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**IMPACT OF FORMULATION ON RECONSTITUTION AND FLOWABILITY
OF SPRAY-DRIED MILK POWDERS**

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ABSTRACT

The present study focuses on the impact of lipid content (1.5, 13.8, and 26 % (w/w)) ~~and air~~
~~incorporation in~~ and pretreatment of dairy concentrates (air incorporation by whipping prior to
spray-drying) on the physicochemical and technofunctional characteristics of dairy powders. Spray-
drying was performed according to the following conditions: 200 °C inlet air temperature and 85 °C
outlet air temperature. Physicochemical properties (particle size distribution, shape factors, moisture
content, water activity, and surface composition) of spray-dried powders were characterized in
order to explain their reconstitution properties and flowability.

The first part of the study revealed that changing the lipid content induced no significant difference
of powder reconstitution. It was evidenced that mean particle size of fatty powders was higher, thus
counteracting the negative effect of powder surface hydrophobicity on its reconstitution behavior.
Air incorporation in feed dairy concentrates did not significantly affect powder reconstitution
properties, even if surface lipid content and mean particle size were both decreased. Standard
wettability and dispersibility tests were not relevant to evaluate the reconstitution ability of

23 investigated powders and all studied powders were classified as non-wettable and poorly
24 dispersible.

25 The second part of the study revealed that changing the lipid content induced a decrease in powder
26 flowability both in unconfined conditions and under high normal stress. Powders were cohesive due
27 to their low mean particle size and their surface lipid coverage, nevertheless feed concentrates
28 aeration induced an improvement of powder flowability by the decrease of in surface lipid content.
29 The bulk density (BD) and the compressed bulk density (CBD) both decreased with the lipid
30 content, and the aeration of feed concentrates did not impact the BD but led to a decrease in CBD.
31 Last, the compressibility increased with the lipid content and the aeration of feed concentrates, as
32 the latter induced the formation of smaller particles.

33

34 **KEYWORDS**

35 Dairy powder; formulation; spray-dryer; powder reconstitution; powder flowability

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45 1. INTRODUCTION

46 Spray-drying is a widely used process in the dairy powder industry, as it allows the production of
47 large amounts of powders with a good yield. Powder technofunctional properties are affected by the
48 physicochemical properties of powders, which are strongly dependent from the drying conditions
49 and formulation of feeding concentrates (dry extract, lipid and protein contents, whey protein/casein
50 ratio, pretreatments, drying conditions, etc.).

51 The pretreatment of feeding concentrates by whipping, leading to air incorporation, ~~seems to have~~
52 ~~never-been~~ has recently begun to be studied in the milk powder field [1]. Air incorporation is
53 mainly used for dairy matrices in the ice cream industry with the objective to monitor product
54 texture: whipping is used to produce a foam stabilized by dairy proteins and retaining fat globules
55 under the form of agglomerates at the surface of bubbles (i.e. at air/water interfaces); dairy proteins
56 also help to stabilize fat globule agglomerates [2,3].

57 Surface composition of milk powders is markedly dependent from the composition of feeding
58 concentrates. During the spray-drying of whole milk powder, lipids migrate to the surface of
59 droplets by buoyancy (resulting from differences between water and lipid density), high
60 hydrophobicity, and slow diffusion in water, ~~and~~ thus result~~ing~~ in an overrepresentation of lipids at
61 particle surface. Milk proteins slowly diffuse slowly in water, but faster than lipids, resulting in a
62 lower protein content at particle surface. Lactose and minerals, due to their good water diffusion
63 ability and their hydrophilicity, are mainly found in the particle core. For low-fat powders such as
64 a skim milk powder, proteins are overrepresented at particle surface, because they are the ~~least~~
65 ~~hydrophilic~~ most surface active compounds of skim milk [4,5].

66 Technofunctional properties of dairy powders such as reconstitution properties (wettability,
67 dispersibility, and solubility) and flowability are crucial for the agri-food industry and consumers.
68 The phenomenon of powder reconstitution can be divided in four steps: wetting, sinking,
69 dispersion, and solubilization. The wetting step designates the displacement of the solid/air interface

70 by the solid/liquid interface when the powder comes into contact with the liquid. The sinking step
71 corresponds to the penetration of water in the immersed particle, causing its swelling. Powder
72 dispersion states for the breakage of particle agglomerates into individual particles. The dissolution
73 is the step where the granular structure disappears and the water-soluble components are solubilized
74 [6–10]. Wettability is the ability of powders to immerse without stirring [7]. Powder wettability is
75 sensitive to several powder physicochemical parameters, especially particle size: the smaller the
76 particles, the longer their wetting time, then the poorer the powder wettability. Moreover, powder
77 surface composition also affects the wetting time: an increase in the proportion of surface lipids is
78 often deleterious for powder wettability [11–13]. Powder dispersibility corresponds to the
79 dissociation of particle agglomerates into single particles. For dispersibility indices lower than 95
80 %, the powder is considered as non-dispersible: for example, the dispersibility index of whole milk
81 powder (containing 26 % lipids) is around 90 % [6,7,13].

82 Solubility represents the ability of particles to dissolve into water and corresponds to the complete
83 loss of granular structure [6,7,9]. Like wettability, solubility decreases with surface lipid content or
84 mean particle size. Powder reconstitution depends on powder physicochemical properties: chemical
85 composition (protein, lipid, lactose, and minerals) and physical parameters such as particle size and
86 shape distributions [11,12]. Furthermore, the reconstitution behavior is affected by temperature,
87 stirring conditions, mineral content of the water solvent, etc. [14,15].

88 Powder flowability represents another big challenge for industrials: it is of utmost importance for
89 the control of the handling and transport of powders in pipes and hoppers. Powder flowability is
90 very dependent from powder physicochemical parameters: the flow is impaired by
91 particle cohesion, an increase of lipid content on particle surface (making particles sticky), a high
92 water content (increasing interparticular capillary interactions), a low mean particle size, a high
93 heterogeneity of particle sizes, and an elevated particle surface roughness [16,17]. Environmental
94 parameters also play a significant role: for instance, flowability is generally poorer at high

95 temperature or high relative humidity (both favoring the glass transition phenomenon leading to the
96 apparition of a rubbery outer layer making the particle sticky), as well as with walls of high surface
97 roughness. Moreover, powder flow properties also depend from the type of stress applied to the
98 powder: flowability may differ in low (fluidization) and high stress conditions (rotational shear).
99 Various tests and flow parameters have been developed to evaluate and classify powder flowability,
100 particularly flow factor (ff) [18], cohesion, and basic flowability energy (BFE) [19].

101 The present study focuses on the impact of formulation (composition and pretreatments of feed
102 concentrates) on the reconstitution-ability and flowability of dairy powders. On one hand, dairy
103 powders differing in lipid content (issued from skim, semi-skim, and whole milks) were produced
104 by spray-drying at pilot-scale. On the other hand, the influence of concentrates pretreatment ~~eed~~ (air
105 incorporation by whipping) ~~aeration~~ prior to spray-drying was also investigated. The reconstitution
106 ability of spray-dried powders was evaluated through wettability, dispersibility, and solubility
107 indices which are classically employed by industrials.

108 2. MATERIAL AND METHODS

109 2.1. Material

110 Skim milk (SPRAY JA12 ALIM) and whole milk (SPRAY JG12 ALIM) powders used for the
111 preparation of milk concentrates were purchased from Lactalis Ingredients (Bourgbarré, France).

112 2.2. Preparation of milk concentrates

113 Concentrates were prepared by reconstituting skim and whole milk powders in distilled water at
114 30 % (w/w) dry extract. Industrial powders were poured into distilled water at 20 °C under
115 mechanical stirring (ZR 2102 control, Heidolph, Germany) between 200 and 500 rpm with a 4-
116 bladed impeller (2 cm length, 1 cm width) or a 3-hole square blade impeller (7 cm length, 7 cm
117 width) to produce unaerated or aerated concentrates, respectively (Figure 1). The 3-hole square
118 blade impeller permitted to aerate feed concentrates by whipping until obtaining a foam. Once the

119 powder was totally immersed, mixtures were stirred during 2 h at 20 °C, then overnight at 4 °C
120 under slight agitation to ensure complete powder reconstitution.

121 ~~Six concentrates varying in lipid content were produced: skim milk, semi-skim milk (using equal~~
122 ~~amounts of skim and whole milk powders), and whole milk, each being aerated or not.~~

123 Six concentrates were produced: skim milk, semi-skim milk (using equal amounts of skim and
124 whole milk powders), and whole milk, each formulation being aerated or not. These concentrates
125 were then spray-dried in the same conditions in order to investigate the influence of formulation
126 (proximal composition, but also the way to prepare concentrates, i.e. with or without air
127 incorporation).

128 **2.3. Production of dairy powders by spray-drying**

129 Spray-drying was carried out on a Micra Spray MS 150 single effect spray-dryer (Anhydro,
130 Soeborg, Denmark), equipped with a bi-fluid nozzle (Fluid Cap 60 100 + Air Cap 120, Spraying
131 System, Wheaton, Illinois, USA). Spray-drying conditions were set at 200 °C drying air inlet
132 temperature and 85 °C outlet temperature in view to obtain powders with good preservation abilities
133 (i.e., water activity ranging from 0.2 to 0.3) [20,21]. The concentrate was fed by a FASTLoad
134 peristaltic pump (VWR, Leuven, Belgium; 4.8 mm pipe internal diameter) and the feed concentrate
135 flow rate was regulated at 68.0 mL/min to maintain the drying air outlet temperature at 85 °C [22].
136 Powders were placed in polypropylene bags just after sampling to limit degradations susceptible to
137 occur during storage (such as moisture uptake) and stored at 4 °C.

138 To sum up, six powders were produced by spray-drying of the six concentrates prepared with
139 industrial milk powders (Table 1): unaerated skim milk powder (SMP), unaerated semi-skim milk
140 powder (SSMP), unaerated whole milk powder (WMP), aerated skim milk powder (ASMP), aerated
141 semi-skim milk powder (ASSMP), and aerated whole milk powder (AWMP).

142

145 **2.4. Physicochemical characterization**

146 2.4.1. Particle size distribution

147 Particle size analysis was carried out with a laser granulometer (Mastersizer S, Malvern Instruments
148 Ltd, Malvern, UK) supplied with a 5 mW He-Ne laser operating at a wavelength of 632.8 nm with a
149 300 RF lens. Air dispersion was achieved with the Aero S module (Malvern). The dispersion
150 conditions were adapted to each powder: air pressure between 2 and 3 bar, feed rate between 60 and
151 90 %, and hopper length between 2 and 3 mm. Three replicates were analyzed for each sample. The
152 mean diameter in volume was chosen as size estimator. Classical granulometric parameters (d_{10} , d_{50} ,
153 and d_{90}) were calculated, where d_x designates the diameter for which x % of the particles have a
154 smaller size. The width of the particle size distribution was evaluated with the span (Eq. (1)) [23].

$$span = \frac{d_{90} - d_{10}}{d_{50}} \quad (1)$$

155 2.4.2. Scanning electron microscopy

156 Scanning electron microscopy (SEM) was employed to observe the size, the morphology, and the
157 surface structure of milk powders. Powder samples were sputtered on double-sided carbon tape,
158 fixed to SEM stubs, and coated with carbon in a sputter coater (Polaron SC7640, Thermo VG
159 Scientific, East Grinstead, United Kingdom). The observations were performed at x 500
160 magnification under ~~under~~ vacuum using a scanning electron microscope (Hitachi S-4800, Japan)
161 operating at 1.0 kV acceleration voltage [10].

2.4.3. Particle surface composition by X-ray photoelectron spectroscopy

The atomic surface contents in carbon, oxygen, and nitrogen of spray-dried powders were determined from X-ray photoelectron spectroscopy (XPS) by using a Kratos AXIS Ultra (Kratos Analytical, Manchester, UK) spectrometer with a monochromatic Al X-ray source at 150 W. Samples were outgassed under vacuum during 24 h before analysis. Each analysis was performed in duplicate. XPS analysis provides atomic composition of powder surface up to 10 nm depth; measurement area was about 700 μm x 300 μm in analytical conditions of this study [21,24]. XPS spectra were used to calculate the surface contents in proteins, lipids, and lactose by solving the following equations (Eq. (2)) using the Excel solver (Windows, Microsoft, Redmond, USA). In these equations, numerical values come from the experimental composition of pure milk proteins, pure lactose, and anhydrous milk fat [21].

$$C = 0.685 \times P + 0.608 \times LA + 0.884 \times LI$$

$$O = 0.182 \times P + 0.392 \times LA + 0.113 \times LI$$

$$N = 0.133 \times P + 0 \times LA + 0.003 \times LI$$

$$P + LA + LI = 100 \%$$

(2)

with:

- C, O, and N (%): atomic concentrations of carbon, oxygen, and nitrogen at the powder surface, respectively;

- P, LA, and LI (%): surface content in proteins, lactose, and lipids, respectively.

2.5. Powder interactions with water

179 2.5.1. Water activity

180 Water activity (a_w) was measured with a HygroPalm23-AW portable a_w -meter (Rotronic, France) at
181 room temperature (20 °C) using a 3 cm powder layer (15 ± 1 g) similarly to ISO 21807:2004 [25].

182 2.5.2. Water content

183 The water content was determined following ISO 5537:2004 [26] by the loss of water mass of 2.00
184 g $\pm$ 0.01 g powder after drying during at least 3 h at 103 °C until a constant dry powder mass was
185 obtained.

186 2.5.3 Reconstitution ability

187 Reconstitution ability of spray-dried powders was evaluated through wettability, dispersibility, and
188 solubility indices determined by standard tests.

189 2.5.3.1 Wettability

190 Wettability was evaluated following IDF 87:1979 [13] as the time in seconds taken by 13.0 ± 0.1 g
191 of powder to become wet when gently deposited at the surface of 125.0 ± 0.1 g distilled water in a
192 250 mL beaker.

193 2.5.3.2 Dispersibility

194 Dispersibility index was measured by a method adapted from IDF 87:1979 [13]. 13.0 ± 0.1 g
195 powder was gently deposited on the surface of 125 g water at 20 °C placed in a 400 mL beaker. The
196 mixture was manually stirred for 20 s, then the mixture was allowed to rest for 30 s. After that, the
197 mixture was passed through a 120 μ m sieve. The total dry matter of the liquid was determined by
198 drying at 103 °C for 5 h (Eq. (3)).

199

$$200 \text{ Dispersibility (\%)} = \frac{W \times T}{100 - (E + T)} \times \frac{100}{S} \quad (3)$$

201 with:

- 202 - W: 125 g distilled water (g);
- 203 - T: dry matter content (% w/w) of the liquid;
- 204 - E: dry matter content (% w/w) of sampled powder;
- 205 - S: 13 g sampled powder (g).

206 2.5.3.3 Solubility

207 Solubility was characterized as the proportion of powder able to be dissolved in distilled water
208 following a method adapted from ISO 8156:2005 [9]. It was determined by the addition of 2.5 ± 0.1
209 g powder in 17.5 mL distilled water at 25 °C in a 50 mL centrifugation tube followed by 30 s
210 stirring and centrifugation at $2\,294 \times g$ during 10 min. Then, the supernatant was removed and the
211 pellet was added to 20 mL distilled water, followed by stirring and centrifugation at $2\,294 \times g$
212 during 10 min. The pellet was desiccated at 103 °C overnight to determine the mass of insoluble
213 residues, which allows determining the solubility index (Eq. (4)).

$$214 \text{ Solubility (\%)} = \frac{(S-M) \times 100}{S} \quad (4)$$

215 with:

- 216 - M: undissolved sediment mass after desiccation (g);
- 217 - S: 2.5 g sampled powder (g).

218 2.6. Flow properties of milk powders

219 Flow properties were analyzed using an FT4 powder rheometer with 25 mm accessories (Freeman
220 Technology, Worcestershire, UK).

221 2.6.1. Stability test

222 The stability test was carried out through a series of seven conditioning cycles and measurements of
223 the mixing energy of the powder bed placed in a 25 mm vessel [16,19]. Test cycles were performed
224 by rotating the blade (-5° helix angle, 23.5 mm diameter, 100 mm/s tip speed) into and through the
225 sample of powder bed placed in a measurement cell (25 mm x 25 mL). ~~Stability test cycles were~~
226 ~~realized by the rotating movement of the blade into and through a powder bed contained in the~~
227 ~~measurement cell (50 mm x 260 mL glass cylinder) at a tip speed of 100 mm/s and helix angle of~~
228 ~~-5°.~~

229 The basic flowability energy (BFE) is the energy needed in the seventh test to displace a
230 conditioned powder sample during downwards moving of the blade, it mainly depends on
231 compressibility, consolidation level, and density of the powder bed [16]. Easy-flowing behavior
232 corresponds to a low BFE; on the contrary, a high BFE is generally obtained for poorly-flowing
233 powders.

234 The specific energy (SE) (Eq. (6)) is the energy needed to displace the conditioned powder bed
235 during upwards testing divided by the mass of analyzed powder sample. It depends on powder
236 cohesion, particle size, shape, surface roughness and composition, as well as chemical and physical
237 interactions between particles [16].

$$SE = \frac{\frac{(Up\ energy\ cycle\ 6 + Up\ energy\ cycle\ 7)}{2}}{Powder\ mass} (mJ/g) \quad (6)$$

238 Powder flow behavior can be classified on the basis of the SE value as follows:

- 239 - SE < 5 mJ/g: low cohesion
- 240 - 5 mJ/g < SE < 10 mJ/g: moderate cohesion
- 241 - SE > 10 mJ/g: high cohesion

242 The classification provided by Freeman Technology is specifically adapted for the FT4 powder
243 rheometer.

2.6.2. Compressibility test

Compressibility test allows to measure the evolution of the powder density as a function of the applied normal stress [16,27,28]. The test ~~proceeds by~~ begins with three conditioning cycles in 25 mm vessel with a 23.5 mm blade, then the vessel was split in a measurement cell (25 mm x 10 mL). The blade was replaced by a 23.5 mm vented piston after the powder was subjected to nine levels of normal stress from 0.5 to 15 kPa. The powder compressibility depends on the particle size and shape, surface roughness and composition, powder bed porosity, and interparticular cohesion. The compressibility allows to obtaining information on the powder density before and after compression at 15 kPa, respectively named the bulk density (BD) (Eq. (7)) and the compressed bulk density (CBD) (Eq. (8)) here.

$$\text{Bulk density} = \frac{\text{Powder mass}}{\text{Volume of the measurement cell}} \text{ (g/mL)} \quad (7)$$

$$\text{Compressed Bulk Density} = \frac{\text{Powder mass}}{\text{Volume of the powder bed after compression}} \text{ (g/mL)} \quad (8)$$

2.6.3. Shear cell test

The shear cell test is intended to evaluate flowability under a high shear rate; it also allows determining powder cohesion. The test started with a conditioning cycle to homogenize the powder with the 23.5 mm blade in the 25 mm container. Then the powder was compacted under a normal stress of 9 kPa with a ventilated piston of 23.5 mm. The excess powder was ~~elucidated~~ removed to obtain a measuring cell of 25 mm x 10 mL, and then ~~help the shear of~~ the powder bed ~~to be~~ was subjected to a shear stress of 7 to 3 kPa by 1 kPa steps [16,19,28,29].

The shear test allowed to obtain different powder properties such as cohesion (kPa) ~~;~~ and the flowability index or flow factor (*ff*) (Eq. (9)).

$$ff = \frac{\sigma_1}{\sigma_c} \quad (9)$$

264 With:

265 - σ_1 : unconfined yield strength (Pa)

266 - σ_c : major principle stress (Pa)

267 Powder flowability can be deduced from flow factor values by using Jenike's classification [18]:

268 - $ff < 1$: not flowing

269 - $1 < ff < 2$: very cohesive

270 - $2 < ff < 4$: cohesive

271 - $4 < ff < 10$: easy flowing

272 - $10 < ff$: free flowing

273 2.7. Statistical analysis

274 All analyses were triplicated to ensure a good analytical repeatability and presented values
 275 correspond to means $\pm$ standard deviations. Data were statistically analyzed by one-way ANOVA
 276 performed with Excel version 2016 and means were separated by Tukey's HSD test at $p < 0.05$
 277 significance level.

278

279 3. RESULTS AND DISCUSSION

280 3.1. Particle size distribution

281 The size of spray-dried particles ranged from 1 to 200 μm , in agreement with the low particle sizes
 282 generally obtained with the pilot spray-dryer used in this study [22,30–32]. Their d_{50} was comprised
 283 between 11.6 and 36.7 μm (Figures 2 & Tables 2). All particle size distributions were monomodal
 284 and particle sizes were rather moderately dispersed around the mean particle size (as reflected by

the moderate span values ranging from 2.0 to 3), except for the SMP was composed of particles of heterogeneous sizes as reflected by its higher span (Table 2 & Figure 2). Feed concentrate aeration prior to spray-drying led to a decrease in particle size and a narrowing of the particle size distribution. This may be explained by the effect of air incorporation (as dissolved air or under the form of bubbles) in the concentrate: in fact, it was expected to decrease the density ~~and/or the viscosity~~ of the concentrate, ~~both~~ thus facilitating the breakup of the liquid jet during the spraying operation [32–35]. Therefore, aerated concentrates resulted in smaller droplets in the spray and smaller particles at the outlet of the spray-dryer. ~~Particle size~~ The mean particle size d_{50} decreased with lipid content for SMP, SSMP and WMP powders, but this impact of lipid content was not evidenced for ASMP, ASSMP and AWMP powders. The decrease in particle size for powders coming from unaerated feed concentrates was probably due to the decrease in ~~viscosity and/or~~ density of feed concentrates when the lipid content was increased. This effect of lipid content on particle size was negligible when spray-drying aerated concentrates, probably because the impact of concentrates aeration on its ~~viscosity and~~ density is well higher than the influence of lipid content.

Figure 2

Table 2

3.2. Particle appearance

According to SEM images, spray-dried milk powders (SSMP, ASSMP, WMP, and AWMP) had a smoother appearance than skim milk powders (SMP and ASMP) particles (Figure 3) [5]. This is likely to come from the presence of a lipid layer at the particle surface, formed by lipid migration by buoyancy in the sprayed droplet [36]. In addition, granulometric results were confirmed by microscopic observations: a rather large heterogeneity of particle sizes was observed for SMP. Also, the decrease in particle size induced by the aeration of feed concentrates prior to spray-drying was confirmed by SEM images.

309 Figure 3

310 3.3. Surface composition

311 XPS analyses (Figure 4) permitted to highlight the influence of lipid content on surface composition
312 of milk particles. For WMP and SSMP powders, lipids were overrepresented and lactose was
313 underrepresented, as often observed for milk powders [5]. This phenomenon is well known for
314 powders containing lipids (even in low proportions); in the current study for example, the SSMP
315 powder had 55 % surface lipids for an overall lipid content of 13.8 % (w/w).

316 A marked decrease (over half of surface lipids) in surface lipid content was observed for powders
317 produced from aerated feed concentrates. Indeed, it is well known that during drying, lipids migrate
318 owing to buoyancy to the air/water interfaces, especially at the droplet surface. It can be
319 hypothesized that droplets coming from aerated feed concentrates may have a higher proportion of
320 occluded air, thus embedding more air/water interfaces. After migration due to buoyancy and
321 hydrophobicity, lipids can be found either at the embedded air/water interfaces or at the droplet
322 surface [1–3]. Therefore, the lipid content at the surface of powders produced from aerated feed
323 concentrates was expected to be lower than for powders coming from unaerated feed concentrates,
324 as if lipids were encapsulated [37].

325 Figure 4

326 3.4. Powder interactions with water

327 Spray-dried powders had a low water activity between 0.15 and 0.29 (Table 3), which is favorable
328 to their good preservation; indeed, the ideal water activity for the preservation of food products,
329 especially dairy, ranges from 0.20 to 0.35 [7]. Thus, during storage, produced powders will be
330 almost insensitive to alteration reactions, except for lipid oxidation of powders containing a
331 significant proportion of lipids such as WMP, because its water activity was slightly below 0.2 [38].

332 The water content of all spray-dried powders respected the maximum water content recommended
333 for dairy powders (5 g/100 g on wet basis) [39]. Despite rigorously identical spray-drying
334 conditions, the water content slightly varied, because powder composition changed and their
335 different components vary in hygroscopicity.

336 The wettability of commercial milk powders generally ranges between 24 s for skim milk powders
337 and 120 s for whole milk powders. The dispersibility of commercial milk powders also varies
338 between 90 % for WMP and 95 % for SMP [7]. Poor wettability of studied powders was expected
339 due to the fineness of powders produced by the pilot-scale spray-dryer owing to nozzle geometry
340 (mean particle size d_{50} under 90 μm); in fact, wetting times greater than 90 min were obtained for
341 investigated powders. For the same reason, poor dispersibility was also expected; in this study,
342 spray-dried powders had very low dispersibility below 25 % [14,40]. Then, this tremendous
343 influence of particle size did not make it possible to highlight the impact of the lipid content on
344 powder wettability and dispersibility. ~~Powder wettability was very poor reflected by a wettability~~
345 ~~time over 90 min and dispersibility was also very poor under 25 %, which results in all likelihood~~
346 ~~from the fineness of spray-dried particles. The dispersibility results were inconclusive because all~~
347 ~~the powders were non-dispersible [14,40].~~

348 Solubility of SMP, ASMP, SSMP, ASSMP, WMP, and AWMP did not significantly differ (Table
349 3). Thus, lipid content and concentrate aeration prior to spray-drying did not affect powder
350 solubility, contrary to what was expected for powders varying in lipid content [7,41]. These results
351 evidenced that the low mean particle size had a well more significant effect ~~than milk powder~~
352 ~~composition~~ on its solubility than milk powder composition [7,42,43].

353 Table 3

354 3.5. Flow properties of milk powders

355 3.5.1. Stability test

BFE of spray-dried powders significantly increased with the increase in lipid content both for powders coming from aerated and unaerated feed concentrates (Table 4). This indicates that powder flowability was impaired by the presence of surface lipids, consistently with the findings of Shrestha et al. [44] and Kim [45]. Indeed, fat surface coverage is expected to make particles stickier, thus increasing powder cohesion.

Moreover, aeration of feed concentrates induced a decrease in particle size, thus impairing powder flowability, as illustrated by the higher BFE of ASMP, ASSMP, and AWMP [17,20].

Regarding SE values, three powder groups can be formed: powders of SE close to 5 mJ/g corresponding to rather low cohesion (SMP), powders with a SE around 7 - 8 mJ/g, moderately cohesive (SSMP, WMP, ASMP), and cohesive powders with SE close or above 10 mJ/g (ASSMP, AWMP). It is obvious that a higher lipid content made powders more cohesive, owing to the increase in surface stickiness, whereas aeration of feed concentrates was deleterious for powder flowability owing to the decrease in particle size. Indeed, electrostatic interactions and friction are expected to be promoted between smaller particles, owing to the larger contact area between particles [45,46].

~~Overall, the modification of fat content seemed to impair powder flowability in an unconfined environment, as much as aeration of feed concentrates~~

Table 4

3.5.2. Compressibility test

The compressibility test allowed ~~to~~ obtaining information on conditioned bulk density (CBD), bulk density (BD), and powder compressibility at 15 kPa (Figure. 5, Table 4).

Aeration of feed concentrates did not induce any significant CBD, BD, or compressibility change, ~~but it increased BD and compressibility~~ except for the compressibility of whole milk powder that

380 was slightly increased when the feed concentrate was aerated. This may be explained by the fact
381 that mean particle size and span of powders coming from aerated concentrates were both smaller
382 and have opposite effects on powder compressibility. Indeed, smaller particles are generally more
383 cohesive and thus more compressible, whereas smaller span indicates a better homogeneity of
384 particle sizes, which makes powders less compressible [47,48]. Hence, it may be concluded from
385 these results that aeration of concentrates did not have a great effect on powder cohesion: a small
386 increase in powder compressibility was sometimes denoted, which may be indicative of a small
387 increase in powder cohesion. ~~This is indicative of higher cohesion of powders coming from aerated~~
388 ~~feed concentrates and meets observations formulated from stability test results.~~

389 Besides, powders containing fat (SSMP, WMP, ASSP, and AWMP) had a ~~reduced~~ lower CBD than
390 skim milk powders, as the presence of surface fat is known to increase powder cohesion. Thus,
391 their sensitivity to compression of fat-containing powders was higher, leading to higher BD and
392 compressibility. This may be a sign of higher cohesion of fat-containing powders, which was
393 obviously expected owing to the stickiness of fat material. Then, fat-containing powders appeared
394 to become cohesive when surface fat content exceeded circa 10 % (intermediate value between
395 surface fat contents of SMP and ASSMP).

396
397 ~~The smaller particles size obtained by feed concentrate aeration can explain the higher~~
398 ~~compressibility of ASSMP and AWMP in comparison with SSMP and WMP, respectively [49].~~
399 ~~The higher compressibility of SMP in comparison with ASMP could be explained by its the higher~~
400 ~~span indicating a probable large proportion of large particles, less sensitive to compression (Table~~
401 ~~32) [47,48].~~

402 ~~Overall, the lipid content seemed to affect powder compressibility more than aeration of feed~~
403 ~~concentrates.~~

404 Figure 5

405 3.5.3. Shear cell test

406 Results of the shear cell test are displayed in Figure 6 and Table 5. On one hand, powders
407 containing significant proportions of lipids (SSMP, WMP, ASSMP, AWMP) were highly cohesive,
408 owing to their previously evoked surface stickiness, in agreement with stability and compressibility
409 results. The flow factor of these powders was found near 2, indicating the difficulty to make these
410 cohesive powders flow. Powders almost free from lipids (SMP and ASMP) were rather easy-
411 flowing powders, having a low cohesion. This confirms that the presence of surface fat has a
412 significant influence on powder flowability.

413 On the other hand, aeration of feed concentrates seemed not to have a tremendous effect on powder
414 flow in high shear stress conditions, but it induced a slight improvement (respectively, impairment)
415 of skim milk powder (respectively, semi-skim, and whole milk powders) flowability, as denoted by
416 the small decrease (respectively, increase) in powder cohesion and the small increase (respectively,
417 decrease) in flow factor. These results slightly differ from those of the stability test, which showed
418 that the negative impact of the smaller particle size of ASMP powder on its flowability exceeded
419 the positive impact its lower surface fat content [46].

420 Overall, the modification of fat content seemed to impair powder flowability in high normal stress
421 conditions more than aeration of feed concentrates. The higher impact of lipid content in high stress
422 conditions than in unconfined ones could be a consequence of the release of free fat at powder
423 surface under high normal stress.

424 Figure 6

425 4. CONCLUSION

426 In the present study, the link between formulation (composition and pretreatment) of the feed
427 concentrate used for spray-drying, powder physicochemical properties, and reconstitution ability

were explored. First, the single effect pilot-scale dryer led to the production of small spray-dried particles, owing to nozzle geometry. It was evidenced that the feed concentrate aeration prior to spray-drying resulted in a great decrease in particle size. ~~For the first time, the use of this processing step (aeration of feed concentrates) was evidenced to impact particle surface composition (lipid surface content was decreased) along with particle size distribution. As powder functional properties (reconstitution ability, stability during storage, flowability) are related to powder physicochemical properties, particle size and lipid location are of great importance and must be managed by industrials.~~

Concentrate aeration prior to spray-drying has been evidenced to allow reducing the surface fat content. Thus, industrial may interestingly reduce interparticular cohesion and thus increase powder flowability by aerating concentrates prior to spray-drying. The decreased surface fat content may also be beneficial for improving powder reconstitution ability and decrease powder sensitivity to caking. In order to avoid lipid oxidation, nitrogen could be incorporated instead of air.

The lipid content was not clearly linked to powder reconstitution behavior as its influence was overwhelmed by the impact of the small particle size of investigated powders. The small particle size of powders obtained by spray-drying at pilot scale induced the formation of non-wettable and non-dispersible powders. It would be interesting to work on the geometry of the bi-fluid nozzle used for spray-drying with the objective to produce larger particles of size comparable to commercially available powders.

The modification of lipid content seemed to impair powder flowability in an unconfined environment as much as aeration of feed concentrates. A higher lipid content seemed to increase powder compressibility more than aeration of feed concentrates. Skim milk powders were rather easy-flowing and had a low cohesion, whereas all other powders, containing a significant proportion of lipids, were cohesive, thus having a poor flowability. Overall, the modification of fat

content seemed to impair powder flowability in high stress conditions and more than aeration of feed concentrates. This greater influence of lipid content in high normal stress conditions may result from release of free fat at particle surface under compression.

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623 I came from an initial training in manipulations of biotechnology.

624 Following my arrival at the Laboratoire d'Ingénierie des Biomolécules in 2006 I have adapted
625 and trained myself in the production and analysis of physical and chemical characterization of food
626 powders. Since then I mobilize my technical skills to collaborate through various research projects
627 in this area.

628



629 Prof Claire Gaiani Since my recruitment in 2007, I have received a total of
630 2 000 k€ in funding for projects on which I was the principal investigator. Among other important
631 activities, I successfully managed fundamental competitive programs including the French ANR
632 (French National Agency for Research) and European projects (Excellent science section with
633 a Marie Skłodowska-Curie action and Interreg). Finally, I manage many industrial partnerships with
634 leading international food companies (including Nestle, Lactalis, Bel, Bongrain, and Senoble). All
635 these projects are directly related to my research theme.



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He now teaches process engineering and physicochemistry. His research theme concerns the impact of formulation and process conditions on physicochemistry and functionality of food powders.

Table 1. Proximate composition on dry basis of ~~powders produced by concentrates used to feed~~ spray-drying, deduced from calculations on the basis of technical datasheet of commercial powders ~~used to prepare feed concentrates~~ employed in the preparation of concentrates.

Powders	Proteins (%)	Lipids (%)	Lactose (%)	Minerals (%)
Commercial skim milk powder/aerated skim milk powder (SMP/ASMP)	> 32.9	< 1.5	< 56.0	< 8.5
Semi-skim milk powder*/aerated semi skim milk powder (SSMP/ASSMP)	> 28.8	> 13.8	< 48.5	< 7.5
Commercial whole milk/aerated whole milk powder (WMP/AWMP) powder	> 24.7	> 26.0	< 41.0	< 6.5

* Mix between 50 % of commercial skim milk powder and 50 % of whole milk powder

Table 2. Granulometer parameters (d_{10} , d_{50} , d_{90} , span) of SMP, SSMP, WMP, ASMP, ASSMP and AWMP.

Sample	d_{10} (μm)	d_{50} (μm)	d_{90} (μm)	Span (-)
SMP	$8.7^b \pm 0.1$	$36.7^a \pm 0.2$	$137.0^a \pm 0.0$	$3.5^a \pm 0.0$
SSMP	$9.2^a \pm 0.0$	$29.6^b \pm 0.1$	$94.3^b \pm 0.4$	$2.9^c \pm 0.0$
WMP	$9.1^a \pm 0.1$	$27.7^c \pm 0.2$	$92.2^b \pm 2.0$	$3.0^b \pm 0.1$
ASMP	$7.4^c \pm 0.1$	$21.0^d \pm 0.1$	$49.8^d \pm 0.1$	$2.0^f \pm 0.0$
ASSMP	$5.9^d \pm 0.0$	$19.3^e \pm 0.1$	$47.3^e \pm 0.2$	$2.1^e \pm 0.0$
AWMP	$6.1^d \pm 0.1$	$20.8^d \pm 0.0$	$58.8^c \pm 0.1$	$2.5^d \pm 0.0$

Means with different superscripted letters in the same column were significantly different according to Tukey’s HSD test ($p < 0.05$; $n = 3$).

Table 3. Techno-functional characterization (reconstitution) of SMP, SSMP, WMP, ASMP, ASSMP and AWMP.

Sample	Water content (g/100 g)	Water activity a_w (-)	Wettability (min)	Dispersibility (%)	Solubility (%)
SMP	4.98 ^a ± 0.05	0.25 ^c ± 0.00	> 90 < 90	23.81 ^a ± 0.09	93.28 ^a ± 5.27
SSMP	3.14 ^c ± 0.02	0.21 ^d ± 0.00	> 90 < 90	14.89 ^d ± 0.45	93.83 ^a ± 2.10
WMP	2.04 ^d ± 0.07	0.15 ^f ± 0.00	> 90 < 90	19.79 ^b ± 0.08	92.65 ^a ± 4.20
ASMP	3.78 ^b ± 0.09	0.18 ^e ± 0.00	> 90 < 90	20.03 ^b ± 0.28	91.97 ^a ± 3.20
ASSMP	3.64 ^b ± 0.13	0.28 ^b ± 0.00	> 90 < 90	12.65 ^e ± 0.06	88.96 ^a ± 1.71
AWMP	3.82 ^b ± 0.17	0.29 ^a ± 0.00	> 90 < 90	15.94 ^c ± 0.22	93.33 ^a ± 2.75

Means with different superscripted letters in the same column were significantly different according to Tukey's HSD test ($p < 0.05$; $n = 3$).

Table 4. Flowability characterization (Stability test, compressibility test, shear cell test) of SMP, SSMP, WMP, ASMP, ASSMP and AWMP.

Sample	BFE (mJ)	SE (mJ/g)	CBD (g/mL)	BD (g/mL)	Compressibility at 15 kPa (%)	Cohesion (kPa)	$ff(-)$
SMP	$48.74^c \pm 5.55$	$5.27^d \pm 0.37$	$0.52^a \pm 0.01$	$0.62^b \pm 0.01$ $0.62^{ab} \pm 0.02$	$16.13^c \pm 0.91$	$0.77^b \pm 0.06$	$5.59^b \pm 0.36$
SSMP	$72.63^b \pm 7.53$	$7.03^c \pm 0.08$	$0.38^b \pm 0.02$	$0.67^{ab} \pm 0.00$ $0.51^b \pm 0.02$	$42.50^{ab} \pm 3.54$	$2.07^a \pm 0.07$	$2.30^c \pm 0.07$
WMP	$91.97^b \pm 2.71$	$7.80^c \pm 0.58$	$0.39^b \pm 0.01$	$0.65^{ab} \pm 0.05$ $0.74^a \pm 0.12$	$39.44^b \pm 3.67$	$2.20^a \pm 0.39$	$2.28^c \pm 0.34$
ASMP	$76.33^b \pm 7.63$	$7.67^c \pm 0.63$	$0.55^a \pm 0.03$	$0.66^{ab} \pm 0.03$ $0.62^{ab} \pm 0.01$	$14.37^c \pm 3.13$	$0.59^b \pm 0.13$	$7.41^a \pm 1.41$
ASSMP	$135.64^a \pm 14.02$	$9.94^b \pm 0.35$	$0.37^b \pm 0.02$	$0.63^b \pm 0.02$ $0.63^{ab} \pm 0.03$	$42.20^{ab} \pm 1.64$	$2.41^a \pm 0.14$	$2.01^c \pm 0.07$
AWMP	$156.15^a \pm 8.44$	$11.68^a \pm 0.06$	$0.36^b \pm 0.01$	$0.70^a \pm 0.02$ $0.70^{ab} \pm 0.17$	$48.79^a \pm 1.47$	$2.59^a \pm 0.29$	$1.86^c \pm 0.13$

Means with different superscripted letters in the same column were significantly different according to Tukey's HSD test ($p < 0.05$; $n = 3$).

Figure(s)



Fig. 1. 3-hole square and 4-bladed impellers.

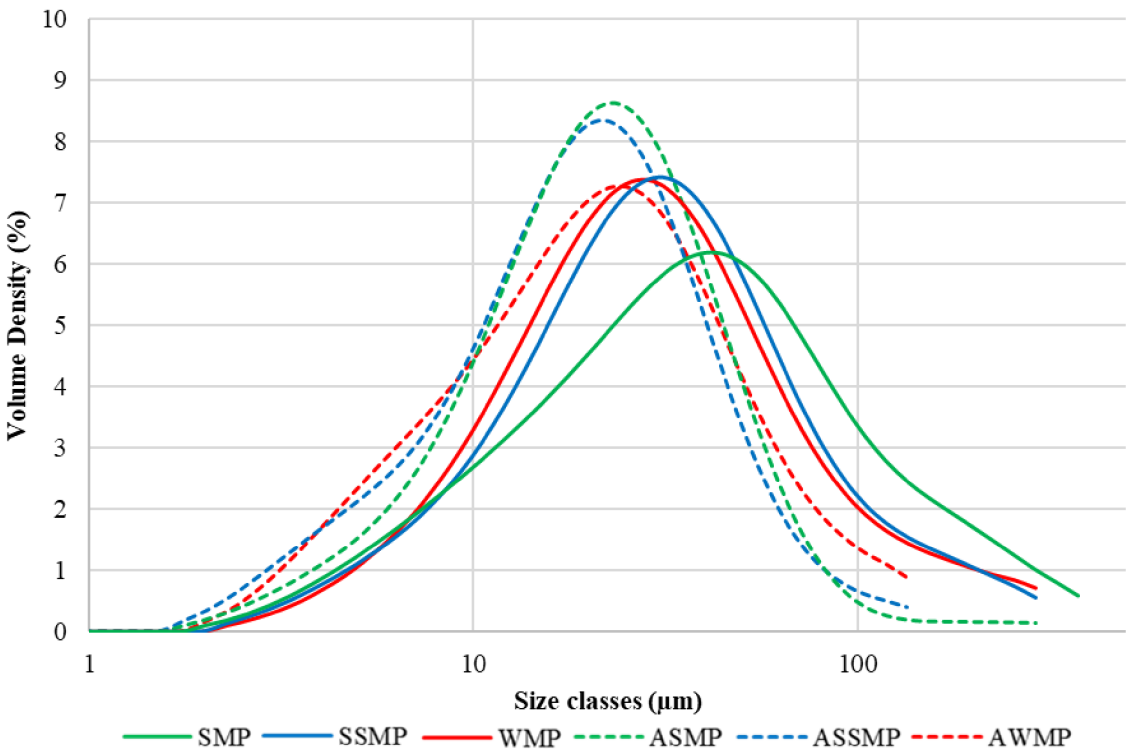


Fig. 2. Particle size distributions (in volume) of SMP, SSMP, WMP ASMP, ASSMP and AWMP.

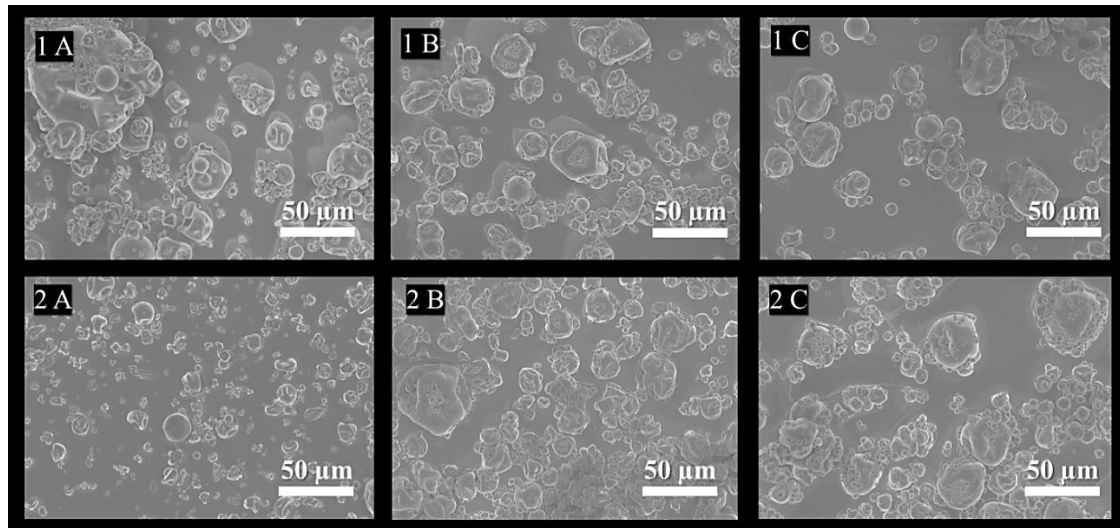


Fig. 3. SEM micrographs of spray-dried milk powders varying in lipid content (1 A: SMP; 2 A: ASMP; 1 B: SSMP; 2 B ASSMP; 1 C: WMP; 2 C: AWMP). Magnification: $\times 500$.

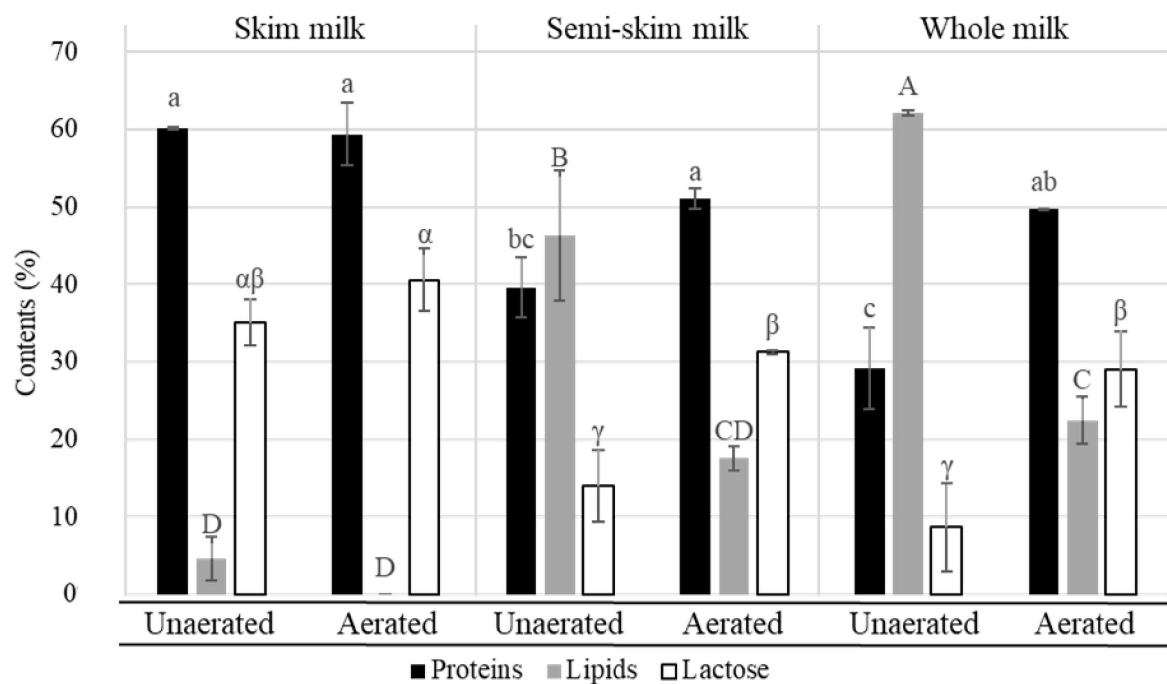


Fig. 3. Surface composition on dry basis deduced from XPS analysis of skim, semi-skim and whole milk powders with or without aeration. For each component separately, bars topped with different letters were significantly different according to Tukey's HSD test ($p < 0.05$; $n = 2$).

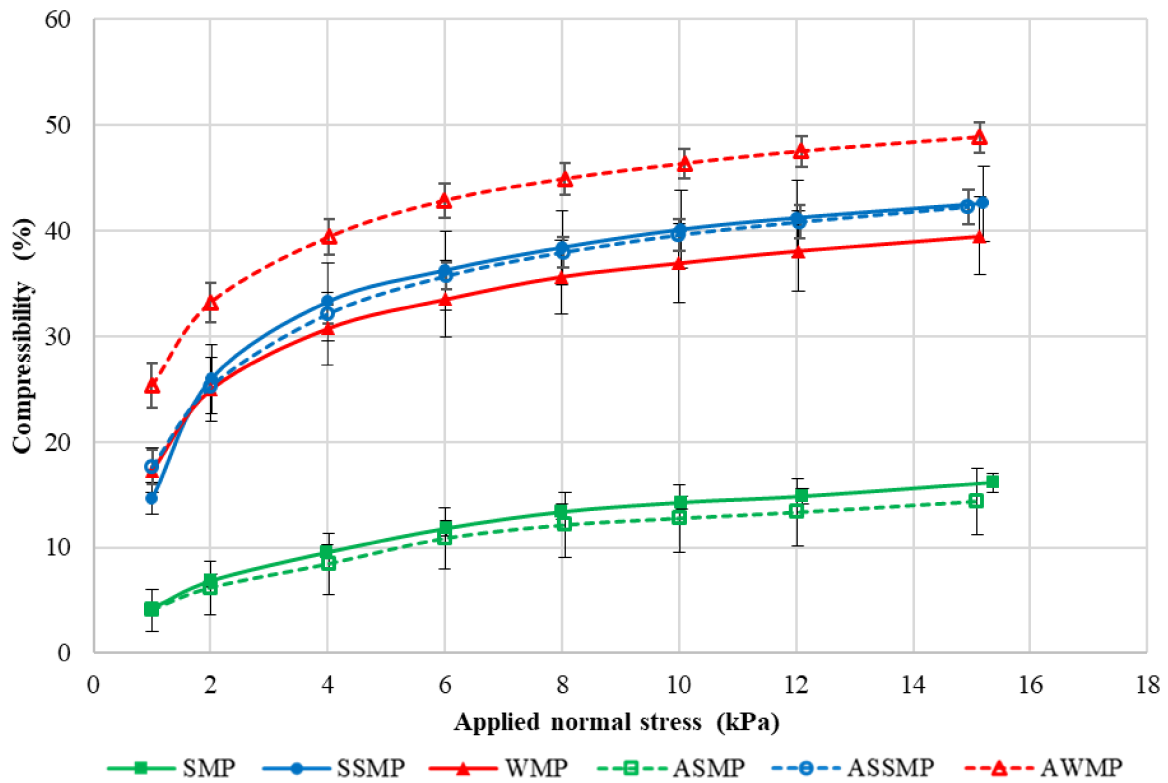


Fig. 4. Compressibility results for SMP, SSMP, WMP, ASMP, ASSMP and AWMP.

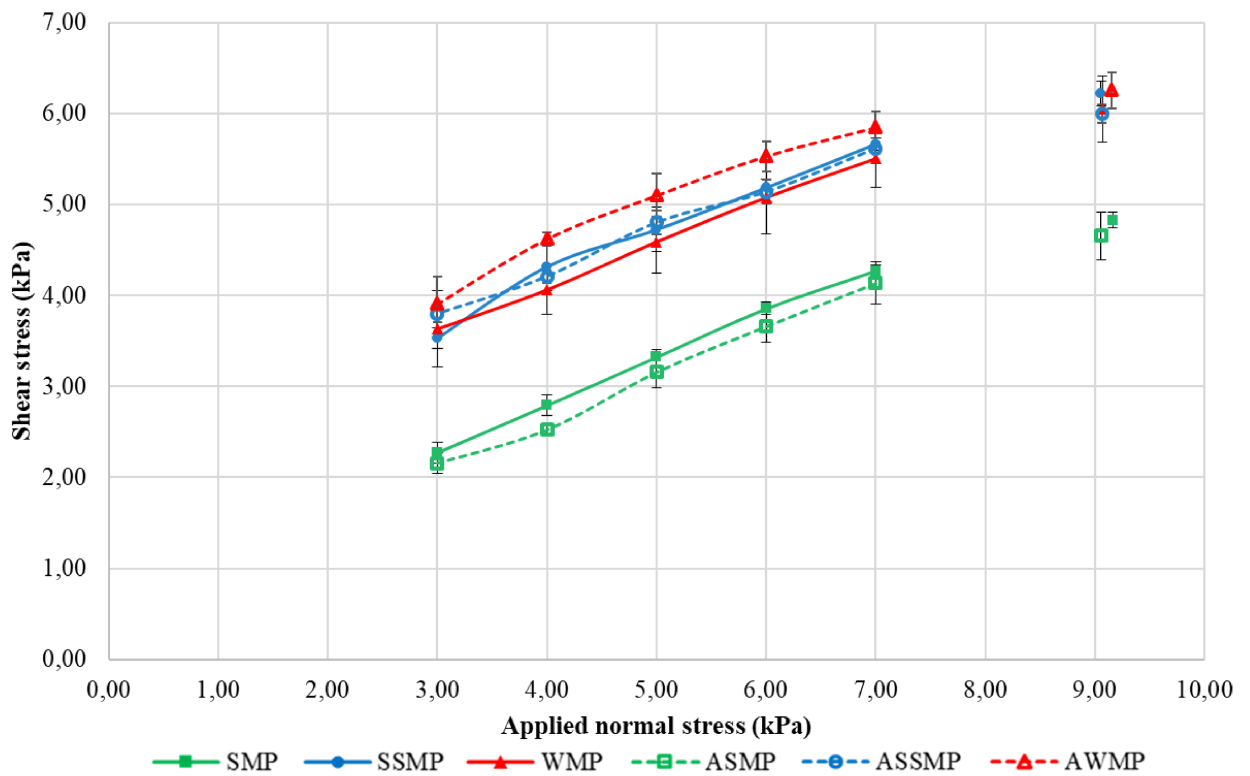


Fig. 5. Shear test results for SMP, SSMP, WMP, ASMP, ASSMP and AWMP.