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1 **Ultrasound-assisted drying of orange peel in atmospheric freeze-dryer**  
2 **and convective dryer operated at moderate temperature**

3  
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23

24 **Abstract:** Atmospheric freeze-drying (AFD) at -10 °C and moderate temperature  
25 convective drying (MTD) at 50 °C without and with ultrasound application (20.5kW/m<sup>3</sup>)  
26 were carried out. Alcohol insoluble residue (AIR) and its swelling capacity (SC), water  
27 retention capacity (WRC) and fat retention capacity (FRC) were measured in the dried  
28 product. Ultrasound significantly shortened the drying time in both processes, the  
29 intensification effect being more significant in atmospheric freeze-drying (57 % and 27  
30 % reduction in atmospheric freeze-drying and convective drying, respectively). As  
31 regards AIR and WRC, no effect was observed of either the drying temperature or  
32 ultrasound application. On the contrary, SC was significantly lower in AFD samples. The  
33 FRC of MTD samples was similar to that of the fresh ones and higher than the values  
34 obtained for atmospheric freeze-dried samples. Therefore, convective drying at moderate  
35 temperature preserved the AIR properties better than atmospheric freeze-drying.

36

37 **Keywords:** By-product; process intensification; fiber; alcohol insoluble residue

38

## 39 1. INTRODUCTION

40 Citrus fruits are among the most-heavily harvested fruits in the world. Most of the  
41 production is destined for to the juice industry, where approximately 50% of the total fruit  
42 weight is discarded, generating a large amount of waste. These residues, mainly peels,  
43 can be used as a source of valuable bioactive compounds [1] with commercial and  
44 technological applications, such as dietary fiber [2, 3] used to fortify food products. In  
45 this sense, citrus peels have a high moisture content which made them very susceptible  
46 to the degradation reactions. Drying processes represent an optional means of producing  
47 a stable and high quality by-product, which becomes raw matter for later processing with  
48 convective drying, at high (HTD) or moderate temperatures (MTD), being the most  
49 commonly-used conventional technique. However, this operation may induce undesirable  
50 structural damage, color alterations and content reduction of nutritional compounds [4,  
51 5]. As a result, there is growing interest in applying alternative techniques, which imply  
52 higher quality products, such as atmospheric freeze-drying (AFD) [5]. AFD consists of  
53 water removal by sublimation at atmospheric pressure using drying air at low temperature  
54 and relative humidity, keeping the product frozen while being dried [5, 6]. This process  
55 is dependent on the air drying characteristics (temperature, velocity and relative humidity)  
56 and the food properties (dimensions, porosity, initial moisture content, etc) [7]. AFD can  
57 be used to dry different foods, obtaining high quality dried products [6, 8].

58 Both elevated air temperatures (HTD and MTD) and long processing times (AFD)  
59 can cause quality loss in foods, affecting, for example, the properties of the fiber. In order  
60 to reduce these impacts caused by convective drying processes, combined techniques,  
61 such as the application of power ultrasound (US), may be considered. US can induce a  
62 reduction in the external and internal mass transfer resistance with only a mild thermal  
63 effect [5]. In a solid porous product, US causes a series of rapid compressions and

64 expansions (sponge effect) facilitating the exit of water through the microchannels  
65 created by the propagation of the waves [6, 8]. The influence of US application has been  
66 addressed in order to shorten the processing time of fruits and vegetables [10-12].

67 Therefore, the main objective of this study was to address the influence of process  
68 characteristics, atmospheric freeze-drying (AFD) and convective drying at moderate  
69 temperature (MTD), and power ultrasound application on the drying kinetics and  
70 functional properties of orange peel.

71

## 72 **2. MATERIAL AND METHODS**

### 73 **2.1. Raw Material**

74 Valencia Late var. oranges (*Citrus sinensis*) were purchased in a local market  
75 (Valencia, Spain). Homogeneity of size and color was the criterion considered when  
76 choosing the fruits. The oranges were washed and superficially dried. Rectangular shell  
77 samples (containing only flavedo and albedo tissue) of  $48\pm 1 \times 26\pm 1 \times 3.18\pm 0.04$  mm  
78 were obtained using sharp knives. The initial moisture content was measured by placing  
79 the samples in a vacuum oven at 70 °C and 200 mmHg until constant weight [13].

80

### 81 **2.2. Drying experiments**

82 Two convective drying techniques were examined, AFD (water removal by  
83 sublimation) and MTD (water removal by evaporation), and the influence of ultrasound  
84 application was addressed in both cases. Every kind of drying condition considered was  
85 tested in triplicate.

86

87

88

### 89 **2.2.1. Atmospheric freeze-drying experiments**

90 Before the AFD process, 18 orange peel samples were placed in a tree-shaped  
91 sample holder, previously described [5, 6], that ensured free-flowing air around them and  
92 a homogenous ultrasonic treatment. The set was covered with a plastic waterproof film  
93 and placed in a blast freezer (HIBER, model ABBBBF051, Italy) at  $-35\pm 1$  °C for 1 h. This  
94 was long enough to reach a temperature of  $-18$  °C in the center of the samples.  
95 Immediately after this, the samples were unwrapped and transferred to an ultrasound-  
96 assisted convective dryer with air recirculation adapted to work at low temperatures [6].  
97 The drying chamber is a cylinder (internal diameter 100 mm, height 310 mm, thickness  
98 10 mm) attached to a piezoelectric transducer (21.9 kHz) that produces an internal high  
99 intensity ultrasonic field. The drying air is recirculated in the system, controlling both the  
100 air velocity and temperature by means of two PID control algorithms. The drying  
101 experiments were performed at  $-10\pm 1$ °C and 1 m/s, without (AFD) and with (AFD-US;  
102  $20.5$  kW/m<sup>3</sup>) ultrasound application. In order to keep the relative humidity low (maximum  
103 value of 15%, measured with a KDK sensor, Galltec+Mela, Germany), the air is forced  
104 to flow through a tray containing desiccant material (Activated Alumina AC14,  
105 Alfphachem, Spain) which is periodically regenerated. The drying kinetics were  
106 determined from the initial moisture content of orange peel samples and the variation in  
107 sample weight during the process. The experiments were performed until the samples lost  
108 60% of their initial weight.

109

### 110 **2.2.2. Convective drying experiments at moderate temperature**

111 Fresh orange peel samples (18 pieces) were placed in a similar sample holder to  
112 that used in AFD experiments. MTD experiments were performed at a temperature of 50  
113 °C and an air velocity of 1m/s, without (MTD) and with (MTD-US;  $20.5$  kW/m<sup>3</sup>)

114 ultrasound application until the samples lost 60% of their initial weight. The  
115 ultrasonically-assisted dryer used for this purpose has been described previously [14].  
116 The characteristics of the drying chamber and the ultrasonic field applied were similar to  
117 those tested for AFD experiments.

118

### 119 **2.3. Modeling of drying kinetics**

120 The modeling of the experimental data permits the comparison and quantification  
121 of the influence of the process variables on the kinetics; the theoretical models, like the  
122 diffusion-based models, are the most adequate for this purpose because they permit  
123 insight to be gained from the phenomena involved in the drying. However, the different  
124 mechanisms of moisture removal involved in the experiments considered, evaporation in  
125 MTD experiments and sublimation in AFD ones, makes the application of this kind of  
126 model difficult. Moreover, while the moisture movement inside the MTD samples can be  
127 assumed to be due to diffusion in the overall volume, in the AFD samples it only takes  
128 place in the external dried layer whose thickness increases at the expense of the internal  
129 frozen core. Since the main aim of this study was not the development of a model but  
130 rather the quantification of the influence of process variables on drying kinetics, the  
131 empirical Weibull model, a model widely used in drying [15], was considered (Equation  
132 1)

$$133 \quad \Psi = \frac{X_{eq} - X_t}{X_{eq} - X_0} = e^{\left(-\frac{t}{\beta}\right)^\alpha} \quad (1)$$

134 Where  $\Psi$  is the dimensionless moisture content;  $X_t$  is the moisture content (kg  
135 water/kg dm) at a drying time  $t$  (s);  $X_0$  is the initial moisture content of samples (kg  
136 water/kg dm);  $X_{eq}$  is the moisture content at equilibrium, which was determined from the  
137 relative humidity of the drying air and the orange peel isotherm reported by Garau et al.  
138 [16]; and  $\alpha$  and  $\beta$  are the parameters of the Weibull model. The parameter  $\alpha$  is the shape

139 factor and represents a behavior index of the product: the higher its value, the lower the  
140 initial velocity of the process. Values of more than 1 predict downtimes in the process  
141 and when the value is 1, the Weibull model becomes a first order kinetic model.  $\beta$ , on the  
142 other hand, is related with the kinetics of the process, showing a reverse relationship with  
143 the drying rate. This parameter includes the effects on the kinetics of variables, such as  
144 the temperature, air velocity or, in this case, ultrasound application.

145 The Weibull parameters were identified by minimizing the sum of squared  
146 difference between the experimental and calculated moisture contents of the samples. For  
147 this purpose, the SOLVER tool of Microsoft Excel (Excel from Microsoft Office  
148 Professional Plus 2016 TM) was used to apply the optimizing method of the Generalized  
149 Reduced Gradient.

150 The percentage of explained variance (% VAR) was used to evaluate the fit of the  
151 model, following Equation (2).

$$152 \quad VAR = \left[ 1 - \frac{S_{calc}^2}{S_{ex}^2} \right] \cdot 100 \quad (2)$$

153 Where  $S_{calc}^2$  and  $S_{ex}^2$  are the calculated and experimental variances, respectively  
154 [5].

155

#### 156 **2.4. Alcohol insoluble residue (AIR)**

157 In order to evaluate the product's functional properties, the alcohol insoluble  
158 residues (AIR) were obtained according to Garau et al. [17], with some adaptations. For  
159 this purpose, 1.5 g of the ground dried sample (5 g in the case of the fresh sample) were  
160 placed in an ethanol-water solution (85% v/v) and homogenized with an ultraturrax (mod.  
161 T25, dispersion tool S25N-18 G; IKA Labortechnik). After a boiling-cooling cycle, the  
162 sample was filtered. These steps were repeated twice with 85 and 96% v/v ethanol-water  
163 solutions. The residue contained in the filter was washed with acetone (99% v/v) and kept

164 in a vacuum oven at 60 °C for moisture removal. The AIR was expressed as g AIR/100 g  
165 dm.

166

## 167 **2.5. Functional Properties of AIR**

168 The swelling capacity (SC), water retention capacity (WRC) and fat retention  
169 capacity (FRC) were determined for the purposes of addressing the influence of the kind  
170 of water removal mechanism (evaporation or sublimation) and ultrasound application  
171 during drying on the quality of the dried orange peel. All of the determinations were  
172 carried out in triplicate, at least.

173

### 174 **2.5.1. Swelling capacity (SC)**

175 The SC was measured according to Daou and Zhang [18], but adapted to the  
176 product. To this end, 0.2 g of AIR were placed in a graduated test tube, 10 mL of distilled  
177 water were added and the tubes were left to stand for 24 h at room temperature (25±1 °C).  
178 The SC was calculated from the difference between the final and initial volumes of the  
179 sample and expressed as mL /g of AIR dm.

180

### 181 **2.5.2. Water retention capacity (WRC)**

182 The WRC was measured according to Garau et al. [17]. For this purpose, the AIR  
183 samples (0.2 g) were hydrated in 10 mL of distilled water for 24 h in centrifuge tubes.  
184 Afterwards, the samples were centrifuged (Medifriger BL-S, Selecta, Spain) at 10,000  
185 r.p.m. for 15 min at 25 °C. The excess supernatant was decanted and the WRC expressed  
186 as g water/g of AIR dm.

187

188

189 **2.5.3. Fat retention capacity (FRC)**

190 The AIR samples (0.2 g) were immersed in 10 mL of sunflower oil for 24 h at  
191 room temperature ( $25\pm 1$  °C) and then centrifuged (Medifriger BL-S, Selecta, Spain) at  
192 6,000 r.p.m. for 15 min at 25 °C, according to Garau et al. [17]. The FRC was expressed  
193 as g oil/g of AIR dm.

194

195 **2.6. Statistical analysis**

196 For the statistical analysis,  $\alpha$ ,  $\beta$ , AIR, SC, WRC, and FRC were considered as  
197 dependent variables and the drying process (AFD or MTD) and the application of  
198 ultrasound as factors. The analysis of variance (one way ANOVA) was calculated using  
199 Statgraphics Centurion XVI (StatPoint Technologies, Inc), to check the significance  
200 ( $p<0.05$ ) of the differences between the values of each dependent variable. The Least  
201 Significant Difference (LSD) intervals were also estimated to determine the significance  
202 of the differences between treatments. Moreover, the values from the replicates of the  
203 different kinds of experiments carried out were averaged and represented as mean and  
204 standard deviation.

205

206 **3. RESULTS AND DISCUSSION**

207 **3.1. Experimental drying kinetics**

208 The initial moisture content of the orange peel was  $2.47\pm 0.08$  (kg water/kg dm),  
209 similar to that found by Tasirin et al. [2]. The air drying temperature and ultrasound  
210 application influenced the length of the drying process (Figure 1). The average time  
211 required to reach a moisture content of 0.5 kg of water/kg of dm in the MTD experiment  
212 ( $3.8\pm 0.3$  h) was 95% shorter than that needed in the AFD experiment ( $93\pm 18$  h). The  
213 increase in the process temperature and the liquid state of water promote a higher heat

214 transfer rate between the heat source, which is the drying air, and the product, leading to  
215 faster moisture removal [19]. During low temperature drying, there is less energy  
216 available to promote moisture loss through the sublimation process and transport the  
217 moisture from the product to the surface. However, losses in nutritional and technological  
218 properties can occur as the drying temperature rises [11, 20].

219         Ultrasound application led to an intensification of the drying process, promoting  
220 a significantly shorter drying time in every condition analyzed ( $p < 0.05$ ) (Figure 1). Thus,  
221 in the AFD-US experiment, the processing time required to reach 0.5 kg of water/kg dm  
222 ( $40 \pm 6$  h) was 57% shorter than in the AFD ( $93 \pm 18$  h). In the case of the MTD  
223 experiments, the application of ultrasound meant that by 23% shorter drying time was  
224 required to attain the same moisture content ( $3.0 \pm 0.4$  h for MTD-US vs.  $3.8 \pm 0.3$  h for  
225 MTD). The most significant effect of US application occurred at low temperatures. This  
226 can be explained by the fact that the mechanical energy generated by US is constant in  
227 every case, and the lower the drying temperature, the higher the proportion that it  
228 represents in relation to the total energy available in the drying system [11, 21, 22].

229         The influence of temperature and ultrasound application was also found in the  
230 evolution of the drying rate. Thus, as can be observed in Figure 2, the drying kinetics  
231 occurred in the falling rate period for every condition considered. As the drying  
232 progressed, however, the drying rate fell more quickly in the MTD experiments than in  
233 the AFD and in the ultrasonically-assisted samples than in the conventional ones (MTD-  
234 US and AFD-US compared to MTD and AFD, respectively).

235

### 236 **3.2. Modeling**

237         The Weibull model fitted the experimental evolution of the moisture content  
238 during drying adequately, as shown by the similar trend of the calculated and

239 experimental drying kinetics (Figure 1) and the values of the percentage of explained  
240 variance achieved, over 99 % in every case (Table 2).

241 The figures identified for the shape factor,  $\alpha$ , demonstrated the differences  
242 between the MTD and AFD experiments. Thus, in the case of the AFD experiments,  $\alpha$   
243 was lower than 1.0, indicating the process was mainly controlled by the internal resistance  
244 to mass transfer. At this temperature, -10 °C, the moisture movement inside the material  
245 is very slow, and the influence of external resistance becomes negligible. On the contrary,  
246 the values of  $\alpha$  identified in the MTD experiments were over 1.0, indicating the existence  
247 of downtimes during the process. Thus, the low air velocity used, 1 m/s, did not reduce  
248 the boundary layer thickness enough to compensate for the faster internal moisture  
249 transport that took place at 50°C (MTD experiments) compared to -10°C (AFD  
250 experiments). Therefore, in the MTD conditions tested, both internal and external  
251 resistances influenced the moisture removal. The application of ultrasound did not  
252 significantly affect the shape factor, meaning that ultrasound was not observed to exert  
253 any significant influence on the relative importance of internal and external resistances.

254 The identified values of the  $\beta$  parameter also demonstrated the big difference  
255 between the two drying temperatures tested, being two orders of magnitude larger in the  
256 AFD experiments than the MTD ones (Table 1). The reverse relationship between this  
257 parameter and the kinetics must be highlighted. These results show that there is big  
258 resistance to mass transport in the low temperature process, leading to a long processing  
259 time. During AFD, the removal of the moisture in the orange peel took place by  
260 sublimation, whereas in MTD this process was by evaporation. This, and the differences  
261 in the amount of energy available in the system due to the different drying-air  
262 temperatures, may explain the differences in the magnitude.

263           Ultrasound application also significantly affected the drying velocity in both kinds  
264 of drying experiments. Thus, the  $\beta$  parameter identified in ultrasonically-assisted AFD  
265 experiments was 38% lower than the one identified in the non-assisted ones. In the case  
266 of the MTD experiments, the reduction was 31%. These results showed that the increase  
267 in drying kinetics produced by ultrasound was greater at the lowest drying temperature  
268 tested. This coincides with what has been reported by other authors [22, 23] and can be  
269 explained, as pointed before, by the fact that the mechanical energy supplied by US is  
270 constant in every experiment. So, at low temperatures, the proportion of ultrasonic energy  
271 in relation to the total energy available is greater than at higher temperatures [22].  
272 Ultrasound can affect external mass transport, by inducing microstirring at interfaces, and  
273 internal mass transport, due to the mechanical stress provoked by the compression and  
274 expansion acoustic forces. In the latter, it must be taken into account that while ultrasound  
275 influences the whole sample in the case of MTD experiments, it only affects the external  
276 dried layer in the AFD because no movement of molecules can take place in the frozen  
277 core. In any case, the Weibull model does not permit a clear distinction between the  
278 ultrasound effects on internal and external resistances.

279

### 280 **3.3. Alcohol insoluble residue (AIR)**

281           The average AIR value of the fresh sample ( $52.89 \pm 3.08$  g AIR/100 g dm) was  
282 similar to that reported by Garau et al. [17] (48.30 g AIR/100 g dm). The small difference  
283 observed can be attributed to the different variety of orange (Canoneta vs. Valencia Late  
284 variety) and the natural variability of the raw matter. The drying processes considered did  
285 not produce changes in the AIR content of the samples. Thus, the AIR values measured  
286 in the samples dried under the different conditions were not significantly ( $p < 0.05$ )

287 different from those measured in the fresh samples. In this sense, Garau et al. [17]  
288 reported no influence of drying temperature on the AIR of orange peel.

289

### 290 **3.4. Functional properties**

291 The functional properties can be correlated with the quality of dietary fiber and  
292 the processing, such as drying, can affect both the physical properties of the fiber's matrix  
293 and also the hydration capacity [24]. For that reason, and for the purposes of evaluating  
294 the effects caused by the processing on the structure of the cell wall-forming  
295 polysaccharides of orange peel samples, the swelling capacity (SC), water retention  
296 capacity (WRC) and fat retention capacity (FRC) were measured.

297 Drying produced a marked reduction in the SC of the AIR from orange peel  
298 (Figure 3). Thus, the SC was by 65% lower in the AIR from the MTD experiments  
299 ( $18.52 \pm 1.45$  mL/g AIR dm) than in the AIR from the fresh samples ( $52.53 \pm 1.13$  mL/g  
300 AIR dm). The application of US in these conditions (MTD-US) did not significantly  
301 affect the observed SC ( $17.34 \pm 1.01$  mL/g AIR dm). The swelling capacity is an important  
302 property of fibers and is related with a satiating effect. Therefore, maintaining this  
303 characteristic may not only be beneficial for human health but may also lead to  
304 improvements in the food industry applications [25].

305 As for the AIR from the AFD experiments ( $10.51 \pm 5.61$  mL/g AIR dm), the SC  
306 reduction was significantly greater than that observed in the AIR from the MTD and  
307 MTD-US experiments ( $p < 0.05$ ). Garcia-Amezquita et al. [26] reported a higher SC in the  
308 powder of orange peel obtained by convective drying at 55 °C than in the powder of  
309 orange peel obtained by vacuum freeze-drying. The lower values of the SC during the  
310 AFD experiments can be attributed to the structural changes caused by the formation of  
311 the ice crystals in the food matrix, which leads to a gradual collapse in the tissue

312 organization and cellular destructuration during the long process time needed by this kind  
313 of drying. Besides, the effect of ultrasound application in these conditions (AFD-US)  
314 produced an AIR with a SC ( $16.98 \pm 1.50$  mL/g AIR dm) similar to that observed in the  
315 MTD and MTD-US experiments. This can be explained by the fact that the ultrasonic  
316 effects are more pronounced in a more rigid and porous matrix, such as that provided by  
317 the freezing and sublimation of the orange peel during the atmospheric freeze-drying  
318 process. These effects significantly shorten the drying time and this may contribute to the  
319 lower degree of degradation of the SC in the AFD-US than in the AFD experiments.

320         The water retention capacity (WRC) was also analyzed in the AIRs of both fresh  
321 and dried orange peel. Thus, the average measured WRC (g of water/g AIR dm) values  
322 were  $18.12 \pm 1.71$  for the fresh sample,  $18.43 \pm 3.70$  for the AFD;  $16.86 \pm 3.01$  for the AFD-  
323 US,  $19.23 \pm 1.19$  for the MTD and  $19.18 \pm 1.01$  for the MTD-US. The determination of  
324 Least Significance Intervals ( $p < 0.05$ ) demonstrated that the small differences between  
325 treatments were non-significant ( $p < 0.05$ ). Abou-Arab et al. [27] reported small  
326 differences in the WRC of orange peel powder obtained from solar, convective and  
327 microwave drying (no temperature data is provided). Garcia-Amezquita et al. [26] also  
328 found small differences in the WCR between hot air dried ( $55$  °C) and vacuum freeze-  
329 dried orange peel. These results could indicate that the different drying conditions tested  
330 do not significantly affect the WRC, which is of interest, as the processed product is  
331 similar to the fresh one. The WRC is an important factor because, according to Nesrine  
332 et al. [28], it allows these by-products to be used as functional ingredients by reducing  
333 the amount of calories ingested, preventing syneresis in dairy products and modifying the  
334 viscosity and texture of others.

335         The FRC values were affected by the processing (Figure 4), as it has been  
336 previously reported by Garau et al. [17]. After drying, the AFD experiments showed a

337 significant 31% reduction in the FRC ( $6.5\pm 0.5$  g of oil/g AIR dm) compared to the fresh  
338 sample ( $9.5\pm 0.4$  g of oil/g AIR dm), while the reduction in the MTD experiments ( $9.4\pm 0.8$   
339 g of oil/g AIR dm) was negligible. The differences between the AFD and MTD  
340 experiments were significant ( $p<0.05$ ), indicating a trend toward a better fat retention  
341 capacity in the samples processed at higher temperatures. Similar behavior has been found  
342 by Garcia-Amezquita et al. [26] after the freeze-drying and convective drying ( $55^{\circ}\text{C}$ ) of  
343 orange peel. In the same sense, Garau et al. [17] also observed a higher FRC in the orange  
344 peel samples dried at  $50^{\circ}\text{C}$  than in others dried at lower temperatures. The application of  
345 power ultrasound promoted an increase by 17% in the FRC value of the AIR from the  
346 AFD-US experiments ( $7.6\pm 0.8$  g of oil/g AIR dm) compared to the AFD ones, these  
347 differences not being significant ( $p<0.05$ ) probably due to the great variability. The  
348 shortening of the atmospheric freeze-drying process produced by ultrasound could limit  
349 FRC degradation. On the contrary, the FRC observed in the AIR from the MTD and  
350 MTD-US experiments was the same ( $9.4\pm 0.8$  g vs.  $9.4\pm 0.3$  of oil/g AIR dm, respectively).  
351 The results show that convective drying at moderate temperature was more effective than  
352 atmospheric freeze-drying as a means of preserving the FRC of the AIR obtained from  
353 orange peel. In atmospheric freeze-drying conditions, ultrasound application could  
354 contribute to this preservation. This preservation is important for industrial applications  
355 because it can promote flavor retention, increase the yield of food products and impart  
356 greater stability to the products and emulsions [28].

357

#### 358 **4. Conclusions**

359 The process characteristics linked to temperature and ultrasound application  
360 significantly influenced both the orange peel drying kinetics and the quality of the alcohol  
361 insoluble residue obtained from dried products. The processing time was highly

362 dependent on the mode of moisture removal (sublimation or evaporation) and ultrasound  
363 application. Even with the intensification of the process resulting from the application of  
364 ultrasound, atmospheric freeze-drying required a very long time to reach the expected  
365 final moisture content. The drying conditions tested were found to exert no significant  
366 influence on either the alcohol-insoluble residue obtained from dried product or their  
367 WRC. On the contrary, atmospheric freeze-drying generated samples with slightly  
368 reduced SC and FRC when compared to those obtained with convective drying at 50 °C.  
369 Ultrasound application did not significantly affect the fiber quality. Therefore, in the case  
370 of orange peel, the AFD did not represent a viable alternative to convective drying at  
371 moderate temperatures, neither in terms of drying time nor fiber quality. Moreover,  
372 ultrasound application enhanced the drying rate without reducing the functional  
373 properties of the fiber. This could be linked to energy saving and consequently to a  
374 reduction in process costs. However, this requires further research.

375

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382

## 383 **6. NOMENCLATURE**

### 384 **Acronyms**

AFD	Atmospheric freeze-drying
AIR	Alcohol insoluble residues (g AIR/100 g dm)

FRC	Fat retention capacity (g oil/g of AIR dm)
HTD	High temperature drying
MTD	Convective drying at moderate temperature
PID	Proportional-Integral-Derivative
SC	Swelling capacity (mL water/g of AIR dm)
US	Ultrasound
WRC	Water retention capacity (g water/g of AIR dm)

### 385 **Variables**

dm	Dry matter
$S^2_{calc}$	Calculated variance
$S^2_{ex}$	Experimental variance
$t$	Time (s)
$X_{eq}$	Moisture content at equilibrium (kg water/kg dm)
$X_t$	Moisture content at time t (kg water/kg dm)
$X_0$	Initial moisture content (kg water/kg dm)

### 386 **Greek Letters**

$\alpha$	Shape factor of Weibull model
$\beta$	Kinetics factor of Weibull model ( $s^{-1}$ )
$\psi$	Dimensionless moisture content

387

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- 474  
475

476 **FIGURE CAPTIONS**

477 **Figure 1.** Experimental and calculated evolution of dimensionless moisture during drying  
478 of orange peel at: (A) -10 °C without (AFD) and with ultrasound application (AFD-US);  
479 (B) 50 °C without (MTD) and with ultrasound application (MTD-US).

480 **Figure 2.** Evolution of drying rate during drying of orange peel at: (A) -10 °C without  
481 (AFD) and with ultrasound application (AFD-US); (B) 50 °C without (MTD) and with  
482 ultrasound application (MTD-US).

483 **Figure 3.** SC – Swelling capacity of AIRs from fresh and dried orange peel. Same letter  
484 shows homogeneous groups determined by Least Significant Difference ( $p < 0.05$ )  
485 intervals.

486 **Figure 4.** FRC – Fat retention capacity of AIRs from fresh and dried orange peel. Same  
487 letter shows homogeneous groups determined by Least Significant Difference ( $p < 0.05$ )  
488 intervals.

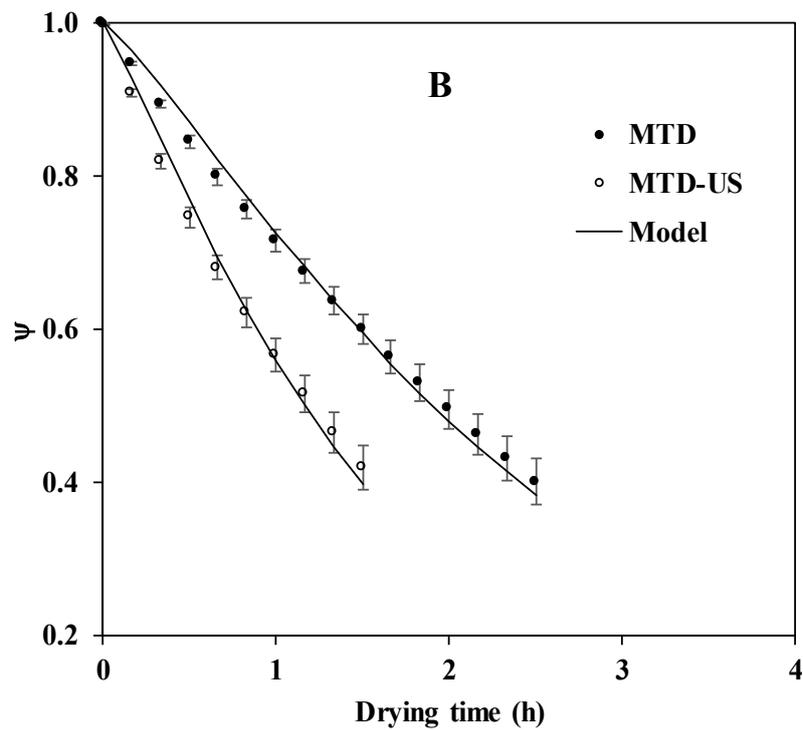
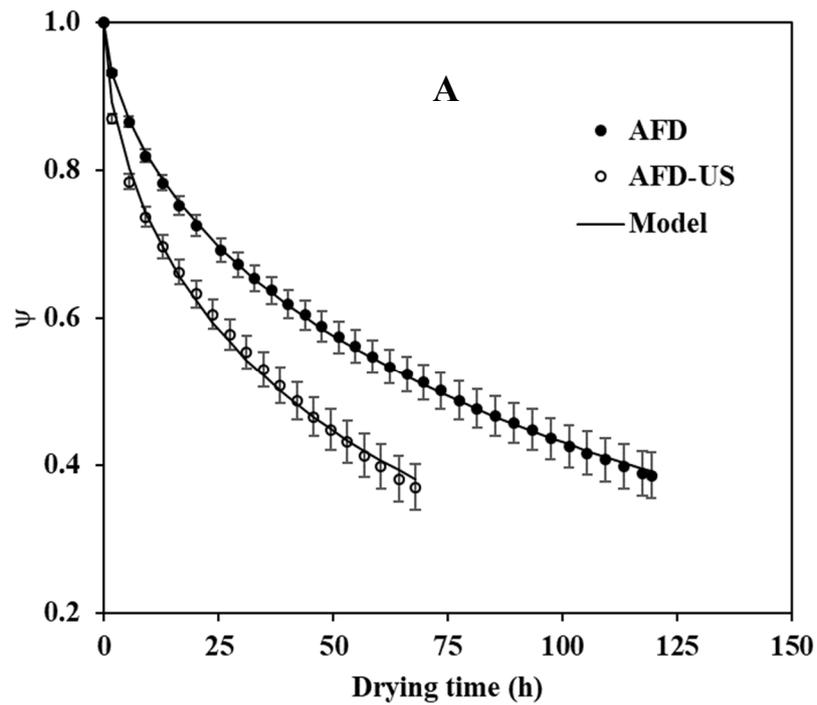
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490 **TABLE CAPTIONS**

491 **Table 1.** Weibull model parameters ( $\alpha$  and  $\beta$ ) identified for the drying of orange peel  
492 (Valencia Late var.) at different temperatures, without and with ultrasound (20.50kW/m<sup>3</sup>;  
493 21.9 kHz) application.

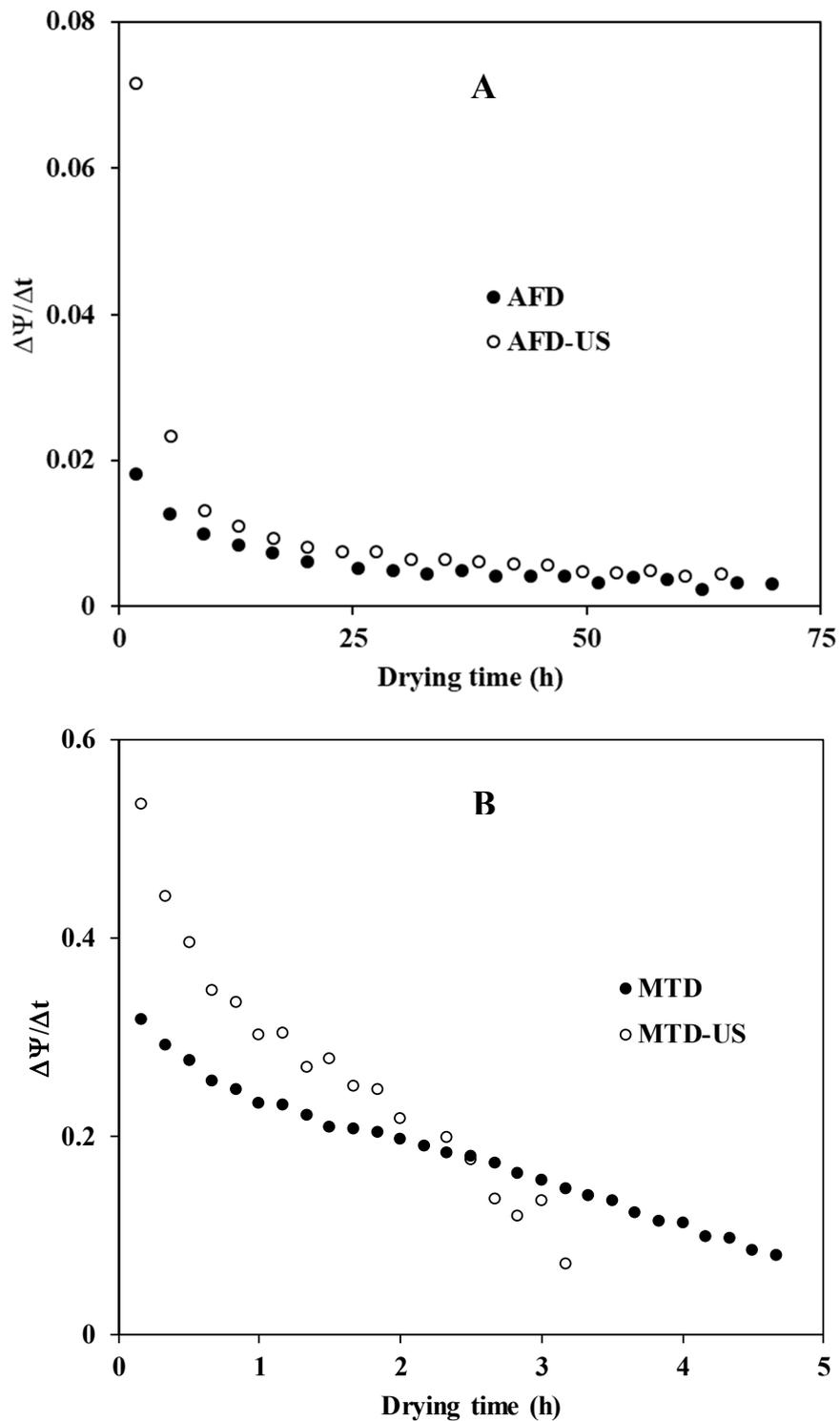
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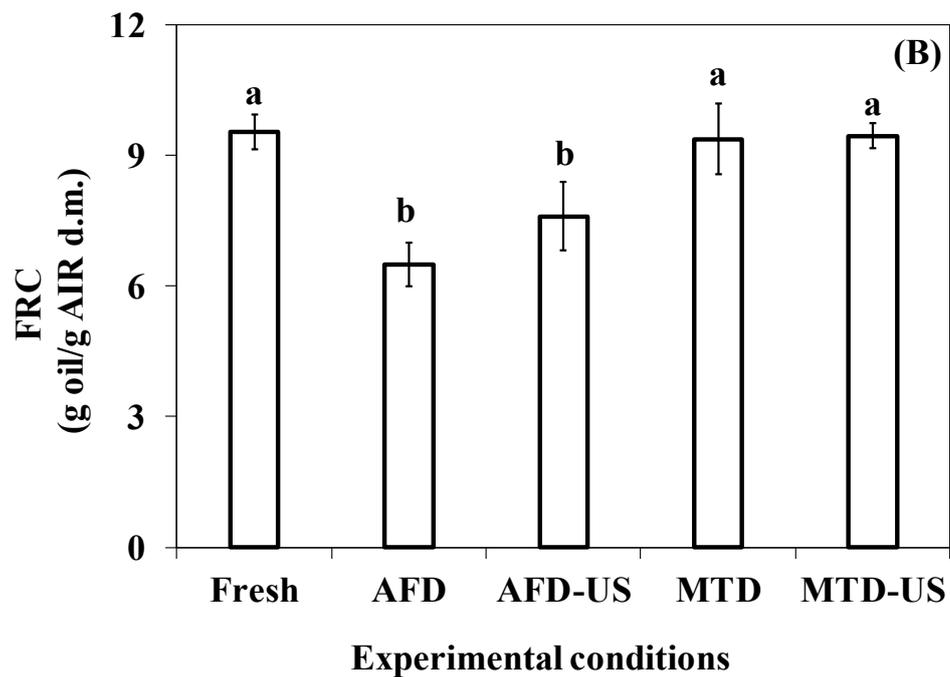
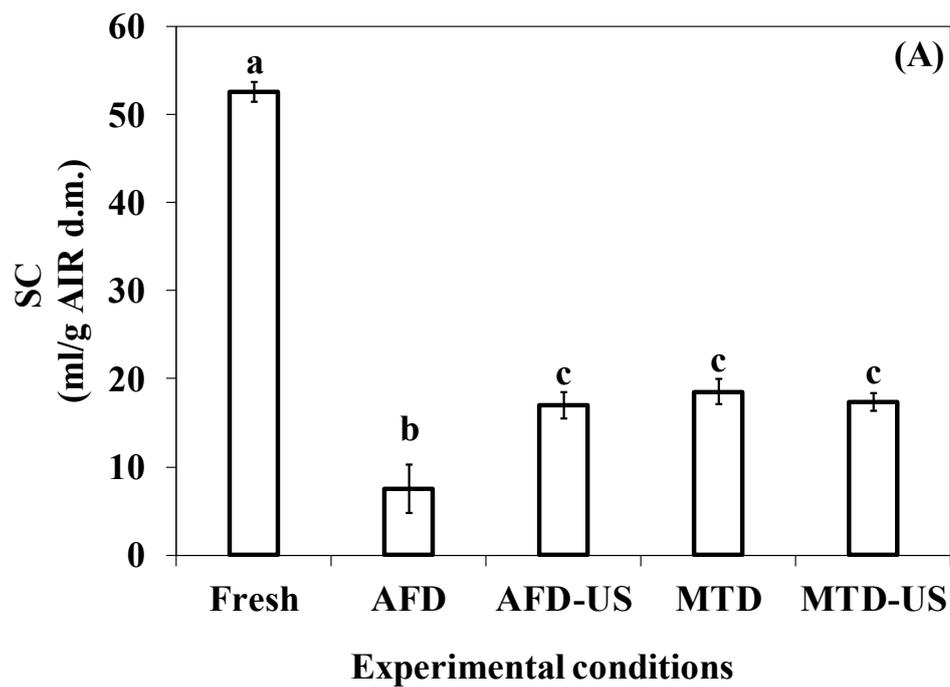
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503



504 **Figure 2.** Evolution of drying rate during drying of orange peel at: (A) -10 °C without  
505 (AFD) and with ultrasound application (AFD-US); (B) 50 °C without (MTD) and with  
506 ultrasound application (MTD-US).

507



508

509 **Figure 3.** Functional properties of AIRs from fresh and dried orange peel. (A) SC –  
 510 Swelling capacity; (B) FRC – Fat retention capacity. Same letter shows homogeneous  
 511 groups determined by Least Significant Difference ( $p < 0.05$ ) intervals.  
 512

513 **Table 1.** Weibull model parameters ( $\alpha$  and  $\beta$ ) identified for the drying of orange peel  
 514 (Valencia Late var.) at different temperatures, without and with ultrasound (20.5kW/m<sup>3</sup>;  
 515 21.9 kHz) application.  
 516

Treatment	$\alpha$	$\beta$ (s <sup>-1</sup> )	VAR
AFD	0.63±0.02 <sup>a</sup>	395569±73199 <sup>a</sup>	99.73
AFD-US	0.60±0.06 <sup>a</sup>	244711±43912 <sup>b</sup>	99.64
MTD	1.14±0.08 <sup>b</sup>	9290±183 <sup>c</sup>	99.40
MTD-US	1.3±0.2 <sup>b</sup>	6398±531 <sup>d</sup>	99.16

517 AFD (atmospheric freeze-drying; -10 °C), AFD-US (ultrasound assisted atmospheric  
 518 freeze-drying; -10 °C; 20.5kW/m<sup>3</sup>), MTD (convective drying at moderate temperature;  
 519 50 °C) and MTD-US (ultrasound assisted convective drying at moderate temperature; 50  
 520 °C; 20.5kW/m<sup>3</sup>). Letters in the same column show homogeneous groups determined by  
 521 Least Significant Difference (p<0.05) intervals.  
 522  
 523