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# Main powder physicochemical characteristics influencing their <br> reconstitution behavior 

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#### Abstract

Thirty-six food powders corresponding to a wide range of physicochemical characteristics (bulk and surface chemical composition, median particle size, span, water activity) and manufacturing processes (grinding, freeze-drying, spray-drying, crystallization) were investigated for their reconstitutability. Their reconstitution profiles were acquired by conductivity measurements and fitted with Hill model. Physicochemical characteristics of investigated powders were correlated to their wetting and reconstitution times by principal component analysis. Four powder categories were identified on the basis of wetting and reconstitution times; i.e. green group (good reconstitutability); yellow and orange groups (intermediate reconstitutability); red group (poor reconstitutability). Long wetting times were associated to high particle surface hydrophobicity, small median particle size, as well as high protein and lipid contents in the powder bulk. Long reconstitution times were linked to the powder manufacturing process (i.e. grinding), as well as to low sugar content in the powder bulk.


Keywords: powder reconstitution; food powder; wettability; particle size; chemical composition.

## 1. Introduction

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

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

Powder wetting is the phenomenon defined by the replacement of the gaseous phase by water at the powder surface. It can be experimentally determined by using standard methods of the International Dairy Federation [7] developed for milk powders. Previous studies evidenced that powder wetting is more efficient for large particles (at least over $90 \mu \mathrm{~m}$ ) or agglomerated powders [8-11]. High particle size induces small particle-solvent contact angle, thus facilitating wetting. High porosity and spherical particles also have a good wetting behavior [9,12]. In
addition to physical properties, chemical composition can also influence powder wettability. For instance, high lipid or protein contents makes powder surface hydrophobic, leading to poor wettability $[11,13,14]$. On the contrary, hydrophilic components located at powder surface like carbohydrates or surfactants enhance its wettability [12,14-16].

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

Dispersibility is defined as the ability of a powder to disperse into water. Powder dispersion consists in two simultaneous mechanisms [20,21]: lumps and agglomerates breaking into individual particles and individual particles fragmentation, both leading to size reduction and specific surface area increase, further facilitating interactions of the solid matrix with water. Dispersion occurs when the forces exerted by the solvent to separate particles overcome particle cohesion [11,22,23]. The dispersion rate is enhanced for high porosity, low density, or lowly cohesive particles [8,23,24].

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

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

## 2. Material and methods

### 2.1. Investigated powders

Thirty-six powders, varying in chemical composition (water, lipids, proteins, carbohydrates and minerals contents), physical properties (particle size distribution) and manufacturing process (spray-drying, drum-drying, freeze-drying, crystallization), were investigated (Supplementary Material Table S1). In order to change the particle size distribution while keeping the same powder bulk composition, polenta and instant coffee powder were ground (to reach 1.0 mm and 0.08 mm , respectively) with a ZM 200 (Retsch, France) ultra- centrifugal mill at 8000 rpm using small powder amounts in order to limit the heating of the grinding system and thus of produced powder [29]. This approach allowed obtaining a wide range of reconstitution profiles.

### 2.2. Physicochemical characterization

### 2.2.1. Particle size distribution

Laser diffraction (Mastersizer S, Malvern Instruments Ltd, Malvern, UK) was used to evaluate particle size distribution with a $5 \mathrm{~mW} \mathrm{He}-\mathrm{Ne}$ laser operating at 632.8 nm wavelength and a 300

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

$$
\begin{equation*}
\operatorname{span}=\frac{D_{90}-D_{10}}{D_{50}} \tag{Equation1}
\end{equation*}
$$

### 2.2.2. Water content

The water content was determined by the mass loss of 2.5 g powder after drying at $103{ }^{\circ} \mathrm{C}$ during at least 3 h following ISO 5537:2004 [32].

### 2.2.3. Water activity

Water activity ( $\mathrm{a}_{\mathrm{w}}$ ) was determined with a HygroPalm23-AW portable water activity-meter (Rotronic, France) in a temperature-controlled room at $20^{\circ} \mathrm{C}$ using a 3 cm powder layer following ISO 21807:2004 [33].

### 2.2.4. Chemical composition of powder bulk

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

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

Total lipids were quantified with a method adapted from [34]. 5 g powder was dissolved into a 2:1 (v/v) mixture of chloroform/methanol for lipid extraction. The mixture was stirred at 700 rpm during 15 min . Then, the soluble phase containing sample lipids was separated by filtration on a sintered glass filter of $10-16 \mu \mathrm{~m}$ porosity and recovered in a round-bottom boiling flask that was previously dried and weighted. The solvent of the soluble phase was removed with a rotary vacuum evaporator (Heidolph Laboratora 4000) at low stirring and $40^{\circ} \mathrm{C}$. The flask empty from residual solvent was dried at $103{ }^{\circ} \mathrm{C}$ for 15 min to ensure complete solvent evaporation and finally weighted.

Minerals content was determined by calcining 2 g sample at $550^{\circ} \mathrm{C}$ during 12 h .

Lactose content was obtained by difference with other components (Equation 2):
$\%$ lactose $=100-(\%$ water $+\%$ proteins $+\%$ minerals $+\%$ lipids $)$
(Equation 2)

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

XPS measurements are performed in ultra-high vacuum $\left(10^{-8} \mathrm{~Pa}\right)$. The surface of the powder is irradiated, which causes a complete transfer of X-ray photon energy to atomic electrons of the sample. When the electron binding energy Eb is lower than the photon energy ho, with h the Planck constant $\left(6.63 \times 10^{-34} \mathrm{~m}^{2} . \mathrm{kg} \cdot \mathrm{s}^{-1}\right)$ and $v$ the radiation frequency $\left(\mathrm{s}^{-1}\right)$, an electron is emitted from the atom with a typical kinetic energy $\mathrm{E}_{\mathrm{k}}$ related to $\mathrm{E}_{\mathrm{b}}$ (Equation 3):

$$
\begin{equation*}
E_{b}=h v-E_{k} \tag{Equation3}
\end{equation*}
$$

Analyses were realized with a Kratos Axis Ultra (Kratos Analytical, Manchester, UK) X-ray photoelectron spectrometer with a monochromatic Al X-ray source at 150 W . The analytical area was about $700 \mu \mathrm{~m} \times 300 \mu \mathrm{~m}$ and the analytical depth 10 nm . Powder samples were attached to a sample holder with double-sided conductive tape. XPS spectra were then recorded and
treated using the Kratos Vision software. Quantification was performed using the photoemission cross-sections and transmission coefficients given in the Kratos Vision package. The main elements (C, O, N, P, Ca) detected at powder surface were identified by means of their respective binding energy. Also, the carbon peak was deconvoluted into its main chemical bonds (C-(C, H); C-(O, N); C=O; O-C=O).

For milk powders, it was assumed that nitrogen was present in proteins only; therefore, the surface composition can be calculated using the matrix method based on theoretical atomic composition of main components $[8,13,35,36]$. Briefly, the atomic proportions of carbon, oxygen, and nitrogen were used in a matrix formula (Equations 4.1-4.3) in order to deduce the surface proportions in the different sample compounds: $\alpha_{\text {La }}$ for lactose (La), $\alpha_{\mathrm{P}}$ for proteins (P), and $\alpha_{\text {Li }}$ for lipids (Li).
$I^{C}=\alpha_{\mathrm{P}} \times I^{C . P}+\alpha_{\mathrm{La}} \times I^{C . L a}+\alpha_{L i} \times I^{C . L i}$
(Equation 4.1)
$I^{O}=\alpha_{\mathrm{P}} \times I^{O . P}+\alpha_{\mathrm{La}} \times I^{0 . L a}+\alpha_{L i} \times I^{O . L i}$
(Equation 4.2)
$I^{N}=\alpha_{\mathrm{P}} \times I^{N . P}+\alpha_{\mathrm{La}} \times I^{N . L a}+\alpha_{L i} \times I^{N . L i}$
$I^{\mathrm{C}}, I^{\mathrm{O}}$, and $I^{\mathrm{N}}$ are the mole fractions of carbon, oxygen, and nitrogen at the sample surface (these values were directly obtained by XPS from the C1s, O1s, and N1s peak areas ); $I^{\text {C.P }}, \mathrm{I}^{\mathrm{C} . \mathrm{La}}$, and $I^{\text {C.Li }}$ are the mole fractions of carbon in proteins, lactose, and lipids, respectively; $\mathrm{I}^{\mathrm{O} P}, \mathrm{I}^{\mathrm{O} . \mathrm{La}}$, and $I^{0 . L i}$ are the mole fractions of oxygen in proteins, lactose, and lipids, respectively; $I^{\text {N.P }}, I^{\text {N.La }}$, and $I^{\text {N.Li }}$ are the mole fractions of nitrogen in proteins, lactose, and lipids.

### 2.4. Powder reconstitution behavior

### 2.2.4. Wettability

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

### 2.2.5. Reconstitutability followed by conductimetry

Experimental setup. An experimental setup (Figure 1) was developed to follow powder reconstitution kinetics by conductimetry. It was constituted of a jacketed glass reactor (GL18, Grosseron) thermostated by a heated water bath (Eco Gold, Lauda), a mechanical stirrer (ZR 2102 control, Heidolph), and a 5.08 cm diameter impeller (Pitch blade turbine 2" 32Ra, Fusion fluid equipment). Powder reconstitution kinetics were followed by conductimetry (SevenCompact S230, Mettler Toledo) with a conductivity probe (InLab 731-ISM, Mettler Toledo) ranging from 0.01 to $1000 \mathrm{mS} . \mathrm{cm}^{-1}$. Powder was fed by a vibratory feeder (DR 100 Model, Retsch, France). Vessel design and reconstitution conditions were based on the literature (Figure 1) [2,19,24,28,37]. Powder reconstitution experiments were performed with distilled water at $25.0 \pm 0.2^{\circ} \mathrm{C}$ in the following conditions: $10.0 \%(\mathrm{w} / \mathrm{w})$ powder/water mass ratios with a total mass (powder + water) fixed at 2.650 kg , 800 rpm stirring rate, and $3.6 \mathrm{~g} / \mathrm{s}$ feeding rate. Experiment duration was adapted for each powder between 15 min and 2 h .

Conductivity normalization. Upon powder reconstitution, conductivity evolved from an initial value of $0 \mu \mathrm{~S} / \mathrm{cm}$ (the real value was inferior to the conductimeter sensitivity) to a final value that depended from powder nature (Figure 2 - left). Therefore, to be able to fit conductivity curves and determine reconstitution times, conductivity values averaged from the three experimental replicates were normalized following Equation 5:

$$
c(t)=\frac{\kappa(t)-\kappa_{i n i}}{\kappa_{f i n}-\kappa_{i n i}}
$$

Where:

- $\mathrm{c}(\mathrm{t})$ designates normalized conductivity (-);
$-\kappa(\mathrm{t})$ stands for conductivity at time $\mathrm{t}\left(\mu \mathrm{S} . \mathrm{cm}^{-1}\right)$;
$-\kappa_{\text {ini }}$ represents the initial conductivity, i.e. distilled water conductivity (close to $0 \mu \mathrm{~S} . \mathrm{cm}^{-1}$ );
- $\kappa_{\text {fin }}$ corresponds to the final conductivity $\left(\mu \mathrm{S} . \mathrm{cm}^{-1}\right)$.

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

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

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

$$
\begin{equation*}
c(t)=c_{0}+\left(c_{\infty}-c_{o}\right) \frac{t^{n}}{k^{n}+t^{n}} \tag{Equation6}
\end{equation*}
$$

With:

- $c_{0}$, initial normalized conductivity (\%);
- $c_{\infty}$, final normalized conductivity (\%);
- t, time (s);
$-\mathrm{k}(\mathrm{s})$ and $\mathrm{n}(-)$, Hill model time constant and power, respectively.
For all powders, initial and final normalized conductivity being equal to $0 \%$ and $100 \%$, respectively, the Hill model could simply be rewritten as (Equation 7):

$$
\begin{equation*}
c(t)=\frac{t^{n}}{k^{n}+t^{n}} \tag{Equation7}
\end{equation*}
$$

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

### 2.5. Statistical analysis

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

## 3. Results and discussion

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

To highlight the impact of particle size distribution and lipids content on powder reconstitution behavior, a focus is carried out here on instant coffee and milk powders.

### 3.1.1. Powders physicochemical characterization

Raw and ground instant coffee powders presented similar chemical composition and differed in median particle size $\mathrm{D}_{50}$ (around $1630 \mu \mathrm{~m}$ and $31 \mu \mathrm{~m}$, respectively) (Tables $\mathrm{S} 1 \&$ S2, Supplementary Material). Raw instant coffee powder had a monomodal particle size distribution with a span around 1 , whereas the particle size distribution of ground instant coffee powder was bimodal with a span around 13 , which was consistent for an agglomerated powder.

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

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

### 3.1.2. Particle surface characterization

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

### 3.1.3. Powder reconstitution kinetics

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

Impact of lipids content on reconstitution. Whole and skim milk powders presented similar median particle sizes but differed in chemical composition through their lipids content. Their reconstitution behavior is presented in Table 1 and Figure 5B. The bulk and surface compositions of these powders markedly differed. Whole milk powder, having a well higher surface lipid content, had a well higher surface hydrophobicity [10,41]. This surely caused that
whole milk powder had a wetting time ( 68 s ) longer than skim milk powder ( 35 s ), the latter having a more hydrophilic surface which is known to improve wettability. These results met literature regarding the negative influence of surface hydrophobicity on powder wetting [14,16,42,45]. The reconstitution time ( $\mathrm{t}_{55 \%}$ ) was shorter for the skim milk powder, confirming the deleterious effect of surface lipids on reconstitution. This may be related to wettability that is well impaired at higher surface lipid content. Hill model parameters (time constant k and power $n$ ) were greater for skim milk powder, indicating a higher reconstitution rate and a shorter reconstitution time. This was confirmed by the calculation of the slope of the normalized conductivity curve at half-height (which is an indicator of reconstitution rate as previously mentioned): $\frac{n}{4 k}$ equaled 0.029 and $0.018 \mathrm{~s}^{-1}$ for skim and whole milk powders, respectively. This confirms the slower reconstitution rate of whole milk powder.

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

On the best knowledge of the authors, food powder surface analysis by XPS has been performed only on few powders (i.e. dairy or wheat powders) [42,46]. For the first time, a large investigation was performed on thirty-six food powders in order to collect surface atomic proportions (in carbon, oxygen, and nitrogen), as well as $\mathrm{C} / \mathrm{O}$ and $\mathrm{C}-\mathrm{C} /($ other carbon bonds) ratios as indicators of surface hydrophobicity (Table S2, Supplementary Material). It appears that surface chemical composition was highly variable for investigated powders. Three hypothesized powder groups were constituted on the basis of surface hydrophilicity/hydrophobicity (Figure 6). The most hydrophilic surface powders were associated with the lowest ranges of $\mathrm{C} / \mathrm{O}$ and C -C/other C bonds ratios of $0-2$ and $0-1$, respectively. Intermediate (slightly hydrophobic) powders corresponded to ranges of $\mathrm{C} / \mathrm{O}$ and C-C/other C bonds ratios of 2-4 and 1-2, respectively. The most hydrophobic surface powders were defined for the highest ranges of $\mathrm{C} / \mathrm{O}$ and C -C/other C bonds ratios of $4-7$ and 2-4, respectively. Sugars, acerola juice, lactose, agar, xanthan, pectin and baking soda were then
sorted as the most hydrophilic surface powders of the study. On the opposite, hazelnut, cocoas, milk protein concentrate, whole milk and whey proteins powders were classified as the most hydrophobic surface powders of the study. Many investigated food powders (cereal powders, flours, coffee powders, couscous powders, instant mash potatoes, etc.) fell into the intermediate category of slightly hydrophobic surface.

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

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

The loading plot shows how strongly each powder physicochemical characteristic influences the variability amongst powders, represented according to the two principal components PC1 and PC2 (Figure 7). PC1, corresponding to the horizontal axis and representing $34 \%$ of powder characteristics variability, was linked to reconstitution times ( $\mathrm{t} 95 \%, \mathrm{k}, \mathrm{n}$ ) and manufacturing process. PC2, associated with the vertical axis and corresponding to $27 \%$ powder variability, was correlated to median particle size (D50), wetting time, and surface chemical composition. Hence, a long wetting time was related to a small median particle size and an elevated surface hydrophobicity $[42,47]$. The relationship between median particle size and wettability had already been demonstrated on milk powders [48], the current work thus makes it possible to generalize this relationship to food powders whatever their composition and manufacturing
process. Hill model parameters logically appeared to be correlated with the reconstitution time: a high $k$ value or a low $n$ value (both inducing a low slope of the normalized conductivity curve at half maximal normalized conductivity $\frac{\mathrm{n}}{4 \mathrm{k}}$ value) were related to a high reconstitution time, i.e. slow reconstitution kinetics. The score plot locates investigated powder samples on the $\mathrm{PC} 1 \times$ PC2 plot (Figure 8). Powders situated on the bottom side (negative PC2) had longer wetting times, whereas powders found in the right side (positive PC 1 ) were linked to long reconstitution times. Therefore, four groups of powders were defined on the basis of their reconstitution behavior: short wetting and reconstitution times (green); long wetting time and short reconstitution time (yellow); short wetting time and long reconstitution time (orange); long wetting and reconstitution times (red). The sorting of investigated powders into these four groups were in great accordance with the literature.

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

## 4. Conclusion

For the first time, the main factors influencing powder reconstitution behavior (median particle size, chemical composition, surface composition, and manufacturing process) have been
identified for a large number of food powders. The current study permitted to confirm and generalize the knowledge developed so far on a limited number of powders and identify four reconstitution behaviors. The industrial interest of the proposed classification resides in the fact that powder reconstitution properties can be modulated by changing powder formulation (particle size distribution, surface composition, bulk composition, etc.) without modifying the reconstitution conditions (stirring speed, temperature, etc.). It would be interesting to go one step further by linking powder physicochemical characteristics and manufacturing process to the different reconstitution steps.

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## 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,

## Figure captions

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

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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## Tables

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

|  | Wetting <br> time (s) | Reconstitution <br> time $t_{95 \%}(\mathrm{~s})$ | Hill model <br> time constant <br> $\mathrm{k}(\mathrm{s})$ | Hill model <br> power $\mathrm{n}(-)$ | Hill model coefficient <br> of determination $\mathrm{R}^{2}$ |
| :---: | :---: | :---: | :---: | :---: | :---: |
| Skim milk powder <br> Whole milk <br> powder | $35 \pm 4$ | $86.0 \pm 10.0$ | $27.2 \pm 2.92$ | $2.76 \pm 0.13$ | 0.95 |
| Instant coffee <br> powder | $22 \pm 2$ | $34.7 \pm 1.1$ | $8.9 \pm 0.2$ | $2.16 \pm 0.08$ | 0.98 |
| Ground instant <br> coffee powder | $51 \pm 4$ | $106.5 \pm 6.8$ | $26.7 \pm 3.7$ | $2.15 \pm 0.31$ | 0.98 |

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Tristan Fournaise : Writing, Investigation, Methodology, Review \& Editing
Claire GAIANI : Conceptualization, Supervision, Review \& Editing, Funding acquisition
Jérémy PETIT : Conceptualization, Supervision, Review \& Editing,

