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Additional Information

1	ULTRASONICALLY ASSISTED LOW-TEMPERATURE DRYING OF DESALTED
2	CODFISH
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18	

### 19 Abstract

Low-temperature drying (LTD) constitutes an interesting means of dehydrating foodstuffs, thus preserving the quality of the product. Power ultrasound (US) generates several mechanical effects that could help to shorten the long drying times associated with LTD. In this work, the feasibility of using US in LTD of desalted cod was assessed.

For this purpose, desalted cod slices (50x30x5 mm) were dried (2 m/s) at different temperatures (10, 0 and -10°C) without (AIR) and with (AIR+US, 20.5 kW/m<sup>3</sup>) US application. Afterwards, the dried samples were rehydrated in distilled water (25°C). A diffusion model was used to describe both drying and rehydration kinetics. The color and hardness of both dried and rehydrated cod samples were also measured.

The application of US increased the drying rate at every temperature tested, shortening the drying time by 16% at 0°C and up to 60% at -10°C. The ultrasonically assisted dried samples presented a rehydration rate which was slightly lower than that of those that had been conventionally dried, but they were harder and whiter, which is more suited to consumer preferences. Therefore, power ultrasound could be considered an affordable technology with which to accelerate LTD of desalted cod, providing high quality dried products.

36

37 *Keywords:* Ultrasound; Dehydration; Rehydration; Texture; Color

38

## 39 **1. Introduction**

40 Dried and salt-cured cod (Gadus morhua) is a highly-appreciated product due to its 41 high nutritional value (high protein and low fat content) and its particular sensory 42 properties. It is mainly produced in Norway and Iceland and primarily consumed in the 43 Southern European countries, such as Spain and Portugal (Martínez-Álvarez and 44 Gómez-Guillén, 2013; Oliveira, Pedro, Nunes, Costa, and Vaz-Pires, 2012). The high 45 salt concentration of this product (approximately 20% w/w) prevents its degradation but 46 limits its direct consumption; for this reason salted cod must be desalted (Ozuna, Puig, 47 Garcia-Perez, and Cárcel, 2014a), a process that takes approximately 24h. This slow 48 salt diffusion constrains the consumption of salted cod for both domestic use and the 49 catering industry. In addition, the desalting converts the cod into a highly perishable 50 product (Fernández-Segovia, Escriche, Fuentes, and Serra, 2007) and, in fact, the fish 51 must be either immediately consumed, chilled or frozen (Lauritzsen et al., 2004). 52 Therefore, it could be interesting to explore alternative preservation methods, such as 53 drying, that ensure both the desalted product's stability and the retention of the sensory 54 attributes (Andrés, Rodríguez-Barona, and Barat, 2005). The desalted and dried cod 55 may be used as an ingredient in prepared foods, such as instant meals or ready-to-use 56 products, due to its low salt content and rehydration ability.

57 Convective drying constitutes a traditional dehydration method for foodstuffs (Garcia-58 Perez, Ozuna, Ortuño, Cárcel, and Mulet, 2011). The use of high air temperatures 59 accelerates the drying kinetics, but causes chemical and physical changes that can 60 affect the quality traits of the dried product (Soria et al., 2010). Consumer demand for 61 high quality products has encouraged research into alternative techniques to minimize 62 quality degradation during processing. In this sense, low temperature drying (LTD) 63 could be an interesting method. However, the long drying times linked to LTD could 64 limit its use on an industrial scale.

65 Power ultrasound (US) has been used to speed up the convective drying of several 66 foodstuffs (Cárcel, Garcia-Perez, Riera, and Mulet, 2011; Gallego-Juárez et al., 2007; 67 Garcia-Perez et al., 2011), mainly by introducing mechanical energy. The ultrasonic 68 waves generate alternating expansions and contractions when travelling across a 69 medium, which have a similar effect to that found in a sponge when it is repeatedly 70 squeezed and released (Gallego Juárez et al., 2007). This mechanical stress helps the 71 water move from the inner parts of the product to the surface and could create 72 microscopic channels that reduce the internal resistance to mass transfer (Gallego-73 Juárez, 2010). Moreover, in solid/gas systems, the application of US also produces 74 oscillating velocities, micro-streaming and pressure variation at the interfaces, which 75 reduce the boundary layer and, as a consequence, improve water movement from the 76 solid surface to air. Therefore, US could help to reduce both the external and the 77 internal mass transfer resistance without introducing a significant amount of thermal 78 energy during drying (Cárcel et al., 2011). In this sense, the feasibility of US application 79 during the LTD process of different products, such as apple (Santacatalina et al., 2014, 80 Garcia-Perez, Cárcel, Riera, Rosselló, and Mulet, 2012), salted cod (Ozuna, Cárcel, 81 Walde, and Garcia-Perez, 2014b), green peas (Bantle and Eikevik, 2011), carrot or 82 eggplant (Garcia-Pérez et al., 2012) has been proved. More research has been done 83 on analyzing the effect of US on the drying kinetics than on the product quality (Pingret, 84 Fabiano-Tixier, and Chemat, 2013). Therefore, the aim of this work was to evaluate the 85 feasibility of using US in LTD of desalted cod, analyzing not only drying and rehydration 86 kinetics but also quality parameters, such as color and texture.

87

### 88 2. Materials and methods

89 2.1. Raw material and sample preparation

90 A homogeneous batch of salted cod (*Gadus morhua*) was provided by a local supplier
91 (Carmen Cambra S. L., Spain). The pieces of salted cod averaged 1.5±0.25 kg.

92 Parallelepiped-shaped samples (50x30x5 mm) were obtained from the central part of 93 the cod loin using a sharp knife and, afterwards, were wrapped in plastic waterproof 94 film and kept refrigerated at 2±1°C (maximum storage time 120 h) until the desalting 95 process took place. For that purpose, the slices of salted cod were immersed in water 96 (70 g cod/L water) of low mineral content (Cortes S.A., Spain) at 4±1°C for 24 h. After 97 desalting, the surface water was removed with tissue paper. Then the samples were 98 wrapped in plastic waterproof film and separated into three batches. Two of them were 99 kept in refrigeration at 2±1°C (maximum storage time 4 h) until the drying experiments 100 were conducted (samples dried at 0 and 10°C). The third (samples dried at -10°C) was 101 frozen by placing samples at -18±1°C until processing (at least 72 h).

102 The moisture and the NaCl content of the cod samples were measured before and 103 after desalting following standard methods 950.46 and 971.27, respectively (AOAC, 104 1997). Thus, the moisture content was obtained by the difference of weighting 105 between salted or desalted cod samples and the same cod samples dried at 105°C 106 until they achieved constant weight (24 h approximately). For the NaCl measurement, 107 approximately 0.5 g of ground sample was placed into 100 mL of distilled water and 108 homogenised at 9500 r.p.m. for 5 min with an ultra-turrax mod. T25 provided with a 109 dispersion tool mod. S25N-18G (IKA Labortechnik, Janke & Kunkel GMBH & Co, 110 Staufen, Germany). The chloride content of the extract was determined in triplicate 111 using a chloride meter (Ciba Corning, mod. 926. L; Halstead, Essex, United Kingdom). 112 Thus, the average value of the moisture content of desalted cod was 4.42±0.02 kg 113 water/kg dry matter of desalted cod (dmdc) and the NaCl content was 0.023±0.001 kg 114 NaCl/kg dmdc.

115

116 2.2. Drying experiments

117 Drying experiments were carried out in a convective drier with air recirculation (Figure 118 1), already described in the literature (Garcia-Perez et al., 2012). The system provides 119 an automatic temperature and air velocity control. Moreover, an ultrasonically activated 120 cylindrical radiator generates a high intensity ultrasonic field (155 dB) in the drying 121 chamber. Drying experiments were conducted using a constant air velocity (2 m/s) at 122 three different temperatures (10, 0 and -10°C), without (AIR) and with (AIR+US, 20.5 123 kW/m<sup>3</sup>) US application. Drying kinetics were obtained by weighing samples at preset 124 times (interval of 15 min) and considering the initial moisture content. In every case, the 125 initial mass load was of 138.7±6.9 g (10 cod slices) and the relative humidity of drying 126 air was maintained below 10±3% during the whole drying process.

The drying experiments were replicated at least three times for each drying condition tested and extended until samples lost  $65\pm3\%$  of the initial weight. After drying, the moisture content of the samples was also measured following standard method 950.46 (AOAC, 1997). Finally, the dried samples were vacuum-sealed and stored in refrigeration (0±1°C; maximum storage time 4 days) until the quality analyses (rehydration, color and texture) were carried out.

133

### 134 2.3. Modeling of drying kinetics

A diffusion model was used to describe the drying kinetics. The mass transport was considered to be one-dimensional due to the fact that sample thickness (5 mm) was 1/6 (30 mm) and 1/10 (50 mm) shorter than the other dimensions. Thus, the approach of considering the samples as infinite slabs can be considered as appropriate (Garau, Simal, Femenia, and Rosselló, 2006). Assuming the effective moisture diffusivity as constant and the solid to be isotropic and homogeneous, the diffusion equation (equation 1) is written as follows:

142 
$$\frac{\partial W_{p}(\mathbf{x},t)}{\partial t} = D_{ed} \left( \frac{\partial^{2} W_{p}(\mathbf{x},t)}{\partial x^{2}} \right)$$
(1)

where  $W_p$  is the local moisture (kg water/kg dmdc), t is the time (s),  $D_{ed}$  is the effective moisture diffusivity (m<sup>2</sup>/s) during drying and x represents the characteristic mass transport direction in the slab geometry (m).

146 In order to solve equation (1), the following assumptions were considered: solid 147 symmetry, uniform initial moisture content and temperature, constant shape during 148 drying and negligible external resistance to mass transfer. The analytical solution of the 149 diffusion equation, expressed in terms of the average moisture content, is shown in 150 equation (2) (Crank, 1975).

151 
$$W(t) = W_{e} + (W_{0} - W_{e}) \left[ 2 \sum_{n=0}^{\infty} \frac{1}{\lambda_{n}^{2} L^{2}} e^{-D_{ed} \lambda_{n}^{2} t} \right]$$
(2)

where,  $\lambda_n$  are the eigenvalues calculated as  $\lambda_n L = (2n + 1)\frac{\pi}{2}$ , W is the average moisture content (kg water/kg dmdc), L the half-thickness of the sample (m) and subscripts 0 and e represent the initial and equilibrium state, respectively.

The diffusion model was fitted to the experimental drying kinetics in order to identify the effective moisture diffusivity. The identification was carried out by minimizing the sum of the squared differences between the experimental and the calculated average moisture content. For that purpose, the Generalized Reduced Gradient (GRG) optimization method, available in Microsoft Excel<sup>™</sup> spreadsheet (Microsoft Corporation, Seattle, WA, USA) was used. The goodness of the fit was determined by calculating the percentage of explained variance (VAR, equation 3).

162 
$$VAR = \left[1 - \frac{S_{xy}^2}{S_y^2}\right] \cdot 100$$
(3)

where S<sub>xy</sub> and S<sub>y</sub> are the standard deviation of the estimation and the sample,
respectively.

## 166 2.4. Rehydration experiments

167 The rehydration capacity was determined by immersing the dried cod samples in 168 distilled water at 25±1°C. In order to obtain the rehydration kinetics, the samples were 169 taken out of the bath at preset times, blotted with tissue paper to remove the surface 170 water and weighed. The rehydration tests were carried out in triplicate for each drying 171 condition considered, using 10 samples (16.5±1.5 g) of dried cod in each run. The 172 experiments were extended until the difference in sample weight between two 173 consecutive measurements (60 min) was lower than 0.5 g, assuming that this point 174 was close to the equilibrium weight. The rehydration kinetics were modeled using the 175 same diffusion model described in section 2.3. for the drying kinetics. In this case,  $W_0$ 176 represents the moisture content of the dried samples and We the equilibrium moisture 177 content of the rehydrated samples and the term Ded was replaced by Der to differentiate 178 the effective moisture diffusivity  $(m^2/s)$  during drying and rehydration.

179

180 2.5. Color

181 The color of both dried and rehydrated cod samples was determined by measuring the 182 CIE L\*a\*b\* color coordinates (Bai, Sun, Xiao, Mujumdar, and Gao, 2013) using a 183 colorimeter (Minolta CM-2500d, Konica Minolta Optics, Inc., Japan), provided with a 184 standard illuminant D65, an observation angle of 10° and calibrated using a standard 185 white. In every case, the measurements were carried out directly on the sample 186 surface, in triplicate and at room temperature (20±1°C). Thus, for each type of dried 187 sample, a minimum of 90 color measurements were carried out. The overall color 188 difference ( $\Delta E$ , equation 4) was computed as the difference between AIR+US (L\*, a\*, 189 b<sup>\*</sup>) and AIR ( $L_0^*$ ,  $a_0^*$ ,  $b_0^*$ ) samples. In the case of the rehydrated samples,  $\Delta E$  indicates

190 the color difference between the rehydrated samples (L\*, a\*, b\*) and the desalted cod 191 before drying ( $L_0^*$ ,  $a_0^*$ ,  $b_0^*$ ).

192 
$$\Delta \mathsf{E} = \sqrt{\left(\mathsf{L}^* - \mathsf{L}^*_0\right)^2 + \left(\mathsf{a}^* - \mathsf{a}^*_0\right)^2 + \left(\mathsf{b}^* - \mathsf{b}^*_0\right)^2} \tag{4}$$

193

### 194 *2.6. Texture*

195 The hardness of both dried and rehydrated cod samples was measured using a 196 Texture Analyzer (TAX-T2, Stable Micro System, Godalming, United Kingdom) with a 197 load cell of 25 kg. The penetration tests were conducted with a 2 mm flat cylinder probe 198 (SMS P/2N), at a crosshead speed of 1 mm/s and a strain of 75% (penetration distance 199 3.5 mm). The hardness was characterized as the maximum penetration force achieved. 200 In each sample, the penetration tests were carried out at 16 points following a preset 201 pattern. For each drying run, at least three dried and three rehydrated samples were 202 analyzed. Because each drying conditions was tested by triplicate, this means that nine 203 dried and nine rehydrated samples was used to assess the hardness in each case.

204

## 205 2.7. Statistical analysis

Analyses of variance (ANOVA) (p<0.05) were carried out and LSD (Least Significant Difference) intervals were estimated using the statistical package, Statgraphics Centurion XVI (Statpoint Technologies Inc., Warrenton, VA, USA), in order to assess the significance of the influence of the different operating conditions (temperature and US application) on the identified  $D_{ed}$  and  $D_{er}$ , as well as on the color and hardness of both the dried and rehydrated samples.

212

## 213 **3. Results and discussion**

### 3.1. Drying experiments

215 The drying kinetics of desalted cod without (AIR) and with (AIR+US) ultrasound 216 application are shown in Figure 2. In both AIR and AIR+US, the lower the drying 217 temperature, the longer the drying time. Thus, in AIR experiments, 69% less time was 218 needed for drying at 10°C (18.1±1.9 h) than at -10°C (57.7±5.9 h). It should be noted 219 that at 0 and 10°C, the water was removed from the solid matrix by evaporation. On the 220 contrary, at -10°C, water removal took place by sublimation due to the fact that the 221 water remains frozen during drying; therefore, under these conditions it could be 222 considered as atmospheric freeze drying (AFD) (Claussen, Ustad, Strommen, and 223 Walde, 2007).

224 The application of US increased the drying rate at every temperature tested (Figure 2). 225 The shortening of the drying time depended on the temperature being higher at -10°C 226 (60%) than at 0 and 10°C (16 and 29%, respectively). As observed in Figure 2, the 227 influence of temperature on AIR+US drying kinetics was less remarkable than in AIR 228 experiments. Thus, the difference in drying time between the AIR experiments carried 229 out at -10 and 10°C was 39.6 h, while this difference was only 10.2 h in the case of 230 AIR+US experiments. Using the same US device, Ozuna et al. (2014b) succeeded in 231 shortening the drying time by between 35 and 54% when US was applied during the 232 drying of salted cod (from -10 to 20°C). In the case of apple drying, and under similar 233 experimental conditions (from -10 to 10°C), Santacatalina et al. (2014) found time 234 savings of between 60 and 77%. Likewise, Garcia-Perez et al. (2012) reported drying 235 time reductions of around 70% in the drying of carrot and eggplant at -14°C. Bantle and 236 Hanssler (2013) reduced the drying time by over 90% when drying salted codfish at 237 10°C using a commercial ultrasonic plate-like emitter (20kHz; DN 20/200, Sonotronic, 238 Karnsbald, Germany), but considering only the initial drying period (until samples 239 reached a moisture content of 45%). Schössler, Jäger, and Knorr (2012) reported that, 240 when freeze-drying red bell pepper cubes using a contact ultrasonic system, the drying 241 time was shortened by 11.5%.

# 243 3.2. Modeling of drying kinetics

244 The proposed model was adequate for describing the drying kinetics of desalted cod 245 slices at 0 and 10°C, obtaining percentages of explained variance (VAR) of over 99% 246 (Table 1). The goodness of the fit at 0 and 10°C is illustrated in Figure 2, where the 247 similar trend of the experimental and calculated moisture content can be observed. On 248 the contrary, a lower VAR value (98.5%) was found in the experiments carried out at -249 10°C, probably because the samples remain frozen during drying. Under these 250 conditions, the water is removed by sublimation and two layers can be found in the 251 product: a frozen inner core and a dry outer layer. Therefore, the product is not 252 homogeneous, as is assumed in the diffusion model.

At the drying temperatures tested, the fit of the diffusion model was poorer when US was applied. This is probably due to the fact that US application partially modifies the mechanisms of mass transport that could affect the relationship between internal and external mass transport resistance, meaning that diffusion was not the only mechanism controlling mass transfer, as assumed in the model.

258 In any case, in the proposed model, any effect on the drying rate was included in the 259 D<sub>ed</sub>. Therefore, this parameter can be used to compare and assess the overall effect of 260 the different conditions tested (temperature and/or US application) on the drying rate. 261 In the case of temperature, the higher the air drying temperature applied, the higher the 262 identified D<sub>ed</sub> (Table 1). The application of US during LTD of desalted cod also involved 263 a significant (p<0.05) increase in the  $D_{ed}$  at the three temperatures studied (Table 1). 264 This influence of US on the identified diffusivities were similar to those reported by 265 Ozuna et al. (2014b) for US-assisted drying kinetics of salted cod at temperatures 266 between 20 and -10°C. The mechanical stress caused by the alternating compressions 267 and expansions (sponge effect) produced by US could improve the internal diffusion of 268 moisture (Gallego-Juárez et al., 2007). Moreover, this stress could create microscopic

channels that help to make the movement of the water towards the product surface
easier (Gallego-Juárez, 2010). At the solid-air interface, US produces alternating
pressures and microstirring that could also help to speed-up the convective moisture
transport.

273 As can be observed in Table 1, the increase in D<sub>ed</sub> produced by US application was 274 significantly (p<0.05) larger in the experiments conducted at -10°C (123.5%) than in 275 those carried out at 0 and 10°C (17.4 and 35.4%, respectively). As stated before, while 276 evaporation was responsible for the water removal at 0 and 10°C, at -10°C it took place 277 through sublimation which can be assumed to be atmospheric freeze drying. This 278 makes the outer porous dry layer developed during this kind of drying more prone to 279 the ultrasonic effects than the more compact structure developed during drying by 280 evaporation at 0 and 10°C. In this sense, Ozuna, Gómez, Riera, Cárcel, and Garcia-281 Perez, (2014c) observed that the product porosity influences the extension of the 282 ultrasound effects during drying. Thus, highly porous materials present higher values of 283 impedance, closer to the surrounding air, than materials with a hard and closed-284 compact structure. The fact that the coupling of the air-porous structure is better makes 285 easier the ultrasound transmission and helps ultrasound effects to be more intense 286 (Ozuna et al., 2014c).

287

## 288 3.3. Rehydration experiments

Since rehydration potential is an important quality attribute for products that need to be reconstituted before consumption, the influence of the drying method on the rehydration kinetics of dried samples ( $0.54\pm0.07$  kg water/kg dmdc) was experimentally determined. The results obtained showed that the AIR samples dried at -10°C rehydrated significantly (p<0.05) faster than those dried at 10 and 0°C (Figure 3). Thereby, the average rehydration time for AIR samples dried at 0 and 10°C was 22.0±0.9 h, while for AIR samples dried at -10°C it was 8.7±0.3 h. The moisture content

reached at the end of the rehydration process was also significantly (p<0.05) higher for samples dried at -10°C (3.05±0.44 kg water/kg dmdc) than for those dried at 10 and 0°C (2.61±0.24 and 2.41±0.10 kg water/kg dmdc, respectively). These results could be explained by the fact that drying at -10°C leads to a minimum shrinkage and a highly porous structure (Stawczyk, Li, Witrowa-Rajchert, and Fabisiak, 2007). So, the high porosity makes it easier for water to enter the dried matrix.

302 The application of US during drying did not significantly (p<0.05) affect the moisture 303 gain rate during the rehydration of samples dried at 0 and 10°C (Figure 3). On the 304 contrary, for samples dried at -10°C, US application slightly reduced the rehydration 305 rate and the final moisture gain. US could affect the microstructure of cod samples, 306 provoking ruptures in the cod fibers and causing the formation of wider spaces 307 between myofibrils (Ozuna et al., 2014c). The extension of these effects were enough 308 to modify the rehydration capacity of cod slices dried at -10°C, but not for those dried at 309 0 and 10°C. It is likely that this is due to the combined impact of freezing and US 310 application on the structure of the samples dried at -10°C, which results in a softer and 311 more unstructured matrix where the water intake and its retention during the 312 rehydration process is more difficult. In this sense, Nowacka, Wiktor, Śledź, Jurek, and 313 Witrowa-Rajchert (2012) reported that a short ultrasonic pretreatment of apple cubes 314 before drying reduced their moisture content after 60 minutes of rehydration due to 315 changes in the product's microstructure. However, Schössler et al. (2012) reported no 316 differences in the rehydration characteristics of the US assisted freeze-dried red bell 317 pepper in comparison with those conventionally freeze-dried, probably due to the lower 318 efficiency of the contact ultrasonic system used in this work.

The experimental rehydration kinetics were also modeled, taking Equation 2 into account. A satisfactory description (VAR>98%) (Table 2) of the rehydration kinetics was only obtained for AIR dried samples at 0 and 10°C (Figure 3). In samples dried at -

322 10°C, mechanisms other than diffusion, and probably linked to the high porosity and323 bulk water input, could be involved.

The D<sub>er</sub> identified for samples dried at 0 and 10°C was similar (Table 2). At these temperatures, US application during drying caused a slight but not significant (p<0.05) increase in the D<sub>er</sub>. However, the D<sub>er</sub> identified for AIR samples dried at -10°C was five times greater than that identified for those dried at 0 and 10°C. In this case, AIR+US samples dried at -10°C showed a significantly (p<0.05) lower D<sub>er</sub> than AIR samples.

329

330 3.4. Color

### 331 3.4.1. Dried samples

332 The average values of the color coordinates of desalted cod before drying were 333 63.7±1.9 for L\*, -3.85±0.52 for a\* and 0.95±0.78 for b\*. In general terms, as can be 334 observed in Table 3, the drying increased the value of the three coordinates. AIR dried 335 samples at 0 and 10°C showed higher a\* and b\* coordinates and lower L\* than those 336 dried at -10°C. These results suggest that the moisture removal by evaporation (0 and 337 10°C) caused a yellowing (higher b\*) and a darkening (lower L\*), while the removal by 338 sublimation (-10°C) leads to brighter and whiter samples. According to Asli and 339 Morkore (2012), cod should preferably have a high lightness value (L\*-value), as the 340 color white is considered positive by consumers. Bjorkevoll, Reboredo, and Fossen 341 (2014) also associated high quality with a whiter and less yellow surface in the sensory 342 evaluation of the heavy salted cod. However, Lauritzsen et al. (2004) reported that the 343 reduction in the water content causes changes in the color of the fish. Brás and Costa 344 (2010) reported an increase in both the lightness (L\*) and yellowness (b\*) of the cod 345 caused by drying, which was more marked as drying progressed.

346 The application of US during drying only significantly (p<0.05) affected the L\* 347 coordinate for samples dried at -10°C, reducing their lightness as compared with AIR

348 samples. Moreover, the overall color difference ( $\Delta E$ ) between samples dried without 349 (AIR) and with (AIR+US) (Table 3) showed negligible differences in the case of 350 samples dried at 0 and 10°C due to the fact that, as reported by Francis and 351 Clydesdale (1975),  $\Delta E$  values lower than 2 are not detected by the human eye. On the 352 contrary, the  $\Delta E$  obtained for the samples dried at -10°C was significantly (p<0.05) 353 higher; this indicated that, at this drying temperature, US application caused a 354 meaningful color difference. This was probably due to the slight thermal effect 355 generated by US on the sample's surface, which could be more marked in this case 356 and bring about a little darkening.

357

## 358 3.4.2. Rehydrated samples

The drying temperature did not affect the color of the rehydrated samples (Table 4) and no significant (p<0.05) differences were found for samples previously dried at -10, 0 or 10°C. In a similar way, US application during drying did not cause noticeable changes in the color coordinates of the rehydrated samples (Table 4).

363 In this case,  $\Delta E$  was calculated by considering the desalted cod prior to drying as 364 reference. In general terms, the dried and rehydrated cod samples did not recover the 365 color of the desalted samples, the  $\Delta E$  ranging from 8.8 to 11.7 (Table 4).

366

367 3.5. Texture

### 368 3.4.1. Dried samples

The initial hardness of the desalted cod was 1.55±0.53 N. Therefore, the drying process provoked a hardening of the samples (Figure 4), regardless of the drying conditions used. For the AIR samples, the hardness was dependent on the drying temperature (Figure 4), so, the lower the air temperature, the harder the dried cod sample. No influence of the air drying temperature on the hardness was observed in

374 the case of AIR+US samples. However, when comparing ultrasonically assisted dried 375 samples with those conventionally dried, it was observed that the AIR+US samples 376 dried at 0 and 10°C were significantly (p<0.05) harder than the AIR ones (Figure 4). 377 This fact could be attributed to the successive compression and expansion cycles of 378 the material produced by US, which could affect cod proteins thus causing a hardening 379 of the samples. At -10°C, the hardening of AIR and AIR+US samples was similar. The 380 previous freezing step and the fact that water removal occurred by sublimation could 381 provoke changes in the sample's structure that may mask the structural US effect.

382

### 383 3.4.2. Rehydrated samples

384 In general, both the drying and the later rehydration process produced samples that 385 were slightly harder than the initial desalted cod (1.55±0.53 N), but their final hardness 386 depended on the drying temperature and US application. Thus, in the case of AIR 387 samples, the effect of drying temperature on rehydrated samples was to the opposite of 388 that found in dried samples; so, the higher the air temperature, the harder the 389 rehydrated sample (Figure 5). As regards the application of US, the rehydrated 390 AIR+US samples dried at 0 were significantly (p<0.05) harder than AIR samples 391 (Figure 5). The textural changes caused by US application could be linked to the 392 denaturation of proteins (Lee and Feng, 2011). In the case of samples dried at 10°C, 393 no significant (p<0.05) differences were observed between AIR and AIR+US, which 394 could be explained by the fact that the shorter drying time at 10°C prevented a lengthy 395 action of US on the internal structure. In the case of -10°C experiments, the effects of 396 freezing on structure can mask the effects of ultrasound.

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398

399

## 400 **4. Conclusions**

401 The application of US during the low temperature drying of desalted cod improved the 402 drying rate at every temperature tested, but it was particularly noticeable when drying 403 took place at -10°C, which is when water removal took place by sublimation. As far as 404 quality attributes are concerned, the cod dried at -10°C rehydrated faster and gained 405 more water than that dried at higher temperatures. Moreover, these samples were 406 brighter, whiter and slightly softer than those dried at 0 and 10°C. US application 407 slightly reduced the rehydration rate and increased the sample's hardness, but allowed 408 whiter samples to be obtained, which are usually preferred by consumers. Therefore, 409 power ultrasound could be considered an interesting technology to speed-up the low 410 temperature drying of desalted cod without greatly affecting the guality of the obtained 411 product.

412

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419

## 420 References

- 421 Andrés, A., Rodríguez-Barona, S., & Barat, J.M. (2005). Analysis of some cod-422 desalting process variables. *Journal of Food Engineering, 70,* 67-72.
- Asli, M., & Morkore, T. (2012). Brines added sodium bicarbonate improve liquid
  retention and sensory attributes of lightly salted Atlantic cod. *LWT-Food Science and Technology, 46,* 196-202.

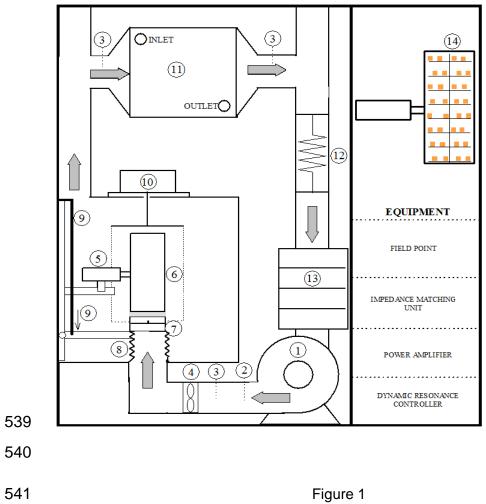
- 426 Association of Official Analytical Chemists (AOAC) (1997). *Official methods of analysis*.
  427 Association of Official Analytical Chemists, Arlington, Virginia, USA.
- Bai, J.W., Sun, D.W., Xiao, H.W., Mujumdar, A.S., & Gao, Z.J. (2013). Novel highhumidity hot air impingement blanching (HHAIB) pretreatment enhances drying
  kinetics and color attributes of seedless grapes. *Innovative Food Science* & *Emerging Technologies*, 20, 230-237.
- Bantle, M., & Eikevik, T.M. (2011). Parametric study of high intensity ultrasound in the
  atmospheric freeze drying of peas. *Drying Technology*, *29*, 1230-1239.
- Bantle, M., & Hanssler, J. (2013). Ultrasonic convective drying kinetics of clipfish during
  the initial drying period. *Drying Technology*, *31*, 1307-1316.
- Bjorkevoll, I., Reboredo, R.G., & Fossen, I. (2014). Methods for phosphate addition in
  heavy salted cod (*Gadus morhua* L.). *LWT-Food Science and Technology, 58,*502-510.
- Brás, A., & Costa, R. (2010). Influence of brine salting prior to pickle salting in the
  manufacturing of various salted-dried fish species. *Journal of Food Engineering,*100, 490-495.
- 442 Cárcel, J.A., Garcia-Perez, J.V., Riera, E., & Mulet, A. (2011). Improvement of
  443 convective drying of carrot by applying power ultrasound-Influence of mass load
  444 density. *Drying Technology, 29*, 174-182.
- Claussen, I.C., Ustad, T.S., Strømmen, I., & Walde, P.M. (2007). Atmospheric freeze
  drying A review. *Drying Technology*, 25, 957-967.
- 447 Crank, J. (1975). *The Mathematics of Diffusion*. Oxford University Press, London, UK.
- Fernández-Segovia, I., Escriche, I., Fuentes, A., & Serra, J.A. (2007). Microbial and
  sensory changes during refrigerated storage of desalted cod (*Gadus morhua*)
  preserved by combined methods. *International Journal of Food Microbiology*, *116*, 64-72.

- 452 Francis, F.J., & Clydesdale, F.M. (1975). *Food colorimetry: Theory and applications*.
  453 AVI Publishing Co. Inc., New York, USA.
- Gallego-Juárez, J.A., Riera, E., de la Fuente Blanco, S., Rodríguez-Corral, G., AcostaAparicio, V.M., & Blanco, A. (2007). Application of high-power ultrasound for
  dehydration of vegetables: processes and devices. *Drying Technology, 25*,
  1893-1901.
- 458 Gallego-Juárez, J.A. (2010). High-power ultrasonic processing: Recent developments
  459 and prospective advances. *Physics Procedia*, *3*, 35-47.
- Garau, M.C., Simal, S., Femenia, A., & Rosselló, C. (2006). Drying of orange skin:
  drying kinetics modelling and functional properties. *Journal of Food Engineering*, 75, 288-295.
- Garcia-Perez, J.V., Ozuna, C., Ortuño, C., Cárcel, J.A., & Mulet, A. (2011). Modeling
  ultrasonically assisted convective drying of eggplant. *Drying Technology, 29*,
  1499-1509.
- 466 Garcia-Perez, J.V., Cárcel, J.A., Riera, E., Rosselló, C., & Mulet, A. (2012).
  467 Intensification of low temperature drying by using ultrasound. *Drying*468 *Technology*, *30*, 1199-1208.
- Lauritzsen, K., Akse, L., Johansen, A., Joensen, S., Sørensen, N.K., & Olsen, R.L.
  (2004). Physical and quality attributes of salted cod (*Gadus morhua* L.) as
  affected by the state of rigor and freezing prior to salting. *Food Research International, 37*, 677-688.
- 473 Lee, H., & Feng, H. (2011). Effect of Power Ultrasound on Food Quality. In: *Ultrasound*474 *Technologies for Food and Bioprocessing,* Feng, H., Barbosa-Cánovas, G.V., &
  475 Weiss, J., Eds., Springer, New York, 559-582.

- 476 Martínez-Álvarez, O., & Gómez-Guillén, C. (2013). Influence of mono- and divalent
  477 salts on water loss and properties of dry salted cod fillets. *LWT-Food Science*478 and Technology, 53, 387-394.
- Nowacka, M., Wiktor, A., Śledź, M., Jurek, N., & Witrowa-Rajchert, D. (2012). Drying of
  ultrasound pretreated apple and its selected physical properties. *Journal of Food Engineering*, *113*, 427-433.
- 482 Oliveira, H., Pedro, S., Nunes, M.L., Costa, R., & Vaz-Pires, P. (2012). Processing of
  483 salted cod (*Gadus* spp.): A review. *Comprehensive Reviews in Food Science*484 and Food Safety, 11, 546-564.
- 485 Ozuna, C., Puig, A., Garcia-Perez, J.V. & Cárcel, J.A. (2014a). Ultrasonically enhanced
  486 desalting of cod (*Gadus morhua*). Mass transport kinetics and structural
  487 changes. *LWT-Food Science and Technology*, 59, 130-137.
- 488 Ozuna, C., Cárcel, J.A., Walde, P.M. & Garcia-Perez, J.V. (2014b). Low-temperature
  489 drying of salted cod (*Gadus morhua*) assisted by high power ultrasound:
  490 Kinetics and physical properties. *Innovative Food Science and Emerging*491 *Technologies, 23*, 146-155.
- 492 Ozuna, C., Gómez, T., Riera, E., Cárcel, J.A., & Garcia-Perez, J.V. (2014c). Influence
  493 of material structure on air-borne ultrasonic application in drying. *Ultrasonics*494 *Sonochemistry, 21*, 1235-1243.
- 495 Pingret, D., Fabiano-Tixier, A.S., & Chemat, F. (2013). Degradation during application
  496 of ultrasound in food processing: A review. *Food Control, 31,* 593-606.
- 497 Santacatalina, J.V., Rodríguez, O., Simal, S., Cárcel, J.A., Mulet, A., Garcia-Perez, J.V.
  498 (2014). Ultrasonically enhanced low-temperature drying of apple: Influence on
  499 drying kinetics and antioxidant potential. *Journal of Food Engineering, 138*, 35500 44.

- Schössler, K., Jäger, H., & Knorr, D. (2012). Novel contact ultrasound system for the
  accelerated freeze-drying of vegetables. *Innovative Food Science and Emerging Technologies, 16,* 113-120.
- Soria, A.C., Corzo-Martínez, M., Montilla, A., Riera, E., Gamboa-Santos, J., & Villamiel,
  M. (2010). Chemical and physicochemical quality parameters in carrots
  dehydrated by power ultrasound. *Journal of Agricultural and Food Chemistry*,
  507 58, 7715-7722.
- 508 Stawczyk, J., Li, S., Witrowa-Rajchert, D., & Fabisiak, A. (2007). Kinetics of 509 atmospheric freeze-drying of apple. *Transport in porous media, 66,* 159-172.

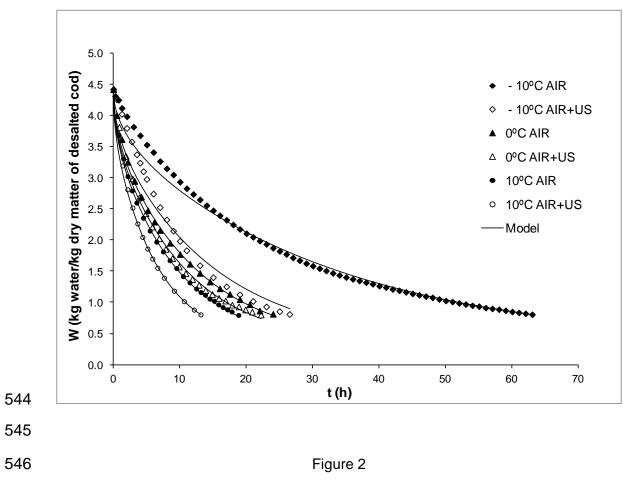
511	Figure captions
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513 514 515 516 517 518 519	<b>Figure 1.</b> Diagram of the ultrasonically assisted convective dryer: 1, fan; 2, Pt-100; 3, temperature and relative humidity sensor; 4, anemometer; 5, ultrasonic transducer; 6, vibrating cylinder; 7, sample load device; 8, retreating pipe; 9, slide actuator; 10, weighing module; 11, heat exchanger; 12, heating elements; 13, desiccant tray chamber; 14, details of the sample load on the trays.
520	Figure 2. Experimental and calculated (diffusion model) drying kinetics (2 m/s) of
521	desalted cod at different temperatures (-10, 0 and 10°C), without (AIR) and with
522	(AIR+US, 20.5 kW/m <sup>3</sup> , 21 kHz) ultrasound application.
523	
524	Figure 3. Experimental and calculated (diffusion model) rehydration kinetics of
525	desalted and dried cod (2 m/s) at different temperatures (-10, 0 and 10°C), without
526	(AIR) and with (AIR+US, 20.5 kW/m <sup>3</sup> , 21 kHz) ultrasound application.
527	
528	Figure 4. Hardness of desalted and dried cod at different temperatures (-10, 0 and
529	10°C), without (AIR) and with (AIR+US, 20.5 kW/m <sup>3</sup> , 21 kHz) ultrasound application.
530	Average values $\pm$ LSD intervals (p<0.05) are plotted. Different letters show significant
531	differences according to LSD intervals (p<0.05).
532	
533	Figure 5. Hardness of rehydrated cod previously desalted and dried at different
534	temperatures (-10, 0 and 10°C), without (AIR) and with (AIR+US, 20.5 kW/m <sup>3</sup> , 21 kHz)
535	ultrasound application. Average values ± LSD intervals (p<0.05) are plotted. Different
536	letters show significant differences according to LSD intervals (p<0.05).
537 538	

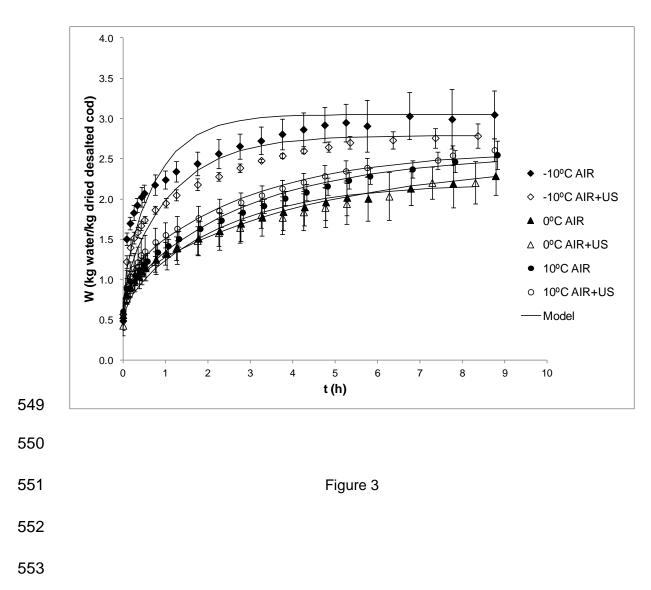






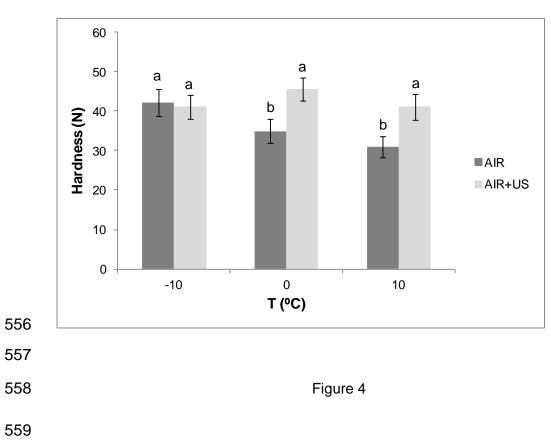












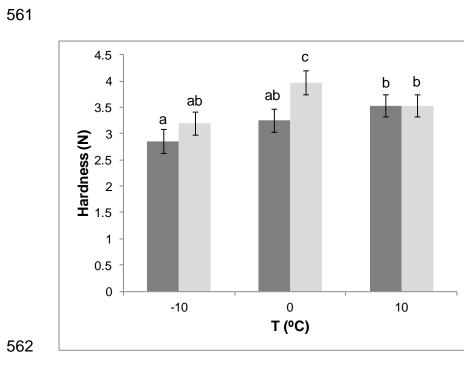




Figure 5

■AIR AIR+US

567

Table 1. Effective moisture diffusivity ( $D_{ed}$ ) for the drying kinetics of desalted cod at different temperatures (10, 0 and -10°C), without (AIR) and with (AIR+US, 20.5 kW/m<sup>3</sup>, 21 kHz) ultrasound application. Average values ± standard deviation are shown. VAR (%) is the percentage of explained variance.  $\Delta D_{ed}$  shows (in percentage) the increase in effective moisture diffusivity produced by ultrasonic application.

	AIR		AIR+US		
T (ºC)	D <sub>ed</sub> (10 <sup>-11</sup> m <sup>2</sup> /s )	VAR (%)	D <sub>ed</sub> (10 <sup>-11</sup> m <sup>2</sup> /s )	VAR (%)	$\Delta D_{ed}$ (%)
10	7.77±0.62 <sup>e</sup>	99.7	10.51±0.33 <sup>f</sup>	99.1	35.4
0	5.65±0.35°	99.3	6.63±0.09 <sup>d</sup>	99.2	17.4
-10	2.17±0.20ª	98.5	4.84±0.60 <sup>b</sup>	94.4	123.5

573 Superscript letters (a, b, c, d, e, f) show homogeneous groups established from LSD (Least Significance Difference) intervals (p< 0.05).

575 576

Table 2. Effective moisture diffusivity ( $D_{er}$ ) for the rehydration kinetics of desalted cod dried at different temperatures (10, 0 and -10°C), without (AIR) and with (AIR+US, 20.5 kW/m<sup>3</sup>, 21 kHz) ultrasound application. Average values ± standard deviation are shown. VAR (%) is the percentage of explained variance.

	AIR		AIR+US		
T (ºC)	D <sub>er</sub> (10 <sup>-10</sup> m <sup>2</sup> /s )	VAR (%)	D <sub>er</sub> (10 <sup>-10</sup> m <sup>2</sup> /s )	VAR (%)	
10	1.99±0.66 <sup>a,b</sup>	99.1	2.26±0.87 <sup>a,b</sup>	97.5	
0	1.89±0.52ª	98.3	2.34±0.58 <sup>a,b</sup>	95.5	
-10	9.93±4.35°	86.7	5.60±2.17 <sup>b</sup>	94.3	

Superscript letters (a, b, c) show homogeneous groups established from LSD (Least Significance Difference) intervals (p< 0.05).

Table 3. CIELab (L\*, a\*, b\*) color coordinates for desalted cod dried at different temperatures (10, 0 and -10°C), without (AIR) and with (AIR+US, 20.5 kW/m<sup>3</sup>, 21 kHz) ultrasound application. Average values  $\pm$  standard deviation are shown.  $\Delta$ E represents the overall color differences between AIR+US and AIR samples.

		-10ºC	0°C	10⁰C
L*	AIR	79.9±4.4ª	57.9±4.1°	54.1±2.3 <sup>c,d</sup>
Ľ	AIR+US	67.0±4.3 <sup>b</sup>	55.3±4.2 <sup>c,d</sup>	55.9±5.0 <sup>d</sup>
a*	AIR	-1.2±0.4 <sup>m</sup>	-1.0±1.3 <sup>m</sup>	-0.1±1.2 <sup>n</sup>
ď	AIR+US	-0.9±0.5 <sup>m</sup>	-1.0±0.8 <sup>m</sup>	-1.0±1.0 <sup>m</sup>
<b>b</b> *	AIR	13.6±3.0 <sup>x</sup>	16.1±3.0 <sup>y</sup>	16.1±2.9 <sup>y</sup>
b*	AIR+US	12.6±2.6 <sup>x</sup>	15.8±2.6 <sup>y</sup>	15.6±3.0 <sup>y</sup>
$\Delta \mathbf{E}$	AIR+US vs AIR	12.9	2.6	2.1

Superscript letters (a, b, c, d), (m, n) and (x, y) show homogeneous groups, established from LSD (Least Significance Difference) intervals (p<0.05) for L\*, a\* and b\*, respectively.