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# Physicochemical, Functional and Bioactive Properties of Pea (Pisum sativum L.) Pods Microwave and Convective Dried Powders

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# **Research Article**

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1	Physicochemical, functional and bioactive properties of pea (Pisum sativum L.) pods
2	microwave and convective dried powders
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### 36 Abstract

37 As the primary by-product of pea processing industry, pea pods represent a rich source of various high-value 38 molecules. The aim of this work is to investigate the effect of nine different microwave output powers (200 to 39 1000 W) and five different ventilated oven temperatures (40, 60, 80, 100 and 120 °C) on the quality of pea pod 40 such as physical-chemical (water activity, total color change), functional properties (water retention capacities 41 and swelling capacity) and bioactive properties (total phenolic content, total flavonoid content and antioxidant 42 capacity). The results show that microwave drying reduced significantly the drying time. Convective drying led 43 to a greater color change than microwave drying. Convective drying at 80 °C for 135.33 min appeared the best 44 process in terms of functional properties giving the highest values of water retention capacity and swelling 45 capacity. Both microwave and convective drying methods could increase phenolic and antioxidant compounds 46 content of pea pods. It was suggested that the microwave method can be a promising technology for drying the 47 pea pods due to the shorter drying time, and higher product quality. 48

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# 49 Graphical Abstract



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# 52 Statement of Novelty

53 Drying of by-products from pea processing industry was investigated as a critical step prior to their 54 valorization for energy and fuel production as well as chemicals recovery. The drying of pea pods has never been 55 investigated before which is the case of the present study. Our research work reported that microwave drying 56 technique can be used for conservation of this valuable product by preserving quality of fresh materials and to 57 economizing energy and time.

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### 1. Introduction

- 68 Currently, about 1300 million tons of food wastes are produced per year due to a lack of proper processing and 69 preservation technique [1]. On the other hand, according to the United Nations (UN) food agency, everyday 70 18,000 children die of hunger and malnutrition [2]. Therefore, proper food processing must be emphasized to 71 reduce this macrine lace
- 71 reduce this massive loss.

Peas are marketed after a simple industrial process in which the pod is removed to prepare the seed as frozen, tinned or fresh [3]. The industrial process generates abundant residues which represent an inexpensive material

that has been undervalued until now, used mainly as fertilizer or for animal feeds [4]. The pea by-products pose

75 an economic and environmental problem due to their high volumes, organic load and disposal costs [5], but they

can also be a promising source of functional compounds [6]

Pea pods are by-products emanating from pea processing industry. These pods represent a rich source of various

high-value molecules. They are very rich in dietary fiber (more than 50% content, mainly insoluble in water

79 dietary fiber), proteins, potassium and iron[7] and phenolic compounds. The latter, if derived from a low-cost

80 resource, such as green pea pods, have great potential, may find an application in the food, feed, cosmetic and

81 pharmaceutical industries.

Recent applications of pea pods include bioethanol and bio-char production [8], polysaccharide and carbohydrate recovery [9], in addition to their use as a source of dietary fibers [5]. The presence of dietary fiber and other nutritive compounds provides an important added value to pea by-products and the possibility to balance food according to nutritional requirements and consequently an advantage against the use of isolate functional ingredients[10].

87 There is a continuous demand to develop innovative approaches for the valorization of pea pod by-products by88 applying environmentally and economically sustainable processes. One of the critical steps for by-products

89 stabilization is the drying operation since, due to their high moisture content, they are highly perishable and need

- 90 to be stabilized for further processing.
- 91 In the drying industry, one of the goals is to minimize the energy consumption, both for environmental and cost
- 92 reasons [11], preserving at the same time the product quality[12], with the quality of dried biomaterial being one
- 93 of the fundamental indicators in the assessment of the drying process effectiveness[13]. In addition to the

94 nutritional quality, which should be ideally preserved, sensory properties are to be considered and evaluated,

95 either directly through a tasting, or indirectly through objective analyses, such as color, water retention and

- 96 swelling capacity, content in antioxidant compounds [14].
- 97 Conventional drying techniques are still extensively employed due to unquestionable advantages such as simple
- 98 apparatus and a very well-known drying mechanism [15], with satisfactory efficiency in the initial period of
- 99 drying caused by relatively high drying rates and large capacity [16]. But, on the other hand, a long-lasting hot
- 100 air drying has a detrimental effect on quality of the obtained product and a higher cost of operation [17].
- 101 Freeze and vacuum drying have been considered as an most effective drying methods to obtain superior quality
- 102 products, however, it is too time-consuming and high cost make those unpopular [18, 19]. For instance, freeze
- 103 drying requires a longer drying time, and the cost is about 200–500% higher than that of hot air drying [20].

104 Whereas, vacuum drying needs higher initial and operating cost to maintain low pressure throughout the drying

105 process [21]. Consequently, novel drying methods are emerging to deal with the weakness of conventional,

106 freeze and vacuum drying and create high-quality products.

107 Microwave drying, a relatively new technology, has been recommended as a fast and effective alternative drying

108 technique [22]. Microwave-assisted thermal conversion of biomass has received significant attention in recent

109 years as it is capable of improving the process efficiency and product quality through rapid volumetric heating

110 [23].

In the literature several vegetables were dried using the microwave drying methods. Some of them are; Green Bean Slices [24], asparagus [25], mushroom [26], eggplant [27] green peas [28]. As it seen from the literature the drying effect on the quality of pea pods has never been investigated before which is the case of the present study whose objectives were: (i) to determine and evaluate quality of pea pods powders obtained by convective and microwave drying methods, (ii) to compare two processes: convection drying and microwave drying of pea pods, and finally (iii) to find the most suitable drying conditions among the applied ones for achieving highquality pea pods powders.

### 118 **2.**Materials and methods

119 2.1 Raw material

Good quality fresh and matured fruits of green peas (*Pisum sativum* L.) were purchased from the local market (Bejaia, Algeria). The fruits were sorted, washed with tap water followed by distilled water to remove the dust, shelled and the two recovered half-pods cut into approximately equal parts with dimension of about 1 cm2 and 0.3 cm thickness.

### 124 2.2 Drying procedures

125 For microwave drying (MWD), a domestic microwave (Maxipower, Model MASMO23S, China) with 2450 kHz 126 working frequency was used. The microwave was equipped with cavity dimensions of  $22.5 \text{ cm} \times 37.5 \text{ cm} \times 38.6$ 127 cm and a digital control system for microwave power (the latter linearly adjustable from 100 to 1000 W) and 128 irradiation time. Approximately, 100 g of pea pods pieces were spread uniformly in a monolayer on the plate and 129 dried at nine different microwave powers (200, 300, 400, 500, 600,700, 800, 900 and 1000 W) until constant 130 weight. Conventional drying (CD) experiments were performed in a ventilated oven (Memmert, ULE-600, 131 Germany). Approximately100 g of pea pods pieces were spread uniformly in a monolayer on the oven tray 132 (38.5 cm × 27 cm) and dried at five different temperatures (40, 60, 80, 100 and 120 °C) and air velocities of 1.0 133 m/s until constant weight. Three repetitions were performed for each temperature and power, and the data 134 provided were an average of these results.

135 The dried pod pieces were crushed using an electric grinder (IKA model A11 basic), and sieved using an

- 136 automatic sieve shaker (Rheinische Str.36 model D-42781 Haan) in order to obtain a fine and homogeneous
- 137 powder. The resulting powders were stored in glass jars, hermetically sealed, protected from light, until use.
- 138

139 The moisture content (MC) of the fresh and dried pea pods samples was evaluated according to the Schulze,140 Hubbermann [29]. The results were expressed as a percentage of the wet base.

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# 143 2.1 Physicochemical properties

144 Determination of water activity (aw)

Water activity (aw) was measured at 25 °C using an AquaLab Series 4TEV water activity meter (Decagon
Devices, Inc., Pullman, Washington, United States).

### 147 Color measurement

148 The surface color of fresh and dried samples was determined by a colorimeter (Minolta CR-400, Konica 149 Minolta, Japan) in five different points on the sample surface. The CIE *Lab* color parameters were used to 150 describe the color of samples, where  $L^*$  indicates lightness,  $a^*$  indicates chromaticity on a green (–) to red (+) 151 axis and  $b^*$  indicates chromaticity on a blue (–) to yellow (+) axis. The total color change  $\Delta E$  between fresh and 152 dried product was calculated with Eq. (1)[30]: 153

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

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## 157 2.4 Functional properties

158 Swelling Capacity (SC)

The Swelling Capacity (*SC*)was analyzed by the method described by Robertson, de Monredon [31]. One hundred milligrams of dry weight of sample were hydrated in a measuring cylinder with 10mLdistilled water at room temperature. After 18 h, the final volume (mL) was recorded, and SC was expressed as volume (mL)/g of original substrate dry weight.

163 Water-Holding Capacity (WHC)

The Water-Holding Capacity (WHC) was according to the method described byLv, Liu [32]: distilled water (15 mL) was transferred into centrifuge tubes containing 250 mg of sample. The mixture was agitated and kept at room temperature for 24 h. After centrifugation at 3641rpm for 1 h, the mixture was filtrated using quantitative filter paper and residue fresh weight was recorded. WHC was expressed as grams of water per gram of dry weight, respectively.

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## 170 2.5 Bioactive properties

171 2.5.1 Total Phenolic Content (TPC)

- 172 To evaluate the total phenolic content, dried pea pods powders were extracted using an ultrasonic processor
- 173 (SonicsVCX 500, Connecticut, USA) according to the protocol proposed by Dahmoune, Boulekbache [33].
- 174 Briefly, one gram of dried pea pods powder was extracted with 40 mL (w/v) of 64% ethanol (v/v) at 77.79% of
- 175 amplitude during 15.05 min of holding time. The extracts were filtered using filter paper Whatman n°3 and
- 176 transferred to 100 mL volumetric flask.
- 177 Total phenolic content was determined spectrophotometrically using the Folin-Ciocalteu colorimetric method. A
- 178 2.5 mL sample of distilled water-diluted Folin-Ciolcateu reagent (1/10) was added with 1 mL of the extract.
- 179 After incubation for 2 min at room temperature, 2 mL of sodium carbonate  $(75g \cdot L^{-1})$  were added the mixture
- 180 was incubated for 15 min at 50 °C and finally cooled in a water-ice bath. The absorbance at 760 nm
- (SHIMADZU UV-1800, Japan) was immediately measured [34] against the blank (obtained with the same 182 protocol but with ethanol instead of the extract). Results were expressed as gallic acid equivalents content of pea
- 183 pods  $(mg_{GAE}/g_{DW})$  by means of a calibration curve obtained with a standard of gallic acid dissolved in ethanol in
- 184 the 0.02 to 0.12 mg/mL concentration range. The same extract was used to evaluate the content of flavonoids and
- 185 antioxidant activity.
- 186

181

#### 187 2.5.2 Flavonoid content

188 Flavonoid content was estimated by the AlCl<sub>3</sub> (Aluminium chloride) method [35]: 1 mL of the extract solution 189 was added to 1 ml of 2% methanolic AlCl<sub>3</sub>·6H<sub>2</sub>O. The absorbance was read 10 min later at 430 nm against a 190 blank. The flavonoid contents were expressed as equivalents (mg quercetin/gDW) by means of a calibration 191 curve obtained with standard quercetin (dissolved in ethanol concentration range 0.01to 0.06 mg/mL) treated in 192 the same conditions.

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#### 194 2.5.3 DPPH° Radical-scavenging activity assay

195 The free radical scavenging activity was determined according to the method described by Mishra, Ojha [36] and 196 Sharma and Bhat [37]. Briefly, 0.1 mL of the extract were mixed with 3.9 mL of a  $6 \times 10^{-5}$  M methanol DPPH 197 solution. The mixtures were vigorously shaken and left standing at room temperature in the dark for 30 min, and 198 the final absorbance was measured at 517 nm. The results were expressed as a percentage effect (I%) of control 199 absorbance reduction (Eq. 2)

Percentage effect (I%) = 
$$\left(\frac{Abs_{Control} - Abs_{sample}}{Abs_{Control}}\right) \times 100$$
 (2)

200 Different sample dilutions in methanol were used in order to obtain anti radical curves and calculate the 201 IC50 values, which is the extract concentration for a 50% percentage effect. The I% was plotted versus pea pods 202 powder concentration.

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#### 204 Ferric reducing power assay (FRAP) 2.5.4

205 The reducing power was performed according to the method reported byFerreira, Baptista [38]. Various 206 concentrations of pea pod ethanolic extracts (2.5 mL) were added to 2.5 mL of phosphate buffer (200 mM, pH 6.6) followed by 2.5 mL of 1% potassium ferricyanide. The reacting mixture was then incubated in a water bath at 50 °Cfor 20 minutes. The reaction was stopped by adding2.5 mL of 10% (w/v) trichloroacetic acid and the content was centrifuged at 650 rpm for 10 min. To 5 mL of the supernatant was added 5 mL of distilled water and 1 mL of 0.1% ferric chloride and the resulting mixture was vortexed and finally the absorbance was read at 700 nm. The reductive potential was expressed as EC50 (µg/mL) related to the pea pods powder corresponding to 50% absorbance intensity.

### 213 2.6 Parameter estimation and statistical methods

Three replications of each drying experiment were carried out and triplicate samples of each experiment were analyzed. All assays were carried out in triplicate and the results are expressed as mean values  $\pm$  standard deviations. The effect of microwave drying power and oven drying temperature on the estimated parameters of the best fitted model was statistically evaluated using analysis of variance (ANOVA). ANOVA was performed using JMP7 Statistics Software. Tukey's multiple range test (HSD) was used to compare means of the estimated kinetic parameters. Evaluations were based on the  $\alpha$ =0.05 level Move

220

# 221 **3.** Results and discussion

# 222 3.1 Physicochemical properties

Drying of the pea pods started with an initial moisture content around  $87.79 \pm 0.195\%$  and continued until no further changes in their mass were observed, e.g., to a final moisture content of about 10% which was then taken as the equilibrium moisture content in the next computations. The changes in MR with time during drying of the pea pods are shown in Figure 1.

227 Drying temperature and microwave power have a significant effect on drying time with drying time decreasing 228 considerably with increasing oven temperature or microwave power. The required drying time to equilibrium 229 moisture in conventional drying at 40, 60, 80, 100 and  $120^{\circ}$ C was about  $850 \pm 22.91$ ,  $206.33 \pm 4.61$ , 230  $135.33 \pm 6.42$ ,  $86.33 \pm 5.68$  and  $50.16 \pm 1.25$  min, respectively. At higher drying temperatures, the heat transfer 231 rate and mass transfer rate increase leading to faster moisture evaporation and shorter drying duration[39]. In 232 microwave drying, time to reach equilibrium moisture at 200, 300, 400, 500, 600, 700, 800, 900 and 1000 W was 233 about  $217.11 \pm 0.67$ ,  $99.05 \pm 2.04$ ,  $72.91 \pm 0.79$ ,  $53.88 \pm 0.29$ ,  $44 \pm 0.94$ ,  $37.55 \pm 0.34$ ,  $32.16 \pm 0.36$ , 234  $27.38 \pm 0.39$  and  $22.36 \pm 0.42$  min, respectively, suggesting higher mass and heat transfer rates. In fact, in MWD 235 there is a rapid MW penetration in the product volume with fast heat generation, followed by a great vapor 236 pressure difference between the center and the surface of the products, resulting in forced expulsion of gases and 237 fast water vapor diffusion[40].

238 3.1.1 Water activity

Water activity  $(a_w)$  is a critical parameter that allows determining the product stability and safety. it is worth mentioning that above the value of 0.4 the relative speed of hydrolysis, lipid oxidation, enzymatic, microbiological and Maillard reactions increases considerably [41]. The overall dry products obtained by different drying conditions were characterized by the  $a_w$  lower than 0.4 (Table 1). The lowest  $a_w$  value was reached after microwave drying at 1000 W for 22min and the highest a<sub>w</sub> value was reached for sample dried in conventional oven at 40 °C for 850 min.

245 *3.1.2 Color* 

246 The color parameters of fresh and dried pea pods including the  $L^*$ ,  $a^*$ ,  $b^*$  and  $\Delta E$ , under different drying 247 conditions, are shown in Table 2.

The  $L^*$  value increases with drying indicating formation of a darker color. A reduction in drying time reduced the lightness variation. The darker color may be due to exposure to oxygen and to enzymatic browning reaction [42]. The statistical analysis showed differences between fresh and dried pea pods at different drying conditions (p < 0.05) for the two drying methods. Also the relatively high content of sugars, proteins, and chlorophyll may favor color changes in dried pea pods [28].

253 The initial sample showed a negative  $a^*$  value indicating greenness, while it increased towards positive (red) 254 values together with the drying treatment severity in terms of high temperatures or microwave powers. The 255 statistical analysis showed significant differences between fresh and dried pea pods at different drying conditions 256 (p < 0.05) for both drying methods, except for CD at 40 °C. Temperatures above 120 °C expediting molecular 257 changes towards the creation of brown compounds as the result of diverse reactions, such as for example 258 decomposition of chlorophyll and other pigments such as carotenoid, polyphenols polymerization and Maillard 259 reaction [43] or even pigment concentration due to water reduction [44]. However, the  $b^*$  values slightly changed 260 and in conventional drying it increased more the longer the treatment revealing in this case decomposition of 261 different compounds probably more sensitive to oxygen exposition, rather than heat (such as carotenoids). In 262 microwave drying  $b^*$  variation was not so correlated with drying time.

The total color change of dried pea pods ( $\Delta E$ ), showed the same trend as  $b^*$  and lower values for MD confirming that a long-lasting hot air drying causes a meaningful color alteration and application of microwave can shorten the drying time limiting color change [13].

# 266 3.2 Functional properties

In order to evaluate possible modifications affecting the structural arrangement of cell wall polysaccharides from pea pods samples, hydration-related properties such as swelling and water retention capacity were measured on fresh and dried samples (Table3).

# 270 3.2.1 Swelling capacity (SC).

271 Convective drying decreased this property showing anyway an increase from the treatment at 40°C to 272 that at 80°C (with a value close to that of fresh sample) and then a decrease with increasing temperature. The 273 statistical analysis showed significant differences between fresh and dried pea pods for both microwave and 274 convective drying methods (p < 0.05), except for CD at 80°C. At lower temperatures, the drying time necessary 275 to decrease moisture content is considerably high affecting functional properties. On the flip side, high 276 temperatures cause some structural changes in final products, such as changes to carbohydrate granules, in 277 particular starch, protein denaturation. So, the optimal conditions for functional properties are a compromise 278 betwixt temperature and drying time [14]. For microwave drying, SC values were on average lower and the 279 increase in microwave power led to the decrease of SC until 400 W and then an increase from 500 to 1000 W. It 280 is reported that MD affects the fibrous matrix modifying the structural characteristics and the chemical

composition of the fiber (water affinity components) due to the high internal pressure produced by microwaveheating which can cause expansion and *SW* of the sample structure [45].

## 283 3.2.2 Water retention capacity (WRC).

284 In general, dehydration promoted a decrease of the WRC for all the tested conditions, with a correlation with 285 temperature / microwave power similar to that commented for SC which is expected due to the same compounds 286 involved in SC and WRC. Statistical analysis showed significant differences between fresh and dried pea pods 287 for both microwave and convective drying methods (p < 0.05). Similar investigations reported that drying 288 temperature is the main factor affecting the WRC [46]. The results for MD showed that volumetric heating 289 during microwave caused higher internal pressure at the start of the drying stage and faster evaporation, resulting 290 in cell swelling, but with the reduction of moisture content, the volume shrinkage is increased which decreases 291 the rehydration capacity[47]. According to Hodge and Osman [48], flours with high WRC have more hydrophilic 292 constituents such as fibers. Apple, wheat, pea, and carrot fibers have a WRC ranging from 2.5 to 10 g/g dry 293 weight. From a technological point of view, fibers are used as texturing agent or additive to increase volume, 294 essentially due to their capacity to absorb water [49]. Thanks to the high value of WRC, pea byproduct could be 295 utilized as a functional ingredient: to reduce calories intake, avoid syneresis, modify the viscosity and texture of 296 formulated foods.

# 297 3.3 Total polyphenolic, total flavonoids compounds and antioxidant activity

The content in antioxidant compounds and antioxidant activity of plant materials was generally lower in the fresh material in agreement with some recent studies that have demonstrated that dehydrated plant materials contain larger amounts of antioxidants when compared to fresh plant materials [50].

# 301 3.3.1 Antioxidant compounds

302 In the present study, the total polyphenols content (TPC) and total flavonoids (TFC) in ethanol extracts of pea 303 pods increased significantly after both conventional and microwave drying and with decreasing drying time. The 304 statistical analysis showed significant differences between fresh and dried pea pods at different drying conditions 305 (p < 0.05) using both drying methods, except for CD at 40 and 60 °C and MD at 200 W. This increase was 306 probably due to changes in their extractability due to the disruption of the plant cell wall(as already commented 307 for MD) enhancing the release of bound polyphenolic and flavonoid compounds [51]. Additionally, the 308 formation of Maillard reaction products may add antioxidant activity and interfere with the Folin-Ciocalteu, 309 DPPH and FRAP test. A decrease in total phenolics and flavonoids was observed only for CD at 40 and 60 °C 310 and MD at 200 W for which overexposure to atmospheric oxygen for long times might have brought to 311 compounds oxidation [52]. Another possibility concerning the low level of phenolic compounds in the fresh 312 sample could be the presence of active enzyme polyphenol oxidase which is faster inactivated at higher 313 temperatures [53].

# 314 3.3.2 Antioxidant activity

The effect of drying method on the antioxidant capacity of ethanol extracts from pea by-product was assessed by the DPPH° radical scavenging assay and the ferric-reducing power estimation. In agreement with what observed for antioxidant compounds, results showed that drying significantly enhanced the overall antioxidant activities of samples, except for the samples dried at 40 °C in CD. The maximum radical scavenging activity values were found after microwave treatment at 1000W. The ferric reducing ability of the extract showed a similar trend to 320 the results observed from the DPPH° assay. Firstly, this can be atributable to the increased amount of antioxidant 321 compounds in the extracts. High correlations were found between DPPH° and total flavonoids and total 322 phenolics. Secondly, there might also be the formation of novel compounds having antioxidant activity during 323 drying, as Maillard compounds[54].

### 324 **4.** Conclusion

- 325 In this study, the effects of microwave output powers and oven temperatures on the different factors including 326 color, functional and bioactive properties of pea pods were investigated to find suitable drying conditions among 327 the applied ones for this valuable product. The main findings of this study can be summarized as:
- 328 ✓ Increasing the microwave power level or drying temperature resulted in a reduction in drying time and
   329 that this reduction is more significant when applying microwave drying.
- 330  $\checkmark$  The lowest water activity  $a_w$  value was reached after microwave drying at 1000 W for 22min.
- 331 ✓ The convective dried pea pods samples exhibited a greater color change than microwave dried pea pods
   332 samples.
- 333 ✓ Conventional drying at 80 °C during 135.33 min was the most efficient combination that provided the
   334 highest swelling and water retention capacity.
- 335 ✓ Drying could increase functional compounds, in terms of extractable phenolic and antioxidant
   336 compounds, with microwave drying at 1000 W for 22.36 min appearing the best process from this
   337 point of view.
- 338

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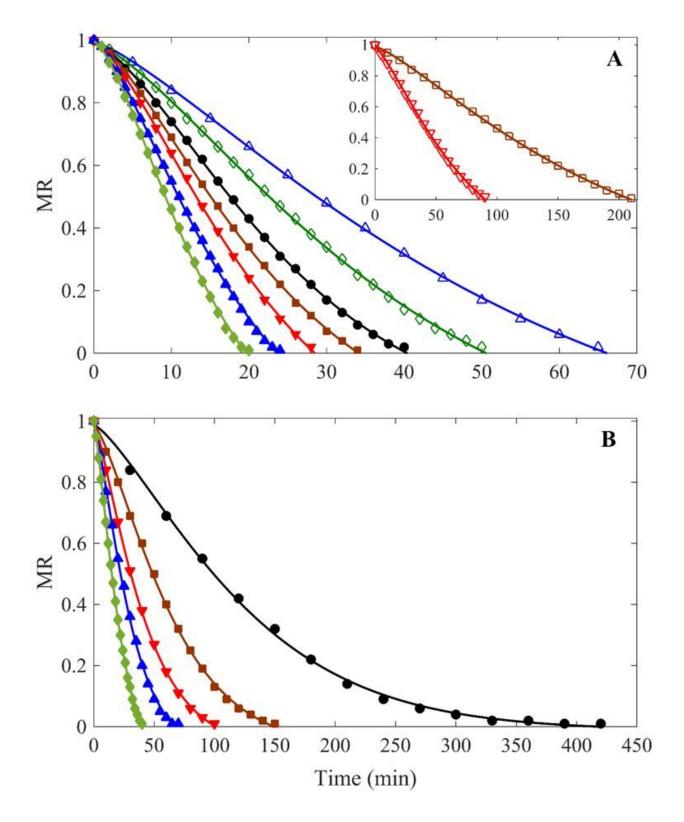
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- 488 Figure Legends
- 489 Fig. 1– Experimental (symbols) and simulated (solid lines) decrease in moisture ratio (MR) of pea pods during
- 490 microwave drying (A) at different powers ( $\Box 200, \nabla 300, \Delta 400, \diamond 500, \bullet 600, \blacksquare 700, \lor 800, \blacktriangle 900$  and
- 491  $\Box$  1000 W) and oven drying (**B**) at different temperatures (• 40,  $\blacksquare$  60,  $\checkmark$  80,  $\blacktriangle$  100 and  $\Box$  120 °C). Solid lines
- 492 are calculated MR using the Midilli *et al.* model.
- 493

# Figures



# Figure 1

Experimental (symbols) and simulated (solid lines) decrease in moisture ratio (MR) of pea pods during microwave drying (A) at different powers ( $\[mathbb{N}\] 200, \[mathbb{N}\] 300, \[mathbb{\Delta}\] 400, \[mathbb{N}\] 500, \[mathbb{N}\] 600, \[mathbb{N}\] 700, \[mathbb{N}\] 800, \[mathbb{\Delta}\] 900$  and \[mathbb{N}\] 1000

W) and oven drying (B) at different temperatures ( $\[M]$  40,  $\[M]$  60,  $\[M]$  80,  $\[D]$  100 and  $\[M]$  120 °C). Solid lines are calculated MR using the Midilli et al. model.

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