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Additional Information

1 **Low-temperature drying of salted cod (*Gadus morhua*) assisted by high**  
2 **power ultrasound: kinetics and physical properties**

3  
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24

25 **ABSTRACT**

26 Low-temperature convective drying could be considered an affordable alternative to  
27 conventional freeze-drying for foodstuffs. The process intensification should be based on  
28 non-thermal technologies, such as power ultrasound. Thereby, the aim of this work was to  
29 evaluate the air-borne application of power ultrasound on the low-temperature drying of  
30 salted cod. For that purpose, drying experiments were carried out at -10, 0, 10 and 20 °C on  
31 salted cod slabs at 2 m/s with (AIR+US, 20.5 kW/m<sup>3</sup>) and without ultrasonic application  
32 (AIR). In the dried-salted cod, its rehydration capacity was analyzed, as were the  
33 microstructural, textural and color changes. At every temperature tested, ultrasound  
34 application increased the drying rate; thus, an average increase of 74% was observed in the  
35 effective diffusivity. AIR+US dried samples were softer and exhibited a higher rehydration  
36 capacity than AIR ones, which was linked to the microstructural changes produced by  
37 ultrasound. In addition, color changes were induced by ultrasound application.

38

39 *Keywords:* Ultrasonic; non-thermal processing, dehydration; microstructure; texture; color.

40

## 41 **Introduction**

42 Dried-salted cod (*Gadus morhua*) or klipfish has long been highly appreciated due to its  
43 high nutritional value and specific sensory properties (Walde, 2003). Although, dried-salted  
44 cod presents a water content of under 45% (wet basis), its salt content may reach 20% (wet  
45 basis) (Barat, Rodríguez-Barona, Andrés, & Fito, 2003). The processing includes the  
46 following steps: salting, washing, pre-drying by keeping the green salted cod for several  
47 days in piles outside the drying chambers, drying, grading and packaging (Oliveira, Pedro,  
48 Nunes, Costa, & Vaz-Pires, 2012). The drying of salted cod is performed at temperatures of  
49 around 20 °C and at relative humidities of under 70% (Walde, 2003). Two main products  
50 can be found on the market depending on the intensity of the drying process: semi-dried or  
51 extra-dried cod.

52 The quality of dried-salted cod is greatly influenced by the salting and drying operation  
53 (Kilic, 2009). Salting induces changes in the muscle protein, generating modification in the  
54 texture, weight and water holding capacity (Oliveira et al., 2012). As far as drying is  
55 concerned, the use of high temperatures entails chemical and microbiological changes  
56 (Ortiz et al., 2013), structural, physical and mechanical modifications (Duan, Jiang, Wang,  
57 Yu, & Wang, 2011), crust formation on the surface (Bellagha, Sahli, Farhat, Kechaou, &  
58 Glenza, 2007) and the reduction of the hydration capacity of proteins (Brás & Costa 2010).  
59 The use of low-temperature convective drying constitutes an interesting alternative means  
60 of improving the quality of the dried-salted cod due to the fact that it provides products  
61 with similar quality characteristics than conventional freeze-drying at lower cost (Kilic,  
62 2009). The term “low temperature” makes reference to the use of air temperatures below  
63 standard room conditions, which includes figures below or close to the product’s freezing  
64 point. Despite its great potential, the use of low-temperatures in convective drying is mostly

65 limited by the low drying rate, which retards the dehydration process and directly increases  
66 the processing costs (Walde, 2003). In this regard, it is of great interest to deal with the  
67 process intensification in order to improve the drying rate. For that purpose, coupling non-  
68 thermal technologies, such as power ultrasound (Ortuño, Martínez-Pastor, Mulet, &  
69 Benedito, 2012), to convective drying so as to achieve a higher yield, a lower energy use  
70 and high product quality and processing safety (Garcia-Perez, Carcel, Riera, Rosselló, &  
71 Mulet, 2012a) is worth exploring.

72 The application of power ultrasound may be accomplished by direct-contact or air-borne  
73 transmission (Schössler, Jäger, & Knorr, 2012a; Ozuna, Cárcel, García-Pérez, & Mulet,  
74 2011). In direct-contact applications, there is an intimate contact between ultrasonic source  
75 and product, while in air-borne applications, ultrasound is transmitted through the air.  
76 Despite air-borne applications are less efficient than direct-contact ones in terms of energy  
77 yield, its lower heating effect and better adaptability to convective driers have largely  
78 contributed to its development. The feasibility of the ultrasonic application should be  
79 evaluated considering both kinetic and quality issues (Ozuna, Puig, García-Pérez, Mulet, &  
80 Cárcel, 2013; Vilkhua, Mawsona, Simonsa, & Bates, 2008).

81 Air-borne ultrasound applications have been reported to assist conventional hot-air drying  
82 (Ozuna et al., 2011; Garcia-Perez, Ortuño, Puig, Carcel, & Perez-Munuera, 2012b) and,  
83 more recently, for the convective freeze-drying (Bantle & Eikevik, 2011; Garcia-Perez et  
84 al., 2012a). Previous results have shown that air-borne ultrasound application during drying  
85 may greatly accelerate water removal. However, the effectiveness of ultrasound greatly  
86 depends on the process and product variables, such as temperature, air velocity, acoustic  
87 power applied, and product porosity (Cárcel, García-Pérez, Benedito, & Mulet, 2012). In  
88 addition, as far as we are concerned, the most recent air-borne applications have been

89 focused on the drying of fruits and vegetables and few works have addressed the treatment  
90 of protein matrices, such as meat or fish products (Nakagawa, Yamashita, & Miura, 1996).  
91 According to literature, ultrasound is able to produce modifications in food quality  
92 parameters such as texture, color, flavor and nutrients (Pingret, Fabiano-Tixier, & Chemat,  
93 2013). To understand the effect of high power ultrasound on food quality, it is important to  
94 know the interactions between acoustic energy and the food structure (Jaeger, Reineke,  
95 Schoessler, & Knorr, 2012). Although several papers have focused on the quality changes  
96 brought about by ultrasonic applications in liquid media (Ahmad-Qasem et al., 2013 Wu,  
97 Hulbert, & Mount, 2000), few references have addressed this issue in gas media  
98 applications. In hot air drying and direct-contact ultrasonic application, Soria et al. (2010)  
99 and Schössler, Thomas, and Knorr (2012b) have studied the changes in chemical and  
100 physical quality parameters induced by a direct-contact ultrasonic application during the  
101 dehydration of carrot (20, 40 and 60 °C, 1.2 m/s) and potato (70 °C), respectively. Garcia-  
102 Perez et al. (2012b) and Puig, Perez-Munuera, Carcel, Hernando, and Garcia-Perez (2012)  
103 reported that the application of ultrasound during drying (40 °C and 1 m/s) of orange peel  
104 and eggplant, respectively, could contribute to a better preservation of the quality (internal  
105 food structure) due to the shortening of the drying time. Schössler, Jager, and Knorr  
106 (2012c) reported that bulk density, color, ascorbic acid content and rehydration  
107 characteristics of red bell pepper was not affected by an ultrasonically (direct-contact)  
108 accelerated freeze-drying .

109 The main aim of this work was to evaluate the air-borne application of power ultrasound on  
110 the low-temperature drying of salted cod, quantifying its influence on the drying kinetics  
111 and on the physical properties of the final dried product.

## 113 **2. Materials and methods**

### 114 2.1. Raw material and shaping samples

115 Salted cod (*Gadus morhua*) was provided by a local supplier (Carmen Cambra S. L.,  
116 Spain), to better ensure the homogeneity of the raw material. According to supplier  
117 specifications, cod fish were caught in high seas and processed immediately in the fishing  
118 boat (bled, gutted, beheaded, split and salted). On average, the pieces of salted cod weighed  
119  $1.5 \pm 0.25$  kg.

120 Parallelepiped-shaped samples (length 50 mm, width 30 mm and thickness 10 mm) were  
121 obtained from the central part of the salted cod loin using a sharp knife. The samples were  
122 wrapped in plastic waterproof film and stored at  $-18 \pm 0.5$  °C until the drying experiments  
123 were carried out (maximum storage time 120 h). The initial moisture and the NaCl content  
124 were measured following standard methods 950.46 and 971.27, respectively (AOAC,  
125 1997).

126

### 127 2.2. Drying experiments

128 The drying experiments of salted cod slabs were conducted in a convective drier with air  
129 recirculation and temperature and air velocity control. Air temperature and velocity are  
130 controlled using a PID algorithm. A cooper tube heat exchanger (Frimetal, Spain), installed  
131 in the air duct, cooled down the air to temperatures close to  $-20$  °C, which was subsequently  
132 heated using electrical devices (3000 W). The air temperature and relative humidity were  
133 measured at three points in the air duct using a combined sensor (KDK, Galltec+Mela,  
134 Germany).

135 The drier includes an ultrasonically activated drying chamber, already described in  
136 literature (Garcia-Perez et al., 2012a). The air-borne ultrasound application system

137 consisted of a cylindrical radiator (internal diameter 100 mm, height 310 mm, thickness 10  
138 mm) driven by a power ultrasonic transducer (frequency 21.9 kHz, impedance 369  $\Omega$ ,  
139 power capacity 90 W). The ultrasonic system provided an average sound pressure level in  
140 the drying chamber of 155 dB. A resonance dynamic controller was connected to a PC by  
141 the RS-232 interface to adequately monitor the main electric parameters of the system  
142 during the air-borne ultrasonic application (power, intensity, voltage, phase, frequency and  
143 impedance).

144 The cod samples were weighed at preset times using an industrial weighing module  
145 (6000 $\pm$ 0.01 g; VM6002-W22, Mettler-Toledo, USA). An application was developed using  
146 LabVIEW 2011 programming code (National Instruments, USA) to provide overall control  
147 and monitoring of the ultrasonically intensified drying process, integrating information on  
148 the air flow, the sample and the ultrasonic parameters.

149 Air-drying (AIR) and ultrasonically assisted air-drying (AIR+US) experiments were  
150 conducted at -10, 0, 10 and 20  $\pm$  1  $^{\circ}$ C, 2  $\pm$  0.1 m/s and an average relative humidity of 9  $\pm$   
151 4%. In the AIR+US experiments, an acoustic power density of 20.5 kW/m<sup>3</sup> was applied  
152 (Ozuna et al., 2011). Prior to the drying experiments, the sealed samples (9 slabs per  
153 experiment) were tempered at the drying temperature for 24 h. Then, the cod slabs were  
154 unwrapped and placed on the sample holder, which consists of a metallic frame where  
155 samples are suspended to allow free airflow around the slabs, and introduced into the  
156 drying chamber. The sample weight was automatically measured and recorded at regular  
157 time intervals (15 min). The drying experiments were replicated at least three times for  
158 each condition tested and extended until the samples lost 20% of the initial weight, which is  
159 a usual figure for klipfish (Oliveira et al., 2012).

160



### 2.3. Rehydration experiments

Rehydration experiments of dried-salted cod (AIR and AIR+US at -10, 0, 10 and 20 °C) were carried out by immersing the samples in distilled water at  $4 \pm 1$  °C for 27 h. The ratio of cod and water volume was kept as 1:20. In this study, the rehydration kinetics were studied globally from the evolution of the net sample weight that includes both moisture and salt transport, since samples both gained water and lost salt. For that purpose, samples were taken at regular time intervals, superficially drained with absorbent paper to remove surface water and weighed. Thus, the net weight change was monitored (Eq. (1)).

$$\Delta M_t^0 = \frac{M_t - M_0}{M_0} \quad (1)$$

For each drying condition tested, a minimum of 6 rehydration experiments were carried out.

### 2.4. Modeling

#### 2.4.1 Drying

A diffusion model, based on Fick's law, was used to mathematically describe the drying kinetics (AIR and AIR+US) of cod samples. The differential equation of diffusion is obtained by combining Fick's law and the microscopic mass balance. For infinite slab geometry, the diffusion equation is shown in Eq. (2), assuming the effective moisture diffusivity as constant and the solid to be isotropic.

$$\frac{\partial W_p(x, t)}{\partial t} = D_w \left( \frac{\partial^2 W_p(x, t)}{\partial x^2} \right) \quad (2)$$

In order to solve Eq. (2), some further assumptions were considered: solid symmetry, a uniform initial moisture content and temperature, constant shape during drying and a

183 negligible external resistance to water transfer. Taking these assumptions into account, the  
 184 analytical solution of the diffusion equation is expressed in terms of the average moisture  
 185 content in Eq. (3) (Crank, 1975).

$$186 \quad W = W_{eq} + (W_0 - W_{eq}) \left[ 2 \sum_{n=0}^{\infty} \frac{1}{\lambda_n^2 L^2} e^{-D_w \lambda_n^2 t} \right] \quad (3)$$

187 where,  $\lambda_n$  are the eigenvalues calculated as  $\lambda_n L = (2n + 1) \frac{\pi}{2}$

188 The equilibrium moisture data were obtained from the desorption data of salted cod at 25  
 189 °C reported by Walde (2003).

190

#### 191 2.4.2 Rehydration

192 The evolution of sample weight during rehydration was modeled by means of Peleg's  
 193 empirical equation (Peleg, 1988) (Eq. (4)).

$$194 \quad M_t = M_0 + \frac{t}{k_1 + k_2 t} \quad (4)$$

195 where  $1/k_1$  and  $1/k_2$  are the model's parameters. The rate constant,  $1/k_1$ , is related to the  
 196 weight gain rate at the very beginning,  $t = t_0$ , and represents the initial rehydration rate (Eq.  
 197 (5)).

$$198 \quad \frac{dM(t = t_0)}{dt} = \frac{1}{k_1} \quad (5)$$

199 The capacity constant,  $1/k_2$ , is related to the equilibrium weight ( $M_e$ ). Thus, when  
 200 rehydration time is very long, Eq. (4) becomes Eq. (6), giving the relationship between  $M_e$   
 201 and  $1/k_2$ .

$$202 \quad M_e = M_0 + \frac{1}{k_2} \quad (6)$$

203

### 204 2.4.3 Model fitting

205 The model parameters ( $D_w$  of diffusion model and  $1/k_1$  and  $1/k_2$  of Peleg model) were  
206 identified by using an optimization procedure that minimized the sum of the squared  
207 differences between the experimental and calculated average data. For that purpose, the  
208 non-linear optimization algorithm of the Generalized Reduced Gradient (GRG), available  
209 in Microsoft Excel<sup>TM</sup> spreadsheet from MS Office 2010 (Microsoft Corp., USA), was used.  
210 The goodness of the fit was determined by the percentage of explained variance, %VAR  
211 (Eq. (7)).

$$212 \quad \% \text{VAR} = \left[ 1 - \frac{S_{xy}^2}{S_y^2} \right] \cdot 100 \quad (7)$$

213

### 214 2.5. Texture

215 Hardness, characterized as the maximum penetration force, was evaluated in the dried and  
216 rehydrated samples using a Texture Analyzer (TAX-T2<sup>®</sup>, Stable Micro System, United  
217 Kingdom). Penetration tests were conducted with a 2 mm flat cylinder probe (SMS P/2N),  
218 at a crosshead speed of 1 mm/s and a strain of 70% (penetration distance 7 mm). In each  
219 sample, penetration tests were carried out at 12 points following a preset pattern.

220

### 221 2.6. Microstructure. Scanning Electron Microscopy (SEM)

222 Cubes (side 3 mm) from samples dried at 0 °C with and without ultrasound application  
223 were immersed in liquid N<sub>2</sub> and then freeze-dried at 1 Pa for 3 days (LIOALFA-6, Telstar,  
224 Spain). Then, samples were vacuum sealed in vials in the same freeze-drier, so that they  
225 would remain stable (Ozuna et al., 2013). After that, they were individually placed on SEM

226 slides with the aid of colloidal silver and then gold-coated with carbon (SCD005, Baltec,  
227 Germany) at  $10^{-2}$  Pa and an ionization current of 40 mA. The samples were observed in a  
228 scanning electron microscope (JSM-5410, Jeol, Japan) equipped with a LINK data-  
229 processing system (INCA 4.09, Oxford Instruments, England) at an acceleration voltage of  
230 10-20 kV.

231

## 232 2.7. Color

233 The color of dried-salted cod was measured by computing the CIE  $L^*a^*b^*$  color  
234 coordinates using a colorimeter (Minolta CR-200, Konica Minolta Optics, Inc., Japan). In  
235 each slice, color test was conducted at 6 points following a preset pattern. According to CIE  
236  $L^*a^*b^*$  system,  $L^*$  measures the lightness on a 0 to 100 scale from black to white;  $a^*$ , (+)  
237 red or (-) green; and  $b^*$ , (+) yellow or (-) blue (Bai, Sun, Xiao, Mujumdar, & Gao, 2013).  
238 The overall color differences ( $\Delta E$ ) between AIR and AIR+US samples were also  
239 determined by Eq. (8).

$$240 \Delta E = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (8)$$

241

## 242 2.8. Statistical analysis

243 In order to identify whether ultrasound and the air temperature significantly ( $p < 0.05$ )  
244 influenced the drying, rehydration properties and the texture and color of dried-salted cod,  
245 analysis of variance (ANOVA) ( $p < 0.05$ ) was carried out and the least significant difference  
246 (LSD) intervals were identified using the Statgraphics Plus 5.1. statistical package  
247 (Statistical Graphics Corp., USA).

248

### 249 **3. Results and discussion**

#### 250 3.1 Experimental drying kinetics

251 The experimental drying kinetics (AIR, AIR+US) of salted cod slabs at -10, 0, 10 and 20 °C  
252 are shown in Fig. 1. The average initial moisture and NaCl contents of salted cod were  
253  $1.23 \pm 0.07$  kg W/kg dry matter and  $0.41 \pm 0.03$  kg NaCl/kg dry matter, respectively. Both the  
254 fact that the raw matter used constitutes a partially-dried material and also the high  
255 concentration of NaCl in cod flesh limits the presence of free water. For this reason, no  
256 constant rate drying period was observed and drying occurred in the falling rate period  
257 (Bellagha et al., 2007). Thus, the initial moisture content was considered as the critical one  
258 for modeling purposes.

259 As stated in Section 2.2, drying experiments were extended until the weight loss reached  
260 20% of the initial weight. Thereby, dried-salted samples presented final moisture and NaCl  
261 contents of  $0.77 \pm 0.04$  kg W/kg dry matter and  $0.41 \pm 0.02$  kg NaCl/kg dry matter,  
262 respectively. The results obtained for the AIR experiments showed that the air temperature  
263 affected drying kinetics, shortening the drying time. For example, the drying time for the  
264 lowest temperature tested, -10 °C, was over 14 h, while at 20 °C, it was only 5 h. Air  
265 temperature was found to have a similar influence in AIR+US experiments.

266 The application of power ultrasound sped up the drying kinetics, as can be observed if the  
267 AIR and AIR+US experiments are compared (Fig. 1). Ultrasound application led to a  
268 shortening of the drying time of between 35 and 54% as compared to AIR experiments. The  
269 greatest time reduction under ultrasound application was found at the lowest and the  
270 highest temperatures tested (-10 and 20 °C). Using the same ultrasonic set-up and under  
271 similar experimental conditions, Garcia-Perez et al. (2012a) found drying time reductions  
272 of 68 and 70% in the drying of carrot and eggplant cubes (-14 °C and 2 m/s), respectively.

273 When drying green peas at -3 °C, Bantle and Eikevik (2011) showed that, with ultrasound  
274 application (20 kHz; DN 20/2000, Sonotronic), there was a maximum reduction in the  
275 drying time of around 10%. On the other hand, drying surimi slabs at 20 °C and 2.8 m/s,  
276 Nakagawa et al. (1996) observed that the drying time was 80% shorter when acoustic  
277 waves (155.5 dB, 19.5 kHz) were applied.

278

### 279 3.2 Modeling drying kinetics

280 In order to quantify the influence of power ultrasound application and temperature on the  
281 drying kinetics of salted cod slabs, the proposed diffusion model (Eq. (3)) was fitted to the  
282 experimental data.

283 The diffusion model provided a percentage of explained variance (%VAR) close to 99% for  
284 AIR and AIR+US experiments at 0, 10 and 20 °C (Table 1). This fact suggests that, in these  
285 cases, the drying process followed a clear diffusion pattern. In the case of AIR+US  
286 experiments at -10 °C, the explained variance achieved by the model was much lower  
287 (95.4%) than at other temperatures, and also lower than that found in AIR experiments at  
288 the same temperature (98.3%). This indicates that there is some influence of ultrasound on  
289 the mass transfer control mechanisms and diffusion was not the only significant mechanism  
290 controlling water transport. In this regard, Garcia-Perez et al., (2012a) observed the same  
291 behavior when analyzing the low-temperature drying (-14 °C, 2 m/s) of carrot and  
292 highlighted that, under such particular conditions, ultrasound application led to a larger  
293 improvement in the diffusion coefficient (407-428%) than it did in the mass transfer  
294 coefficient (96-170%). Therefore, the application of power ultrasound reduced the internal  
295 resistance to mass transfer more than the external, which means that diffusion does not

296 prevail as the most significant ( $p < 0.05$ ) water transfer controlling mechanism and  
297 convection grows in importance.

298 In AIR experiments,  $D_w$  values ranged from  $0.14 \times 10^{-10}$  to  $0.35 \times 10^{-10} \text{ m}^2/\text{s}$ . Temperature  
299 affected the effective moisture diffusivity, so, the higher the temperature applied, the  
300 greater the identified effective moisture diffusivity (Table 1). The effective moisture  
301 diffusivities identified for the AIR experiments are close to other reported values. Park  
302 (1998) obtained values of around  $0.87\text{-}1.61 \times 10^{-10} \text{ m}^2/\text{s}$  for salted fish muscle dried at 20-  
303 40 °C. In a data compilation of the moisture diffusivity of foodstuffs, Zogzas, Maroulis, and  
304 Marinos-Kouris (1996) reported  $D_w$  values ranging between  $0.13 \times 10^{-10}$  and  $3.1 \times 10^{-10}$   
305  $\text{m}^2/\text{s}$  for the drying of unsalted fish muscle (30 °C).

306 The application of high power ultrasound significantly increased ( $p < 0.05$ ) the effective  
307 moisture diffusivity at every temperature tested. The increase in this parameter ( $\Delta D_w$ )  
308 ranged from 110% at the highest temperature tested, 20 °C, to 42% at 0 °C (Table 1). The  
309 improvement in the  $D_w$  values is mainly linked with the mechanical effects brought about  
310 by applying ultrasound to the material being dried (Puig et al., 2012). Ultrasound  
311 introduces a series of rapid and cyclic compressions and expansions of the material that can  
312 be compared to a sponge being squeezed and released repeatedly, thus improving the water  
313 diffusion in the particle. Moreover, acoustic energy also introduces pressure variations,  
314 oscillating velocities, and microstreaming on the solid-gas interfaces, reducing boundary  
315 layer thickness and, therefore, improving the water transfer rate from the solid surface to  
316 the air medium (Cárcel et al., 2012).

317 The influence of air temperature on  $D_w$  identified for AIR and AIR+US experiments  
318 followed an Arrhenius type relationship (Fig. 2) (Bai et al., 2013). A similar activation  
319 energy of 20.46 and 21.79 kJ/mol was obtained for AIR and AIR+US drying experiments,

320 respectively. In such a way, the energy needed for water removal was not affected by  
321 ultrasound application. The activation energy figures are similar to those proposed by other  
322 authors for fish drying. Thus, Jason (1958) reported an activation energy of 30 kJ/mol for  
323 cod muscle (unsalted) and Park (1998) a value of 21.94 kJ/mol for the drying of salted  
324 shark muscle.

325 From Fig. 2, the  $D_w$  values for AIR and AIR+US experiments are easily compared. It may  
326 be observed that the  $D_w$  for AIR+US experiments at -10 °C was similar to the figure found  
327 in AIR experiments at 0 °C. Similar values were also found for AIR+US and AIR  
328 experiments at 0 and 20 °C, respectively. This fact suggests that the increase in  $D_w$   
329 produced by US application could be equivalent to a temperature rise of between 10 and 20  
330 °C.

331

### 332 3.3. Rehydration kinetics

333 Samples dried under the different conditions tested were rehydrated in distilled water at 4  
334 °C. The net weight change was used to monitor the process kinetics ( $\Delta M^0_t$ ), in which a  
335 coupled water and salt transfer exists. The experimental data showed that the drying air  
336 temperature influenced the rehydration kinetics (Fig. 3). For example, after 27 h of  
337 rehydration, AIR samples dried at 20 °C gained 38% more weight than that observed in  
338 samples dried at -10 °C. Duan et al. (2011) reported that the rehydration ratio of dried  
339 tilapia fish fillets increased with the rise in drying temperature. Likewise, Russo, Adiletta,  
340 and Di Matteo. (2013) connected the drying temperature with a marked influence on the  
341 microstructure of dried products; high drying temperatures led to an increase in porosity  
342 and the collapse of the structure, causing a rise in the initial water uptake during  
343 rehydration.



344 The application of ultrasound during drying affected the rehydration ability of dried-salted  
345 cod samples. The AIR+US samples exhibited a higher final weight gain than AIR ones  
346 (Fig. 3) and, therefore, a higher final water content (Table 2). As regards the final NaCl  
347 content, no significant ( $p>0.05$ ) changes were observed between AIR and AIR+US  
348 samples.

349 The higher water gain in AIR+US dried samples could be linked to changes produced by  
350 ultrasound in the microstructure during drying (Fig. 4). According to the SEM micrograph  
351 obtained from the longitudinal section of salted cod dried at 0 °C, AIR+US samples (Figs. 4  
352 D and E) showed a more damaged and collapsed structure than AIR ones (Figs. 4 A and B).  
353 Ultrasound application provoked ruptures in the cod fibers (Fig 4 E) and a greater migration  
354 of salt to the fiber surface (Fig. 4 D). In addition, micrographs obtained from a cross section  
355 of AIR+US dried-salted cod exhibited the formation of wider spaces between myofibrils  
356 and a more intense salt redistribution on the surface (Fig. 4 F). Hence, these structural  
357 differences which are induced by mechanical effects linked to ultrasound can explain the  
358 greater weight gain of AIR+US samples during rehydration. In addition, the fact that  
359 ultrasound application leads to a shorter drying time can lessen the damage to the protein  
360 structure (denaturation), contributing to a greater water holding capacity, thereby increasing  
361 the rehydration capacity of AIR+US samples (Brás & Costa, 2010).

362

### 363 3.4. Modeling rehydration kinetics.

364 The Peleg model was used to analyze and quantify the influence of both drying temperature  
365 and ultrasound application on the net weight gain ( $\Delta M^0_t$ ) of dried-salted cod slabs during  
366 rehydration. As can be observed in Table 3, the model adequately described the rehydration  
367 kinetics, providing percentages of explained variance ranging between 96.8 and 99.3%.

368 The identified Peleg parameters, related to the initial mass transfer rate ( $1/k_1$ ) and the  
369 equilibrium weight ( $1/k_2$ ), are shown in Table 3. As far as the effect of the drying  
370 temperature is concerned, the identified model parameters for AIR samples slightly  
371 increased when the temperature rose (Table 3). However, these increases were only  
372 significant ( $p < 0.05$ ) for the equilibrium constant,  $1/k_2$ . Air temperature was also observed  
373 to have a similar effect on AIR+US samples.

374 The application of ultrasound during drying significantly modified ( $p < 0.05$ ) the Peleg  
375 parameters at every drying air temperature tested. There was a faster and more substantial  
376 rehydration of AIR+US samples than AIR samples, which could be observed in the  
377 increase in both  $1/k_1$  and  $1/k_2$  parameters. Thereby, when comparing the rehydration  
378 patterns of AIR and AIR+US samples, the changes observed should be related to the  
379 structural changes brought about by ultrasound and depicted in Section 3.3. As can be  
380 observed in Table 3, ultrasound had a greater effect on Peleg parameters,  $\Delta 1/k_1$  and  $\Delta 1/k_2$ ,  
381 at the lowest temperatures tested (-10 and 0 °C) than at the highest ones (10 and 20 °C).  
382 This fact could be linked to the length of time samples are exposed to the ultrasonic energy.  
383 While drying experiments carried out at the lowest temperatures (-10 and 0 °C) lasted  
384 approximately 6 to 8 h, at the highest temperatures (10 and 20 °C), the drying time was  
385 reduced by almost half (Fig. 1). Therefore, the longer the exposure time to ultrasound  
386 application, the more intense the ultrasound effects on the cod structure (Fig. 4).

387

### 388 3.5. Texture

389 The hardness of dried and rehydrated cod was evaluated by computing the maximum  
390 penetration force. The initial hardness value of salted cod was  $3.62 \pm 0.35$  N, thus, the  
391 drying process provoked a hardening of the samples (Table 4). The measurements taken

392 from AIR samples showed that the sample hardness was dependent on the drying air  
393 temperature used (Table 4); the higher the air temperature applied, the harder the dried cod.  
394 Thus, AIR samples dried at 10 and 20 °C were significantly ( $p<0.05$ ) harder than those  
395 dried at 0 and -10 °C. The temperature rise could induce a greater denaturation of the  
396 connective tissues and the myofibrillar proteins (myosin and actin) promoted by NaCl,  
397 leading to the sample hardening. (Ortiz et al., 2013; Brás & Costa, 2010).

398 As is shown in Table 4, AIR+US dried samples were significantly ( $p<0.05$ ) softer than AIR  
399 ones. This fact could be linked to the mechanical effects caused by ultrasound application  
400 on salted cod fibers (Figs. 4 D, E and F). The fact that the structure of the AIR+US samples  
401 is more collapsed and porous than the AIR samples (Figs. 4 A, B and C) explains the low  
402 degree of hardness found. In addition, the application of ultrasound is linked to a reduction  
403 in the drying time which could contribute to some mild damage in the protein structure,  
404 causing a lesser degree of hardening (Oliveira et al., 2012; Kilic, 2009). Moreover, the  
405 hardness of AIR+US samples was not affected by the drying air temperature (Table 4).

406 The hardness was also measured in rehydrated samples (4 °C, 27 h). As can be observed in  
407 Table 4, the rehydration produced a softening of dried-salted samples. After rehydration,  
408 AIR samples dried at -10, 10 and 0 °C were significantly ( $p<0.05$ ) softer than those dried at  
409 20 °C. This fact suggests that drying at 20 °C affected the structure of the dry material,  
410 limiting the softening of rehydrated samples. At every temperature tested, AIR+US  
411 rehydrated samples were softer than AIR ones, which is consistent with the effect observed  
412 in rehydration kinetics (Fig. 3, Table 2) produced by structural changes (Fig. 4).

413

414 3.6. Color

415 The color of dried-salted cod is generally considered to be one of the most relevant quality  
416 traits. In order to analyze the influence of ultrasound application and drying temperature on  
417 the color changes of dried-salted cod, CIE L\*a\*b\* color coordinates were measured  
418 directly on dried samples under different conditions (AIR and AIR+US at -10, 0, 10 and 20  
419 °C). The average values of chromatic coordinates (L\*, a\*, b\*) for raw salted cod were  
420  $L^*=58.93\pm 2.00$ ,  $a^*=-3.66\pm 0.49$  and  $b^*=4.86\pm 1.50$ . Thus, as observed in Fig. 5, the drying  
421 process provoked changes in the color of fish muscle (Fig.5). In general terms, L\*, a\* and  
422 b\* increased, which indicates that the drying process caused both the yellowing (higher b\*)  
423 of the samples and the increase in their lightness index (higher L\*) (Brás & Costa, 2010,  
424 Lauritzen et al., 2004).

425 In AIR samples, L\* and b\* coordinates values were significantly ( $p<0.05$ ) affected by the  
426 drying air temperature; the higher the temperature applied, the lower the values of L\* and  
427 b\* (Figs. 5 A, C). This may be explained by the fact that a temperature rise induces, in the  
428 cod muscle, the contraction of myotomes due to protein aggregation (Fernandez-Segovia,  
429 Camacho, Martinez-Navarrete, Escriche, & Chiralt, 2003), the oxidation of phospholipids  
430 and reactions which stem from the presence of other ions in the salt composition (Oliveira  
431 et al., 2012). This leads to an increase in the opacity of the fish tissue (Lauritzen et al.,  
432 2004) and contributes to the color degradation of dried-salted cod samples. The  
433 experimental measurements obtained coincide with those reported by Ortiz et al. (2013),  
434 who studied the influence of drying air temperature on the color of dried salmon (*Salmo*  
435 *salar* L.) fillets and found a significant ( $p<0.05$ ) decrease in L\* and b\* values when  
436 comparing samples dried at 40 and 60 °C. On the contrary, the a\* value for AIR samples  
437 did not show any significant ( $p<0.05$ ) temperature-linked trend (Fig. 5 B).

438 As observed in Figure 5 D, there were found to be changes between the color ( $\Delta E$ ) of  
439 AIR+US and AIR samples, which were dependent on the drying temperature (Fig. 5 D);  
440 thus, the lower the drying temperature, the greater the color differences between AIR and  
441 AIR+US samples. These differences are obviously linked to specific changes in chromatic  
442 coordinates ( $L^*$ ,  $b^*$  and  $a^*$ ), although no common pattern was found. As regards  $L^*$  and  $b^*$   
443 coordinates, AIR+US samples dried at -10 and 0 °C exhibited higher lightness ( $L^*$ ) and  
444 lower yellowness ( $b^*$ ) values in comparison to AIR samples, which could be interesting for  
445 the cod industry which requires products with high whiteness values (Oliveira et al., 2012).  
446 Moreover, AIR+US samples dried at 10 and 20 °C did not show significant ( $p<0.05$ )  
447 changes in  $L^*$  and  $b^*$  coordinates compared to AIR samples (Figs. 5 A, C). According to  
448 the  $a^*$  coordinate (Fig. 5 B), there was a significant ( $p<0.05$ ) effect of ultrasound on  
449 samples dried at -10, 0 and 10 °C; however, this effect did not exhibit a significant  
450 temperature-linked trend.

451

#### 452 **4. Conclusions**

453 The application of power ultrasound during the drying of salted cod improved the drying  
454 rate, shortening the drying time by an average of 35-50%. Water removal at 0, 10 and 20 °C  
455 showed a diffusion pattern, while at -10 °C convection was also significant, especially  
456 when ultrasound was applied. Microstructural analyses showed that the application of  
457 ultrasound during drying brought about changes in cod fibers, which led to a higher  
458 rehydration capacity and softer samples. Ultrasound also promoted changes in dried-salted  
459 cod, particularly an increase in lightness ( $L^*$ ) at low temperatures. Therefore, the feasibility  
460 of power ultrasound, a non-thermal technology, to improve the low-temperature drying of  
461 salted cod has been highlighted and further studies should address whether the kinetic

462 improvement is coupled to an energy reduction, which could bring this technology closer to  
463 a potential industrial use.

464

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470 University College.

471 **NOMENCLATURE**

$D_w$	Effective moisture diffusivity, $m^2/s$
$\Delta D_w$	Increase in effective moisture diffusivity produced by ultrasound application, %
$L$	Half thickness, m
$x$	Characteristic coordinate in slab geometry, m
$\lambda_n$	Eigenvalues
$S_y$	Standard deviation of the sample
$S_{yx}$	Standard deviation of the estimation
$t$	Time, s
$M_0$	Initial weight, g
$M_t$	Weight at time $t$ , g
$M_e$	Equilibrium weight, g
$\Delta M_t^0$	Net weight change, g
$W$	Average moisture content, kg water/kg dry matter
$W_0$	Initial moisture content, kg water/kg dry matter
$W_p$	Local moisture content, kg water/kg dry matter
$W_{eq}$	Equilibrium moisture content, kg water/kg dry matter
$1/k_1$	Peleg rate constant, g water/g dry matter $\times$ s
$1/k_2$	Peleg capacity constant, g
$\Delta 1/k_1$	Increase in Peleg rate constant produced by ultrasound application, %
$\Delta 1/k_2$	Increase in Peleg capacity constant produced by ultrasound application, %
$L^*$	Chromatic coordinate, lightness 0 (black) to 100 (white)

a\* Chromatic coordinate, (+) red or (-) green

b\* Chromatic coordinate, (+) yellow or (-) blue



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579

580 **Figure captions**

581 **Figure 1.** Drying kinetics of salted cod slabs (length 50 mm, width 30 mm and thickness 10  
582 mm) at -10, 0, 10 and 20 °C (2 m/s) with (AIR+US, 20.5 kW/m<sup>3</sup>) and without (AIR)  
583 ultrasound application.

584 **Figure 2.** Influence of drying air temperature on the average effective moisture diffusivities  
585 identified for salted cod drying with (AIR+US, 20.5 kW/m<sup>3</sup>) and without (AIR) ultrasound  
586 application.

587 **Figure 3.** Rehydration kinetics (4 °C) of salted cod slabs (length 50 mm, width 30 mm and  
588 thickness 10 mm) dried at -10, 0, 10 and 20 °C (2 m/s) with (AIR+US, 20.5 kW/m<sup>3</sup>) and  
589 without (AIR) ultrasound application.

590 **Figure 4.** Longitudinal and cross-section (x100) observed by SEM. Dried salted cod at 0  
591 °C and 2 m/s with (AIR+US, 20.5 kW/m<sup>3</sup>: D, E and F) and without ultrasound application  
592 (AIR: A, B and C).

593 **Figure 5.** CIELAB coordinates (L\*, a\*, b\*) of salted cod (length 50 mm x width 30 mm x  
594 thickness 10 mm) dried at -10, 0, 10 and 20 °C with (20.5 kW/m<sup>3</sup>, AIR+US) and without  
595 (AIR) ultrasound application.  $\Delta E$  represents the overall color change between AIR+US and  
596 AIR samples. Average values  $\pm$  LSD intervals at a confidence level of 95% are plotted.

597

Figure 1

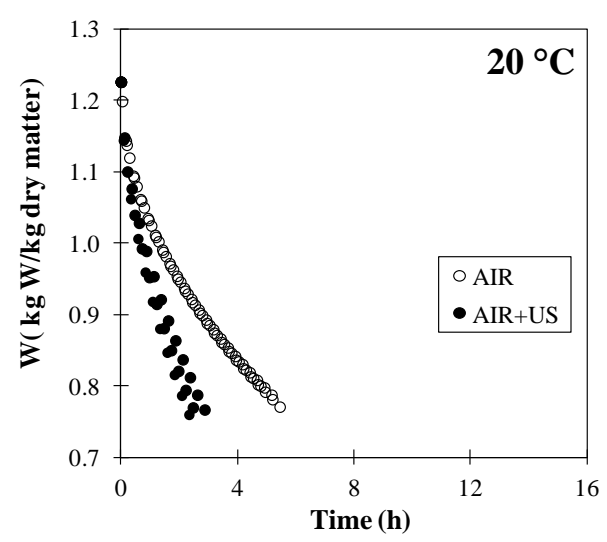
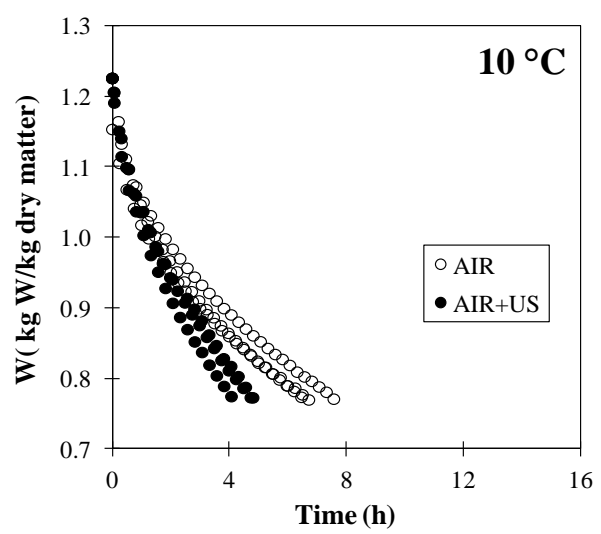
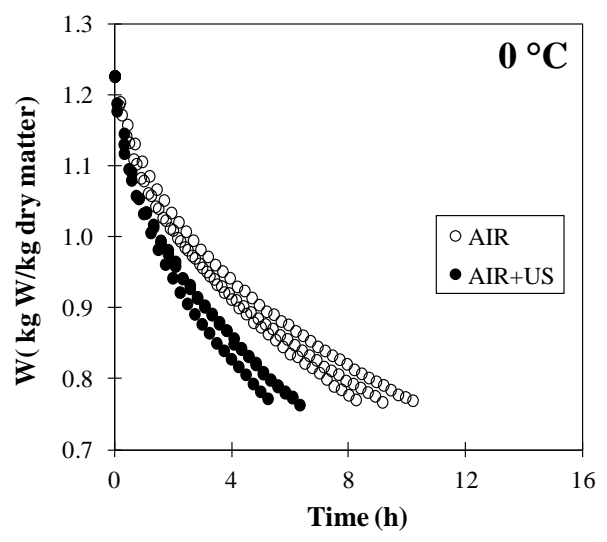
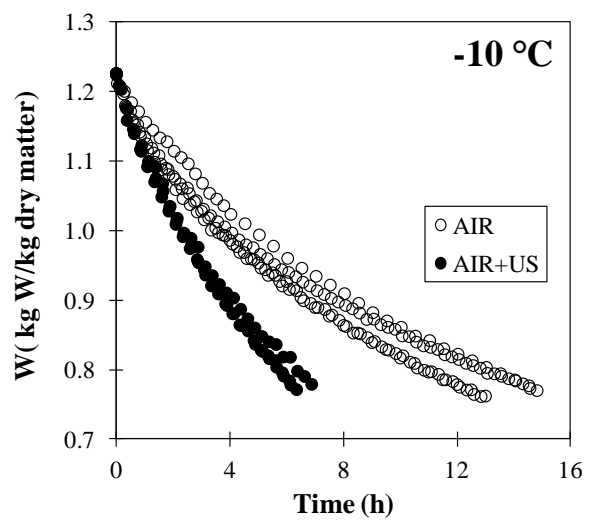


Figure 2

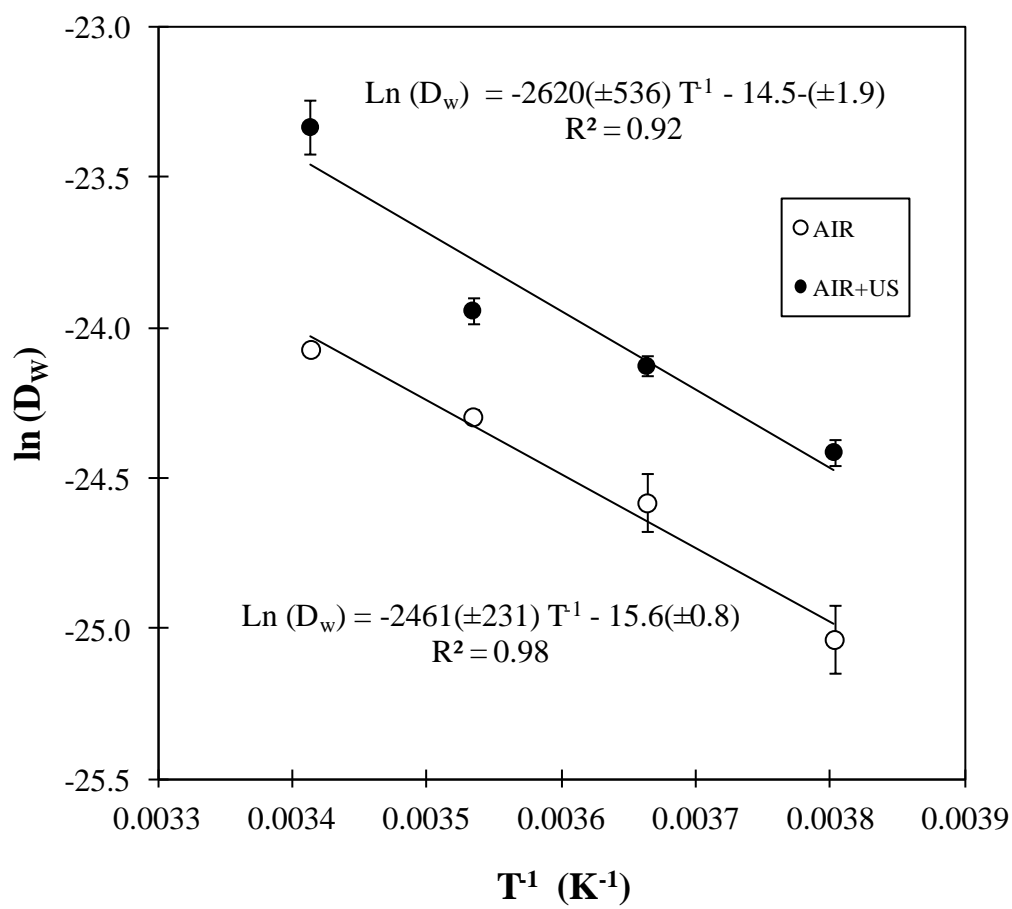




Figure 3

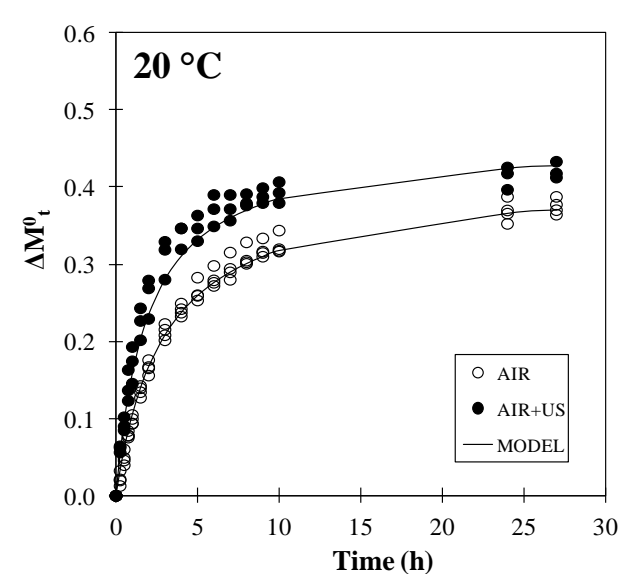
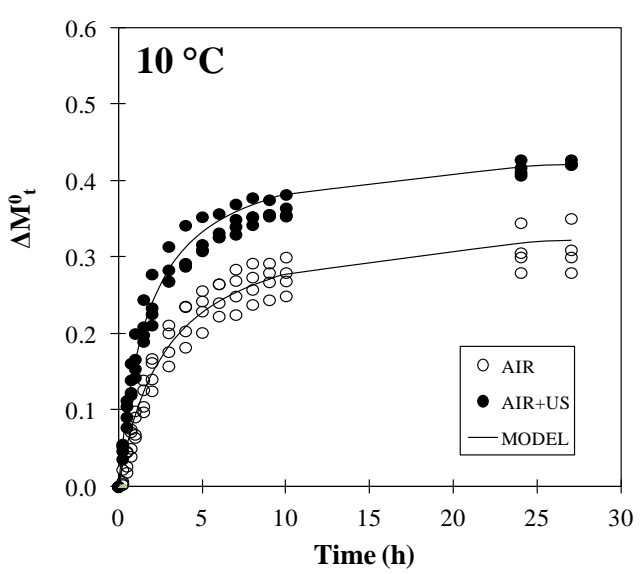
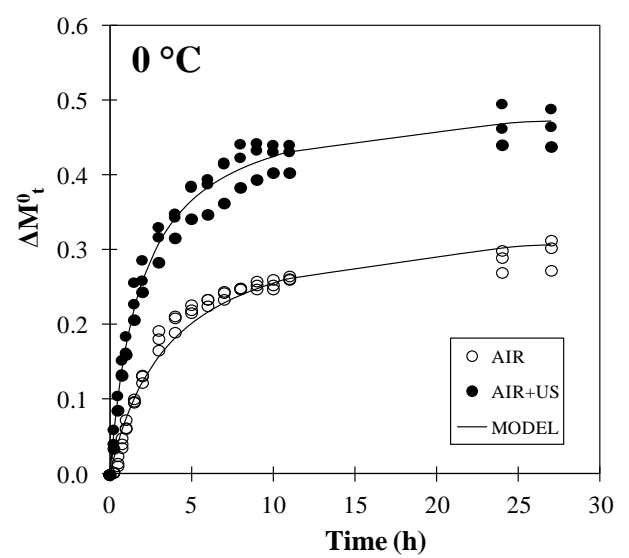
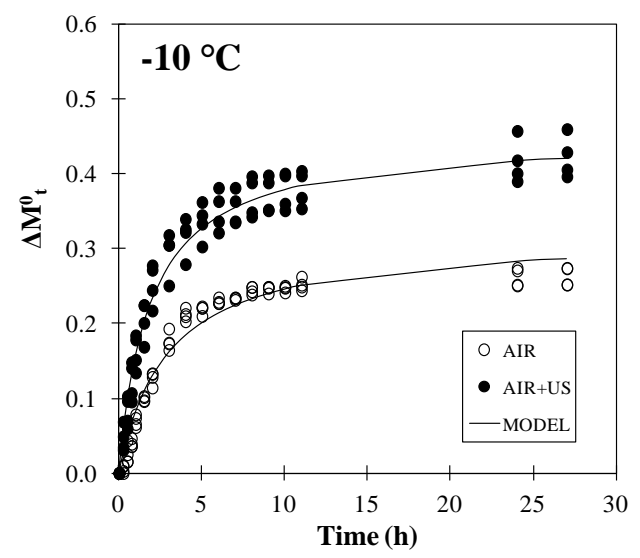


Figure 4

AIR

AIR+US

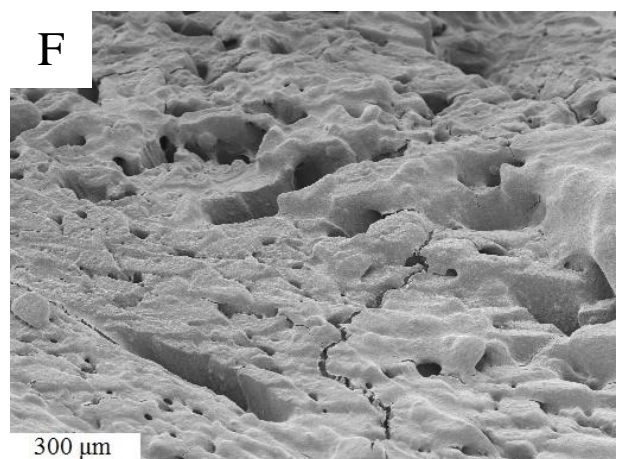
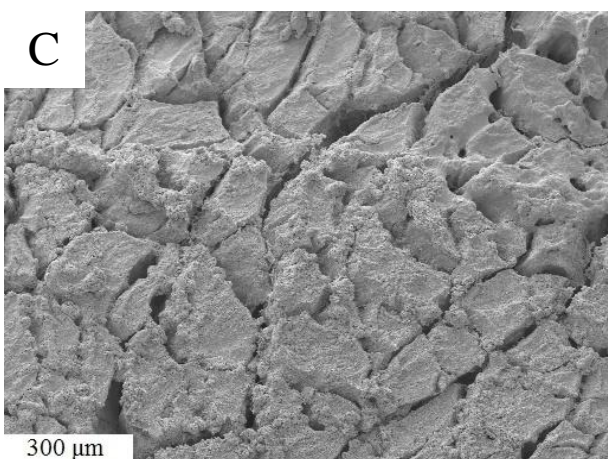
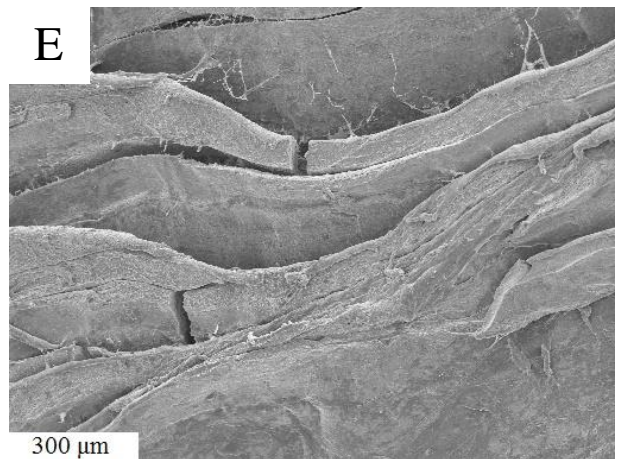
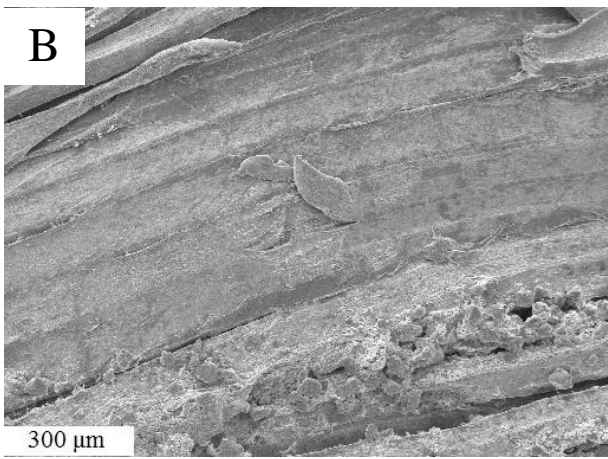
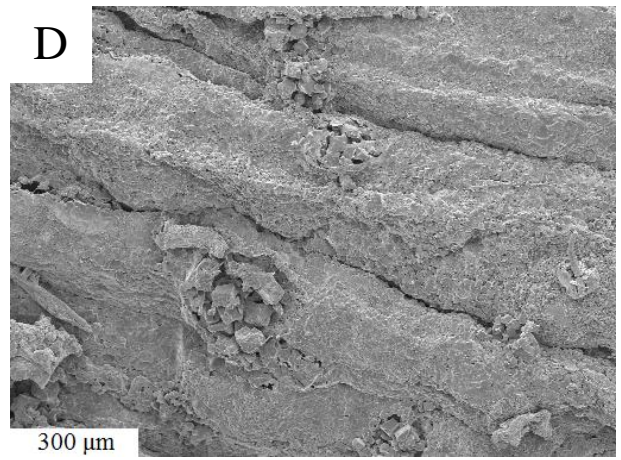
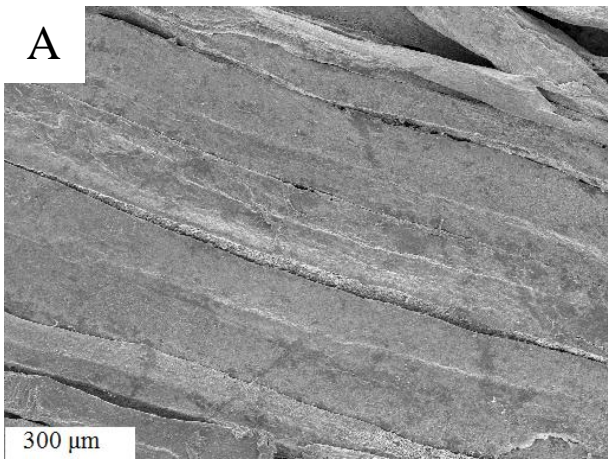
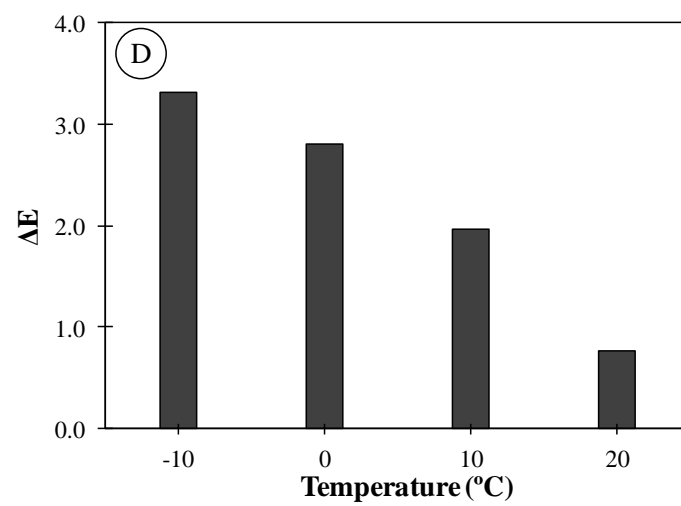
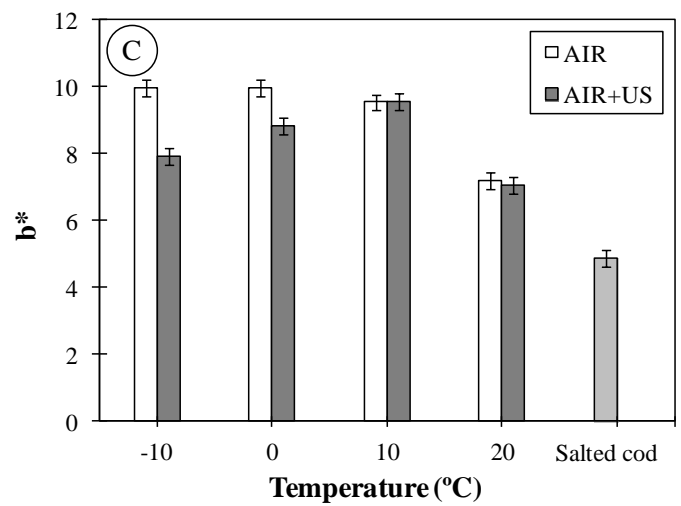
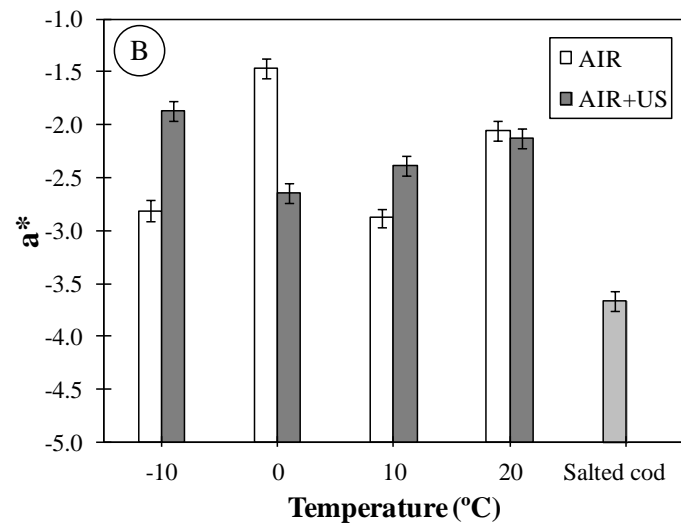
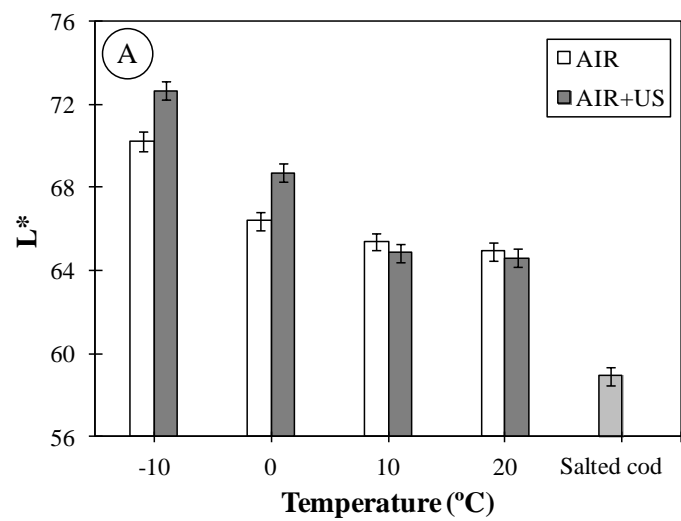


Figure 5



**Table 1.** Average values and standard deviation of effective diffusivity,  $D_w$ , on salted cod drying. Increase in effective moisture diffusivity,  $\Delta D_w$  (%), produced by ultrasound application and percentage of explained variance, VAR (%). The superscripts a, b, c, d and e show homogeneous groups established from LSD (least significance difference) intervals ( $p < 0.05$ ).

T (°C)		$D_w (10^{-10} \text{ m}^2/\text{s})$	VAR (%)	$\Delta D_w$ (%)
-10	AIR	$0.14 \pm 0.01_a$	98.3	-
	AIR+US	$0.25 \pm 0.02_b$	95.4	85.5
0	AIR	$0.21 \pm 0.02_b$	99.8	-
	AIR+US	$0.33 \pm 0.03_c$	99.6	57.3
10	AIR	$0.28 \pm 0.02_{b,c}$	99.6	-
	AIR+US	$0.40 \pm 0.04_d$	99.4	42.4
20	AIR	$0.35 \pm 0.01_{c,d}$	99.6	-
	AIR+US	$0.74 \pm 0.04_e$	99.9	110.1

**Table 2.** Experimental moisture and NaCl content of rehydrated (27 h) dried salted cod at -10, 0, 10 and 20 °C with (20.5 kW/m<sup>3</sup>, AIR+US) and without (AIR) ultrasound application. Average  $\pm$  standard deviations are shown. The superscripts a, b and c (in moisture) and x (in NaCl) show homogeneous groups established from LSD (least significance difference) intervals ( $p < 0.05$ ).

T (°C)		W (kg W/kg dry matter)	NaCl (kg NaCl/kg dry matter)
-10	AIR	3.38 $\pm$ 0.11 <sub>a</sub>	0.037 $\pm$ 0.004 <sub>x</sub>
	AIR+US	3.80 $\pm$ 0.20 <sub>c</sub>	0.036 $\pm$ 0.004 <sub>x</sub>
0	AIR	3.37 $\pm$ 0.19 <sub>a</sub>	0.041 $\pm$ 0.005 <sub>x</sub>
	AIR+US	3.81 $\pm$ 0.17 <sub>c</sub>	0.040 $\pm$ 0.007 <sub>x</sub>
10	AIR	3.39 $\pm$ 0.15 <sub>a</sub>	0.040 $\pm$ 0.007 <sub>x</sub>
	AIR+US	3.76 $\pm$ 0.12 <sub>c</sub>	0.040 $\pm$ 0.005 <sub>x</sub>
20	AIR	3.00 $\pm$ 0.13 <sub>b</sub>	0.040 $\pm$ 0.009 <sub>x</sub>
	AIR+US	3.37 $\pm$ 0.18 <sub>a</sub>	0.041 $\pm$ 0.003 <sub>x</sub>

**Table 3.** Modeling of the net weight gain,  $\Delta M^0_t$ , by means of the Peleg model during rehydration of dried salted cod at -10, 0, 10 and 20 °C with (20.5 kW/m<sup>3</sup>, AIR+US) and without (AIR) ultrasound application. Increase in Peleg parameters,  $\Delta 1/k_1$  and  $\Delta 1/k_2$  (%), produced by ultrasound application and percentage of explained variance, VAR (%). The superscripts x and y (in  $1/k_1$ ) and a, b, c, d and e (in  $1/k_2$ ) show homogeneous groups established from LSD (least significance difference) intervals ( $p < 0.05$ ).

T (°C)		$1/k_1$ ( $10^{-3}$ g W/g dry matter $\times$ s)	$1/k_2$ (g)	VAR (%)	$\Delta 1/k_1$ (%)	$\Delta 1/k_2$ (%)
-10	AIR	$1.8 \pm 0.2_x$	$0.32 \pm 0.01_a$	96.8	-	-
	AIR+US	$4.2 \pm 0.9_y$	$0.45 \pm 0.01_d$	98.8	133.0	41.8
0	AIR	$1.8 \pm 0.3_x$	$0.35 \pm 0.01_b$	97.7	-	-
	AIR+US	$4.4 \pm 0.4_y$	$0.51 \pm 0.02_e$	98.6	151.8	46.6
10	AIR	$2.2 \pm 0.3_x$	$0.36 \pm 0.02_b$	98.6	-	-
	AIR+US	$4.4 \pm 0.8_y$	$0.45 \pm 0.02_d$	99.3	104.1	25.8
20	AIR	$2.4 \pm 0.2_x$	$0.41 \pm 0.03_c$	98.7	-	-
	AIR+US	$4.1 \pm 0.7_y$	$0.46 \pm 0.03_d$	99.1	70.6	11.4

**Table 4.** Hardness (average  $\pm$  standard deviations) of dried and dried+rehydrated-salted cod samples at -10, 0, 10 and 20 °C with (20.5 kW/m<sup>3</sup>, AIR+US) and without (AIR) ultrasound application. The superscripts a, b and c (in dried) and x, y and z (in dried+rehydrated) show homogeneous groups established from LSD (least significance difference) intervals ( $p < 0.05$ ).

T (°C)		Dried	Dried+Rehydrated
		Hardness (N)	Hardness (N)
-10	AIR	9.93 $\pm$ 0.80 <sub>a</sub>	1.13 $\pm$ 0.13 <sub>x</sub>
	AIR+US	7.65 $\pm$ 0.48 <sub>b</sub>	0.89 $\pm$ 0.09 <sub>y</sub>
0	AIR	9.93 $\pm$ 0.70 <sub>a</sub>	1.12 $\pm$ 0.11 <sub>x</sub>
	AIR+US	7.30 $\pm$ 0.61 <sub>b</sub>	0.88 $\pm$ 0.10 <sub>y</sub>
10	AIR	11.58 $\pm$ 0.50 <sub>c</sub>	1.17 $\pm$ 0.16 <sub>x</sub>
	AIR+US	7.31 $\pm$ 0.55 <sub>b</sub>	0.88 $\pm$ 0.10 <sub>y</sub>
20	AIR	12.4 $\pm$ 0.96 <sub>c</sub>	2.38 $\pm$ 0.20 <sub>z</sub>
	AIR+US	8.25 $\pm$ 0.57 <sub>b</sub>	1.19 $\pm$ 0.17 <sub>x</sub>