions, according to an oxidation development increase.

### 3.5 Interaction compound formation

In both fluorescence analyses (Tab. 4), no significant differences were obtained as a result of the preliminary icing conditions employed, although in all cases higher mean values were obtained for samples that were previously stored under flake ice. Both icing conditions showed an increasing FR value in the canned product (fish muscle and coating oil) when a 5-day chilled storage was considered. This FR increase agrees with previous research and can be explained as a quality loss increase resulting from the preliminary storage [9, 10]. Compared to the starting raw fish FR value ( $0.18 \pm 0.03$ ), all kinds of canned fish muscle showed a significant increase ( $p < 0.05$ ). According to the FR value of the starting oil ( $2.66 \pm 0.08$ ), a significant increase ( $p < 0.05$ ) under both icing conditions was only obtained after 5 days of preliminary chilling storage.

Previous research on chilled sardines [17] has shown an inhibitory effect on the FR increase for slurry ice treatment when compared to flake ice conditions. However, this effect was not observed in the actual scores as a result of the heat treatment application.

**Tab. 4.** Comparative evolution of interaction compound formation (fluorescence ratio) in canned sardine that was preliminarily chilled under slurry ice and flake ice conditions<sup>§</sup>.

Parameter	Preliminary chilling time [days]	Preliminary icing conditions	
		Slurry ice	Flake ice
Fluorescence ratio (fish muscle)	0	0.83 <sup>a</sup> (0.11)	0.83 <sup>a</sup> (0.11)
	2	0.77 <sup>a</sup> (0.20)	0.87 <sup>a</sup> (0.16)
	5	1.10 <sup>b</sup> (0.17)	1.19 <sup>b</sup> (0.11)
Fluorescence ratio (coating oil)	0	2.56 <sup>a</sup> (0.13)	2.56 <sup>a</sup> (0.13)
	2	3.28 <sup>a</sup> (0.28)	3.50 <sup>a</sup> (0.68)
	5	6.20 <sup>b</sup> (2.42)	9.13 <sup>b</sup> (3.92)

§ Mean values of four independent determinations ( $n = 4$ ). Standard deviations are indicated in brackets. For each kind of fluorescence analysis and for each chilling condition, mean values followed by different letters (a, b) denote significant differences ( $p < 0.05$ ) as a result of chilling time. Both fluorescence assessments showed no significant differences ( $p > 0.05$ ) between both preliminary icing conditions.

## 4 Conclusions

In previous research, it has been proven that the use of slurry ice as a chilling storage system exerts an inhibitory effect on the sensory, biochemical and microbiological spoilage mechanisms occurring in marine species. In the present work, a profitable application of slurry ice as a preliminary storage system of sardine prior to its canning processing was explored. Previous processing of sardine in slurry ice led to significantly ( $p < 0.05$ ) less lipid damage as compared to storage in flake ice, as can be concluded from the results obtained for the anisidine and polyene values,  $\alpha$ -tocopherol retention and the induction time of oxidation development (Rancimat method). However, no significant ( $p > 0.05$ ) effects on volatile amine formation (total and trimethylamine), physical properties (cohesivity and firmness) and interaction compound formation (fluorescence ratio) could be assessed when comparing both icing conditions. Because of the important role that lipid oxidation may play in fish food quality loss [48, 49] and since most species employed for canning are of medium and high fat content, the slurry ice storage is recommended instead of the conventional flake ice treatment, to obtain less oxidised canned products and, accordingly, fish products supporting a higher quality degree.

## Acknowledgments

The authors thank Mr. Marcos Trigo for his excellent technical assistance, Kinarca S.A.U. for providing the slurry ice equipment, and Justo López Valcarcel S.A. for kindly lending its equipment to elaborate the canned product. The work was supported by the Secretaría Xeral de I+D from the Xunta de Galicia (Galicia, Spain) (Project PGIDIT02RMA18E).

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