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Additional Information

1	PEF as pretreatment to ultrasound-assisted convective drying:
2	Influence on quality parameters of orange peel
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5	Ronaldo E. Mello <sup>a</sup> , Alessia Fontana <sup>b</sup> , Antonio Mulet <sup>c</sup> , Jefferson Luiz G. Correa <sup>a</sup> , Juan
6	A. Cárcel <sup>c</sup>
7 8	<sup>a</sup> Food Science Department, Universidade Federal de Lavras, Lavras, Minas Gerais,
9	Brazil
10	<sup>b</sup> Applied Science and Technology Department, Politecnico di Torino, Torino, Italy
11 12	<sup>o</sup> Grupo ASPA, Food Technology Department, Universitat Politecnica de Valencia, Valencia Spain
13	valencia, Spani
14	
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25	Corresponding author:
26	Juan A. Cárcel
27	E-mail:jcarcel@tal.upv.es
28	Tf: +34 96 387 93 65
29	

#### 30 Abstract

Pulsed electric field (PEF) pretreatments and ultrasound (US) application are techniques 31 previously used to enhance the drying operation, not only increasing the kinetics but 32 improving product quality. Because PEF pretreatments could affect product structure and 33 US influence depends on the internal structure of products, the combination of both 34 techniques could have a synergistic effect. Thus, the influence of the combined 35 application of pulsed electric field (PEF) pretreatments and ultrasound (US) during drying 36 on the color, total phenolic content, ascorbic acid and antioxidant activity of orange peel 37 was studied. To this end, a series of drying experiments (50 °C) was performed without 38 and with ultrasound application  $(20.5 \text{kW/m}^3)$  and with and without PEF pretreatments 39 (1.20 kV/cm) for two different times, 200 µs (0.37 kJ/kg) and 600 µs (1.12 kJ/kg). Thus, 40 when individually applied, ultrasound significantly shortened the drying time, and PEF 41 pretreatments slightly extended the process. However, the shortest drying time was 42 observed combining 200 µs PEF pretreatment and ultrasound. This combination also 43 provided the more similar color parameter to the fresh samples and significantly increased 44 the percentage of phenolic compound retention. In addition, every treatment with PEF 45 exhibited a similar percentage of ascorbic acid retention, and only the longer pretreatment 46 (600 µs) produced a reduction in the antioxidant activity retention. Therefore, the 47 combined use of a PEF pretreatment and ultrasound application during orange peel drying 48 49 can lead to an interesting way both to shorten the drying process and preserve important compounds. 50

51 Keywords: By-products; drying time; color; antioxidant, phenolic content, vitamin C.

## 53 1. Introduction

One current global priority is the need for healthy foodstuffs, easy-to-prepare and 54 of practical consumption. In this sense, the health benefits of fruit and vegetable 55 consumption have been well established. Orange, produced and consumed worldwide, is 56 highly appreciated for its flavor and for the fact that it has a variety of nutrients. On an 57 industrial scale, orange is mainly processed for the purposes of juice production. This 58 activity generates a massive amount of residue, orange peel being one of the major 59 components. This by-product is a rich source of vitamin C, phenolic compounds and 60 dietary fiber (Slama & Combarnous, 2011; Luengo, Álvarez, & Raso, 2013; Adiamo, 61 Ghafoor, Al-Juhaimi, Babiker, & Mohamed Ahmed, 2018; Garcia-Amezquita, Tejada-62 Ortigoza, Campanella, & Welti-Chanes, 2018) and can be used as flour and seasoning 63 64 (Espinosa-Garza, Antonyan, & Loera-Hernández, 2018), as source of nanofibers (Hideno, Abe, & Yano, 2014), natural colorants (Różyło, 2020) or others. However, like many 65 biological products, orange peel has a high moisture content, making it adequate for 66 microbial and enzymatic degradation reactions. Therefore, there is a need for the 67 68 application of conservation techniques which provide the product with stability (Bejar, Kechaou, & Mihoubi, 2011; Onwude, Hashim, Abdan, Chen, & Oladejo, 2017) 69 permitting a subsequent permitting a subsequent valorization process. 70

One of the most widely-applied conservation techniques is hot-air convective 71 72 drying (HAD), mainly due to its user-friendliness (Valadez-Carmona et al., 2017). This technique promotes the removal of the moisture by creating a vapor pressure gradient 73 74 between the product and the drying air. The energy needed by the process is provided by the high air temperature. Thus, a stable product is obtained with reduced weight and 75 76 volume (Pérez-Won et al., 2016). However, HAD can promote changes in the material structure, color alterations, oxidation reactions, shrinkage or nutritional and functional 77 quality degradation linked to the long exposure time to high temperatures, which also 78 involves great energy consumption (Santacatalina et al., 2014; Pérez-Won et al., 2016; 79 Corrêa, Rasia, Mulet, & Cárcel, 2017; Vallespir, Rodríguez, Cárcel, & Simal, 2019). 80 Therefore, there is great interest in the development and application of techniques that 81 can help to minimize both the decrease in quality and the energy costs. Some of the 82 techniques tested include the application of microwave, osmotic dehydration, power 83 ultrasound or pulsed electric fields (Rojas & Augusto, 2018; Ambros, Mayer, Schumann, 84

& Kulozik, 2018; Martins, Cortés, Eim, Mulet, & Carcel, 2018; Mello Jr, Corrêa, Lopes,
de Souza, & da Silva, 2019).

Pulsed electric field (PEF) is a non-thermal technique that involves the application 87 of short and repeated high voltage pulses to a biological product. This can promote 88 changes in the electrical conformation of the cell membrane, inducing the generation of 89 pores, a phenomenon known as electroporation. These pores can ease both the mass 90 transfer and the exit of the inner components of the cell, which can enhance operations 91 92 such as extraction or drying (Traffano-Schiffo et al., 2017; Risvy, Lyng, Frontuto, Marra, & Cinquanta, 2018). Thus, the use of PEF as a pretreatment has been previously tested in 93 the drying of blueberries, parsnips or carrots and the results showed that PEF application 94 can contribute to shorten the drying time (Yu, Jin, & Xiao, 2017; Risvy, Lyng, Frontuto, 95 96 Marra, & Cinquanta, 2018). However, the effect of PEF application depends on the conditions considered. Thus, Toepfl, Siemer, Saldana-Navarro, & Heinz (2014) found a 97 98 reduction in the drying rate of radishes when an increased number of pulses were applied. As regards the quality of the products, some authors have found a reduction in the 99 100 nutritional quality (Toepfl, Siemer, Saldana-Navarro, & Heinz, 2014; Yu, Jin, & Xiao, 101 2017) and others (Soliva-Fortuny, Balasa, Knorr, & Martín-Belloso, 2009) have reported 102 a reduction in the degree of product damage when compared with other techniques.

103 The use of high intensity airborne ultrasound (US) has been widely studied as a 104 means of intensifying the drying process and preserving the characteristics of dried products (Santacatalina et al., 2014; Clemente, Sanjuán, Cárcel, & Mulet, 2014; 105 106 Santacatalina, Contreras, Simal, & Cárcel, 2016; Corrêa, Rasia, Mulet, & Cárcel, 2017; 107 Cárcel, Castillo, Simal, & Mulet, 2018). The effects induced by US application can help 108 to reduce the internal (sponge effect) and external (microstreaming at interfaces) resistance to mass transport (Cárcel, Castillo, Simal, & Mulet, 2018; Martins, Cortés, 109 110 Eim, Mulet, & Cárcel, 2018). However, US can also affect the quality of dried food (Rodríguez et al., 2018); this influence is dependent on the drying conditions used, as was 111 reported during the drying of passion fruit peel (Nascimento, Mulet, Ascheri, Wanderlei, 112 & Cárcel, 2016) or green pepper (Szadzinska, Łechtanska, Kowalski, & Stasiak, 2017). 113

114 The combination of PEF and ultrasound application in liquid media, both as 115 pretreatments of drying proces have been previsouly studied (Wiktor & Witrowa-116 Rajchert, 2020). However, to best of our knowledge, no previous studies about the 117 combination of PEF pretreatments and the application of airborne ultrasound during 118 drying have been carried out. Therefore, the aim of this study was to assess the influence 119 of the combined use of PEF as a pretreatment and the ultrasonically asisted drying on 120 some quality parameters of orange peel.

121

- 122 2. Material and methods
- 123

124 2.1. Raw material

The oranges used in this study, (*Citrus sinensis, Valencia Late var.*) were purchased in a local market (Valencia, Spain). The fruits selected were homogenous in size and color. The oranges were washed and their surface dried with the aid of absorbent paper. Then, rectangular shaped samples ( $48 \pm 1 \ge 26 \pm 1 \ge 3.18 \pm 0.04$  mm) of orange peel, including both albedo and flavedo tissues, were obtained with a sharp knife. The moisture content was measured in triplicate by measuring the weight difference after maintaining peel samples at 60 °C in a vacuum oven until constant weight (AOAC, 1997).

132

#### 133 2.2. Pulsed electric field (PEF) treatment

The PEF pretreatments were carried out in a laboratory scale system, with a 134 maximum positive pulse voltage ranging up to 10 kV (EPULSUS-PM1-10, Energy Pulse 135 136 System, Lisbon, Portugal). The generated pulses were applied to the samples in a chamber containing electrodes (distance between electrodes of 8.2 cm) (Figure 1). For each 137 138 experiment, the sample holder containing the peel samples (18 samples per run) was filled with tap water (electrical conductivity 1.11 mS/cm) at 20±1 °C as electricity driven 139 140 medium (sample/water ratio of 1:4.7 g/mL). In this study, two PEF treatments were 141 considered: one with 8 pulses and the other 24, of 25 µs each, which means a total 142 treatment time of 200 (PEF 200 µs) and 600 µs (PEF 600 µs), respectively. A frequency of 10 Hz and an electrical field strength (E) of 1.20 kV/cm (Won, Min, & Lee. 2015), 143 144 provided a specific energy input of 0.37 kJ/kg and 1.12 kJ/kg for PEF 200 µs and PEF 145 600 µs, respectively. These parameters were in the range of the used by other authors (Chauhan, Sayanfar, & Toepfl 2018, Won, Min & Lee, 2015) studying the influence of 146

PEF pretreatment in drying. After each treatment, the samples were removed from the
treatment chamber and their surface dried with absorbent paper before the drying
experiments.

150

# 151 2.3. Hot air drying (HAD) experiments

HAD experiments were performed in an ultrasonically assisted convective dryer 152 (Garcia-Perez, Ortuño, Puig, Carcel, & Perez-Munuera, (2012); Santacatalina et al., 153 2015). In this system, the drying chamber is constituted by an airborne ultrasonic device 154 (Figure 1), specifically, an aluminum-vibrating cylinder (height 310 mm, internal 155 diameter 100 mm, thickness 10 mm) attached to a piezoelectric transducer (21.9 kHz) 156 which is able to generate an internal high intensity ultrasonic field. Drying is conducted 157 automatically with the control of temperature and air velocity. Each drying experiment 158 was carried out with a parallel flux of air around 18 samples, randomly placed in a sample 159 holder (Rojas, Augusto, & Cárcel, 2020). A balance permit the weighing of the samples 160 161 at preset times and then, the monitoring of the drying kinetics. Every drying experiment was performed at  $50\pm1$  °C and 1 m/s and they were extended until the samples lost  $70\pm1\%$ 162 163 of the initial weight, which assure the stability of samples after drying. Different conditions were tested by combining PEF pretreatments (200 µs and 600 µs) with the 164 drying without and with (20.5 kW/m<sup>3</sup>; electric power applied to the transducer divided 165 by drying chamber volume) US application. Moreover, conventional HAD experiments 166 167 were carried out as reference (Table 1). Each experimental combination was performed at least in triplicate. 168

169

#### 170 2.4. Quality parameters

171 2.4.1. Color

The color of the dried orange peel samples was determined by measuring the CIELAB spectrum color parameters  $L^*$  (lightness/darkness),  $a^*$  (redness/greenness) and  $b^*$  (yellowness/blueness) using a colorimeter (CM-2500d model, Konica Minolta, Japan) provided with a D65 illuminant reference system and a 10° opening angle. The excluded specular component (SCE) was considered. As a measurement of color saturation, the chroma value ( $C^*$ ) was obtained from Eq. (2) (Wiktor et al., 2016).

178 
$$C^* = \sqrt{(a^{*2} + b^{*2})}$$
 (2)

179 2.4.2. Antioxidant properties

The measurements of the antioxidant properties (TPC, AA, and AC) of the fresh and dried orange peel were taken in an ethanolic extract. For that purpose, 1g of orange peel powder (particle size smaller than 200  $\mu$ m), obtained with the help of a domestic grinder, was placed into 20 mL of ethanol (96% v/v) and homogenized with an ultraturrax for 1 min at 13000 r.p.m. Then, the mix was filtered and stored at 4 ± 0.5 °C, protected from light, until analysis.

186 2.4.2.1 Total phenolic content (TPC)

The TPC was determined through the Folin-Ciocalteu method (Singleton, 187 188 Orthofer, & Lamuela-Raventós, 1999). For this, 100 µL of the ethanolic extract of samples were mixed with 200 mL of Folin-Ciocalteu's phenol reagent (Sigma-Aldrich, 189 190 Madrid, Spain) and 2 mL of distilled water. After 3 min at 25 °C, 1 mL of Na<sub>2</sub>CO<sub>3</sub> (Panreac, Barcelona, Spain) solution (Na<sub>2</sub>CO<sub>3</sub>-water 20:80, w/v) was added and the 191 192 mixture was kept in the dark at room temperature for 1 h. Finally, the absorbance was read at 765 nm using a spectrophotometer (Helios Gamma, Thermo Spectronic, 193 194 Combridge, UK). A standard curve was previously prepared using solutions of a known 195 concentration of gallic acid hydrate (Sigma-Aldrich, Madrid, Spain) in distilled water. Results were expressed as mg of gallic acid (GAE) per g of dry matter of orange peel 196 samples. The measurements were taken in triplicate for each condition tested. 197

198 2.4.2.2 Ascorbic acid content (AA)

The AA was measured according to Jagota & Dani (1982). To this end, 0.5 mL of the ethanolic extract of sample was mixed with 0.5 mL of a trichloroacetic acid solution (7.5%). After 5 min at 4 °C, the mix was filtered. Subsequently, 0.2 mL of extract, 2 mL of distilled water and 0.2 mL of diluted Folin reagent (1:10 v/v) were blended and maintained for 10 min at room temperature (Jagota & Dani, 1982). Afterwards, absorbance was measured at 760 nm in a spectrophotometer (Helios Gamma, Thermo Spectronic, Combridge, UK). The procedure was also performed in triplicate for each condition considered. The concentration of vitamin C was obtained from a calibrationcurve made up of solutions of known ascorbic acid concentration.

208 2.4.2.3 Antioxidant capacity (AC)

The AC was determined by using the Ferric-Reducing Ability Power (FRAP) 209 210 method, which was described by Benzie & Strain (1996). In a spectrophotometer cuvette, 211 30 µL of distilled water, 30 µL of ethanolic extract of sample and 900 µL of FRAP were mixed in this order. The FRAP reagent was prepared by adding 2.5 mL of 10 mM TPTZ 212 (Fluka, Steinheim, Germany) in a 40 mM HCl (Panreac, Barcelona, Spain) solution plus 213 2.5 mL of 20 mM FeCl<sub>3</sub>.6H<sub>2</sub>O (Panreac, Barcelona, Spain) and 2.5 mL of 0.3 M acetate 214 buffer (Panreac, Barcelona, Spain), pH 3.6. For the AC determination, 30 mL of each 215 sample were used completed with 30 mL of distilled water and 900 mL of FRAP reagent 216 and kept at 37 °C for 30 min. Using a spectrophotometer (Helios Gamma, Thermo 217 Spectronic, Cambridge, UK), the absorbance was read at 595 nm. A calibration curve was 218 previously obtained using ethanol solutions of known Trolox (SigmaeAldrich, Madrid, 219 Spain). The procedure was performed in triplicate and concentrations were described as 220 221 millimole Trolox equivalent per gram of dry mass of orange peel.

222

#### 223 2.5. Statistical analysis

224 For the statistical analysis, the color parameters, TPC, AA and AC were considered as process-dependent variables, and the PEF pretreatments and US application 225 as factors. The analysis of variance was calculated using Statgraphics Centurion XVI 226 227 (StatPoint Technologies, Inc) to check the significance (p<0.05) of the differences between the values of each dependent variable. The Least Significant Difference (LSD) 228 229 intervals were also estimated to determine the significance of the differences between treatments. Additionally, the values from the replicates of the different kinds of 230 experiments carried out were averaged and represented as mean and standard deviations. 231

232

#### 233 **3.** Results and discussion

#### 235 3.1. Drying experiments

The initial moisture content of the orange peel was  $2.70 \pm 0.31$  kg water/kg dry 236 matter, this value being similar to that reported in the literature (Angoy et al., 2020). The 237 238 evolution of the dimensionless moisture content of orange peel during drying at the different conditions tested is shown in Figure 2. As can be observed, the application of 239 PEF as a pretreatment did not accelerate the drying process, but slightly delayed it. Thus, 240 241 for example, the time needed to reach a moisture content of 0.6 kg water/kg dry matter in 242 HAD experiments  $(3.5 \pm 0.3 \text{ h})$  was 19 and 17% shorter than in HAD-200 µs  $(4.4 \pm 0.2 \text{ h})$ h) and HAD-600  $\mu$ s (4.3  $\pm$  0.1 h), respectively. A similar effect was observed by Liu et 243 244 al. (2016) during the drying of radish. However, the opposite results can be found in the literature, namely a shortening of the drying time when PEF was applied as a pretreatment 245 246 to the drying processes of apple (Wiktor et al., 2013) or red pepper (Won, Min, & Lee, 2015). The fact that studies can give different results may be related with both the 247 248 characteristics of each product studied but also with the operational conditions of the PEF 249 treatment. In the case of orange peel, there is two main tissues, flavedo, the external layer 250 of orange peel, and albedo, the internal one, which can be differently affected by the PEF 251 pretreatment. Thus, the longer drying process in PEF pretreated samples could be related with a possible partial sealing effect in flavedo layer, which can make the inner moisture 252 transport to the sample surface difficult. 253

254 The application of ultrasound during the drying of orange peel samples accelerated the process in every condition tested (Figure 2). Thus, for instance, to reach 255 256 a moisture content of 0.6 kg water/kg dry matter, the time needed was 25% shorter in HAD-US experiments  $(2.6 \pm 0.5 \text{ h})$  than in HAD ones  $(3.5 \pm 0.3 \text{ h})$ . Similar behavior has 257 258 been previously reported in the literature for the ultrasonically-assisted drying of various fruits and vegetables. Thus, Nascimento et al. (2016) found a drying time reduction of 259 260 49% for passion fruit drying (50 °C, 1 m/s) when ultrasound was applied (30.8 kW/m<sup>3</sup>). Rojas, Augusto, & Cárcel (2020) reported a 41% reduction in the case of apple drying 261 262 (50 °C, 1 m/s and 20.5 kW/m<sup>3</sup>) and Ortuño et al. (2010) observed that the drying time for orange peel (40 °C, 1 m/s and 37 kW/m<sup>3</sup>), Navel variety, was 45 % shorter. 263

The effects on the drying kinetics of the combined application of the PEF pretreatment and ultrasonically-assisted drying depended on the PEF pretreatment applied. Thus, compared with the HAD experiments, HAD-US-600 µs meant only an 8%

shorter drying time, while in the case of HAD-US-200 µs this reduction reached 33% 267 (Table 2). How much an effect ultrasound has on the drying rate is related with the 268 structural properties of product, such as porosity (Ozuna, Álvarez-Arenas, Riera, Cárcel, 269 & Garcia-Perez, 2014). PEF could induce changes in the internal structure of albedo 270 271 tissue, affectig it porosity, and therefore the magnitude of the US effects. In this sense, the less intense PEF treatment of the HAD-US-200 µs experiments (only 8 pulses of 1.2 272 273 kV/cm) could enhance the ultrasound effects. On the contrary, the more intense PEF treatment applied in HAD-US-600 µs (24 pulses) could partially degrade the inner 274 275 structure; this would make it difficult for ultrasound to have significant effects and could also hinder the drying itself. 276

277

### 278 3.2. Quality parameters

279 3.2.1. Color

280 Color is a very important parameter in the sensory evaluation of dried fruits and vegetables. Therefore, it is essential to understand the influence of the drying processes 281 and the pretreatments on possible changes in the color of the products. Thus, the color 282 parameters of fresh and dried orange peel samples were measured to determine the 283 influence of the process variables studied the PEF pretreatments and the application of 284 US during drying. Compared with fresh samples, HAD promoted a decrease of color 285 parameters which was significant (p<0.05) for  $b^*$  and chroma (Table 2). These results 286 were no significant different than those obtained in HAD-200 µs experiments (Table 2) 287 but significantly (p<0.05) greater than the  $L^*$ ,  $b^*$  and  $C^*$  values observed for HAD-600 µs. 288 289 These changes may be related to the leaching of compounds responsible for the 290 characteristic coloring of the orange peels, which occurs during the pretreatment, this effect being proportional to the intensity of the PEF applied. Wiktor et al. (2016) found 291 no changes in the lightness of PEF treated carrot samples. On the contrary, they observed 292 changes in the  $a^*$  parameter, probably linked with electroporation. Furthermore, Rizvi et 293 294 al., (2018) observed that the effect of the pre-treatment promoted reductions in the value of  $L^*$  in carrots. Wiktor et al. (2016) also reported that the increase in the intensity of the 295 PEF treatments promoted reductions in  $b^*$  and  $C^*$ . While studying the influence of PEF 296 on pumpkin samples, Rahaman et al. (2019) also observed that the PEF pretreatment 297 developed changes in the  $b^*$  and  $C^*$  parameters. The fact that several studies into the 298

application of PEF have obtained this variability of results could be a consequence of
several factors, including differences in equipment, varying process conditions, the
application of different pretreatments and even the intrinsic characteristics of each
product (Raso et al., 2016).

Ultrasound application during drying induced changes in all the analyzed color 303 parameters (p<0.05). Thus, the  $L^*$ ,  $a^*$ ,  $b^*$  and  $C^*$  figures obtained in HAD-US experiments 304 were significantly lower than those in HAD ones (Table 3). When studying the drying of 305 306 apple peel in similar conditions of temperature and ultrasound power applied, Martins et al. (2018) did not observe differences between the  $L^*$  and  $b^*$  figures of ultrasonically and 307 non-ultrasonically assisted dried samples but did report a significant increase in  $a^*$  and  $C^*$ 308 after applying ultrasound. Nowacka & Wedzik (2016) identified several effects as a result 309 310 of ultrasound application during the drying of carrot slices, and reported a reduction in  $L^*$ and an increase in  $b^*$  due to ultrasound application in every case studied. 311

As for the color parameters of experiments carried out with the combination of PEF and US application, the values obtained were similar (p<0.05) to those obtained in HAD experiments. Only small, but significant (p<0.05), decreases in both  $b^*$  and  $C^*$  were found in the HAD-US-600 µs, which was similar to that observed in the HAD-600 µs samples. Therefore, the PEF pretreatment seemed to reduce the effect of ultrasound application on color parameters.

318

319 3.2.2. Antioxidant properties

320 3.2.2.1. Total phenolic content (TPC)

The TPC of fresh orange peel was 0.30±0.03 mg GAE/g dry matter. This content 321 was similar to that reported by Teixeira et al. (2020), 0.31±0.06 mg GAE/g dry matter, 322 and higher than those found by Montero-Calderon, Cortes, Zulueta, Frigola, & Esteve 323 (2019), 0.16±0.06 mg GAE/g dry matter. In addition, Park, Lee, & Park (2014) reported 324 a TPC for orange peel ranging from 1.39 to 1.85 mg GAE/g dry matter. Drying reduced 325 326 the initial TPC of fresh sample in every condition tested. However, the lower TPC retention, 27%, was observed in the HAD experiments, as shown in Figure 3. As Wiktor 327 et al. (2019) reported, this can be attributed to the long drying time of these experiments, 328 which also means a long thermal treatment. 329

The PEF pretreatments significantly (p < 0.05) increased TPC retention compared 330 with HAD experiments, however the level achieved depended on the intensity of the 331 treatment. Thus, the TPC retention of the HAD-200 µs samples (48%) was significantly 332 (p<0.05) greater than the observed in HAD-600 µs ones (37%) (Figure 3). Application of 333 334 PEF can favor the extraction of intracellular compounds, including phenolic compounds (Kim, Kwon, & Lee, (2019). However, this fact also favor degradation reaction of these 335 compounds. These results could indicate the existence of a suitable range of PEF 336 intensity, which could provide the greater TPC retention. Additional experiments at 337 different PEF conditions should be carried out to identify the value of this optimum 338 339 intensity.

The application of ultrasound during drying (HAD-US) significantly (p<0.05) increased the TPC retention compared with the HAD experiments (38% vs 27%, respectively). A similar positive effect of ultrasound application on TPC was reported by Nascimento et al. (2016) during the ultrasonically-assisted drying (30.8 kW/m<sup>3</sup>) of passion fruit peel at a moderate temperature (50 °C). Such effect can be related to the reduction of the polyphenols degradation due to the shorter exposure time of the samples to the drying air (Nascimento et al., 2016).

347 The TPC retention obtained when combining PEF and US was similar to the same experiments carried out without US application. Thus, the HAD-US-200 µs experiments 348 349 promoted a 48% retention of phenolic compounds (vs 48% of the HAD-200 µs) and 33% in the case of the HAD-US-600 µs (vs 37% of the HAD-600 µs), both values being 350 significantly greater from the HAD experiment (p < 0.05) (Figure 3). This indicates that, 351 in the case of TPC, the effects of both techniques can be complementary. The PEF 352 353 pretreatment contributes to a better TPC preservation and US contributes to a significant 354 increase in the drying rate.

355 3.2.2.2. Ascorbic acid (AA)

Ascorbic acid is a compound of great nutritional relevance, which is highly sensitive to thermal processes. This is the main reason why it is used as an indicator of thermal treatment damage. The ascorbic acid content of fresh orange peel samples was  $0.25 \pm 0.01$  mg ascorbic acid/g dry matter, this value lying in the range of that found in the literature. Thus, Tasirin et al. (2014) reported a content of  $0.50 \pm 0.02$  mg ascorbic acid/g dry matter and Hernández-Carranza et al. (2016) observed values between 0.18 to
1.02 mg ascorbic acid/g dry matter, according to the extraction parameters used.

363 Drying process induce a significant degradation of AA. Thus, in the case of HAD experiments, a retention of 51% was observed (Figure 4). The retention was similar in the 364 other conditions tested, ranging from 52% to 45% of the initial content which indicates 365 the main damage to AA content was produced by the drying itself. The experiments 366 carried out with PEF pretreatments, exhibited slightly but significant lower (p<0.05) 367 368 retentions values (45 and 46% for HAD-200 µs and HAD-600 µs experiments respectively) than HAD experiments. These results can be explained by the 369 370 electroporation mechanism and the side effects that the PEF can cause in the matrix, resulting in a slightly increase in the exposure of the AA to the drying air, according to 371 372 Wiktor et al. (2019).

373 The application of ultrasound during the drying process did not directly affect the 374 percentage of AA retention in the samples under study, this (51% in the HAD-US) not 375 being significantly different (p<0.05) than those obtained in the HAD experiments, even 376 with the reduction of the exposure time of the samples to the drying process which US 377 application meant. Martins et al. (2018) reported that the application of ultrasound during 378 the drying of apple peel did not affect the retention of ascorbic acid during drying at low and moderate temperatures (-10, 30 and 50 ° C). Furthermore, the combined application 379 380 of US and PEF did not significantly affect AA retention compared to HAD experiments.

381 3.2.2.3. Antioxidant capacity (AC)

The antioxidant capacity of the samples under study was  $7.40 \pm 0.15$  mg Trolox/g 382 383 dry matter. This was slightly higher than that reported by Hernández-Carranza et al. (2016), who found orange peel AC in the range of 5.01 - 6.03 mg Trolox/g dry matter. In 384 385 this case, the drying also produced an important reduction of AC as showed the 45% of AC retention in HAD experiments (Figure 5). The application of PEF did not influence 386 387 the retention percentage of AC in the HAD-200 µs experiments if compared to the HAD 388 ones (Figure 5). However, the experiments carried out with the more intense pretreatment, 389 the HAD-600  $\mu$ s, exhibited a lower (p <0.05) AC retention (31%) than that of the HAD experiments (45%) (Figure 5). When studying the drying of blueberries at 45 and 60 °C, 390 391 Yu, Jin, & Xiao (2017) found that the PEF (2kV/cm and 96 µs of total time process) 392 pretreatment reduced the values of AC compared to the non-pretreated samples.

Similarly, Wiktor et al. (2015) found that, in general, the application of PEF reduced the antioxidant capacity of apples. The decrease in antioxidant activity may be related to the cell leakage that occurs during pretreatment, which promotes a greater exposure of the bioactive compounds to the drying process, thus leading to a higher degree of degradation (Lammerskitten et al., 2019).

398 Ultrasound application during drying was observed to exert no influence on the antioxidant capacity of the orange peels. Thus, both the HAD and HAD-US experiments 399 showed the same AC retention figure, 45% (Figure 5). Martins et al. (2018) also found 400 that the application of ultrasound ( $20.5 \text{ kW/m}^3$ ) during the drying of apple peel at -10, 30, 401 50 and 70 ° C and under similar conditions of ultrasonic power led to no significant 402 difference. The combination of PEF and US retained less AC than the experiments 403 404 without US (45% in the HAD-200 µs vs 37% in the HAD-US-200µs; 31% in the HAD-405 600 µs vs 25% in the HAD-US-600 µs), both values of the HAD-US-200µs and the HAD-406 600  $\mu$ s being significantly different from those of the HAD experiment (p<0.05). It is worth mentioning the wide variety of compounds present in the orange peel which can 407 408 contribute to the total antioxidant activity. It will need more specific analysis to identify 409 and measure their particular antioxidant activity. Therefore, in relation to the AC, these data indicate that the PEF contributes to a better preservation and the US plays an 410 411 important role in shortening the drying time, both being complementary techniques for 412 the dehydration process of orange peels.

413

#### 414 4. Conclusions

The combination of PEF pretreatment and airborne ultrasound application during 415 drying significantly shorten the drying process of orange peel. Moreover, they contributed 416 to a better preservation of quality characteristics such as color, phenolic content or the 417 antioxidant capacity. Therefore, combining both technologies with conventional drying 418 419 process is a feasible means of obtaining dried products with a lower impact on quality. 420 However, a too intense PEF pretreatment can induce negative effects on both kinetics and 421 product characteristics and it its necessary to find the optimum value of PEF treatment 422 variables which enhance not only the ultrasonic drying but also the quality of the dried 423 product. In this sense, PEF pretreatment can affect other important components of orange peel such as pectin, which must be considered in further research. 424

## 426 5. Acknowledgments

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#### 653 Figure caption

Figure 1. Scheme of the cell used in PEF pre-treatments and the vibrating drying chamberof the ultrasonically assisted dryer.

**Figure 2.** Experimental dimensionless moisture content (moisture content divided initial moisture content) evolution of orange peel non-pretreated and pretreated with PEF (200 and 600  $\mu$ s) during drying (HAD, 50 °C; 1 m/s) without and with ultrasound application (US, 20.5 kW/m<sup>3</sup>).

**Figure 3.** Retention of the total phenolic content (TPC) of orange peel after drying at 50 °C without and with (US) ultrasound (20.5 kW/m<sup>3</sup>) application and PEF pretreatment (200 and 600  $\mu$ s). Mean values and standard deviation are shown. Different letter indicates different least significant difference intervals (p<0.05).

**Figure 4.** Retention of the total ascorbic acid (AA) of orange peel after drying at 50 °C without and with (US) ultrasound (20.5 kW/m<sup>3</sup>) application and PEF pretreatment (200 and 600  $\mu$ s). Mean values and standard deviation are shown. Different letter indicates different least significant difference intervals (p<0.05).

**Figure 5.** Retention of the antioxidant capacity (AC) of orange peel after drying at 50 °C without and with (US) ultrasound (20.5 kW/m<sup>3</sup>) application and PEF pretreatment (200 and 600  $\mu$ s). Mean values and standard deviation are shown. Different letter indicates different least significant difference intervals (p<0.05).

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# **Table captions**

- **Table 1.** Conditions tested in drying experiments
- **Table 2.** CIELAB color parameters  $(L^*, a^* \text{ and } b^*)$  and chroma  $(C^*)$  of orange peel dried
- at 50 °C without (HAD) or with (20.5 kW/m<sup>3</sup>) ultrasound (US) application and PEF (200
- $\mu$ s and 600  $\mu$ s) pretreatment



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- Figure 1. Scheme of the cell used in PEF pre-treatments and the vibrating drying chamber
- 683 of the ultrasonically assisted dryer.



Figure 2. Experimental dimensionless moisture content (moisture content divided initial moisture content) evolution of orange peel non-pretreated and pretreated with PEF (200 and 600  $\mu$ s) during drying (HAD, 50 °C; 1 m/s) without and with ultrasound application (US, 20.5 kW/m<sup>3</sup>).



**HAD HAD-US HAD-200 \mus HAD-600 \mus HAD-US-600 \mus** Figure 3. Retention of the total phenolic content (TPC) of orange peel after drying at 50 °C without and with (US) ultrasound (20.5 kW/m<sup>3</sup>) application and PEF pretreatment (200 and 600  $\mu$ s). Mean values and standard deviation are shown. Different letter indicates different least significant difference intervals (p<0.05).



Figure 4. Retention of the total ascorbic acid (AA) of orange peel after drying at 50 °C without and with (US) ultrasound (20.5 kW/m<sup>3</sup>) application and PEF pretreatment (200 and 600  $\mu$ s). Mean values and standard deviation are shown. Different letter indicates different least significant difference intervals (p<0.05).



Figure 5. Retention of the antioxidant capacity (AC) of orange peel after drying at 50 °C without and with (US) ultrasound (20.5 kW/m<sup>3</sup>) application and PEF pretreatment (200 and 600  $\mu$ s). Mean values and standard deviation are shown. Different letter indicates different least significant difference intervals (p<0.05).

-			
	Experiment	PEF pretreatment time	US application during drying
	code	$(\Box s)$	$(20.5 \text{ kW/m}^3)$
	HAD	0	No
	HAD-US	0	Yes
	HAD-200 μs	200	No
	HAD-US-200 µs	200	Yes
	HAD-600 μs	600	No
	HAD-US-600 µs	600	Yes

707 Table 1. Conditions tested in drying experiments

711 Table 2. CIELAB color parameters ( $L^*$ ,  $a^*$  and  $b^*$ ) and chroma ( $C^*$ ) of orange peel dried 712 at 50 °C without (HAD) or with (20.5 kW/m<sup>3</sup>) ultrasound (US) application and PEF (200

at 50 °C without (HAD) or with (20.5 kW/m <sup>3</sup> ) ultrasound (US) application and PEF (200					
reatment	*	*	4		
$L^*$	a	$b^*$	$C^*$		
$64.17\pm1.77^{\rm a}$	$25.93\pm1.36^{\rm a}$	$39.91\pm3.76^{\rm a}$	$47.60 \pm 3.86$		
$62.32\pm0.63^{ac}$	$27.30\pm0.97^{\rm a}$	$33.53\pm0.68^{\text{b}}$	$43.24\pm0.82$		
$45.61\pm2.34^{\text{b}}$	$11.88\pm4.60^{\text{b}}$	$9.20\pm3.87^{\rm c}$	$34.90 \pm 1.33$		
$62.28\pm1.43^{\text{ac}}$	$27.61 \pm 1.05^{\mathrm{a}}$	$33.96\pm2.05^{\text{b}}$	$43.78\pm2.00$		
$61.98 \pm 1.45^{\text{ac}}$	$27.50\pm0.90^{\rm a}$	$31.45\pm1.66^{\text{bd}}$	$41.80 \pm 1.33$		
$60.61 \pm 1.50^{\circ}$	$27.28 \pm 1.29^{\rm a}$	$28.17 \pm 2.07^{d}$	$39.22 \pm 2.20$		
$60.98\pm2.18^{\text{ac}}$	$26.39\pm0.80^{\rm a}$	$30.21 \pm 1.44^{\text{d}}$	$40.12 \pm 1.10$		
	AD) or with (20.5 reatment $L^*$ $64.17 \pm 1.77^a$ $62.32 \pm 0.63^{ac}$ $45.61 \pm 2.34^b$ $62.28 \pm 1.43^{ac}$ $61.98 \pm 1.45^{ac}$ $60.61 \pm 1.50^c$ $60.98 \pm 2.18^{ac}$	AD) or with (20.5 kW/m <sup>3</sup> ) ultrasour reatment $ \frac{L^{*}}{64.17 \pm 1.77^{a}} 25.93 \pm 1.36^{a} \\ 62.32 \pm 0.63^{ac} 27.30 \pm 0.97^{a} \\ 45.61 \pm 2.34^{b} 11.88 \pm 4.60^{b} \\ 62.28 \pm 1.43^{ac} 27.61 \pm 1.05^{a} \\ 61.98 \pm 1.45^{ac} 27.50 \pm 0.90^{a} \\ 60.61 \pm 1.50^{c} 27.28 \pm 1.29^{a} \\ 60.98 \pm 2.18^{ac} 26.39 \pm 0.80^{a} $	AD) or with (20.5 kW/m <sup>3</sup> ) ultrasound (US) application reatment $ \frac{L^{*}}{64.17 \pm 1.77^{a}} \frac{25.93 \pm 1.36^{a}}{27.30 \pm 0.97^{a}} \frac{39.91 \pm 3.76^{a}}{33.53 \pm 0.68^{b}} $ $ \frac{45.61 \pm 2.34^{b}}{11.88 \pm 4.60^{b}} \frac{9.20 \pm 3.87^{c}}{9.20 \pm 3.87^{c}} $ $ \frac{62.28 \pm 1.43^{ac}}{61.98 \pm 1.45^{ac}} \frac{27.50 \pm 0.90^{a}}{27.50 \pm 0.90^{a}} \frac{31.45 \pm 1.66^{bd}}{31.45 \pm 1.66^{bd}} $ $ \frac{60.61 \pm 1.50^{c}}{60.98 \pm 2.18^{ac}} \frac{26.39 \pm 0.80^{a}}{30.21 \pm 1.44^{d}} $		

714 Same letters in each column show homogeneous groups determined by least significant difference intervals
 715 (p<0.05).</li>