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# Main powder physicochemical characteristics influencing their reconstitution behavior

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## 25 **1. Introduction**

26 **Biologically**-derived powdered materials are involved in the manufacturing of many industrial  
27 products (such as cosmetics, foods, **and** pharmaceuticals). In the food field (but not only), various  
28 raw materials and ingredients, such as flours, spices, and processed products like instant coffee  
29 **powder** or powdered milk are traded in powder form. They originate either from liquid  
30 conversion into powder by various techniques such as spray-drying, freeze-drying, drum-  
31 drying, belt-drying, or crystallization, or from size reduction of **solid materials** [1,2]. Because  
32 of the various powder origins, their particle size and shape distributions, bulk and surface  
33 chemical compositions, and physical properties are highly variable. Therefore, more than one  
34 analytical technique is often required to obtain a full set of information about a given scientific  
35 question [3].

36 The reconstitution of powders is of utmost relevance for industrial applications, considering  
37 that most powdered ingredients are reconstituted or extracted before use. Literature put in  
38 evidence the existence of four main reconstitution steps: wetting and swelling (**which may occur**  
39 **simultaneously depending** on powder composition); sinking; dispersion (including breaking of  
40 lumps and agglomerates and fragmentation of individual particles); and solubilization. The  
41 main scientific obstacle is that these steps can hardly be isolated. Indeed, several reconstitution  
42 steps overlap at different time scales [4] and the entire reconstitution mechanism **is still**  
43 **complicated to state** [2,5,6].

44 Powder wetting is the phenomenon defined by the replacement of the gaseous phase by water  
45 at the powder surface. **It can be experimentally determined by using standard methods of the**  
46 **International Dairy Federation** [7] **developed for milk powders**. Previous studies evidenced that  
47 powder wetting is more efficient for large particles (at least over 90  $\mu\text{m}$ ) or agglomerated  
48 powders [8–11]. High particle size induces small particle-solvent contact angle, thus facilitating  
49 wetting. High porosity and spherical particles also have a good wetting behavior [9,12]. In

50 addition to physical properties, chemical composition can also influence powder wettability.  
51 For instance, high lipid or protein contents makes powder surface hydrophobic, leading to poor  
52 wettability [11,13,14]. On the contrary, hydrophilic components located at powder surface like  
53 carbohydrates or surfactants enhance its wettability [12,14–16].

54 Sinkability is defined as the ability of particles or particle agglomerates to sink below the water  
55 surface [17]. Sinking mainly depends on particle size distribution, porosity, and density: as a  
56 general rule, large and/or high-density particles enhance sinking. However, large particles of  
57 high porosity (low density) exhibit higher sinking rate than small and low porosity (high  
58 density) particles, as sinking is favored by water capillary diffusion into inter- and intra-  
59 particular pores [16,18,19]. Cohesive powders often sink in the form of lumps [2].

60 Dispersibility is defined as the ability of a powder to disperse into water. Powder dispersion  
61 consists in two simultaneous mechanisms [20,21]: lumps and agglomerates breaking into  
62 individual particles and individual particles fragmentation, both leading to size reduction and  
63 specific surface area increase, further facilitating interactions of the solid matrix with water.

64 Dispersion occurs when the forces exerted by the solvent to separate particles overcome particle  
65 cohesion [11,22,23]. The dispersion rate is enhanced for high porosity, low density, or lowly  
66 cohesive particles [8,23,24].

67 Solubilization is often considered as the determinant reconstitution step as it is the final one  
68 [23]. It results in the complete disappearance of soluble granular structures and the release of  
69 all soluble components (e.g. proteins, minerals, etc.) [25,26]. Complete powder solubilization  
70 leads to a homogeneous solution without insoluble particles, which is an important criterion for  
71 the consumer. The chemical composition is crucial for powder solubility. For example, powder  
72 solubilization is generally delayed by the presence of hydrophobic molecules at the particle  
73 surface [27]. Also, it has been observed that intermicellar interactions of caseins significantly  
74 decrease the solubilization rate [21].

75 Countless techniques have been used to describe and characterize the successive powder  
76 reconstitution steps and interesting advancements have been achieved [2,5,19,24,28].  
77 Nevertheless, previous studies have mainly been focused on a limited number of powders  
78 similar in origin and composition (i.e. often milk powders). Therefore, owing to the great  
79 variety of industrially available powders, a larger investigation is still needed in order to identify  
80 **the main** physicochemical characteristics controlling powder reconstitution. For this purpose,  
81 the wettability and reconstitutability of thirty-six powders of various **origins and natures** were  
82 determined and linked to manufacturing process, chemical compositions (bulk and surface),  
83 and physical characteristics (**median particle size**) by principal component analysis.

84

## 85 **2. Material and methods**

### 86 **2.1. Investigated powders**

87 Thirty-six powders, varying in chemical composition (water, lipids, proteins, carbohydrates and  
88 minerals contents), **physical properties (particle size distribution) and manufacturing process**  
89 (spray-drying, drum-drying, freeze-drying, crystallization), were investigated (**Supplementary**  
90 **Material Table S1**). In order to change the particle size distribution while keeping the same  
91 powder bulk composition, polenta and instant coffee **powder** were ground (to reach 1.0 mm and  
92 0.08 mm, **respectively**) with a **ZM 200 (Retsch, France)** ultra- centrifugal mill at 8 000 rpm  
93 **using small powder amounts in order to limit** the heating of the grinding system **and thus of**  
94 **produced powder** [29]. This **approach** allowed obtaining a wide range of reconstitution profiles.

### 95 **2.2. Physicochemical characterization**

#### 96 **2.2.1. Particle size distribution**

97 Laser diffraction (Mastersizer S, Malvern Instruments Ltd, Malvern, UK) was used to evaluate  
98 particle size **distribution** with a 5 mW He-Ne laser operating at 632.8 nm wavelength and a 300

99 RF lens. Dry dispersion was carried out with an Aero S air disperser module (Malvern  
100 Instruments Ltd). The analytical conditions were adapted for each powder: air pressure was  
101 fixed at 2 bar, feed rate between 30 and 60 %, and hopper length between 2 and 3 mm. The  
102 volume-equivalent sphere diameter was chosen as size estimator. Classical granulometric  
103 parameters  $D_{10}$ ,  $D_{50}$ , and  $D_{90}$  were used to characterize powders, where  $D_x$  designates the  
104 diameter for which  $x$  % of the particles have a smaller size. The width of particle size  
105 distribution was estimated by the span, calculated with **Equation 1** [8,30,31].

$$106 \quad span = \frac{D_{90} - D_{10}}{D_{50}} \quad (\text{Equation 1})$$

### 107 **2.2.2. Water content**

108 The water content was determined by the mass loss of 2.5 g powder after drying at 103 °C  
109 during at least 3 h following [ISO 5537:2004](#) [32].

### 110 **2.2.3. Water activity**

111 Water activity ( $a_w$ ) was determined with a HygroPalm23-AW portable water activity-meter  
112 (Rotronic, France) in a temperature-controlled room at 20 °C using a 3 cm powder layer  
113 following [ISO 21807:2004](#) [33].

### 114 **2.2.4. Chemical composition of powder bulk**

115 The bulk composition (i.e. contents in carbohydrates, lipids, proteins, minerals, water) was  
116 determined only for whole and skim milk powders. For all other powders, data about powder  
117 chemical composition were extracted from supplier technical sheets (**Table S1,**  
118 **Supplementary material**).

119 The total nitrogen content and the non-proteic nitrogen content corresponding to the soluble  
120 fraction remaining after precipitation with trichloroacetic acid were determined by the Kjeldahl  
121 method. Nitrogen contents were converted into protein contents using a 6.38 conversion factor.

122 Total lipids were quantified with a method adapted from [34]. 5 g powder was dissolved into a  
123 2:1 (v/v) mixture of chloroform/methanol for lipid extraction. The mixture was stirred at 700  
124 rpm during 15 min. Then, the soluble phase containing sample lipids was separated by filtration  
125 on a sintered glass filter of 10 – 16 µm porosity and recovered in a round-bottom boiling flask  
126 that was previously dried and weighted. The solvent of the soluble phase was removed with a  
127 rotary vacuum evaporator (Heidolph Laborator 4000) at low stirring and 40 °C. The flask  
128 empty from residual solvent was dried at 103 °C for 15 min to ensure complete solvent  
129 evaporation and finally weighted.

130 Minerals content was determined by calcining 2 g sample at 550 °C during 12 h.

131 Lactose content was obtained by difference with other components (**Equation 2**):

$$132 \quad \% \text{ lactose} = 100 - (\% \text{ water} + \% \text{ proteins} + \% \text{ minerals} + \% \text{ lipids}) \quad (\text{Equation 2})$$

### 133 **2.3. Powder surface composition characterization by X-ray photoelectron** 134 **spectroscopy**

135 XPS measurements are performed in ultra-high vacuum ( $10^{-8}$  Pa). The surface of the powder  
136 is irradiated, which causes a complete transfer of X-ray photon energy to atomic electrons of  
137 the sample. When the electron binding energy  $E_b$  is lower than the photon energy  $h\nu$ , with  $h$   
138 the Planck constant ( $6.63 \times 10^{-34}$  m<sup>2</sup>.kg.s<sup>-1</sup>) and  $\nu$  the radiation frequency (s<sup>-1</sup>), an electron is  
139 emitted from the atom with a typical kinetic energy  $E_k$  related to  $E_b$  (**Equation 3**):

$$140 \quad E_b = h\nu - E_k \quad (\text{Equation 3})$$

141 Analyses were realized with a Kratos Axis Ultra (Kratos Analytical, Manchester, UK) X-ray  
142 photoelectron spectrometer with a monochromatic Al X-ray source at 150 W. The analytical  
143 area was about 700 µm x 300 µm and the analytical depth 10 nm. Powder samples were attached  
144 to a sample holder with double-sided conductive tape. XPS spectra were then recorded and

145 treated using the Kratos Vision software. Quantification was performed using the  
146 photoemission cross-sections and transmission coefficients given in the Kratos Vision package.  
147 The main elements (C, O, N, P, Ca) detected at powder surface were identified by means of  
148 their respective binding energy. Also, the carbon peak was deconvoluted into its main chemical  
149 bonds (C-(C, H); C-(O, N); C=O; O-C=O).

150 For milk powders, it was assumed that nitrogen was present in proteins only; therefore, the  
151 surface composition can be calculated using the matrix method based on theoretical atomic  
152 composition of main components [8,13,35,36]. Briefly, the atomic proportions of carbon,  
153 oxygen, and nitrogen were used in a matrix formula (Equations 4.1-4.3) in order to deduce the  
154 surface proportions in the different sample compounds:  $\alpha_{La}$  for lactose (La),  $\alpha_P$  for proteins (P),  
155 and  $\alpha_{Li}$  for lipids (Li).

$$156 \quad I^C = \alpha_P \times I^{C.P} + \alpha_{La} \times I^{C.La} + \alpha_{Li} \times I^{C.Li} \quad \text{(Equation 4.1)}$$

$$157 \quad I^O = \alpha_P \times I^{O.P} + \alpha_{La} \times I^{O.La} + \alpha_{Li} \times I^{O.Li} \quad \text{(Equation 4.2)}$$

$$158 \quad I^N = \alpha_P \times I^{N.P} + \alpha_{La} \times I^{N.La} + \alpha_{Li} \times I^{N.Li} \quad \text{(Equation 4.3)}$$

159  $I^C$ ,  $I^O$ , and  $I^N$  are the mole fractions of carbon, oxygen, and nitrogen at the sample surface (these  
160 values were directly obtained by XPS from the C1s, O1s, and N1s peak areas );  $I^{C.P}$ ,  $I^{C.La}$ , and  
161  $I^{C.Li}$  are the mole fractions of carbon in proteins, lactose , and lipids, respectively;  $I^{O.P}$ ,  $I^{O.La}$ , and  
162  $I^{O.Li}$  are the mole fractions of oxygen in proteins, lactose, and lipids, respectively;  $I^{N.P}$ ,  $I^{N.La}$ , and  
163  $I^{N.Li}$  are the mole fractions of nitrogen in proteins, lactose, and lipids.

## 164 **2.4. Powder reconstitution behavior**

### 165 **2.2.4. Wettability**

166 Wettability measurement was adapted from the IDF (1987) method: in this work, the wettability  
167 was defined as the time in seconds taken by  $1.00 \pm 0.01$  g of powder to become wet when  
168 deposited at the surface of  $10.0 \text{ g} \pm 0.1 \text{ g}$  distilled water at  $25 \text{ }^\circ\text{C}$  in a 20 mL beaker under  
169 magnetic stirring at 600 rpm.

## 170 2.2.5. Reconstitutability followed by conductimetry

171 **Experimental setup.** An experimental setup (**Figure 1**) was developed to follow powder  
172 reconstitution kinetics by conductimetry. It was constituted of a jacketed glass reactor (GL18,  
173 Grosseron) thermostated by a heated water bath (Eco Gold, Lauda), a mechanical stirrer (ZR  
174 2102 control, Heidolph), and a 5.08 cm diameter impeller (Pitch blade turbine 2" 32Ra, Fusion  
175 fluid equipment). Powder reconstitution kinetics were followed by conductimetry  
176 (SevenCompact S230, Mettler Toledo) with a conductivity probe (InLab 731-ISM, Mettler  
177 Toledo) ranging from 0.01 to 1 000 mS.cm<sup>-1</sup>. Powder was fed by a vibratory feeder (DR 100  
178 Model, Retsch, France). Vessel design and reconstitution conditions were based on the  
179 literature (**Figure 1**) [2,19,24,28,37]. Powder reconstitution experiments were performed with  
180 distilled water at 25.0 ± 0.2 °C in the following conditions: 10.0 % (w/w) powder/water mass  
181 ratios with a total mass (powder + water) fixed at 2.650 kg, 800 rpm stirring rate, and 3.6 g/s  
182 feeding rate. Experiment duration was adapted for each powder between 15 min and 2 h.

183 **Conductivity normalization.** Upon powder reconstitution, conductivity evolved from an initial  
184 value of 0 µS/cm (the real value was inferior to the conductimeter sensitivity) to a final value  
185 that depended from powder nature (**Figure 2 - left**). Therefore, to be able to fit conductivity  
186 curves and determine reconstitution times, conductivity values averaged from the three  
187 experimental replicates were normalized following **Equation 5**:

$$188 \quad c(t) = \frac{\kappa(t) - \kappa_{ini}}{\kappa_{fin} - \kappa_{ini}} \quad \text{(Equation 5)}$$

189 Where:

190 - c(t) designates normalized conductivity (-);

191 - κ(t) stands for conductivity at time t (µS.cm<sup>-1</sup>);

192 - κ<sub>ini</sub> represents the initial conductivity, i.e. distilled water conductivity (close to 0 µS.cm<sup>-1</sup>);

193 - κ<sub>fin</sub> corresponds to the final conductivity (µS.cm<sup>-1</sup>).

194 Consequently, all normalized conductivity curves started at 0 and ended at 1. These curves had  
195 a sigmoidal shape (**Figure 2 - right**) which can be related to the different steps of powder  
196 reconstitution:

- 197 • first, upon powder wetting/swelling and sinking, very few powder components were  
198 solubilized and conductivity slightly increased very slightly over a rather long time;
- 199 • then, powder dispersion and solubilization occurred, leading to a marked conductivity  
200 increase;
- 201 • finally, as the amount of powder to reconstitute became low, conductivity still increased  
202 but evolved more slowly, until reaching the final conductivity once all soluble powder  
203 material was solubilized.

204 **Curve fitting.** The normalized conductivity curves were fitted with the OriginPro 2019 software  
205 (OriginLab Corporation) using the least squares method solved by the Levenberg Marquardt  
206 iteration algorithm. It was found that the Hill model (**Equation 6**) was the most suitable and  
207 simple model to fit normalized conductivity curves of all powders [38]:

$$208 \quad c(t) = c_0 + (c_\infty - c_0) \frac{t^n}{k^n + t^n} \quad \text{(Equation 6)}$$

209 With:

210 -  $c_0$ , initial normalized conductivity (%);

211 -  $c_\infty$ , final normalized conductivity (%);

212 -  $t$ , time (s);

213 -  $k$  (s) and  $n$  (-), Hill model time constant and power, respectively.

214 For all powders, initial and final normalized conductivity being equal to 0 % and 100 %,   
215 respectively, the Hill model could simply be rewritten as (**Equation 7**):

$$216 \quad c(t) = \frac{t^n}{k^n + t^n} \quad \text{(Equation 7)}$$

217 Model parameter  $k$  corresponds to the time needed to reach 50 % normalized conductivity. The  
218 slope of normalized conductivity curve at  $t = k$  was found equal to  $\frac{n}{4k}$ , then it can be considered  
219 that the model parameter  $n$  gives an indication of the powder reconstitution rate once wetted,  
220 swelled, and sinked, i.e. in the dispersion and solubilization steps of powder reconstitution.  
221 Last, powder reconstitution time was deduced from obtained Hill model by taking the time  
222 needed to reach 95 % normalized conductivity, it was then noted  $t_{95\%}$  and expressed in seconds.

## 223 **2.5. Statistical analysis**

224 All analyzes were carried out on the thirty-six powders and performed in triplicate.  
225 Principal Component Analysis (PCA) was used to link powder physicochemistry and  
226 manufacturing process to reconstitution behavior using Unscrambler (CAMO software AS,  
227 Norway). [Due to the huge number of powders analyzed, the objective of PCA was to determine](#)  
228 [which main underlying factors could explain the observed differences in reconstitutability.](#)

## 230 **3. Results and discussion**

### 231 **3.1. Impact of particle size distribution and lipids content on powder** 232 **reconstitution**

233 To highlight the impact of particle size distribution and lipids content on powder reconstitution  
234 behavior, a focus is carried out [here](#) on instant coffee and milk powders.

#### 235 **3.1.1. Powders physicochemical characterization**

236 Raw and ground instant coffee powders presented similar chemical composition and differed  
237 in median particle size  $D_{50}$  (around 1 630  $\mu\text{m}$  and 31  $\mu\text{m}$ , respectively) ([Tables S1 & S2,](#)  
238 [Supplementary Material](#)). Raw instant coffee powder had a monomodal particle size  
239 distribution with a span around 1, whereas [the particle size distribution of ground instant coffee](#)  
240 [powder](#) was bimodal with a span around 13, which was consistent for an agglomerated powder.

241 On the opposite, skim and whole milk powders differed in chemical composition and presented  
242 similar median particle sizes  $D_{50}$  (around 80  $\mu\text{m}$ ) and monomodal particle size distributions  
243 (**Tables S1 & S2, Supplementary Material**). Skim milk powder presented a low lipid content  
244 (1.5 % on dry basis), whereas whole milk powder was rich in lipids (26 % on dry basis).

245 The water contents of coffee and milk powders were comprised between 1.6 and 2 % on wet  
246 basis and their water activity ranged between 0.16 and 0.34 (**Table S3, Supplementary**  
247 **Material**), which is favorable to their preservation [40,38].

### 248 **3.1.2. Particle surface characterization**

249 The surface atomic proportions of instant coffee and milk powders are displayed in **Figure 3 -**  
250 **left**. Carbon, oxygen, and nitrogen surface proportions were similar for all powders except for  
251 whole milk powder that had more surface carbon and less surface oxygen and nitrogen. Surface  
252 and bulk compositions of milk powders were compared in **Figure 4**. The bulk composition was  
253 measured by classical chemical analysis techniques whereas the surface composition was  
254 calculated with the matrix (Equations 4). It appears that whole milk powder was mainly covered  
255 by lipids, whereas skim milk powder presented a surface overrepresentation mostly in lipids  
256 but also in proteins in a lesser extent [41]. These differences between bulk and surface  
257 compositions are known to strongly influence powder reconstitution behavior [42]. For coffee  
258 powders, it was not possible to use the matrix method as nitrogen is also present in non-proteic  
259 compounds like caffeine (and not only proteins as for milk powders). Nevertheless, the surface  
260 atomic composition was still informative. It was very close to that of skim milk powder for  
261 carbon and oxygen, but nitrogen contents were slightly lower (**Figure 3 - left**). Carbon bond  
262 ratios of the four powders are presented in **Figure 3 - right**. Again, only whole milk powder  
263 had a clearly more hydrophobic surface than the three other powders.

### 264 3.1.3. Powder reconstitution kinetics

265 **Impact of particle size on reconstitution.** The reconstitution behavior of raw and ground instant  
266 coffee powders, differing in median particle size, is detailed in **Table 1** and **Figure 5A**. The  
267 wetting step is known to be highly linked to the median particle size, as well as bulk and surface  
268 compositions. As previously shown, the bulk and surface composition of instant coffee powders  
269 were identical (**Figure 3** and **Supplementary Material Table S1**). It appeared that wettability  
270 was better for the raw instant coffee powder composed of larger particles (23 s wetting time)  
271 than for the ground instant coffee powder (52 s wetting time). Indeed, a high proportion of large  
272 and/or agglomerated particles is known to improve powder wettability [10,11]. The  
273 reconstitution time was also shorter for raw instant coffee powder probably owing to its shorter  
274 wetting step. Hill model parameters, time constant  $k$  and power  $n$ , were also informative about  
275 the reconstitution kinetics.  $k$ , corresponding to the time needed to reach half of the final  
276 conductivity, was doubled for the ground instant coffee powder, denoting slower reconstitution  
277 kinetics. Moreover, the slope of the normalized conductivity curve at half maximal normalized  
278 conductivity, equal to  $\frac{n}{4k}$ , could be viewed as an indicator of reconstitution rate; for instant  
279 coffee powders,  $\frac{n}{4k}$  equaled 0.050 and 0.020 s<sup>-1</sup> for raw and ground samples, respectively. This  
280 confirms the slower reconstitution rate of ground instant coffee powder, likely due to its lower  
281 median particle size preventing its correct wetting and dispersibility [22,23]; indeed,  
282 interparticular cohesive forces are expected to be stronger between small particles owing to  
283 their higher contact surface [43,44].

284 **Impact of lipids content on reconstitution.** Whole and skim milk powders presented similar  
285 median particle sizes but differed in chemical composition **through their lipids content**. Their  
286 reconstitution behavior is presented in **Table 1** and **Figure 5B**. The bulk and surface  
287 compositions of these powders markedly differed. Whole milk powder, having a well higher  
288 surface lipid content, had a well higher surface hydrophobicity [10,41]. This surely caused that

289 whole milk powder had a wetting time (68 s) longer than skim milk powder (35 s), the latter  
290 having a more hydrophilic surface which is known to improve wettability. These results met  
291 literature regarding the negative influence of surface hydrophobicity on powder wetting  
292 [14,16,42,45]. The reconstitution time ( $t_{95\%}$ ) was shorter for the skim milk powder, confirming  
293 the deleterious effect of surface lipids on reconstitution. This may be related to wettability that  
294 is well impaired at higher surface lipid content. Hill model parameters (time constant  $k$  and  
295 power  $n$ ) were greater for skim milk powder, indicating a higher reconstitution rate and a shorter  
296 reconstitution time. This was confirmed by the calculation of the slope of the normalized  
297 conductivity curve at half-height (which is an indicator of reconstitution rate as previously  
298 mentioned):  $\frac{n}{4k}$  equaled 0.029 and 0.018  $s^{-1}$  for skim and whole milk powders, respectively.  
299 This confirms the slower reconstitution rate of whole milk powder.

### 300 **3.2. Surface composition of the thirty-six food powders**

301 On the best knowledge of the authors, food powder surface analysis by XPS has been performed  
302 only on few powders (i.e. dairy or wheat powders) [42,46]. For the first time, a large  
303 investigation was performed on thirty-six food powders in order to collect surface atomic  
304 proportions (in carbon, oxygen, and nitrogen), as well as C/O and C-C/(other carbon bonds)  
305 ratios as indicators of surface hydrophobicity (**Table S2, Supplementary Material**). It appears  
306 that surface chemical composition was highly variable for investigated powders. Three  
307 hypothesized powder groups were constituted on the basis of surface  
308 hydrophilicity/hydrophobicity (**Figure 6**). **The most hydrophilic** surface powders were  
309 associated **with the lowest** ranges of C/O and C-C/other C bonds ratios of 0 - 2 and 0 - 1,  
310 respectively. Intermediate (slightly hydrophobic) powders corresponded **to ranges** of C/O and  
311 C-C/other C bonds ratios of 2 - 4 and 1 - 2, respectively. **The most hydrophobic** surface powders  
312 were defined **for the highest** ranges of C/O and C-C/other C bonds ratios of 4 - 7 and 2 - 4,  
313 respectively. Sugars, acerola juice, lactose, **agar, xanthan, pectin** and baking soda were then

314 sorted as the most hydrophilic surface powders of the study. On the opposite, hazelnut, cocoas,  
315 milk protein concentrate, whole milk and whey proteins powders were classified as the most  
316 hydrophobic surface powders of the study. Many investigated food powders (cereal powders,  
317 flours, coffee powders, couscous powders, instant mash potatoes, etc.) fell into the intermediate  
318 category of slightly hydrophobic surface.

### 319 **3.3. Principal component analysis linking physicochemical properties,** 320 **manufacturing process, and reconstitution behavior of the thirty-six food** 321 **powders**

322 A principal component analysis was performed to link powder physicochemical properties and  
323 their reconstitution behavior. The main objective was to capture the variability of data in a few  
324 principal components, which encompass the most variation of the dataset. The data taken into  
325 consideration in the principal component analysis are related to powder bulk and surface  
326 composition, manufacturing process, median particle size, and reconstitution properties  
327 (wetting time and reconstitution time) (Tables S1, S2, and S3, Supplementary Material).

328 The loading plot shows how strongly each powder physicochemical characteristic influences  
329 the variability amongst powders, represented according to the two principal components PC1  
330 and PC2 (Figure 7). PC1, corresponding to the horizontal axis and representing 34 % of powder  
331 characteristics variability, was linked to reconstitution times ( $t_{95\%}$ , k, n) and manufacturing  
332 process. PC2, associated with the vertical axis and corresponding to 27 % powder variability,  
333 was correlated to median particle size (D50), wetting time, and surface chemical composition.  
334 Hence, a long wetting time was related to a small median particle size and an elevated surface  
335 hydrophobicity [42,47]. The relationship between median particle size and wettability had  
336 already been demonstrated on milk powders [48], the current work thus makes it possible to  
337 generalize this relationship to food powders whatever their composition and manufacturing

338 process. Hill model parameters logically appeared to be correlated with the reconstitution time:  
339 a high  $k$  value or a low  $n$  value (both inducing a low [slope of the normalized conductivity curve](#)  
340 [at half maximal normalized conductivity](#)  $\frac{n}{4k}$  value) were related to a high reconstitution time,  
341 i.e. slow reconstitution kinetics. The score plot locates investigated powder samples on the  
342 [PC1](#)  $\times$  [PC2](#) plot (**Figure 8**). Powders situated on the bottom side (negative [PC2](#)) had longer  
343 wetting times, whereas powders found in the right side (positive [PC1](#)) were linked to long  
344 reconstitution times. Therefore, four groups of powders were defined on the basis of their  
345 reconstitution behavior: short wetting and reconstitution times (green); long wetting time and  
346 short reconstitution time (yellow); short wetting time and long reconstitution time (orange);  
347 long wetting and reconstitution times (red). The sorting of investigated powders into these four  
348 groups were in great accordance with the literature.

349 The reconstitution behavior of milk powders has been extensively described in the literature  
350 contrarily to other food powders [6,18,21]. For the first time, the main factors influencing the  
351 reconstitution are identified for a large variety of powders. Manufacturing processes, mainly  
352 grinding and spray-drying, were associated to long and short reconstitution times, respectively.  
353 Indeed, cereal powders ([couscous powders](#), flour, semolina, wheat powders), obtained by  
354 grinding, had slow reconstitution kinetics. Besides, small particle and hydrophobic surface  
355 powders were correlated to long wetting times, whereas large particle and hydrophilic powders  
356 were associated with short wetting times. Finally, powder within each of red and green groups  
357 were also similar in terms of bulk composition (high lipids and high sugar contents,  
358 respectively).

## 359 **4. Conclusion**

360 For the first time, the main factors influencing powder reconstitution behavior (median particle  
361 size, chemical composition, surface composition, and manufacturing process) have been

362 identified for a large number of food powders. The current study permitted to confirm and  
363 generalize the knowledge developed so far on a limited number of powders and identify four  
364 reconstitution behaviors. The industrial interest of the proposed classification resides in the fact  
365 that powder reconstitution properties can be modulated by changing powder formulation  
366 (particle size distribution, surface composition, bulk composition, etc.) without modifying the  
367 reconstitution conditions (stirring speed, temperature, etc.). It would be interesting to go one  
368 step further by linking powder physicochemical characteristics and manufacturing process to  
369 the different reconstitution steps.

370

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378

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- 527

## **Highlights**

- Food powders were sorted according to their reconstitution behavior
- Principal component analysis linked powder characteristics to reconstitutability
- Crucial physicochemical factors for wetting and reconstitution were identified

Sample CRediT author statement

Tristan Fournaise : Writing, Investigation, Methodology, Review & Editing

Claire GAIANI : Conceptualization, Supervision, Review & Editing, Funding acquisition

Jérémy PETIT : Conceptualization, Supervision, Review & Editing,



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## Figure captions

**Figure 1.** Experimental setup with a focus on the geometrical characteristics of the A200 Impeller and the reactor (h:d:H:D of 1:1:3:3 with  $H = D = 150$  mm and  $h = d = 50$  mm).

**Figure 2.** Temporal evolution of conductivity upon reconstitution before (left) and after (right) normalization.

**Figure 3.** Surface atomic compositions (left) and carbon bond ratios (right) of instant coffee, ground instant coffee, skim milk, and whole milk powders.

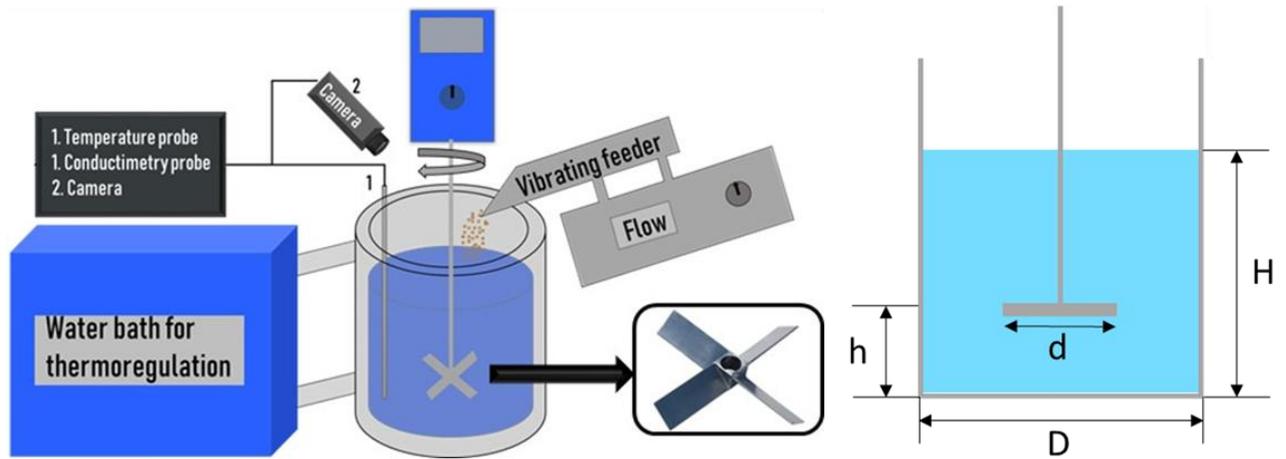
**Figure 4.** Bulk and surface compositions of skim and whole milk powders.

**Figure 5.** Effect of median particle size (A - left) and fat content (B - right) on powder reconstitution kinetics.

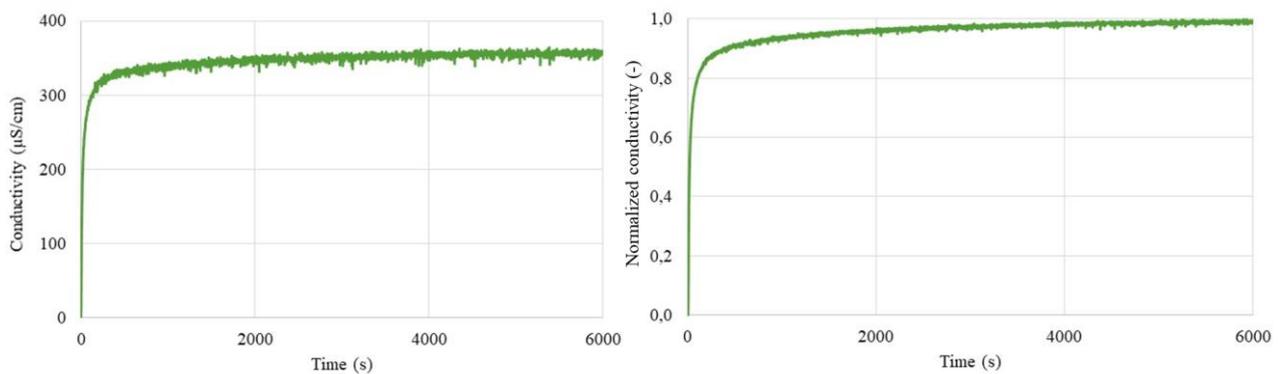
**Figure 6.** Hypothesized classification of investigated powders into three groups (more hydrophobic, intermediate, and more hydrophilic) from their surface atomic composition.

**Figure 7.** Correlation scores obtained from PCA for the physicochemical and reconstitution characteristics of the thirty-six investigated powders.

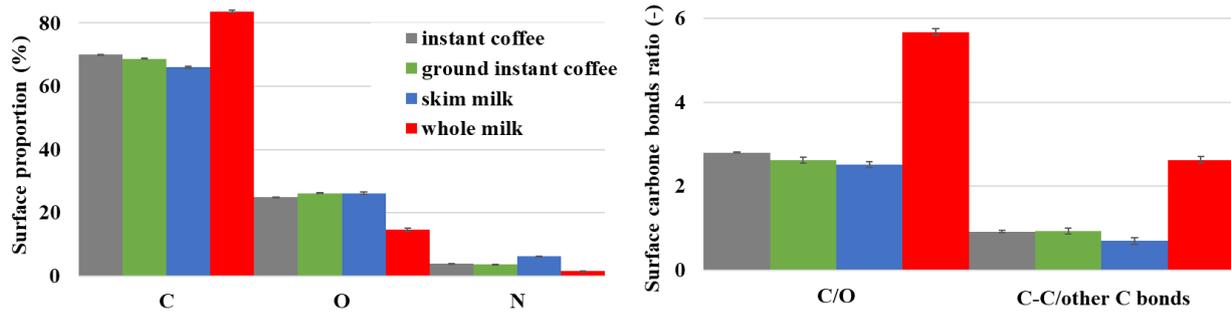
**Figure 8.** Classification of the thirty-six investigated powders according to their wettability and reconstitutability deduced from PCA (loading scores).



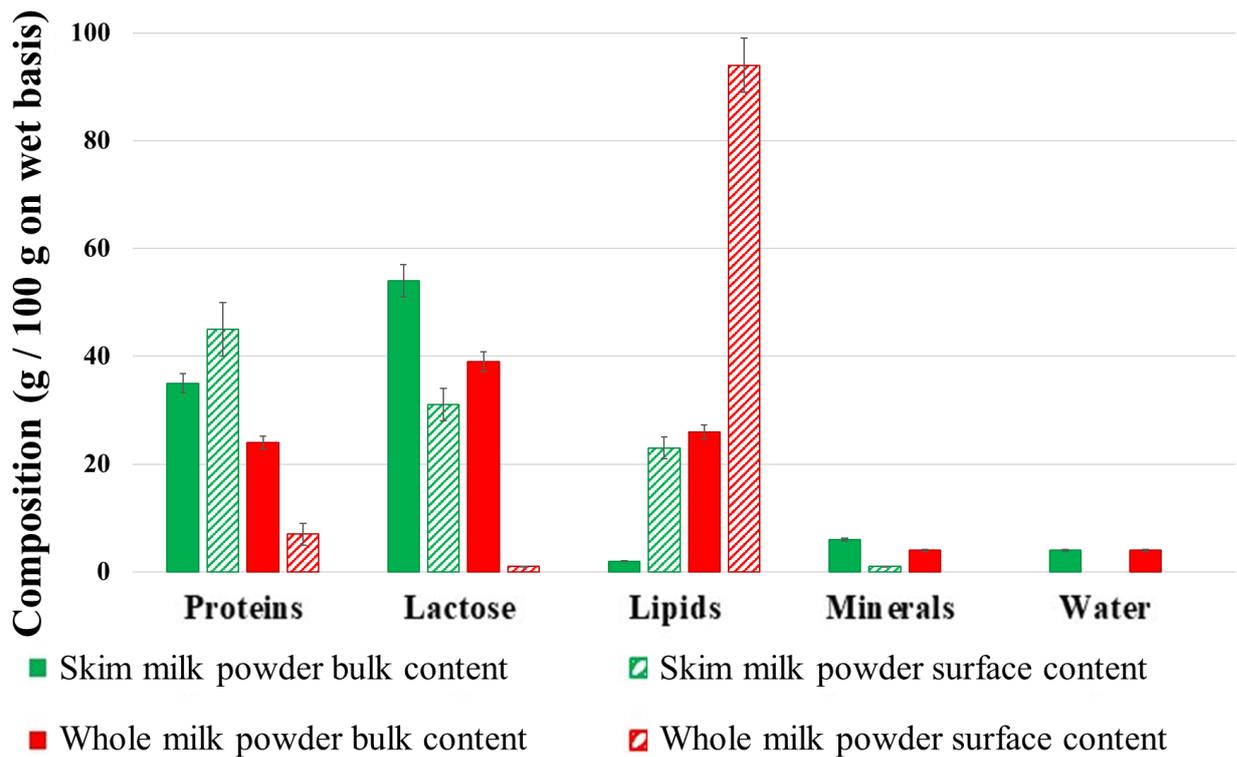
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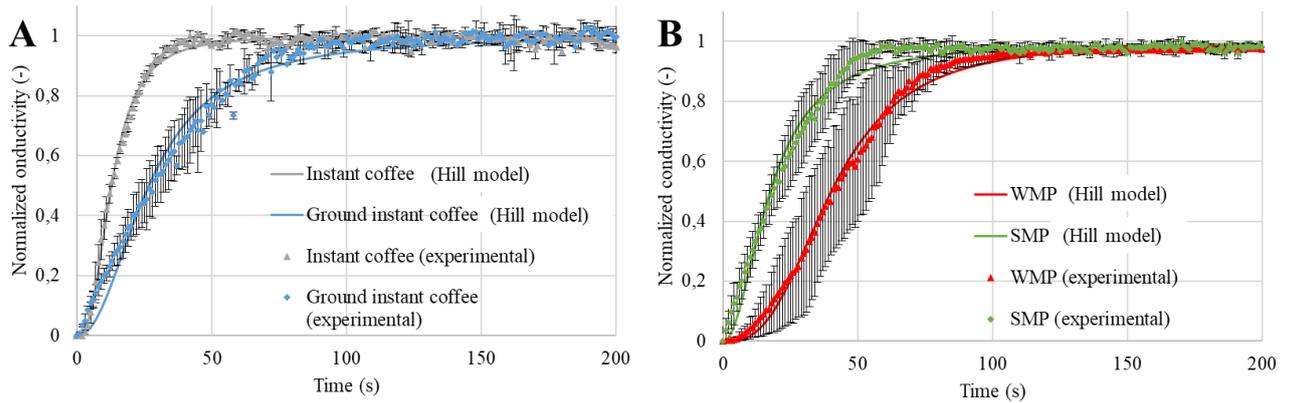
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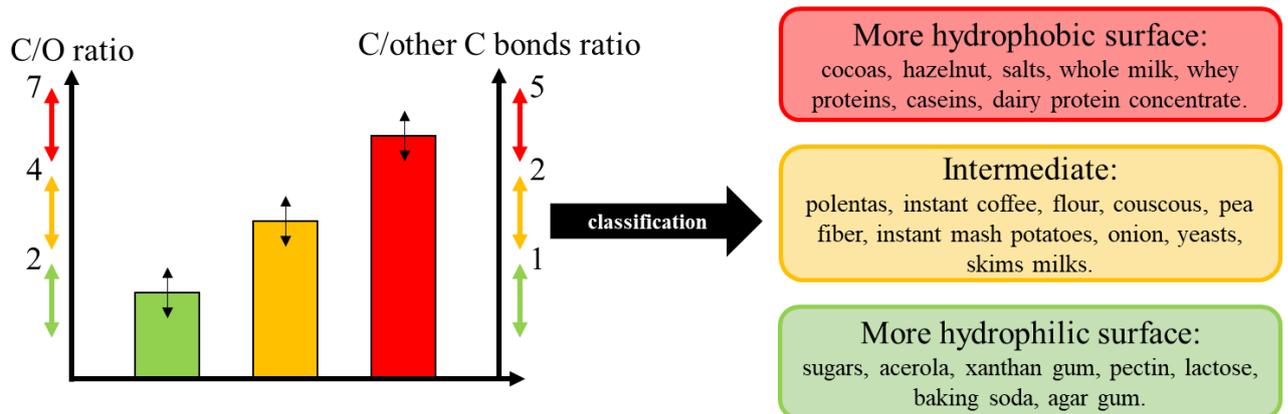
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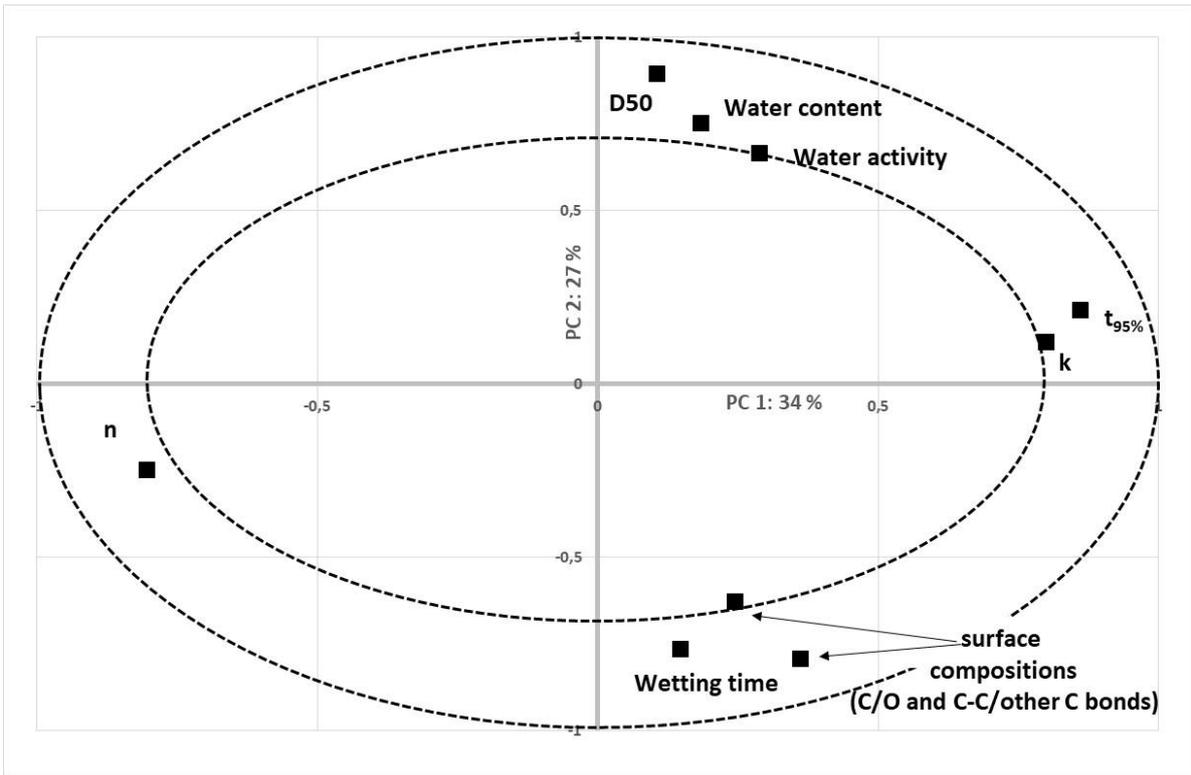
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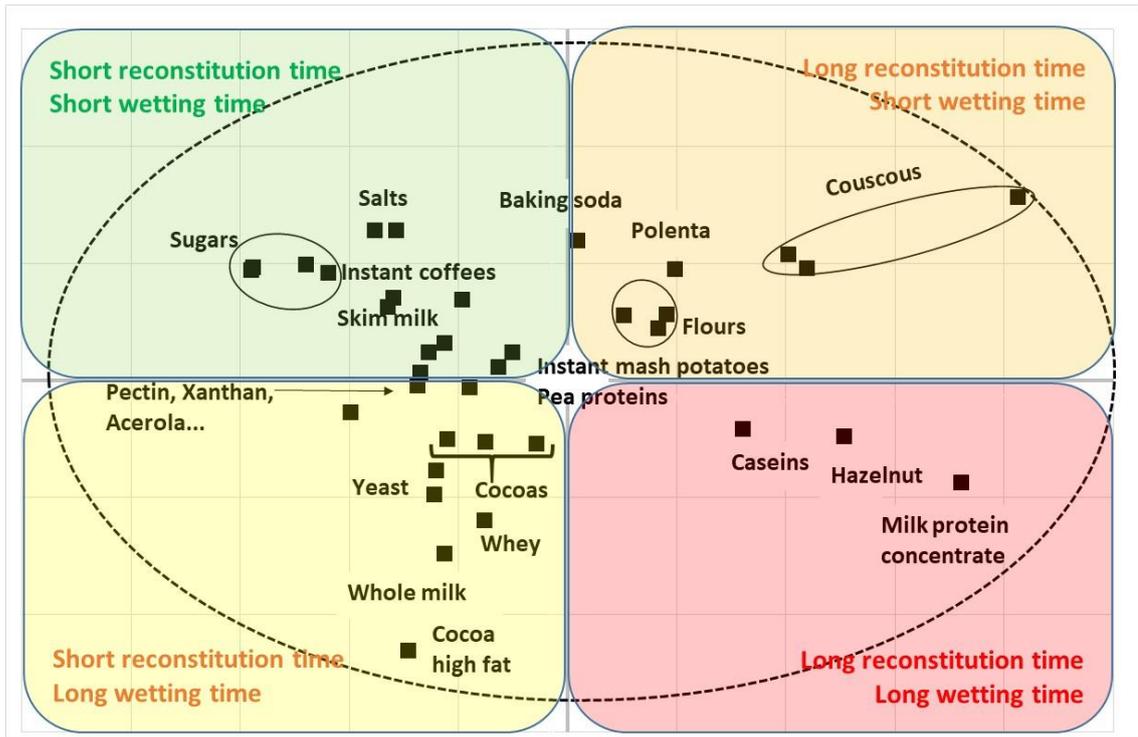
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## Tables

**Table 1.** Wetting and reconstitution times, as well as Hill model parameters (time constant k and power n) and coefficient of determination ( $R^2$ ) of normalized conductivity curve fitting by Hill model of investigated milk and coffee powders.

	Wetting time (s)	Reconstitution time $t_{95\%}$ (s)	Hill model time constant k (s)	Hill model power n (-)	Hill model coefficient of determination $R^2$
Skim milk powder	$35 \pm 4$	$86.0 \pm 10.0$	$27.2 \pm 2.92$	$2.76 \pm 0.13$	0.95
Whole milk powder	$68 \pm 8$	$105.0 \pm 10.4$	$133.6 \pm 9.18$	$0.96 \pm 0.01$	0.98
Instant coffee powder	$22 \pm 2$	$34.7 \pm 1.1$	$8.9 \pm 0.2$	$2.16 \pm 0.08$	0.98
Ground instant coffee powder	$51 \pm 4$	$106.5 \pm 6.8$	$26.7 \pm 3.7$	$2.15 \pm 0.31$	0.98

Sample CRediT author statement

Tristan Fournaise : Writing, Investigation, Methodology, Review & Editing

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