



**HAL**  
open science

## Reconstitution of fruit powders: A process – structure – function approach

Naïma Gaudel, Claire Gaiani, Yogesh Harshe, Jana Kammerhofer, Matthieu Pouzot, Stephane Desobry, Jennifer Burgain

### ► To cite this version:

Naïma Gaudel, Claire Gaiani, Yogesh Harshe, Jana Kammerhofer, Matthieu Pouzot, et al.. Reconstitution of fruit powders: A process – structure – function approach. *Journal of Food Engineering*, 2022, 315, pp.110800. 10.1016/j.jfoodeng.2021.110800 . hal-03664698

**HAL Id: hal-03664698**

<https://hal.univ-lorraine.fr/hal-03664698v1>

Submitted on 16 Oct 2023

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial 4.0 International License

1                    **Reconstitution of fruit powders: a process – structure – function approach**

2                    Naïma GAUDEL<sup>a</sup>, Claire GAIANI<sup>a</sup>, Yogesh M. HARSHE<sup>b</sup>, Jana KAMMERHOFER<sup>b</sup>, Matthieu

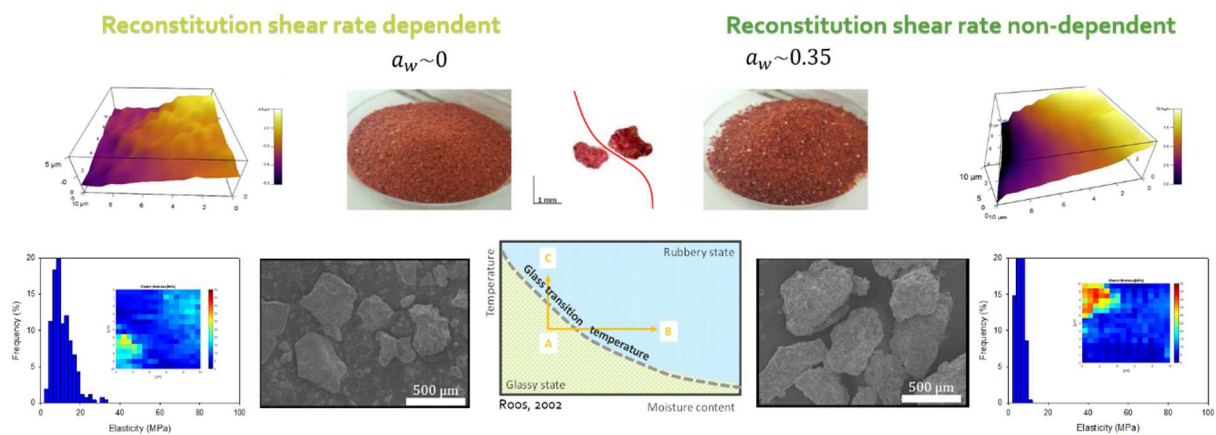
3                    POUZOT<sup>b</sup>, Stephane DESOBRY<sup>a</sup>, Jennifer BURGAIN<sup>a\*</sup>

4                    <sup>a</sup>Université de Lorraine, LIBio, F-54000 Nancy, France

5                    <sup>b</sup>Nestlé Research, P.O. Box 44, CH-1000 Lausanne 26, Switzerland

6                    \*Corresponding author: [jennifer.burgain@univ-lorraine.fr](mailto:jennifer.burgain@univ-lorraine.fr)

7  
8                    **Graphical Abstract**



10                    Schematic explanation of the relationship between water activity and fruit powders behavior  
11                    during reconstitution.

12                    **Abstract**

13                    Reconstitution of six fruit powders, compacted and non-compacted, were studied. It appears  
14                    that powder reconstitution behavior under sheared conditions was significantly different with  
15                    regards to the water activity ( $a_w$ ). At low  $a_w$ , the powders exhibit a glassy state with a stiff  
16                    surface measured by Atomic Force Microscopy and porous/angular shapes were observed by

17 Scanning Electron Microscopy. In that case, increasing the shear rate accelerated the kinetics  
18 of reconstitution by improving water penetration through particle surface pores thus  
19 reducing the reconstitution time. Therefore, the reconstitution is shear rate dependent. At  
20 high  $a_w$ , the powders are under a rubbery state with a soft surface and a smooth shape.  
21 Under these environmental conditions (i.e. above powder's glass transition temperature),  
22 increasing the shear rate did not improve their reconstitution. Therefore, the reconstitution  
23 kinetics is not shear-dependent in the stirring rate studied (from 800 to 1500 rpm). It is  
24 suggested that the viscous layer created on the particle surface blocks the entrance of water  
25 for these powders rich in sugar and presenting low glass transition temperatures.

#### 26 Key words

27 Fruit powder; reconstitution; shear rate; glass transition temperature; compaction; water  
28 activity

29

#### 30 1 Introduction

31 Fresh fruits are usually dried as they contain about 90 % water by weight and reducing the  
32 water activity in the product allows for food preservation (i.e. reducing microbial and  
33 enzymatic activities). Indeed, browning and loss of nutritional value are typical of fresh fruits  
34 even at ambient temperature and relative humidity. In the powder form, the stability and the  
35 functionality of the ingredients composing fruits are maintained (Fitzpatrick and Ahrné, 2005).  
36 However, fruit powders contain a high quantity of low molar mass sugars with low glass  
37 transition temperature ( $T_g$ ) (Fang and Bhandari, 2011). The direct consequence is that fruit  
38 powders are highly hygroscopic and sticky at high temperatures but also at ambient  
39 temperature if the water content is not well mastered. This feature causes the powder

40 adhesion to surfaces and powder caking during the storage, which affects the quality of the  
41 final product (Gabas et al., 2007; Fabra et al., 2011). The  $T_g$  is one of the most important  
42 parameters to take into account during powder storage. Indeed, the phenomenon of glass  
43 transition is the gradual and reversible transition of amorphous materials from a hard and  
44 "glassy" state into a viscous and rubbery state as the temperature and/or the moisture  
45 content is increased. The glass transition is also linked to the water activity and water as a  
46 strong plasticizer decreases the  $T_g$ . Also, the glass transition temperature depends on the  
47 molecular mass as the  $T_g$  of monosaccharides, disaccharides, oligosaccharides and  
48 polysaccharides increases with increasing their molecular mass (Roos, 2002). If the glass  
49 transition is reached during powder storage, unexpected phenomena can happen that affect  
50 powder functionality such as reconstitution ability. One way of influencing this powder  
51 functionality is the granulation process.

52 Granulation is a particle size-enlargement process carried out to improve flowability,  
53 compaction and homogeneity of powders. This is an important process in many industries  
54 including the food, pharmaceuticals and chemical industries. Roller compaction is a process  
55 used for dry granulation typically employed in the food industry to increase the flowability of  
56 a powder by forming granules of large size and density (Kleinebudde, 2004). During this  
57 processing step, a powder blend passes through two counter-rotating rolls that compact the  
58 blend into ribbons. The ribbons subsequently are milled into granules of a desired size  
59 distribution. This granulation process induces structural and physical changes by increasing  
60 the size of the particles and the voids between particles, but also by playing on powder bulk  
61 properties, especially by decreasing the bulk density (Rajniak et al., 2007). As a consequence,  
62 the wetting step is improved by the production of particles with large pores, which facilitate  
63 the entrance of water into particles (Hogekamp and Schubert, 2003). Dispersibility is also

64 related to the increased particle size (Goalard et al., 2006). Thus, granulation will impact the  
65 reconstitution ability.

66

67 Reconstitution of powdered ingredients is of particular importance to both manufacturers  
68 and consumers as a target of consumption quality (Fang et al., 2011). Reconstitution is highly  
69 dependent of both operating conditions and material properties. The objective is to restore  
70 the fresh product functional properties, especially for heat-sensitive food products such as  
71 vegetables and fruits powders (Lopez-Quiroga et al., 2019). Reconstitution of food powder  
72 generally consists of four steps or phases: wetting of powder particles, sinking, dispersing,  
73 and particles completely dissolving in solution (Forny et al., 2011; Mitchell et al., 2015).  
74 Wetting is the first step where the particles contact liquid, the physicochemical properties of  
75 particle surface is of major importance for improving initial step. Dispersing and dissolution  
76 are the critical phases where primary particles start to release materials from the particle  
77 surface into the liquid. Once any of these steps is limited, the time for the whole  
78 reconstitution is prolonged. Particularly, in the case of fruit powders, the dissolution cannot  
79 be fully achieved due to the presence of many insoluble polysaccharides. Additionally,  
80 different steps can overlap that makes understanding the global rehydration process more  
81 complicated (Schubert, 1993).

82 The influence of reconstitution conditions (stirrer speed, mixing systems, temperature, and  
83 viscosity) is also known to influence the reconstitution time. For dairy powders, authors  
84 (Jeantet et al., 2010; Andre et al., 2012) show that increasing temperature and/or agitation  
85 resulted systematically in decreasing the reconstitution time. Also, their respective influences  
86 were compared and increasing the temperature by 4 °C was found to have the same

87 influence on reconstitution time than multiplying by two the stirring speed. On the best  
88 knowledge of the authors, this type of study crossing the impact of the process parameters  
89 were never done on fruit powders. Because of various origins, particle size and shape  
90 distributions, chemical composition, surface composition, and physical properties,  
91 reconstitution is highly variable. Again, many data were acquired for dairy powders (Gaiani et  
92 al., 2006, 2007a, 2007b, 2009), hydrocolloids (Wangler and Kohlus, 2018), biopolymers  
93 (Wangler et al., 2019), but not yet for fruit powders.

94 With regard to process, powder composition and structure complexity, knowledge of fruit  
95 powder reconstitution is clearly insufficient in comparison with other food powders (*i.e.* dairy  
96 powders). Therefore, the objectives of this study were to investigate the impact of structure  
97 (compacted or non-compacted), composition (various fruits), stirring speed and material state  
98 (glassy vs rubbery depending on the  $a_w$ ) on fruit powder reconstitution.

99

## 100 2 Material and methods

### 101 2.1 *Fruit powders*

102 Six industrial fruit powders of different nature were analyzed (**Figure 1**):

- 103 ○ Fruit-based powders non-compacted and compacted, denoted FNC and FC,  
104 respectively.
- 105 ○ Red mix powders non-compacted and compacted, denoted RNC and RC, respectively.
- 106 ○ Purple mix powders non-compacted and compacted, denoted PNC and PC,  
107 respectively.

108 Each formulation contains different mixes of fruit powders. To obtain compacted powders,  
109 each mix of non-compacted powders had undergone a compaction process to form larger

110 particles. Therefore, for one similar chemical composition, two powders were studied: non-  
111 compacted (NC) and compacted (C).

112

## 113 **2.2 Storage conditions**

114 All the powders were stored at two conditions that are  $a_w \sim 0$  and  $a_w \sim 0.35$  before using  
115 them for experiments. In order to maintain a low water activity (*i.e.*  $a_w \sim 0$ ) and prevent  
116 powder caking, powders were stored at 20 °C in a desiccator (Nalgene cabinets) containing  
117 phosphorus pentoxide ( $P_2O_5$  from Sigma Aldrich) as drying agent. For the second condition,  
118 powders were stored at 20 °C in another desiccator containing magnesium chloride salts  
119 ( $MgCl_2$  from Sigma Aldrich) to maintain an  $a_w \sim 0.35$ , in order to mimic ambient conditions.

120 The value of water activity was often verified and determined using a Hygropalm HP23-AW  
121 (Omni Instruments, UK) before using the powders for experiments. This device, calibrated  
122 using salts whose water activity is known, has a measuring range equals 0 to 1 with an  
123 accuracy of  $\pm 0.008$ , over a temperature range comprised between  $-10$  °C and  $60$  °C. A  
124 sample of powder (typically around 15 g) was poured into a polypropylene container, and the  
125 water activity measurements were realized using an electrolytic sensor. Three repetitions  
126 were performed for each sample.

127

## 128 **2.3 Surface properties**

### 129 **2.3.1 Optical imaging**

130 The influence of water activity on visual properties was evaluated with classical imaging.  
131 Pictures of the fruit powders studied were taken with a classic camera. Pictures with an optic  
132 microscope (Dinolite, 5 Megapixel Edge sensor, 10 – 200× magnification, polarizer and  
133 flexible LED control) were also recorded.

### 134 2.3.2 Scanning Electron Microscopy

135 A high-resolution field-emission scanning electron microscope (SEM) type JEOL JSM-7100F  
136 supplied with a hot (Schottky) electron gun (JEOL Ltd., Tokyo, Japan) and having a resolution  
137 around 1 nm at 30 kV was used to investigate the surface morphology and topography of fruit  
138 powders at two different water activity values. The equipment was operated at 1 kV. Samples  
139 were mounted onto SEM stubs by fixing them on a carbon double-sided adhesive tape.  
140 Finally, a coating of iridium was applied to the samples until reaching around 10 nm coating  
141 thickness.

### 142 2.3.3 Atomic force microscopy (AFM)

143 Prior to AFM experiments, fruit powders were glued onto the surface of circular glass. The  
144 surface topography of fruit powder particles was probed in air with a MFP3D-BIO instrument  
145 (Asylum Research Technology, Oxford Instrument, Manheim, Germany) using gold coated  
146 AFM-tips (NPG, Bruker SAS, Palaiseau France). The used scan area was 10  $\mu\text{m}$   $\times$  10  $\mu\text{m}$   
147 (corresponding to 512 lines). Mechanical properties of individual particles were probed at the  
148 nanoscale by AFM nanoindentation technique. Measurements were performed in air and at  
149 room temperature with stiff cantilevers (MPP-13100 with 200 N/m spring constant). The  
150 cantilever was calibrated onto sapphire substrate. The force-distance curves recorded by AFM  
151 were analyzed according to the theoretical Sneddon model to extract the mechanical  
152 properties (*e.g.* elasticity).

153

### 154 2.3.4 X-ray photoelectron spectroscopy

155 The elemental surface composition of FNC and FC (up to 10 nm depth) was measured by X-  
156 ray Photoelectron Spectroscopy (XPS) (Gaiani, 2006; Rouxhet et al., 2008). Before analysis,



157 the samples were outgassed under vacuum for 24 h. Spectra were obtained using a KRATOS  
158 Axis Ultra X-ray photoelectron spectrometer (Kratos Analytical, Manchester, UK) equipped  
159 with a mono-chromatic Al K $\alpha$  X-ray ( $h\nu = 1\,486.6$  eV) operated at 150 W. The analysis area  
160 equaled 700  $\mu\text{m} \times 300 \mu\text{m}$  and spectra were collected at normal take-off angle 90°. Four  
161 repetitions were performed for each sample.

## 162 **2.4 Physical and chemical properties**

### 163 2.4.1 Differential scanning calorimetry

164 Differential scanning calorimetry (DSC 2500, Discovery DSC Series, TA Instruments)  
165 measurements were used to investigate the glass transition in fruit powders. Samples of  
166 powder, pre-weighed (around 10 mg), were sealed in Tzero aluminum hermetic pans with a  
167 mechanical crimp. These pans were placed in parallel to an empty capsule, which was the  
168 reference. The samples were under protective nitrogen and the procedure was fixed to  
169 perform a first round of heating from -20 °C to 50 °C at 10 °C.min<sup>-1</sup> rate, followed by a cooling  
170 from 50 °C to -20 °C at the same rate. A second heating from -20 °C to 50 °C at 10 °C.min<sup>-1</sup>  
171 rate was then performed, and the glass transition temperature ( $T_g$ ) extracted from this  
172 heating were studied. Two repetitions at least were performed for each sample.

### 173 2.4.2 Particle size distribution

174 The particle size distribution was obtained using a laser diffraction granulometer (Mastersizer  
175 3000, Malvern Instruments, UK) equipped with Aero S dry powder dispersion unit. All samples  
176 were dispersed at 1 bar air pressure. The feed rate and the hopper length values were  
177 adjusted to obtain a correct obscuration between 1 and 15 %. The particle size distribution  
178 was calculated from Mie theory, and provided as a volume density function. The particle size  
179 estimator was the  $d_{50}$ , which means that 50 % of particles have a lower diameter. The span  
180 value was used as a polydispersity indicator, and is given by:

181 **Equation 1** 
$$span = \frac{d_{90} - d_{10}}{d_{50}}$$

182 where 10 %, 50 % and 90 % of the sample particles are smaller than the diameter  $d_{10}$ ,  $d_{50}$   
183 and  $d_{90}$ , respectively. Three repetitions were performed for each sample.

## 184 **2.5 Reconstitution properties**

### 185 2.5.1 Conductivity measurements during reconstitution

186 The time evolution of the conductivity was obtained using a portable conductivity S7 -  
187 Standard Kit with a sensor (INLAB 738 ISM from Mettler Toledo Seven2Go). The calibration of  
188 the sensor was made at  $T = 25\text{ }^{\circ}\text{C}$  with a calibration liquid at a conductivity  $1\,413\text{ }\mu\text{S}\cdot\text{cm}^{-1}$ . The  
189 time duration of the measurement was adapted depending on the sample. Conductivity  
190 measurements were realized in a beaker of 200 mL that was put in water bath with circulating  
191 water supply in order to have a constant and fixed temperature ( $25\text{ }^{\circ}\text{C}$ ). A volume of 100 mL  
192 of water was poured into the beaker and the powders were reconstituted under specific TS  
193 conditions ( $10 \pm 0.1\text{ g}$ ). The powders were blended into water, thanks to a bar magnet of  
194 length 3 cm, with a magnetic stirrer in a range of stirring speed values (from 800 to 1 500  
195 rpm). Three repetitions were performed for each sample.

### 196 2.5.2 Particle size distribution during reconstitution

197 The particle size during reconstitution was followed using the laser diffraction granulometer  
198 (Mastersizer 3000, Malvern Instruments, UK) equipped with the Hydro MV unit. The particles  
199 were dispersed in 120 mL dispersant volume with a stirrer ( $0 - 3\,500 \pm 10\text{ rpm}$ ). The  
200 dispersant used was distilled water. The samples passed through a measuring cell using a  
201 centrifugal pump (flow rate  $Q = 0 - 2\text{ L}\cdot\text{min}^{-1}$ ). The particle size was probed with a sequential  
202 combination of measurements with red and blue light sources. Experiments were conducted  
203 with powders stored at  $a_w \sim 0$  and  $a_w \sim 0.35$ . The stirring speed was fixed at 2 000 rpm, and

204 water temperature at  $T = 24 \pm 2$  °C. For one measurement, the integration time of each light  
205 source was adapted depending on the sample and its kinetics (typically between 1 and 5 sec)  
206 in order to have reliable and repeatable results. The time variation of particle size  
207 distributions was assessed choosing the number of measurements and the delay between  
208 each one, depending on the sample. The mass of the studied samples, typically between 0.02  
209 and 0.30 mg, were adapted to obtain obscuration between 1 and 30 % depending on the  
210 particle size. For instance, the mass of powder rehydrated into the device was chosen at  $m =$   
211 0.15 g and  $m = 0.30$  g for FNC and FC, respectively. Two repetitions were performed for each  
212 sample.

213

## 214 3 Results and discussion

### 215 **3.1 Particle size, shape and visual aspects**

216 The particle size distributions of the six fruit powders (three fruit compositions and two  
217 structures per composition) were measured at two water activity values (**Figure 2**).  
218 Information related to these functions such as the mean diameter  $d_{50}$ , the **span** value and  
219 the percentage increase of  $d_{50}$  between two  $a_w$  values are presented in **Table 1**. Visual/optic  
220 microscopy and electronic microscopy images are presented in **Figures 3** and **4**, respectively.

221 **Impact of compaction.** The compaction process leads systematically to a high mean diameter,  
222 and also produces only monodisperse population of particles even if the non-compacted  
223 powder has bimodal distribution (for instance, FNC/FNC and RNC/RC powders). From images,  
224 non-compacted powders seem to have a particle size around a hundred microns, which was  
225 confirmed by the determination of the  $d_{50}$ . Also, compaction process seems to produce more

226 homogeneous size distribution (images), which is confirmed by a smaller **span** value. Finally,  
227 images suggest that compaction process led to angular shapes particles with high rugosity.

228 ***Impact of water activity.*** From size measurements, increasing the water activity value leads to  
229 an increase of the mean diameter, and a decrease in **span** value. This can be explained by the  
230 apparition of two populations: the lowest size population represents the increase in mean  
231 diameter with increasing  $a_w$ , while the higher size population (around  $d \sim 1\ 000\ \mu\text{m}$ ) is due to  
232 the agglomeration of individual particles. An increase in moisture content leading to an  
233 increase in free volume due to the formation of hydrogen bonds may also impact the particle  
234 size (Jadhav et al., 2009; Moghbeli et al., 2019). The non-compacted powders are much more  
235 sensitive to the impact of water activity regarding their mean diameter, in comparison with  
236 the compacted ones as shown by the percentage increase of  $d_{50}$ . Moreover, for compacted  
237 powders, the greater the diameter at  $a_w \sim 0$ , the less it increases with increasing  $a_w$ . Images  
238 suggest a smoother surface at  $a_w \sim 0.35$ , in comparison with the angular shape observed at  
239  $a_w \sim 0$ . This suggests a sticky surface that may explain the agglomerates for non-compacted  
240 powders, and a change in powder surface. Increasing water activity value seems to change  
241 the powder surface and the particle size (Liu et al., 2010). Although it is not visible using SEM  
242 imaging due to the coating, this could also change the porosity at the surface of the particles,  
243 and thus influencing the reconstitution properties (entrance of water into the structure).  
244 Finally, optical images also show a clear difference in color when increasing the water activity  
245 value, in agreement with Jiang et al. (Jiang et al., 2013). These pictures suggest a change in  
246 surface properties between  $a_w \sim 0$  and  $a_w \sim 0.35$ , that may impact later the reconstitution by  
247 clogging pores where water could penetrate. This aspect will be discussed later in the results  
248 and discussion part.

## 249 3.2 Powder surface characterization

250 **Impact of compaction.** XPS analysis was performed on FNC and FC in order to evaluate the  
251 impact of the compaction process on the surface composition for powders having similar bulk  
252 composition. Three common chemical elements were detected at the surface of the powders:  
253 carbon C, oxygen O, nitrogen N with mineral traces (K and P). The detection of nitrogen  
254 suggests the presence of proteins at the surface. For FNC powder, minor elements were  
255 detected in very low concentration: potassium K and phosphorus P (**Figure 5a**). Regarding the  
256 results of the surface atomic concentration of chemical elements, there is no impact of the  
257 compaction process on the surface composition of the studied powders. Moreover, the  
258 analysis of carbon bonds (**Figure 5b**) reveals again no significant influence of the compaction  
259 process on the carbon element. Almost the same atomic concentration of C-C bonds and C-O  
260 bonds (50 % - 50 %) is observed. This suggests that fruit powder surfaces are mainly  
261 composed of polysaccharides. Ratios between carbon element and oxygen element, and C-C  
262 bonds and other C bonds (C-O, C=O, O-C=O) were calculated (**Figure 5c**). These ratios give  
263 information about the hydrophilic/ hydrophobic nature of the powder surface. It appears that  
264 the surface is mostly similar and that the compaction had no impact measured by XPS. The  
265 XPS results suggested that potential differences in reconstitution properties should not come  
266 from a chemical point of view in term of surface composition (Nijdam and Langrish, 2006;  
267 Murrieta-Pazos et al., 2012).

268 **Impact of water activity.** To go further and quantify the impact of water activity on the surface  
269 of fruit powders, surface topography and nanomechanics were performed by AFM (**Figure 6**).  
270 Regarding the measurements at  $a_w \sim 0.35$ , it was very difficult to keep the contact between  
271 the AFM tip and the surface, probably due to a thin layer of water present at the surface as  
272 already reported in literature (Eaton and West, 2010). For nanoindentation, it can be seen

273 that the distribution is centered at 10 MPa for the powders stored at  $a_w \sim 0$  and a shift to  
274 lower values (5 MPa) was observed for the powders stored at  $a_w \sim 0.35$ . This means that  
275 powders stored at higher water activity present a softer surface compared to the ones stored  
276 at  $a_w \sim 0$ . The elasticity maps reveal that some regions are stiffer than the rest of the surface  
277 meaning that nanomechanical properties are inhomogeneous at the particle surface. It  
278 appears that increasing water activity impacts the surface properties of powders. DSC  
279 measurements were obtained for the six fruit powders studied at two  $a_w$ , in order to  
280 determine the influence of water activity on the glass transition temperature evolution (**Table**  
281 **2**). For all powders, a glass transition clearly appeared and the endpoint glass transition  
282 temperature ( $T_g$ ) was extracted. From a general point of view, glass transition occurred at  
283 lower temperature for non-compacted powders than for compacted ones. This result may be  
284 due to both an increase in surrounding pressure and a decrease in water content as already  
285 reported in literature (Jadhav et al., 2009; Lopez-Quiroga et al., 2019; Moghbeli et al., 2019).  
286 Furthermore, a significant decrease of the glass transition temperature  $T_g$  was observed with  
287 increasing water activity value, which could be explained by an increase in free volume (Roos,  
288 2002). The most important result is that all powders (whatever the compaction and the  
289 relative activity) are above or slightly below their glass transition at ambient temperature  
290 ( $T=20$  °C). At  $a_w=0$ , some samples are in the glassy state (FC, PNC and PC powders) and some  
291 (FNC, RNC, RC powders) are just above  $T_g$ , leading to a transition from a glassy state to a  
292 rubbery state. As these samples are close to the transition from glassy to rubbery state, in  
293 these conditions, at microscopic scale the matrix is partly at rubbery and partly at glassy state.  
294 At  $a_w=0.35$ , all samples are at the rubbery state. Also, for material containing sugars, like fruit  
295 powders, this transition from brittle solids to sticky materials is responsible for caking and  
296 particle size increase as previously observed by visual and microscopy images.

297 Therefore, powders at  $a_w \sim 0.35$  are under a rubbery state whereas the one maintained at  
298 low  $a_w$  are partly under a glassy state. As seen in **Figures 3** and **4** and already discussed,  
299 particle surface is rougher and more angular at  $a_w \sim 0$ . It confirms that a material transition  
300 occurred; this transition being correlated to a modification of the nanomechanical properties  
301 at the particle surface. These surface changes should influence the reconstitution, particularly  
302 concerning the initial interaction between particle and water, during the wetting and swelling  
303 steps.

304

### 305 **3.3 Powders reconstitution ability**

306 **Conductivity measurements.** The conductivity values were followed with time during  
307 reconstitution of the six studied powders at a water activity  $\sim 0$  and  $\sim 0.35$  (**Figure 7**). An  
308 example of the typical time evolution of the conductivity and the extraction of the  
309 reconstitution time ( $\tau_R$ ) are plotted in **Supplementary material 1**. The conductivity value  
310 quickly increases and then reaches a constant value. The reconstitution time was then  
311 evaluated by considering that the reconstitution is reached when the conductivity value  
312 equals 90 % of the maximum conductivity value (Mitchell et al., 2015, 2019).

313 The non-compacted powders have systematically faster reconstitution than the compacted  
314 ones, thus resulting in shorter reconstitution times. This is mainly due to the particle size,  
315 which is higher for the compacted powder and add a step of disintegration of the granules.  
316 However, two distinct behaviors appear concerning the evolution of  $\tau_R$  with shear, which  
317 seems to depend on the water activity value. At low water activity, the evolution of  $\tau_R$  with  
318 the stirring speed, obtained by conductivity measurements, exhibits that an increase in  
319 stirring speed leads to a decrease of  $\tau_R$ . This comes from a better water entrance through  
320 particle surface porosity and therefore a faster reconstitution of particles under applied

321 shear. This result is usually observed by authors for food powders and more particularly for  
322 dairy powders (Andre et al., 2012). But at a higher water activity ( $\sim 0.35$ ), the behavior during  
323 reconstitution is significantly and systematically changed. Although reconstitution times are  
324 higher for compacted powders than for non-compacted,  $\tau_R$  does not evolve significantly with  
325 shear for a given fruit powder, in the range of stirring speed studied (800 – 1 500 rpm).  
326 Therefore, it is observed that the reconstitution of these six powders is shear dependent at  
327 low relative humidity and non-shear dependent at a higher relative humidity. This result was  
328 totally unexpected and to the best of our knowledge, it was never observed until now. This  
329 could come from the fact that powder surface at  $a_w \sim 0.35$  is partially hydrated leading to a  
330 swelling of the particles that is reflected by the particle size increase. In that case, the limiting  
331 factor for powder reconstitution is mainly the diffusion of water inside the structure. This  
332 could also explain the observation that for some powders (FNC, RC, PNC and PC) and at low  
333 stirring speed value ( $N = 750 - 800$  rpm),  $\tau_R$  at  $a_w \sim 0$  is lower than  $\tau_R$  at  $a_w \sim 0.35$ . These  
334 results clearly put in evidence that increasing water activity value leads to a non-dependence  
335 of shear on the reconstitution time, in the range of stirring speed studied.

336 **Particle size distribution (PSD).** The experiments of reconstitution by particle sizing were  
337 performed for FNC and FC powders in order to confirm the results obtained by conductivity  
338 measurements and verify the final state of the powders. The results of the time evolution of  
339 the volume density functions are presented in the form of a map with both the initial and the  
340 final PSD at  $a_w \sim 0$  and  $a_w \sim 0.35$  for FNC and FC (**Figure 8**). Although a larger particle size at  
341 the beginning when increasing the water activity, a given composition powder leads to an  
342 identical final volume density function, irrespective of the initial particle structure (compacted  
343 or non-compacted) and the water activity value. Rather, these two parameters (structure and  
344  $a_w$ ) influence the reconstitution time. It can also be noticed that the initial and final volume



345 density functions of FNC at  $a_w \sim 0$  are almost the same, with a slight increase of the volume  
346 density for the main peak ( $d \sim 200 \mu\text{m}$ ) and a slight decrease of the secondary peak ( $d \sim 30$   
347  $\mu\text{m}$ ). This result suggests that mainly dispersion step occurs during reconstitution of this  
348 powder. Indeed, disintegration of small agglomerates into primary particles (from the main  
349 peak) and small particles (from secondary peak) would explain the observations. The time  
350 evolution of the mean diameter  $d_{50}$  extracted from volume density functions is plotted in  
351 **Figure 9**. The data are well fitted to an incomplete dissolution model derived from the Noyes-  
352 Whitney model (Cogswell et al., 2006). This model was proposed and successfully used by  
353 Fang *et al.* (Fang et al., 2011) in the case of the reconstitution of milk protein concentrate by  
354 measuring the particle size through optical images. The model is defined as:

355 **Equation 2** 
$$d_{50}(t) = (d_0 - d_\infty) \exp\left(-\frac{t}{\tau_{PSD}}\right) + d_\infty$$

356 where  $d_0$  is the initial mean diameter (dry case),  $d_\infty$  is the final mean diameter (at the end of  
357 the rehydration) and  $\tau_{PSD}$  is the characteristic reconstitution time of the particle size  
358 distribution. The characteristic reconstitution time and the final mean diameter value  
359 extracted are presented in **Table 3**. As already observed with the conductivity measurements,  
360 the compaction process increases the reconstitution time because a step of granule  
361 destruction adds up compared to non-compacted powders. The results also show that  
362 increasing the water activity value leads to an increase in reconstitution time. The results  
363 from granulometry during reconstitution provide a new piece of information: the final mean  
364 diameter depends neither on the water activity value nor the compaction process. This means  
365 that powders reach the same final state after total reconstitution.

### 366 3.4 Relationships between powder process, structure and function at two water activities

367 The relationships between process (compaction), structure (glassy/rubbery state) and  
368 function (reconstitution ability) is established with this multi-scale approach. From a  
369 macroscopic point of view, fruit powders are particularly sensitive to their storage  
370 environment (Islam et al., 2016). The observations demonstrate a transition from a glassy  
371 state to a rubbery state, easily visible by a color and aspect changes (caking due to the  
372 plasticizing effects of water on the particle surface) with the water activity increase at typical  
373 storage temperature (*i.e.* around 20 °C). The glass transition can appear either by increasing  
374 the temperature and/or the water activity (Roos, 2002). Here, a clear impact of water activity  
375 on macroscopic fruit powders characteristics was observed. It was already shown an inverse  
376 relationship between  $T_g$  and water activity for fruit powders (Tonon et al., 2009; Goula and  
377 Adamopoulos, 2010; Islam et al., 2016). From a microscopic point of view, this transition from  
378 angular particles to smoother ones with a large increase in the mean diameter (swelling of  
379 particles) was observed with increasing water activity. These microscopic modifications are  
380 observed by both SEM and PSD with the calculation of the  $d_{50}$  increase with relative  
381 humidity. Again, similar observations were previously made for sugar rich powders (Tonon et  
382 al., 2009; Goula and Adamopoulos, 2010; Islam et al., 2016). Finally, a transition from a stiff  
383 and brittle surface to a soft surface using nanomechanics was observed with increasing water  
384 activity value. The evolution of the Young's modulus is in agreement with both macroscopic  
385 and microscopic evolution of the powders.

386 It is well admitted that many factors can affect the powder reconstitution. It may be the  
387 method of reconstitution (type of stirrer, configuration, intensity of agitation, water  
388 temperature ...) (Fitzpatrick et al., 2000; Jeantet et al., 2010) or the material properties  
389 (particle size distribution, density, porosity, morphology, surface

390 hydrophilicity/hydrophobicity...) (Gaiani et al., 2007a; Murrieta-Pazos et al., 2012). The  
391 novelty in this study is that it has been shown that shear rate does not affect the  
392 reconstitution behavior at high water activity value ( $\sim 0.35$ ) whereas it impacts at low water  
393 activity value ( $\sim 0$ ). This non-dependence to shear was never observed for food powders and  
394 more particularly dairy powders largely studied at various shear rates (Richard et al., 2013).  
395 Nevertheless, the composition dairy powders is very different than fruit powders.  
396 Furthermore, the glass transition temperature of fruit powders is lower compared to milk-  
397 based powders due to high fraction of sugars. Recently, a study of Wangler *et al.* (Wangler et  
398 al., 2019) on the reconstitution of biopolymer powders with different coating demonstrated  
399 the importance of a viscous layer on the dissolution/non-dissolution rate. The authors  
400 showed that a biopolymer particle exhibiting a layer with low viscous cohesion reconstitutes  
401 first by capillarity and then by diffusion. On the contrary, increasing the layer thickness due to  
402 swelling leads to an entrance of the water mainly by diffusion. Regarding our results on fruit  
403 powders, an analogy can be made with reconstitution of fruit powders depending on the  
404 water activity. Indeed, mainly three reconstitution steps are relevant to our study: the  
405 wetting, the swelling and the dispersion/dissolution. The dispersion/dissolution rate mainly  
406 depends on the specific surface area in contact with water, which is higher for non-  
407 compacted powders. Thus, the effect of compaction process is very important at this  
408 reconstitution step. At  $a_w \sim 0$ , powders are under a glassy state and their porous surface help  
409 both surface erosion and the entrance of water by capillarity, following by water diffusion. In  
410 this case, increasing shear leads to a decrease of the reconstitution time. However, for  
411 particles under a rubbery state (at  $a_w \sim 0.35$ ), the wetting/swelling steps have already begun,  
412 resulting in the collapse of the pore network, making appear a viscous layer. In this case,  
413 increasing shear does not improve water entrance due to the viscous layer on the surface of

414 the particles. Thus, for this elevated relative humidity, diffusion may be the limiting step, *i.e.*  
415 the step with which we can improve the reconstitution. In this particular case, to promote the  
416 rate of reconstitution speed it may be useful to increase the temperature to reduce the  
417 viscosity in the viscous layer rather than increasing the shear rate. The hypothesis proposed in  
418 this work is presented in a schematic in **Figure 10**.

419

#### 420 4 Conclusion

421 In the present work, the reconstitution of fruit powders was evaluated. Three mixes were  
422 used in a non-compacted and a compacted state. First of all, it was found that the compaction  
423 process only delays the entire reconstitution process but the final state was identical for each  
424 mix, irrespective of the initial structure of the powder. Secondly, the crucial role of water  
425 activity was evidenced and its impact on the kinetics of powder reconstitution was evaluated.  
426 In fact, at low water activity, a shear-dependence of reconstitution kinetics was observed  
427 indicating that the increase in the stirring speed improved the entrance of water inside a  
428 particle. In that case, powders were under the glass transition temperature, so they were in  
429 the glassy state. This was evidenced by both DSC measurements and also by imaging (SEM  
430 and AFM), which reveals porous/angular shapes and stiff particles. On the contrary, at a  
431 higher water activity ( $\sim 0.35$ ), the powders were above the  $T_g$  due to the plasticizing effect of  
432 water. The powders were in the rubbery state and the particles appeared smooth and soft.  
433 Under these circumstances, the kinetics of reconstitution was found independent of the  
434 stirring speed. This behavior may have resulted from the formation of a viscous layer at the  
435 particle surface, which is responsible for the closure of the porosity at the particle surface.  
436 The main consequence is that the entrance of water into the internal structure is significantly

437 reduced. Thus, diffusion becomes the rate controlling step for powder reconstitution. Hence,  
438 increasing shear rate which would typically enhance the mass transfer coefficient on the  
439 surface is not observed under these circumstances.

440

#### 441 **Acknowledgements**

442 The authors acknowledge support of the "LIBio" by the "Impact Biomolecules" project of the  
443 "Lorraine Université d'Excellence" (Investissements d'avenir – ANR 15-004). The authors  
444 would like to thank the Laboratoire de chimie du solide minéral (Université de Lorraine,  
445 Nancy) for the SEM observations and the Laboratoire de Chimie Physique et de Microbiologie  
446 pour l'Environnement (Université de Lorraine, Nancy) for XPS analysis and AFM  
447 measurements (Rana OMAR).

448

#### 449 **References**

450 Andre, C., Richard, B., Le Page, J.-F., Jeantet, R., Delaplace, G., 2012. Influence of Mixing  
451 System Design and Operating Parameters on Dissolution Process. Chem. Eng. Technol.  
452 35, 247–254. <https://doi.org/10.1002/ceat.201100441>

453 Cogswell, S., Berger, S., Waterhouse, D., Bally, M.B., Wasan, E.K., 2006. A parenteral  
454 econazole formulation using a novel micelle-to-liposome transfer method: In Vitro  
455 characterization and tumor growth delay in a breast cancer xenograft model. Pharm.  
456 Res. 23, 2575–2585.

457 Eaton, P.J., West, P., 2010. Atomic force microscopy. Oxford University Press, Oxford ; New  
458 York.

459 Fabra, M.J., Márquez, E., Castro, D., Chiralt, A., 2011. Effect of maltodextrins in the water-  
460 content–water activity–glass transition relationships of noni (*Morinda citrifolia* L.) pulp  
461 powder. *J. Food Eng.* 103, 47–51. <https://doi.org/10.1016/j.jfoodeng.2010.09.018>

462 Fang, Y., Selomulya, C., Ainsworth, S., Palmer, M., Chen, X.D., 2011. On quantifying the  
463 dissolution behaviour of milk protein concentrate. *Food Hydrocoll.* 25, 503–510.  
464 <https://doi.org/10.1016/j.foodhyd.2010.07.030>

465 Fang, Z., Bhandari, B., 2011. Effect of spray drying and storage on the stability of bayberry  
466 polyphenols. *Food Chem.* 129, 1139–1147.  
467 <https://doi.org/10.1016/j.foodchem.2011.05.093>

468 Fitzpatrick, J.J., Ahrné, L., 2005. Food powder handling and processing: Industry problems,  
469 knowledge barriers and research opportunities. *Chem. Eng. Process. Process Intensif.*  
470 44, 209–214. <https://doi.org/10.1016/j.cep.2004.03.014>

471 Fitzpatrick, J.J., Weidendorfer, K., Teunou, E., 2000. Reconstitution of dairy powders to high  
472 solids content in a stirred-tank: the effect of agitation. *Milchwissenschaft* 55, 437–440.

473 Forny, L., Marabi, A., Palzer, S., 2011. Wetting, disintegration and dissolution of agglomerated  
474 water soluble powders. *Powder Technol.* 206, 72–78.  
475 <https://doi.org/10.1016/j.powtec.2010.07.022>

476 Gabas, A.L., Telis, V.R.N., Sobral, P.J.A., Telis-Romero, J., 2007. Effect of maltodextrin and  
477 arabic gum in water vapor sorption thermodynamic properties of vacuum dried  
478 pineapple pulp powder. *J. Food Eng.* 82, 246–252.  
479 <https://doi.org/10.1016/j.jfoodeng.2007.02.029>

480 Gaiani, C., 2006. Étude des mécanismes de réhydratation des poudres laitières : influence de  
481 la structure et de la composition des poudres. INPL, Nancy.

482 Gaiani, C., Scher, J., Ehrhardt, J.J., Linder, M., Schuck, P., Desobry, S., Banon, S., 2007b.  
483 Relationships between Dairy Powder Surface Composition and Wetting Properties  
484 during Storage: Importance of Residual Lipids. *J. Agric. Food Chem.* 55, 6561–6567.  
485 <https://doi.org/10.1021/jf070364b>

486 Gaiani, C., Scher, J., Schuck, P., Desobry, S., Banon, S., 2009. Use of a turbidity sensor to  
487 determine dairy powder rehydration properties. *Powder Technol.* 190, 2–5.  
488 <https://doi.org/10.1016/j.powtec.2008.04.042>

489 Gaiani, C., Scher, J., Schuck, P., Hardy, J., Desobry, S., Banon, S., 2006. The dissolution  
490 behaviour of native phosphocaseinate as a function of concentration and temperature  
491 using a rheological approach. *Int. Dairy J.* 16, 1427–1434.  
492 <https://doi.org/10.1016/j.idairyj.2005.12.004>

493 Gaiani, C., Schuck, P., Scher, J., Desobry, S., Banon, S., 2007a. Dairy Powder Rehydration:  
494 Influence of Protein State, Incorporation Mode, and Agglomeration. *J. Dairy Sci.* 90,  
495 570–581. [https://doi.org/10.3168/jds.S0022-0302\(07\)71540-0](https://doi.org/10.3168/jds.S0022-0302(07)71540-0)

496 Goalard, C., Samimi, A., Galet, L., Dodds, J.A., Ghadiri, M., 2006. Characterization of the  
497 Dispersion Behavior of Powders in Liquids. Part. Part. Syst. Charact. 23, 154–158.  
498 <https://doi.org/10.1002/ppsc.200601024>

499 Goula, A.M., Adamopoulos, K.G., 2010. A new technique for spray drying orange juice  
500 concentrate. *Innov. Food Sci. Emerg. Technol.* 11, 342–351.  
501 <https://doi.org/10.1016/j.ifset.2009.12.001>

502 Hoge Kamp, S., Schubert, H., 2003. Rehydration of Food Powders. *Food Sci. Technol. Int.* 9,  
503 223–235. <https://doi.org/10.1177/1082013203034938>

504 Islam, M.Z., Kitamura, Y., Yamano, Y., Kitamura, M., 2016. Effect of vacuum spray drying on  
505 the physicochemical properties, water sorption and glass transition phenomenon of

506 orange juice powder. *J. Food Eng.* 169, 131–140.  
507 <https://doi.org/10.1016/j.jfoodeng.2015.08.024>

508 Jadhav, N.R., Gaikwad, V.L., Nair, K.J., Kadam, H.M., 2009. Glass transition temperature: Basics  
509 and application in pharmaceutical sector. *Asian J. Pharm. AJP Free Full Text Artic.*  
510 *Asian J Pharm* 3.

511 Jeantet, R., Schuck, P., Six, T., Andre, C., Delaplace, G., 2010. The influence of stirring speed,  
512 temperature and solid concentration on the rehydration time of micellar casein  
513 powder. *Dairy Sci. Technol.* 90, 225–236. <https://doi.org/10.1051/dst/2009043>

514 Jiang, H., Zhang, M., Adhikari, B., 2013. Fruit and vegetable powders, in: *Handbook of Food*  
515 *Powders*. Elsevier, pp. 532–552. <https://doi.org/10.1533/9780857098672.3.532>

516 Kleinebudde, P., 2004. Roll compaction/dry granulation: pharmaceutical applications. *Eur. J.*  
517 *Pharm. Biopharm.* 58, 317–326. <https://doi.org/10.1016/j.ejpb.2004.04.014>

518 Liu, F., Cao, X., Wang, H., Liao, X., 2010. Changes of tomato powder qualities during storage.  
519 *Powder Technol.* 204, 159–166. <https://doi.org/10.1016/j.powtec.2010.08.002>

520 Lopez-Quiroga, E., Prosapio, V., Fryer, P.J., Norton, I.T., Bakalis, S., 2019. Model discrimination  
521 for drying and rehydration kinetics of freeze-dried tomatoes. *J. Food Process Eng.*  
522 <https://doi.org/10.1111/jfpe.13192>

523 Mitchell, W.R., Forny, L., Althaus, T., Niederreiter, G., Palzer, S., Hounslow, M.J., Salman, A.D.,  
524 2019. Surface tension-driven effects in the reconstitution of food powders. *Chem.*  
525 *Eng. Res. Des.* 146, 464–469. <https://doi.org/10.1016/j.cherd.2019.04.015>

526 Mitchell, W.R., Forny, L., Althaus, T.O., Niederreiter, G., Palzer, S., Hounslow, M.J., Salman,  
527 A.D., 2015. Mapping the rate-limiting regimes of food powder reconstitution in a  
528 standard mixing vessel. *Powder Technol.* 270, 520–527.  
529 <https://doi.org/10.1016/j.powtec.2014.08.014>



530 Moghbeli, S., Jafari, S.M., Maghsoudlou, Y., Dehnad, D., 2019. Influence of pectin-whey  
531 protein complexes and surfactant on the yield and microstructural properties of date  
532 powder produced by spray drying. *J. Food Eng.* 242, 124–132.  
533 <https://doi.org/10.1016/j.jfoodeng.2018.08.025>

534 Murrieta-Pazos, I., Gaiani, C., Galet, L., Scher, J., 2012. Composition gradient from surface to  
535 core in dairy powders: Agglomeration effect. *Food Hydrocoll.* 26, 149–158.  
536 <https://doi.org/10.1016/j.foodhyd.2011.05.003>

537 Nijdam, J.J., Langrish, T.A.G., 2006. The effect of surface composition on the functional  
538 properties of milk powders. *J. Food Eng.* 77, 919–925.  
539 <https://doi.org/10.1016/j.jfoodeng.2005.08.020>

540 Rajniak, P., Mancinelli, C., Chern, R., Stepanek, F., Farber, L., Hill, B., 2007. Experimental study  
541 of wet granulation in fluidized bed: Impact of the binder properties on the granule  
542 morphology. *Int. J. Pharm.* 334, 92–102.  
543 <https://doi.org/10.1016/j.ijpharm.2006.10.040>

544 Richard, B., Le Page, J.F., Schuck, P., Andre, C., Jeantet, R., Delaplace, G., 2013. Towards a  
545 better control of dairy powder rehydration processes. *Int. Dairy J.* 31, 18–28.  
546 <https://doi.org/10.1016/j.idairyj.2012.07.007>

547 Roos, Y.H., 2002. Importance of glass transition and water activity to spray drying and stability  
548 of dairy powders. *Le Lait* 82, 475–484. <https://doi.org/10.1051/lait:2002025>

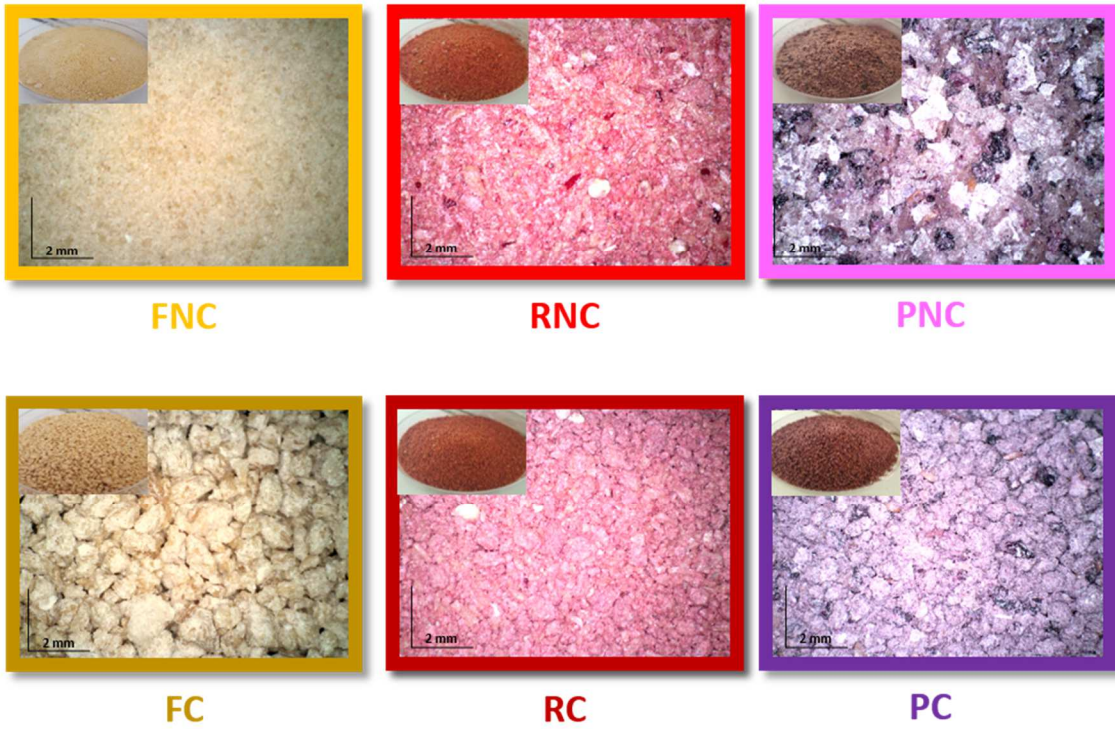
549 Rouxhet, P.G., Misselyn-Bauduin, A.M., Ahimou, F., Genet, M.J., Adriaensen, Y., Desille, T.,  
550 Bodson, P., Deroanne, C., 2008. XPS analysis of food products: toward chemical  
551 functions and molecular compounds. *Surf. Interface Anal.* 40, 718–724.  
552 <https://doi.org/10.1002/sia.2779>

553 Schubert, H., 1993. Instantization of powdered food products. *Int. Chem. Eng.* 33, 28–45.

554 Tonon, R.V., Baroni, A.F., Brabet, C., Gibert, O., Pallet, D., Hubinger, M.D., 2009. Water  
555 sorption and glass transition temperature of spray dried açai (*Euterpe oleracea* Mart.)  
556 juice. *J. Food Eng.* 94, 215–221. <https://doi.org/10.1016/j.jfoodeng.2009.03.009>  
557 Wangler, J., Kohlus, R., 2018. Development and validation of methods to characterize  
558 rehydration behavior of food hydrocolloids. *Food Hydrocoll.* 82, 500–509.  
559 <https://doi.org/10.1016/j.foodhyd.2018.04.018>  
560 Wangler, J., Teichmann, H., Konstanz, E., Kohlus, R., 2019. Experimental investigation and  
561 simulation of rehydration dynamics of biopolymer powders. *Powder Technol.* 355,  
562 461–473. <https://doi.org/10.1016/j.powtec.2019.07.022>  
563

1 **Figures and Captions.**

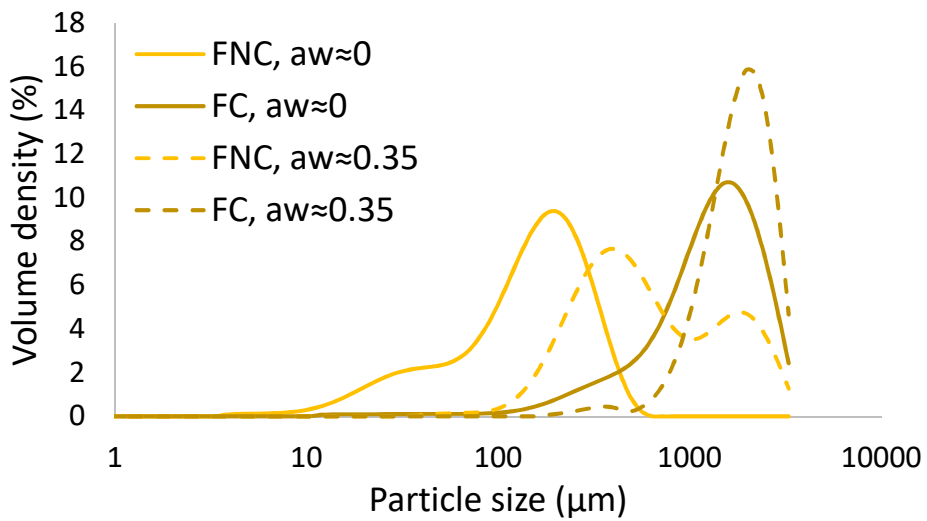
2 **Figure 1.** Macroscopic and microscopic views of the fruit powders studied. Top: powders before  
3 compaction process (FNC, RNC and PNC). Bottom: powders after compaction process (FC, RC  
4 and PC).



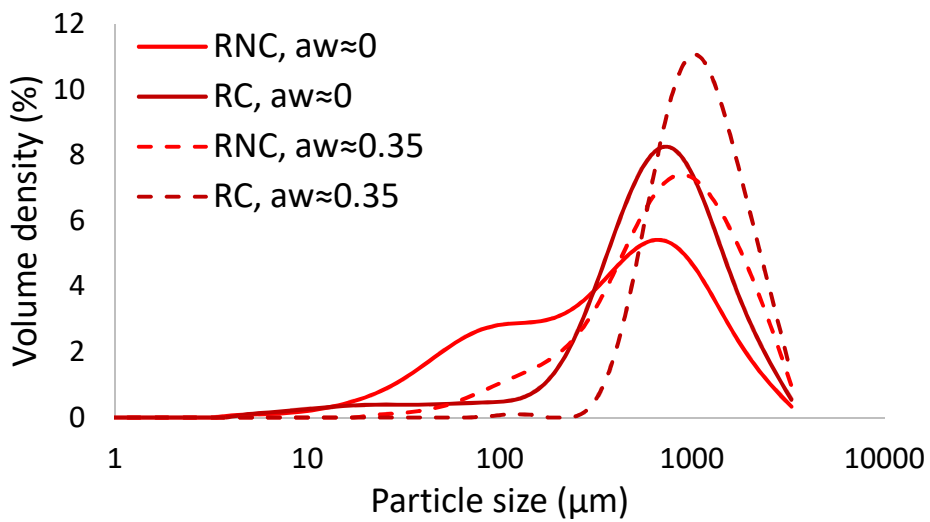
5  
6  
7  
8  
9  
10  
11  
12  
13  
14  
15  
16  
17  
18

19 **Figure 2.** Particle size distribution functions of the six fruit powders studied at the two water  
20 activity values ( $a_w \sim 0$  and  $\sim 0.35$ ).

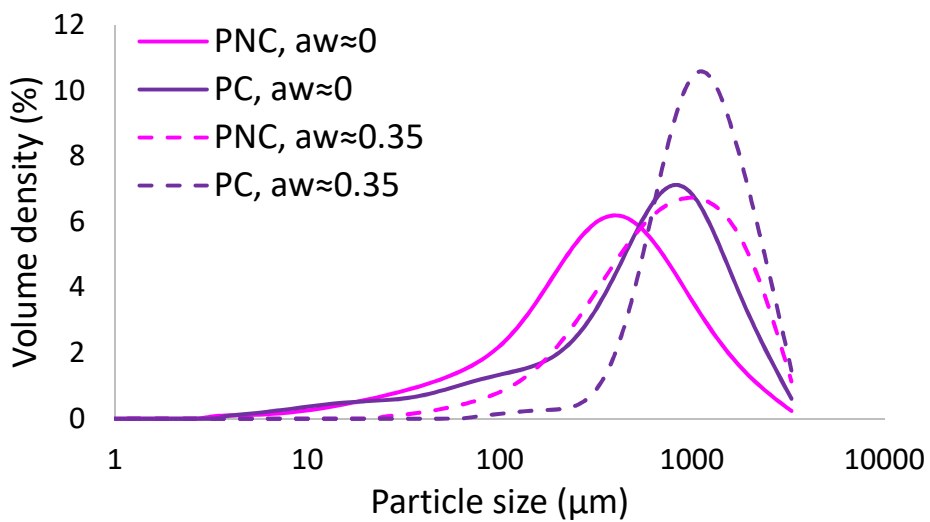
21



22

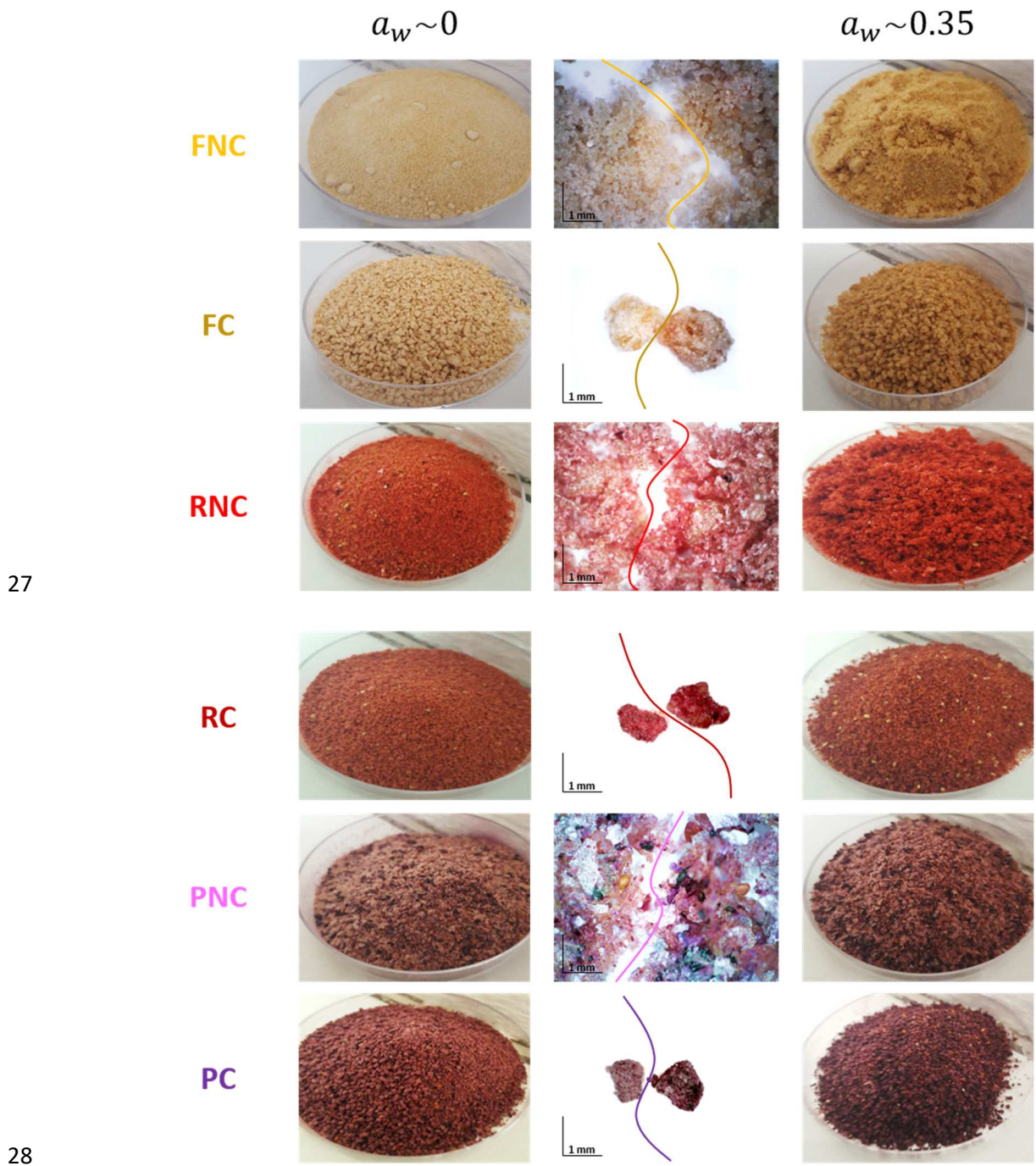


23



24

25 **Figure 3.** Pictures and microscopic images at two water activity values ( $a_w \sim 0$  and  $\sim 0.35$ ) of  
26 the six fruit powders studied (FNC, FC, RNC, RC, PNC and PC).



27

28

29

30

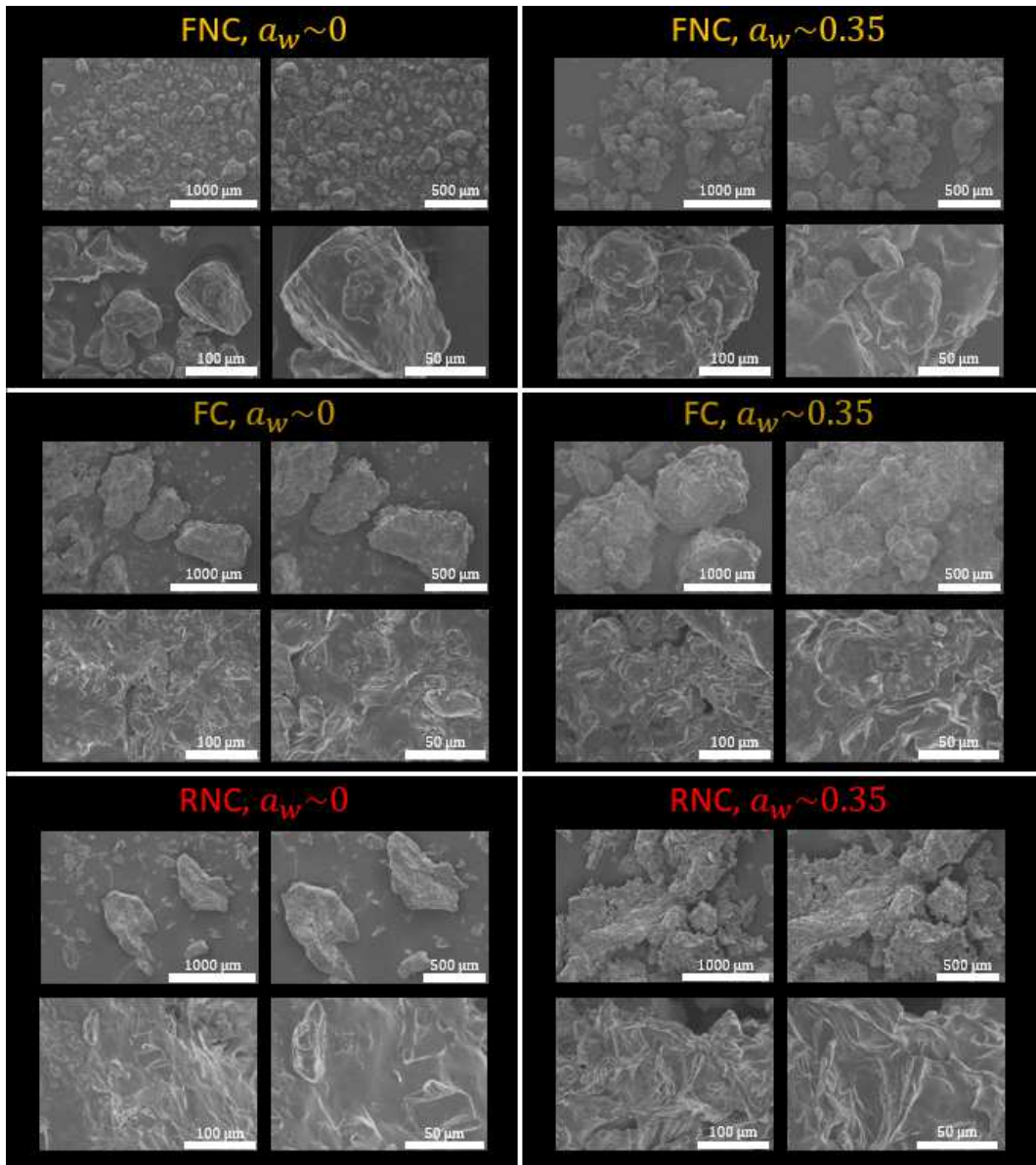
31

32

33

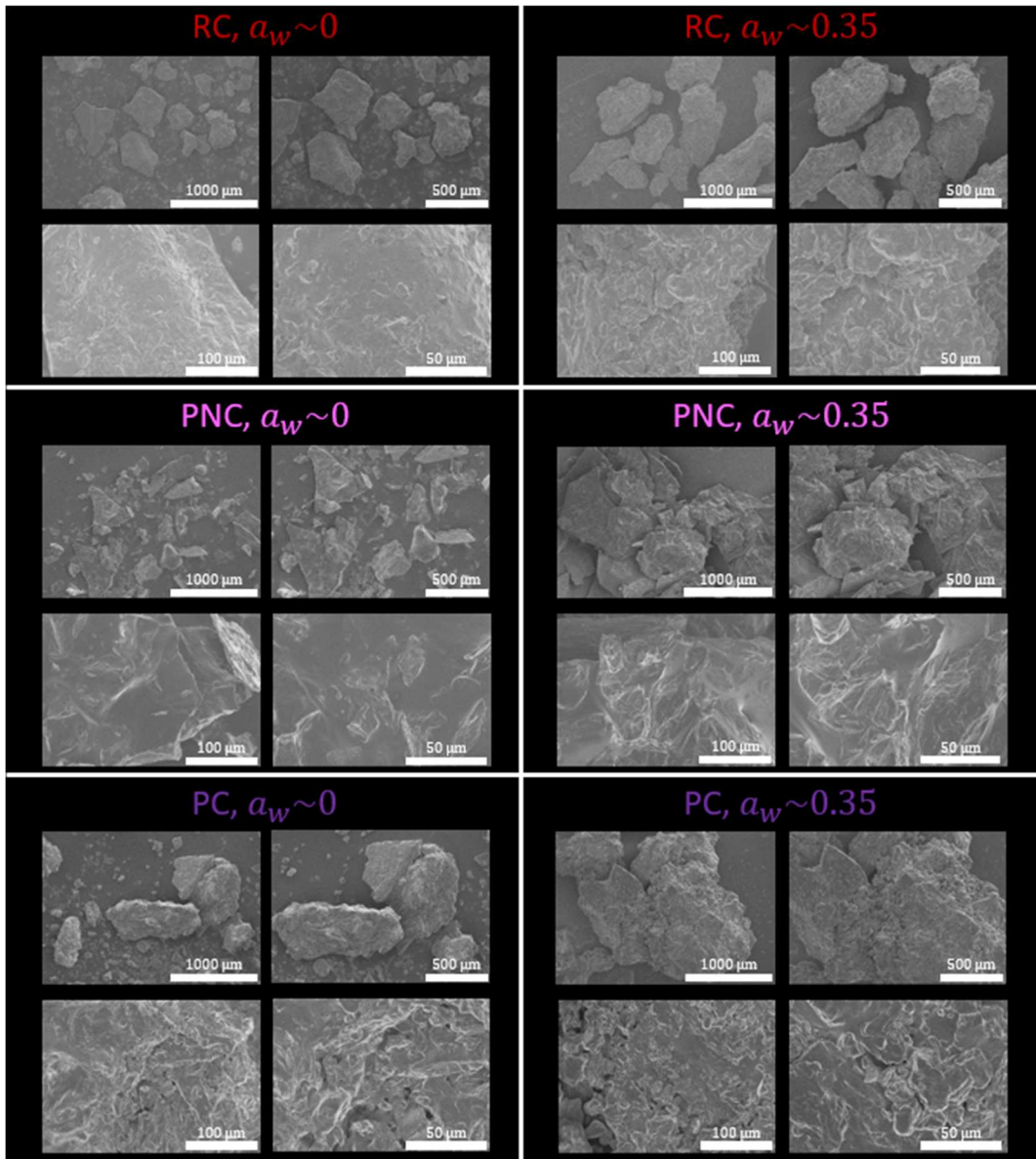
34 **Figure 4.** SEM images at various magnifications for the six fruit powders studied at both  $a_w \sim 0$   
35 and  $a_w \sim 0.35$ .

36



37

38

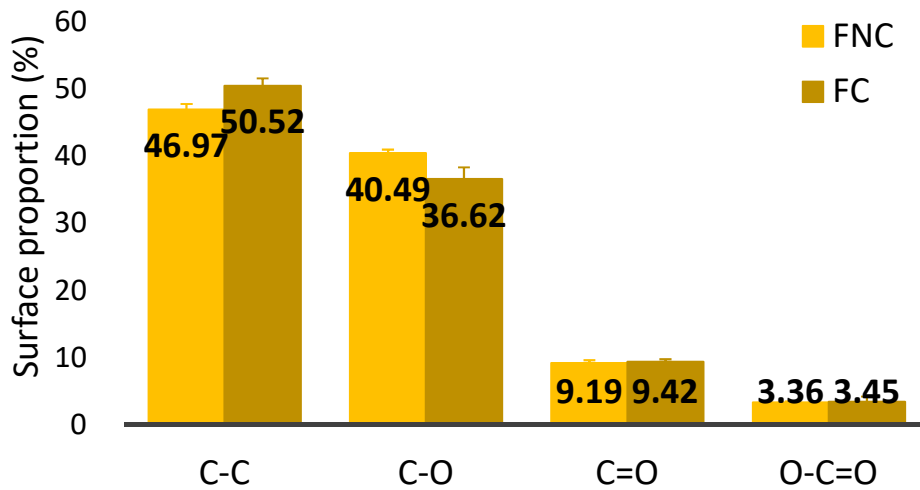


39  
 40  
 41  
 42  
 43  
 44  
 45  
 46  
 47  
 48  
 49  
 50

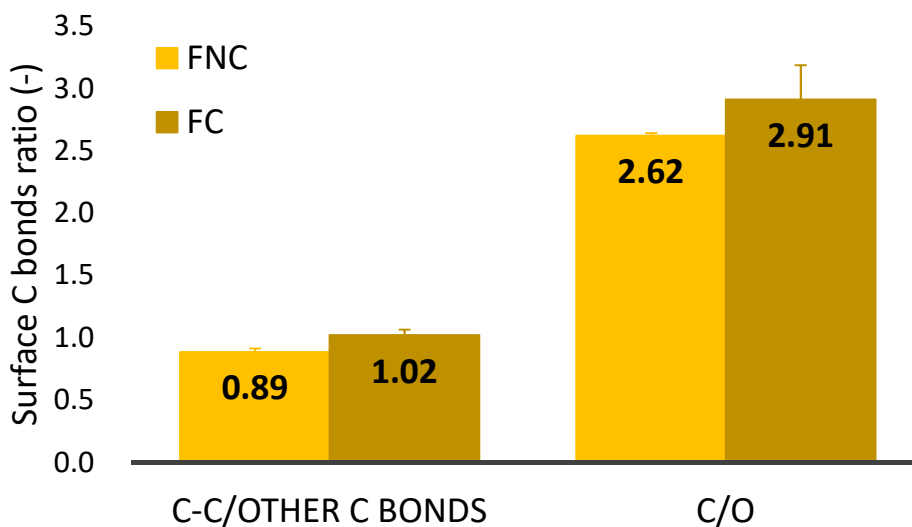
51 **Figure 5.** (a) Surface atomic concentration of chemical elements, (b) proportion of carbon  
 52 bonds and (c) proportion of carbon bonds ratios detected at the surface of FNC and FC  
 53 powders.

| Elements              | FNC          | FC           |
|-----------------------|--------------|--------------|
| <b>C<sub>1s</sub></b> | 70.92 ± 0.13 | 73.38 ± 1.96 |
| <b>O<sub>1s</sub></b> | 27.05 ± 0.18 | 25.39 ± 1.91 |
| <b>N<sub>1s</sub></b> | 1.79 ± 0.10  | 1.23 ± 0.12  |
| <b>K<sub>2p</sub></b> | 0.15 ± 0.03  | --           |
| <b>P<sub>2p</sub></b> | 0.09 ± 0.09  | --           |

54 (a)



55 (b)

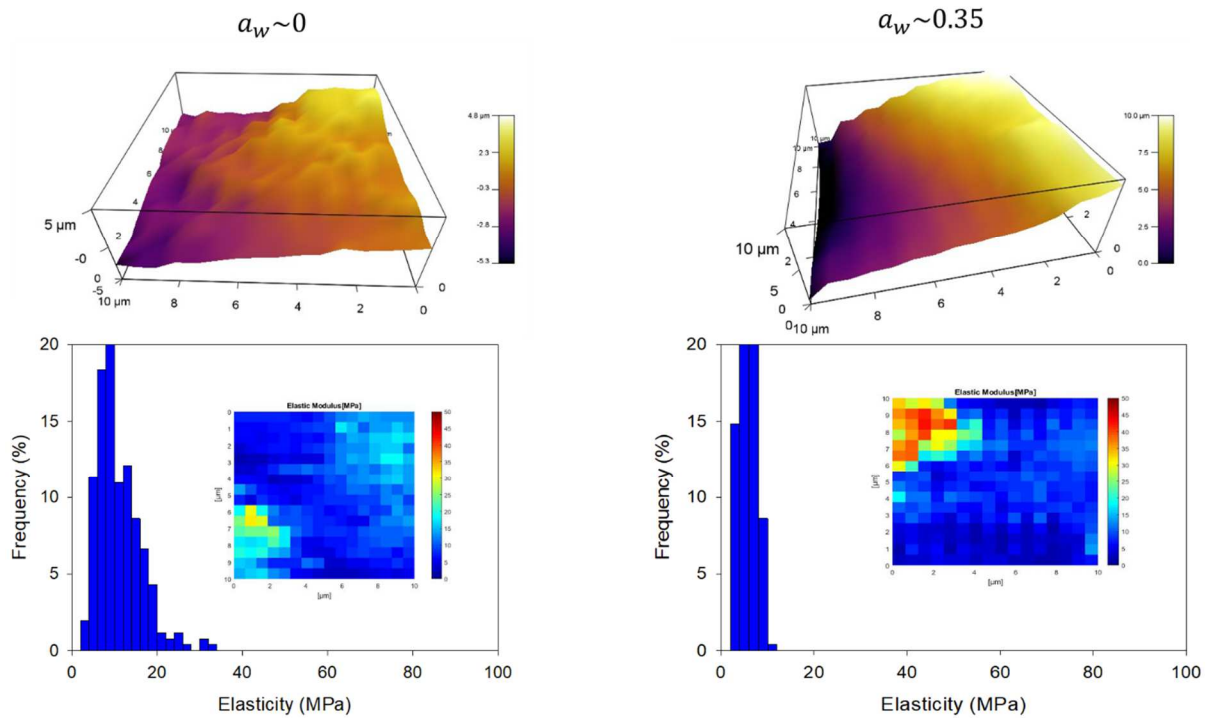


56 (c)



57 **Figure 6.** Surface topography at two water activity values for RC powder and corresponding  
58 distribution of the elastic modulus after curve fitting with the Sneddon model. Inserts in  
59 histograms represent the associated elasticity maps.

60



61

62

63

64

65

66

67

68

69

70

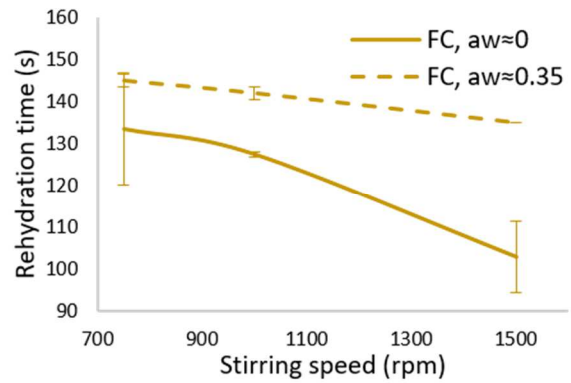
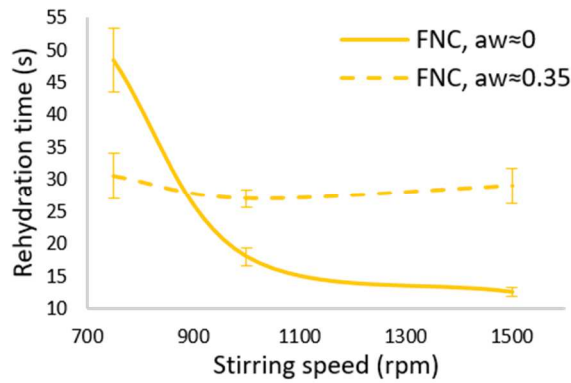
71

72

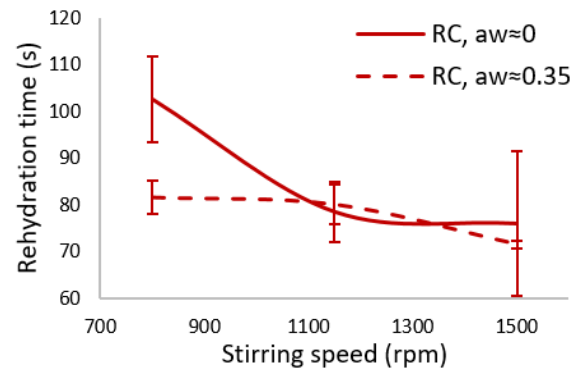
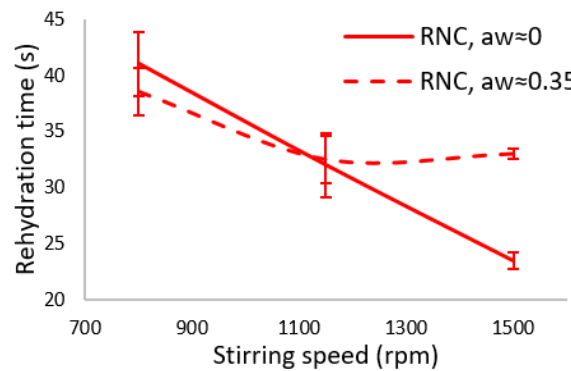
73

74

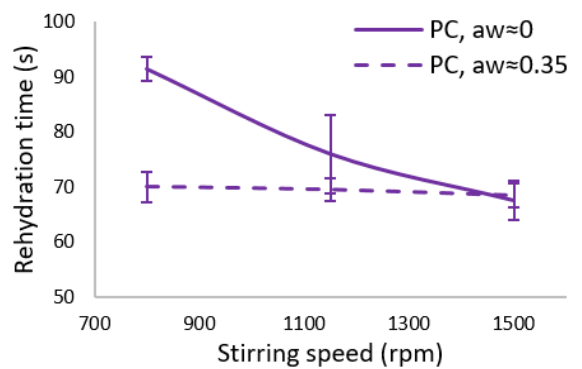
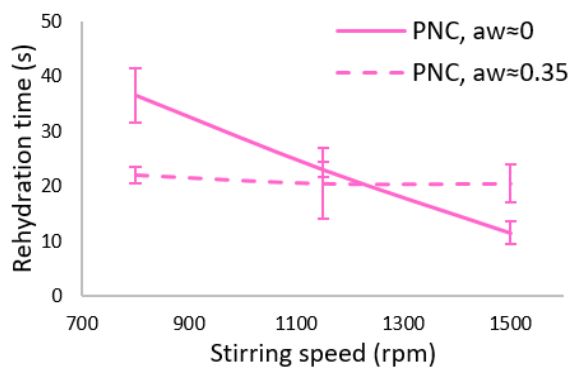
75 **Figure 7.** Evolution of the rehydration time as a function of the stirring speed value  $N$  at a fixed  
 76 water temperature  $T=25\text{ }^{\circ}\text{C}$ , and at different water activity values for (a) FNC and FC powders,  
 77 (b) RNC and RC powders and (c) PNC and PC powders.



78 (a)  
 79



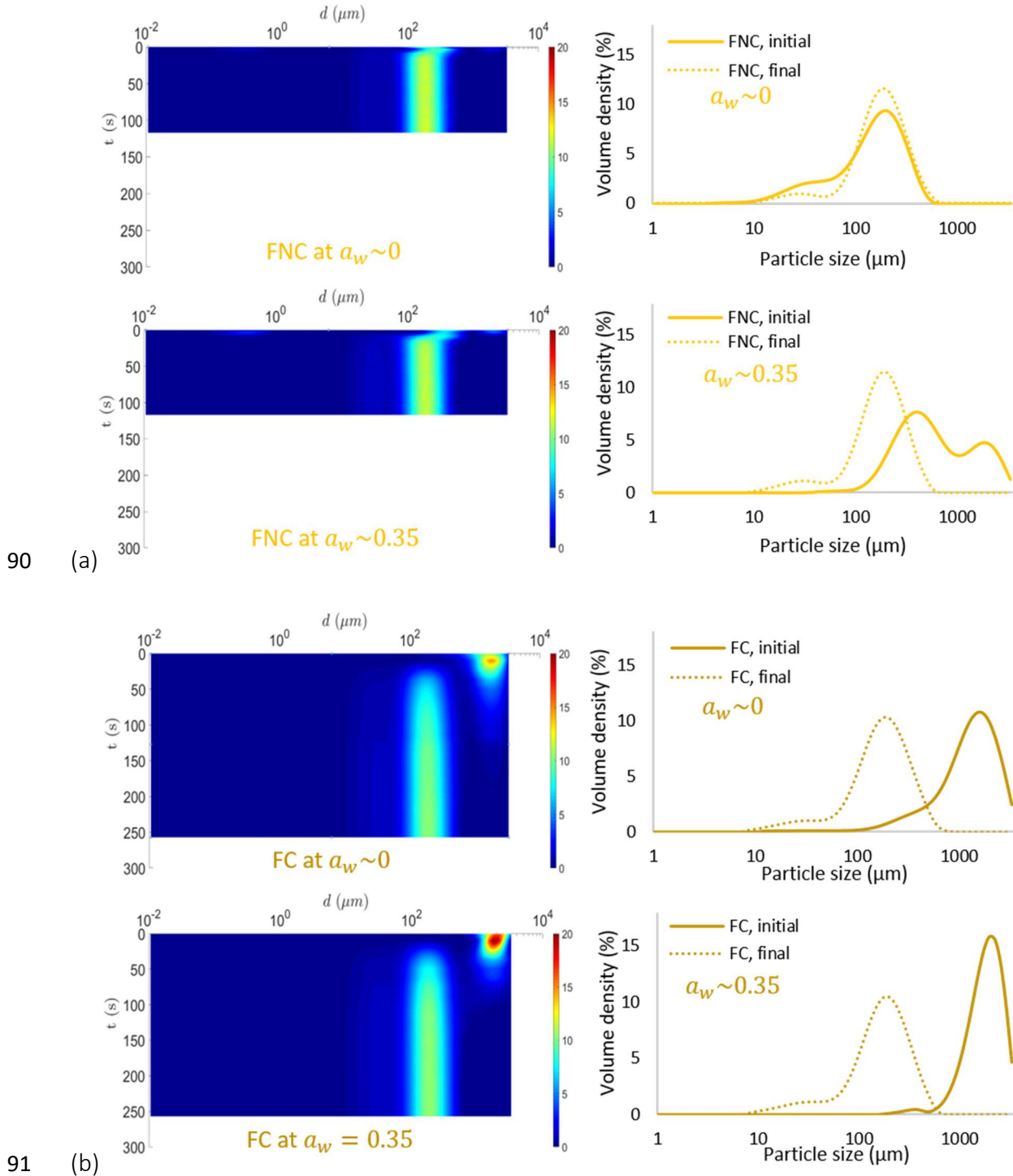
80 (b)  
 81



82 (c)

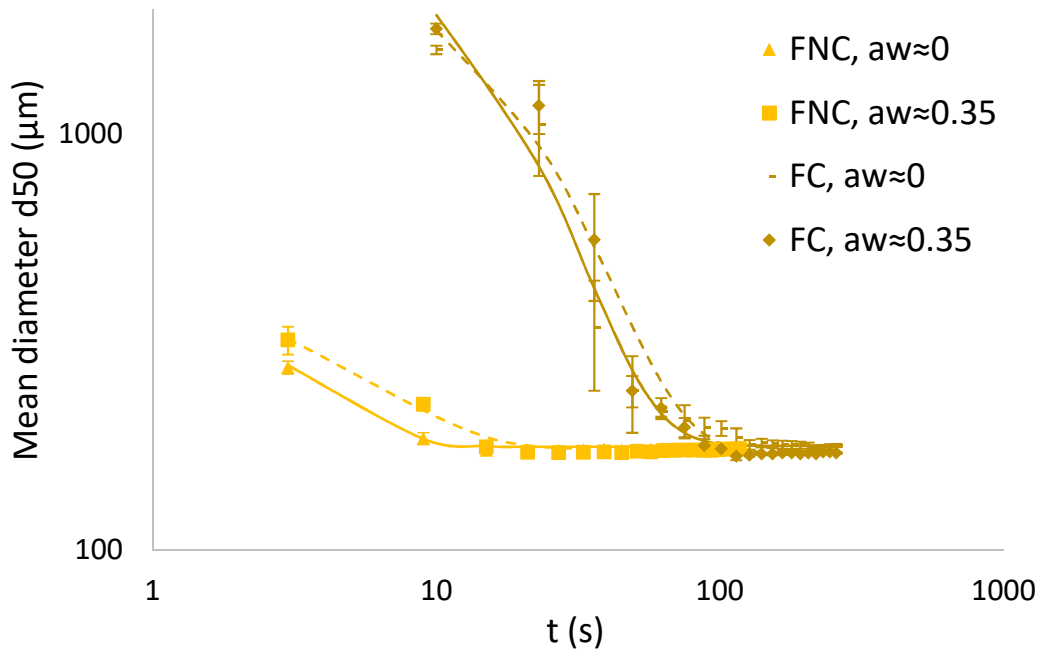
83  
 84  
 85  
 86

87 **Figure 8.** At both  $a_w \sim 0$  and  $a_w \sim 0.35$ , for (a) FNC powder and (b) FC powder. Left: Maps  
 88 presenting the time evolution of the volume density function. Right: Volume density function  
 89 at  $t_{initial}$  (dry powder) and  $t_{final}$  (powder entirely rehydrated).



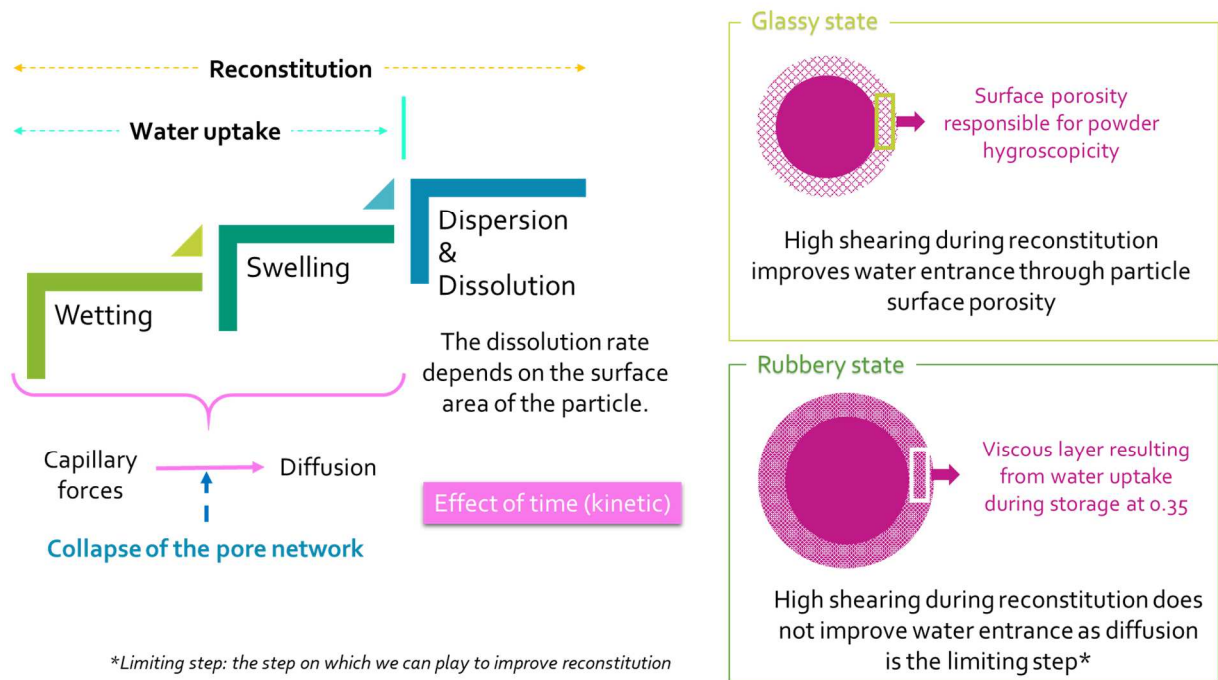
91  
 92  
 93

94 **Figure 9.** Logarithmic plot of the time evolution of the mean diameter  $d_{50}$  extracted from  
95 volume density functions for FNC and FC powders, at  $a_w \sim 0$  and  $a_w \sim 0.35$ . The data are well  
96 fitted to equation 2 (solid lines and dotted lines).  
97



98  
99  
100  
101  
102  
103  
104  
105  
106  
107  
108  
109

110 **Figure 10.** Summary of the relevant results in term of physicochemical properties from macro-  
 111 to nano- scale for fruit powders.



112  
 113  
 114  
 115  
 116  
 117  
 118  
 119  
 120  
 121  
 122  
 123  
 124

125 Table of content

126 **Table 1.** Information related to particle size distribution functions of the six fruit powders  
 127 studied at  $a_w \sim 0$  and  $a_w \sim 0.35$ .

|     |                 | $d_{50}$ ( $\mu\text{m}$ ) | span            | % increase $d_{50}$ with water activity |
|-----|-----------------|----------------------------|-----------------|---|
| FNC | $a_w \sim 0$    | $152 \pm 1$                | $1.83 \pm 0.04$ | 270 %                                   |
|     | $a_w \sim 0.35$ | $563 \pm 136$              | $3.22 \pm 1.34$ |   |
| FC  | $a_w \sim 0$    | $1330 \pm 39$              | $1.55 \pm 0.10$ | 10 %                                    |
|     | $a_w \sim 0.35$ | $1460 \pm 148$             | $1.26 \pm 0.30$ |   |
| RNC | $a_w \sim 0$    | $387 \pm 46$               | $3.36 \pm 0.77$ | 98 %                                    |
|     | $a_w \sim 0.35$ | $765 \pm 3$                | $2.30 \pm 0.09$ |   |
| RC  | $a_w \sim 0$    | $671 \pm 33$               | $2.17 \pm 0.24$ | 59 %                                    |
|     | $a_w \sim 0.35$ | $1070 \pm 53$              | $1.49 \pm 0.18$ |   |
| PNC | $a_w \sim 0$    | $348 \pm 17$               | $3.12 \pm 0.41$ | 121 %                                   |
|     | $a_w \sim 0.35$ | $770 \pm 18$               | $2.38 \pm 0.07$ |   |
| PC  | $a_w \sim 0$    | $634 \pm 31$               | $2.55 \pm 0.18$ | 74 %                                    |
|     | $a_w \sim 0.35$ | $1100 \pm 69$              | $1.54 \pm 0.21$ |   |

128  
 129  
 130  
 131  
 132  
 133  
 134  
 135  
 136  
 137  
 138  
 139

140 **Table 2.** Endpoint glass transition temperatures  $T_g$  (°C) of the six fruit powders studied during  
141 the second heat rate, at  $a_w \sim 0$  and  $a_w \sim 0.35$ .

142

|                 | FNC             | FC              | RNC             | RC              | PNC             | PC              |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| $a_w \sim 0$    | 14.55<br>± 0.18 | 24.92<br>± 0.44 | 13.91<br>± 0.14 | 17.71<br>± 0.54 | 26.53<br>± 1.98 | 24.73<br>± 1.67 |
| $a_w \sim 0.35$ | -1.77<br>± 1.64 | -0.52<br>± 1.02 | -4.96<br>± 1.85 | -2.80<br>± 1.02 | 1.91<br>± 1.00  | -4.28<br>± 1.16 |

143

144

145

146

147

148

149

150

151

152

153

154

155

156

157

158

159

160

161

162

163

164

165 **Table 3.** Parameters values extracted from exponential time decay functions (Equation 2) for  
 166 FNC and FC at  $a_w \sim 0$  and  $a_w \sim 0.35$ : characteristic PSD rehydration time  $\tau_{PSD}$  and final mean  
 167 diameter value  $d_\infty$ .

|     |                 | $\tau_{PSD}$ (s) | $d_\infty$ ( $\mu\text{m}$ ) |
|-----|-----------------|------------------|------------------------------|
| FNC | $a_w \sim 0$    | $2.35 \pm 0.09$  | $177 \pm 7$                  |
|     | $a_w \sim 0.35$ | $4.76 \pm 0.21$  | $175 \pm 8$                  |
| FC  | $a_w \sim 0$    | $13.27 \pm 0.01$ | $178 \pm 1$                  |
|     | $a_w \sim 0.35$ | $17.57 \pm 0.45$ | $171 \pm 5$                  |

168

169

170

171

172

173

174

175

176

177

178

179

180

181

182

183

184