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Powder Technology

Impact of the whey protein/casein ratio on the reconstitution and flow properties of spray-dried dairy protein powders

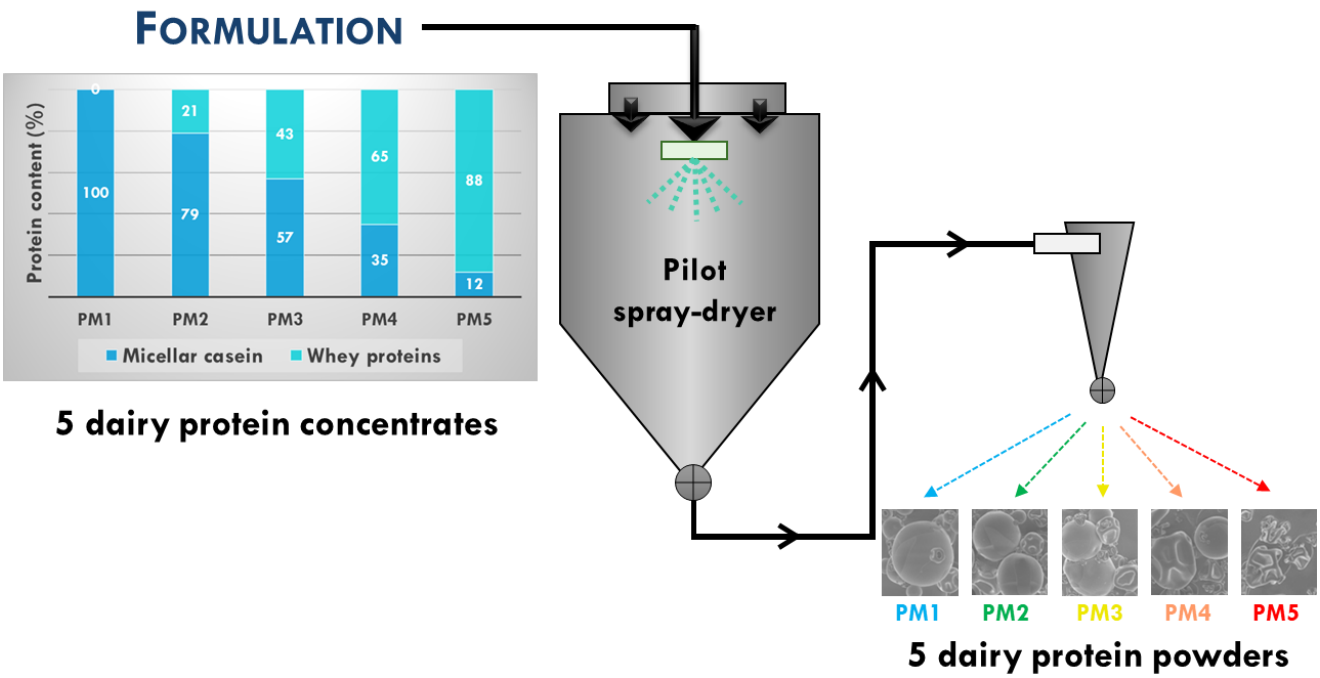
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	Carole Perroud-Thomassin
	Jeremy Petit
Abstract:	This study focuses on the impact of the whey protein/casein ratio on the physicochemical and functional characteristics of dairy protein powders spray-dried at 200 °C inlet and 85 °C outlet air temperatures. Physicochemical properties of powders were characterized to explain their reconstitutability and flowability. Solubility of dairy protein powders linearly varied with the whey protein/casein ratio. Standard wettability and dispersibility indices were not relevant to evaluate the reconstitutability of powders produced at pilot scale, likely owing to the low median particle size of investigated powders. The flowability of powders was poor due to a high level of interparticular cohesion, which derived from their small particle size. The impact of protein composition on flowability was not statistically significant. The small particle size, leading to poor flow properties, overwhelmed the impact of protein composition, but a slight improvement of flowability and a reduction of cohesion observed for higher proportions of whey proteins

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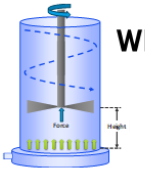
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GRAPHICAL ABSTRACT



TECHNOFUNCTIONAL PROPERTIES

Particle size controls powder flowability



Powder rheometer

Whatever the formulation, same granulometry: small particles = poor flowability

Casein/whey protein ratio impacts on powder solubility

High casein proportion → Poor solubility

High whey protein content → Good solubility

ABSTRACT

This study focuses on the impact of the whey protein/casein ratio (100/0, 79/21, 57/43, 35/65, and 12/88) on the physicochemical and functional characteristics of dairy protein powders. Spray-drying was performed in the following conditions: 200 °C inlet air temperature and 85 °C outlet air temperature. Physicochemical properties (particle size distribution, shape factors, moisture content, and water activity) of spray-dried powders were characterized to explain their reconstitutability, on the basis of the wettability, dispersibility, solubility indices, and their flowability, evaluated with the FT4 powder rheometer.

Reconstitutability analyses revealed that solubility of dairy protein powders linearly varied with the whey protein/casein ratio. It was also highlighted that wettability and dispersibility determined with the standard methods were not relevant to evaluate the reconstitutability of investigated powders: in fact, all studied powders were non-wettable and non-dispersible according to the milk powder standards. This was likely to result from the low median particle size (between 10 and 30 µm) of powders produced by spray-drying at pilot-scale.

The flowability of powders was evaluated as poor due to a high level of interparticular cohesion, which mainly derived from their small median particle size. The impact of the protein composition on the flowability was not statistically significant, despite a shape modification reflected by a decrease in particle sphericity upon casein proportion increase. The small particle size, leading to poor flow properties, overwhelmed the impact the whey protein/casein ratio, but a slight improvement of the flowability and a reduction of cohesion was induced by higher proportions of whey proteins.

Reviewer 1: This study focuses on the impact of the whey protein/casein ratio on the physicochemical and functional characteristics of dairy protein powders, which has been widely investigated.

The novelty of this paper can be improved if the spray-dried samples are furthur investigated from a chemical perspective.

It is true that the link between physicochemical properties and functionalities of powdered whey proteins and caseins has been widely investigated, but the novelty of this study comes from the focus on the impact of formulation (including chemical composition but also the preparation protocol of the concentrate to be spray-dried). The study presented in this manuscript was a part of a larger study who delt with the impact of formulation (pretreatment of concentrates prior to spray-drying, lipid content, protein content, and whey proteins/caseins ratio) on physicochemical and functional properties of spray-dried dairy powders. The part of this larger study dealing with concentrate pretreatment (air incorporation by stirring with a blade usually used for foaming purposes) and lipid content has already been published in Powder Technology (https://doi.org/10.1016/j.powtec.2020.05.085).

1. DSC, XRD, XPS and Raman investigations are suggested to reveal the mechanisms of the known phenomena at a chemical/molecular level.

DSC and XPS have already been carried out on the samples of the present study but showed no differences between samples (from DSC, it was observed that all samples were amorphous and their glass transition temperature was hardly measurable; from XPS, it was shown that their surface was enriched in lipids and proteins, as lipids tend to migrate to the droplet surface by buoyancy during the drying process, whereas proteins, being surface-active molecules, are generally found more concentrated at water/air interfaces (i.e. at the surface of droplets). Thus, it was decided not to present these analyzes in the manuscript.

DRX would not be more informative, as samples were amorphous. As for Raman spectroscopy, it could be useful to elucidate the structure of dairy proteins in produced samples, but the aim of the larger study was focused on physicochemical and functional properties of spray-dried dairy powders, not on the structure of individual particles constituting the powders.

2. Thermal stability effects are suggested to be studied for different whey protein/casein ratio.

This would be interesting, but the objective of the study was to find appropriate formulation conditions to improve reconstitutability and flowing properties of spray-dried dairy powders. Thermal stability, preservation ability, gelation properties, water-holding capacity, etc. are another functional properties of interest for spray-dried dairy powders, but they did not fall into the scope of the larger study.

3. The crumpled surfaces can be calculated with math models with diffusion coefficiency.

The present study was intended to elucidate the most influent parameters (amongst formulation and powder physicochemical properties) on powder reconstitutability and flowing properties. For example, it was evidenced that particle shape can counteract the influence of particle size on powder flowing properties (i.e. samples richer in caseins had larger particles of more wrinkled shape and flowed more poorly). However, the mechanisms leading to particle shape were not the subject of the present study.

Reviewer 2: Manuscript Number: POWTEC-D-21-00864

The subject of research includes in this journal. Research work is interesting. In the paper titled "Impact of the whey protein/casein ratio on the reconstitution and flow properties of spray-dried dairy protein powders".

The authors in their article discuss a very important aspect study focuses on the impact of the whey protein/casein ratio on the physicochemical and functional characteristics of dairy protein powders spray-dried. Research work is interesting.

Thank you for your interest in our work. We hope that modifications performed following your comments will help in improving manuscript quality.

The paper needs minor revision because:

1. The quality of the language is insufficient. Have a native speaker or similar assist you.

The language level was improved by using a more sustained language when possible (cf. modifications in blue in revised manuscript lines 16-18, 20-23, 24, 27, 40, 43, 64, 131, 190, 212, 231, 248, 253, 255, 280 and 282-310).

2. The Introduction is quite random with broad and varying statements and not very well focused.

Indeed, the introduction was a little too vague, as we initially wanted to make the link with the first part of the study already published in Powder Technology (https://doi.org/10.1016/j.powtec.2020.05.085). A better focus on the subject of the study was carried out (cf. modifications in blue in revised manuscript lines 16-18, 20-23 and 28).

3. In my opinion, the final conclusions should also be drawn up as they are not as specific as the scope of work.

The final conclusion was too exhaustive, we have modified it to make it more concise and better focused (cf. modifications in blue in the manuscript lines 280 and 282-310).

4. Please standardise the number of decimal places in the tables. Please use a precision of at least two decimals places.

We initially chose to present results with 3 significant numbers, thus the number of decimals depended on the values. Thus, we followed reviewer #2's advice for table 3 (cf. modifications in blue). For tables 1 and 2, it was not possible to add more decimals, as data of table 1 came from supplier sheets which were not more accurate than presented values and data of table 2 were calculated from date of table 1. Moreover, one decimal was added to water activity results, as the accuracy of the analytical equipment was sufficient to allow this (cf. modifications in blue).

5. Please explain why the authors chose such a statistical analysis?

ANOVA and Tukey's HSD test are very classical statistical analysis methods: the first one allows identifying the presence of significant differences amongst sample results, whereas the second one permits to sort samples according their significant differences. Pearson's correlation coefficients are also rather classically used to link parameters between them: it permits to calculate the degree of linear correlation between two variables; in the present study, the degree of correlation between analyzed parameters was determined to help identifying the main physicochemical parameters influencing each analyzed functional properties.

6. Could an additional PCA analysis have been performed?

Yes, indeed, PCA could have been performed, and it would have given similar information as Pearson's correlation coefficients calculated for all pairs of investigated parameters (as PCA intends to correlate all investigated parameters in a single statistical analysis, whereas Pearson's

correlation coefficients are able to indicate the degree of correlation between two parameters only). We chose to discuss results with Pearson's correlation coefficients that we estimate easier to understand for readers than PCA.

7. Improve the readability of the Figure 3, 4.

The readability of figures 3 and 4 has been enhanced (cf. figures 3 and 4 in figures file).

8. References is incorrectly formatted number [17 - 21 and 31].

The reference 17 has been modified, but it's a French PhD so the title should be kept in French. Other concerned references are norms that were already correctly formatted: these references have already been published in Powder Technology with this format (https://doi.org/10.1016/j.powtec.2020.05.085).

Revised Manuscript Click here to view linked References

IMPACT OF THE WHEY PROTEIN/CASEIN RATIO ON THE RECONSTITUTION AND FLOW PROPERTIES OF SPRAY-DRIED DAIRY PROTEIN POWDERS

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KEYWORDS

Dairy powders; formulation; protein composition; spray-drying; reconstitutability; flowability

1. INTRODUCTION

Technofunctional properties of dairy powders such as reconstitutability (wettability, swelling, dispersibility and solubility) and flowability are crucial for the agri-food industry and consumers.

Wettability is the ability of powders to immerse without stirring [1]. Powder wettability is sensitive to several parameters like particle size: smaller particles generally induce poorer wettability~~Powder surface composition also affects the wetting time: an increase in the proportion of surface lipids is deleterious for powder wettability due to higher hydrophobicity~~[2–4].

Dispersibility corresponds to the dissociation of particle agglomerates into single particles and, in a lesser extent, to the attrition of particle agglomerates and single particles. ~~For dispersibility indices lower than 95 %, the powder is considered as non-dispersible: for example, in the case of whole milk (containing 26 % lipids) for which the high proportion of surface lipids impairs~~

~~interactions with water, the dispersibility index is 89.5 %. Moreover,~~ The presence of caseins has been identified ~~known~~ to strongly reduce dairy powder dispersibility (indeed, the dispersibility index of casein powders is around 26 %) [1].

Solubility represents the ability of powders to dissolve into water, which corresponds to the disappearance ~~complete loss~~ of granular structure once the product is reconstituted [1]. Like wettability, solubility decreases with ~~lipids proportion or~~ particle size. In addition, it was evidenced that a high casein content is related to a poor solubility, whereas whey proteins promote dairy powder solubilization [5–7]. Powder flowability is highly linked to its physicochemical properties: large, smooth, and spherical particles having a low water content are known to have good flow properties [8–10]. As matters the influence of the whey protein/casein ratio, a high proportion of caseins is known to lead to wrinkled and dense particles, with poorer flow properties [11]. The flowability can be characterized in low and high stress conditions by a rotational powder rheometer such as the FT4 in order to obtain several flow parameters, especially basic flowability energy (BFE) and SE (specific energy) in low stress conditions or flow factor (*ff*) and cohesion in high stress conditions [8,12–14].

The present study focuses on the impact of composition on reconstitutability and flowability of dairy protein powders. Investigated powders differed in protein composition (variable casein/whey protein ratio) and were produced by spray-drying at pilot scale. Afterwards~~Then~~, the reconstitutability of spray-dried powders was evaluated through the wettability, dispersibility, and solubility indices classically employed in the dairy industry. Moreover~~Besides~~, flowability of dairy protein powders was assessed through measurement of BFE, SE, compressibility, cohesion, and *ff* with the FT4 powder rheometer. Finally, physicochemical properties of spray-dried powders (particle size distribution, appearance of particle surface, water content, and water activity) were determined and related to powder reconstitution and flow behavior.

2. MATERIAL AND METHODS

2.1. Material

Whey proteins isolate (PRODIET 90 S) and micellar casein powder (PRODIET 87 B) were provided by Ingredia (Arras, France). Their proximate composition in wet basis was calculated from technical sheets (Table 1).

Table 1. Proximate composition expressed on wet basis of whey protein isolate and phosphocaseinate powders employed for the preparation of dairy concentrates to be spray-dried.

2.2. Preparation of milk concentrates

Five concentrates differing in protein composition were produced from 100 % whey protein isolate (protein mixture 1, PM1), 75 % whey protein isolate and 25 % casein powder (protein mixture 2, PM2), 50 % whey protein isolate and 50 % casein powder (protein mixture 3, PM3), 25 % whey protein isolate and 75 % casein powder (protein mixture 4, PM4), and 100 % casein powder (protein mixture 5, PM5). Concentrates were prepared by reconstituting adequate amounts of powders in distilled water at 15 % (w/w) dry extract under mechanical stirring (ZR 2102 control, Heidolph, Germany) between 200 and 500 rpm with a 4-bladed impeller (2 cm length, 1 cm width). Once the powder mixture was completely~~totally~~ immersed, the solution was stirred during 2 h at room temperature (20 °C), then at 4 °C under slight agitation at 300 rpm overnight to ensure complete powder reconstitution.

The proximal composition of spray-dried concentrates was calculated from technical sheets of reconstituted powders (Table 2).

Table 2. Proximate composition on dry basis of spray-dried concentrates.

2.3. Production of dairy powders by spray-drying

Spray-drying was carried out on a Micra Spray MS 150 single effect spray-dryer (Anhydro, Soeborg, Denmark), equipped with a bi-fluid nozzle (Fluid Cap 60 100 + Air Cap 120, Spraying System, Wheaton, Illinois, USA). Spray-drying conditions were set at 200 °C drying air inlet temperature and 85 °C outlet temperature in view to obtain powders with good preservation abilities (i.e., water activity ranging from 0.2 to 0.3) [1,15]. The concentrate was fed by a FASTLoad peristaltic pump (VWR, Leuven, Belgium; 4.8 mm internal pipe diameter) and the feed concentrate flow rate was regulated at circa 68.0 $mL.min^{-1}$ to maintain the drying air outlet temperature at 85 °C [16]. Powders were bagged just after sampling to limit degradations susceptible to occur during storage (such as caking induced by moisture uptake) and stored at 10 °C until analyses.

2.4. Physicochemical characterization

2.4.1. Particle size distribution

Particle size measurements were carried out with a laser granulometer (Mastersizer S, Malvern Instruments Ltd, Malvern, UK) supplied with a 5 mW He-Ne laser operating at a wavelength of 632.8 nm with a 300 RF lens. Air dispersion was achieved with the Aero S module (Malvern). The dispersion conditions were adapted to each powder: air pressure between 2 and 3 bar, feed rate between 60 and 90 %, and hopper length between 2 and 3 mm. Three replicates were analyzed for each sample. The mean diameter in volume was chosen as size estimator. Classical granulometric parameters (D_{10}, D_{50}, and D_{90}) were calculated, where D_x designates the diameter for which *x* % of the particles have a smaller size. The width of the particle size distribution was evaluated with the span (Eq. (1)) [17].

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

2.4.2. Scanning electron microscopy

Scanning electron microscopy (SEM) was employed to characterize size, morphology, and surface structure of milk powders. Powder samples were sputtered on double-sided carbon tape, fixed to SEM stubs, and coated with carbon in a sputter coater (Polaron SC7640, Thermo VG Scientific, East Grinstead, England). Observations were performed at × 500 magnification under partial vacuum using a scanning electron microscope (Hitachi S-4800, Japan) operating at 1.0 kV acceleration voltage [18].

2.5. Powder interactions with water

2.5.1. Water activity

Water activity was measured with a HygroPalm23-AW portable water activity-meter (Rotronic, France) at room temperature (20 °C), using a 3 cm powder layer (15 ± 1 g) in a cup similarly to ISO 21807:2004 [19].

2.5.2. Water content

The water content was measured by the loss of water mass of 2.0 g ± 0.1 mg powder after drying during at least 3 h at 103 °C until a constant dry powder mass was obtained following ISO 5537:2004 [20].

2.5.3. Reconstitutability

Reconstitutability of spray-dried powders was evaluated through wettability, dispersibility and solubility indices.

2.5.3.1.Wettability

The wettability index is defined as the time in seconds taken by 13.0 ± 0.1 g powder to become wet when gently deposited at the surface of 125.0 g ± 0.1 g distilled water in a 250 mL beaker, following IDF 87:1979 [21].

2.5.3.2.Dispersibility

Dispersibility was measured by a method adapted from IDF 87: 1979 [21]. 13.0 ± 0.1 g powder was gently deposited on the surface of 125.0 g water at 20 °C placed in a 400 mL beaker. The mixture was manually stirred for 20 s, then it was allowed to rest for 30 s. After that, the mixture was passed through a 120 µm-mesh sieve. The total dry matter of the liquid, corresponding to the dispersibility index, was determined by drying at 103 ° C for 5 h following (Eq. (2)).

$$Dispersibility\ (\%) = \frac{W \times T}{100-(E+T)} \times \frac{100}{S} \quad (2)$$

with:

- W: distilled water mass (g);

- T: dry matter content (% w/w) of the liquid;

- E: dry matter content (% w/w) of sampled powder;

- S: sampled powder mass (g).

2.5.3.3.Solubility

Solubility was characterized as the percentage of powder able to dissolve in distilled water following ISO 8156:2005 [22]. It was determined by the addition of 2.5 ± 0.1 g powder in 17.5 mL distilled water in a 50 mL centrifugation tube followed by 30 s stirring and centrifugation at 2 294 × g during 10 min (Megafuge 8R, Thermoscientific, France). Afterwards~~Then~~, the supernatant was removed, and the pellet was added to 20 mL distilled water, followed by stirring and centrifugation at 2 294 × g during 10 min. The pellet was

desiccated at 103 °C overnight to determine the mass of insoluble residues, which allows determining the solubility index (Eq. (4)).

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

with:

- M: undissolved sediment mass after desiccation (g);

- S: sampled powder mass (g).

2.6. Flow properties of dairy protein powders

Flowability properties were evaluated using a FT4 powder rheometer with 25 mm accessories (Freeman Technology, Worcestershire, UK).

2.6.1. Stability test

The stability test was performed through a series of seven conditioning cycles by rotating the blade (−5° helix angle, 23.5 mm diameter, 100 $mm.s^{-1}$ tip speed) and measurements of the flow energy of the powder bed [9,13].

This test allows determining two flow indices: BFE and SE. The BFE is linked to the flow behavior of the powder bed, and a low BFE generally corresponds to an easy-flowing behavior, whereas a high BFE is often obtained for poorly-flowing powders. The SE is much linked to powder cohesion [8,9] with high SE values associated with cohesive powders.

Freeman Technology provides a classification of powder flow behavior on the basis of the SE calculated by the FT4 powder rheometer by:

- SE < 5 mJ/g: low cohesion

- 5 mJ/g < SE < 10 mJ/g: moderate cohesion

- SE > 10 mJ/g: high cohesion.

2.6.2. Compressibility test

The compressibility test is used to measure the evolution of powder density as a function of normal stress from 0.5 to 15 kPa applied by a 23.5 mm vented piston to the powder sample placed in a 25 mm × 10 mL measurement cell [9,23,24].

The compressibility test allows obtaining the bulk density (BD), i.e. powder density before compression, similar to aerated density, and the compressed bulk density (CBD) for powder density after compression. Particle size, shape, surface composition, and roughness are known to influence these compressibility parameters [8].

2.6.3. Shear cell test

The shear cell test was carried out to evaluate powder flowability and cohesion under high shear stress after a precompaction at 9 kPa. The powder bed was subjected to normal stresses of 7 to 3 kPa by 1 kPa steps in a 25 mm × 10 mL measurement cell [8,9,13,14,24].

The shear test is used to determine the powder cohesion and the flow factor (*ff*), calculated by the yield locus method from the shear stress vs. normal stress curve [8]. Powder flowability has been classified by Jenike [12] from flow factor values:

- $ff < 1$: not flowing
- $1 < ff < 2$: very cohesive
- $2 < ff < 4$: cohesive
- $4 < ff < 10$: easy flowing
- $f > 10$: free flowing.

2.7. Statistical analysis

All analyses were triplicated to ensure a good analytical repeatability and presented values correspond to means ± standard deviations. Data were statistically analyzed by one-way ANOVA performed with Excel version 2016 and means were separated by Tukey's HSD test at $p < 0.05$ significance level. The degree of correlation between powder physicochemical and functional properties was evaluated by calculating Pearson's correlation coefficients.

3. RESULTS AND DISCUSSION

3.1. Particle size distribution

Dairy protein powders spray-dried at pilot scale were composed of fine particles ranging from 1 to 200 µm (Figure 1 & Table 3), consistently with the particle size range generally obtained with the employed pilot spray-dryer [16,25–27]. Their median particle size D_{50} was comprised between 11.5 and 18.9 µm, particle size distributions were monomodal, and moderate dispersion around the median value was denoted by the span values ranging from 2.1 to 2.5.

Consistently with literature, the median particle size of protein powders increased with casein proportion of spray-dried concentrates (Table 2) [28], as evidenced by the significant~~strong~~ positive correlation obtained between these two parameters according to a Pearson's correlation coefficient of 0.932. This may be explained by the fact that during drying, droplets of concentrates rich in caseins quickly form a crust at their surface [11], which results in larger particles than spray-dried protein powders containing a greater whey protein proportion. No significant influence of protein composition on the span of the particle size distribution was denoted.

Fig. 1. Particle size distributions (in volume) of PM 1, PM 2, PM 3, PM 4, and PM 5.

Table 3. Granulometric parameters of PM 1, PM 2, PM 3, PM 4, and PM 5 powders.

3.2.Particle appearance

SEM images of protein powders (Figure 3) showed that protein composition influenced particle shape: particle sphericity decreased at higher casein proportion, confirming previous literature results [11,29,30]. This may confirm that the surface of droplets of concentrates rich in caseins had rapidly formed a crust when put in contact with the hot drying air and warped during the drying step, leading to non-spherical particles. It may be concluded that whey protein concentrate droplets did not form a crust upon drying, thus the spherical shape of droplets was kept during the drying step, leading to globally spherical whey protein particles.

Fig. 2. SEM micrographs of spray-dried protein powders varying in protein composition (in the increasing order of casein proportion: PM 1 (A); PM 2 (B); PM3 (C); PM 4 (D); PM 5 (E)) acquired at × 500 and × 2000 magnifications.

3.3. Powder interactions with water

Water activity of spray-dried dairy protein powders was low, ranging from 0.16 to 0.26 (Table 4), ensuring an excellent~~good~~ protection against spoilage during storage [31], as the ideal water activity of food products, especially dairy, is comprised between 0.2 and 0.3 [1]. The good preservability of produced powders was confirmed by their water content, not exceeding the threshold of 5 % (w/w) on wet basis generally advised for dairy powders [32]. The slight variation of water content of spray-dried dairy protein powders in spite of identical manufacturing conditions may come from the difference in hygroscopicity of whey proteins and caseins along with the impact of formulation on the drying kinetics.

The fineness of spray-dried dairy protein powders caused their poor wettability, as expected for powders spray-dried at pilot-scale [8,16,33]. Their dispersibility was very low (around 5 - 10 %), highlighting a high level of powder cohesion (which may be an indication of poor flowability). The influence of each dairy protein type could not be deduced from these results,

because all powders were hardly dispersible [1,11], leading to insignificant differences between powder samples [34,35]. The impact of protein type was much more interesting on powder solubility. Indeed, a linear decrease (Eq. 5, R^2 = 98.62 %) of powder solubility was observed when increasing the casein content of spray-dried dairy protein powders (Table 4-B & Figure 5).

$$solubility\ (\%) = 99.554 - 0.698 \times casein\ content\ (\%) \qquad (Eq.\ 5)$$

This can be related to literature indicating that the presence of casein constitutes a limiting factor for powder reconstitutability, especially by impairing solubility [1,5,36,37]. Moreover~~Besides~~, as whey proteins and lactose from dairy protein powders are known to solubilize well more easily and quickly than caseins [7], the decrease in their contents upon increase in casein content (cf. Table 2) may contribute to the observed solubility decrease. As regards the impact of mineral content, which increased with casein content in studied powders (cf. Table 2), Mimouni et al. (2010) [7] showed that calcium, phosphorus, and magnesium, which constitute the majority of dairy minerals as they are principally associated with casein micelles, have rather slow reconstitution kinetics; this also contributes to the solubility decrease of investigated powders upon casein content increase. Last, lipids content in studied powders was low and very close (cf. Table 2), thus the negative impact of these highly hydrophobic components [1, 2, 3] (Gaiani et al., 2006) [1–3,38] on powder solubility could not be observed in the present study.

Table 4. Water content, water activity, wettability, dispersibility, and solubility of spray-dried dairy protein powders.

Fig. 3. Evolution of powder solubility with casein content for spray-dried dairy protein powders.

3.4. Powder flow properties

Flow parameters deduced from the stability, compressibility, and shear cell tests are gathered in Table 5.

BFE of spray-dried dairy protein powders increased in the following order: PM1 < PM5 ≈ PM2 < PM3 < PM4. No significant~~clear~~ influence of protein composition on basic flowability energy was denoted, except that 100 % whey protein powder (PM1) had slightly better flowability. As its smaller median particle size and larger span should be deleterious for flow properties, it can be hypothesized that its better flowability may arise from the more spherical shape of its particles. Specific flow energy is generally more informative regarding flowability classification. In the present study~~Here~~, specific flow energy results indicate that PM1 and PM5 are expected to better flow and be moderately cohesive, followed by PM2, PM3, and PM4 that are expected to flow more poorly and have higher cohesion. Afterwards~~Then~~, powders with better flowability were either composed of very small particles of relatively high heterogeneity but rather spherical shape (PM1) or small particles of moderate heterogeneity and wrinkled shape (PM5). Indeed, powder flowability in generally improved for large particles, of low heterogeneity, and/or of spherical shape [8–11].

Compressibility at 15 kPa was identical for all spray-dried dairy protein powders (Figure 4), presumably owing to their relatively close particle size distributions and their composition dominated by dairy proteins. It can be guessed that their cohesion was not too different, as no marked differences of compressibility have been obtained. In fact, cohesion, deduced from the shear cell test, was comprised between 1.55 and 1.75 kPa for all investigated powders, indicating a rather high level of cohesion, and thus a low flowability. *ff* values were found in the range from 2.78 to 3.12, showing that all investigated powders had close flow properties, exhibiting a cohesive behavior and a low flowability. No significant impact of protein composition was evidenced, but trends indicated that cohesion was slightly raised and *ff* slightly

reduced for powders containing more caseins (Figure 5); this was confirmed by the highly negative Pearson's correlation coefficient between casein proportion and *ff* (R = -0,762) and the moderately positive Pearson's correlation coefficient between casein proportion and cohesion (R = 0,570). Thus, flowability under normal stress was a little improved for powders richer in whey proteins.

Table 5 : Flowability characteristics (from stability, compressibility, and shear cell tests) of PM 1, PM 2, PM 3, PM 4, and PM 5.

Fig. 4: Compressibility results for PM 1, PM 2, PM 3, PM 4, and PM 5.

Fig. 5: Shear test results for PM 1, PM 2, PM 3, PM 4, and PM 5.

4. CONCLUSION

This study is part of a wider research project intending to evaluate the relative influences of formulation (whey proteins/caseins ratio ~~preparation and composition of concentrates~~) and spray-drying conditions on physicochemical and functional properties of spray-dried dairy powders. ~~The final aim of the project is to establish process relationships relating formulation and spray-drying conditions to powder reconstitutability, flowability, and preservability.~~

~~In the present study, a focus was carried out on the influence of protein composition on functional properties of spray-dried dairy protein powders. Thus, dairy protein powders differing in whey protein/casein ratio were spray-dried in identical the same conditions in order to isolate the influence of protein composition.~~

In the present study, a focus was carried out on the influence of whey protein/casein ratio on functional properties in identical spray-drying conditions.

~~All powders presented similar particle size distributions, which were monomodal with a moderate dispersion of particle sizes around the median value. A marked influence of~~

~~formulation was denoted by the small increase in median particle size at higher casein proportion.~~

Particle size distributions of spray-dried powders varying in whey proteins/caseins ratio were similar (they were monomodal and had comparable span), but a marked influence of formulation was denoted by the small increase in median particle size at higher casein proportion.

~~This was explained by the different drying behaviors of droplets of whey protein and casein concentrates: formulations richer in caseins are known to lead to the formation of a crust at the droplet surface, resulting in larger and wrinkled particles, whereas droplets containing more whey proteins are known to lead to more spherical particles of smaller size. These hypotheses of different drying mechanisms, consistent with the literature, were validated by the differences in particle appearance between powders containing more whey proteins and those composed of more caseins.~~

Formulations richer in caseins are known to lead to the formation of a crust at the droplet surface, resulting in larger and wrinkled particles, whereas droplets containing more whey proteins are known to lead to more spherical particles of smaller size.

~~Water content and water activity of all spray-dried powders were low, ensuring their good preservability.~~

Owing to the low median particle size obtained for powders spray-dried at pilot scale, wettability and dispersibility of produced powders were very low according to industrial standards, and no significant difference due to powder formulation was evidenced. Solubility linearly varied with the whey protein/casein ratio (it was presumably impaired by the presence of a casein crust at the particle surface) and this process relationship may help industrials formulating their spray-dried dairy protein powders.

Flowability of spray-dried dairy protein powders was poor and interparticular cohesion was high, presumably due to the small median particle size of powder spray-dried on the employed pilot-scale equipment. For all powders, the influence of their particle size distribution seemed to overwhelm the impact of powder formulation. However, a slight improvement of flow properties was observed for dairy protein powders containing more whey proteins and less caseins. This may arise from the more spherical shape of this kind of powders. Powder flowability, mainly controlled by median particle size, should be improved for powders spray-dried at industrial scale, which are generally well larger.

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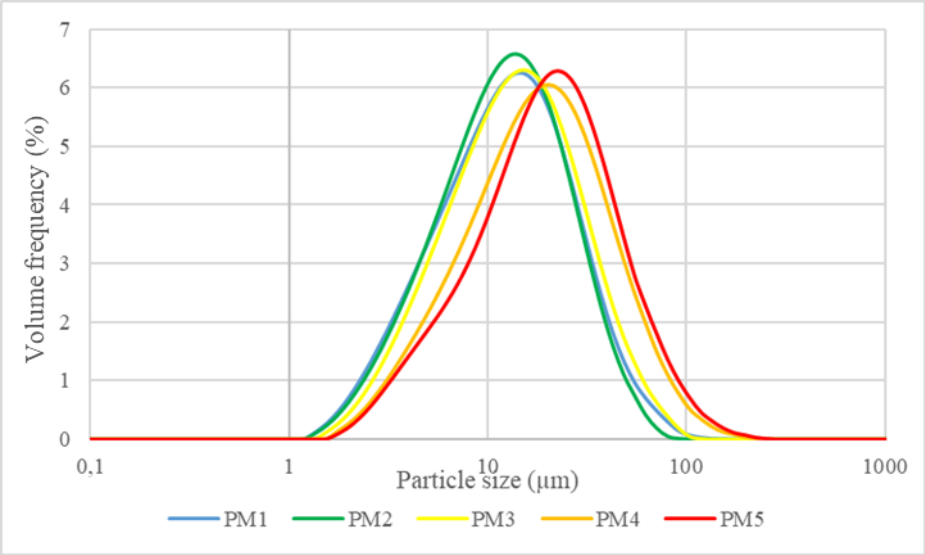


Fig. 1. Particle size distributions (in volume) of PM 1, PM 2, PM 3, PM 4, and PM 5.

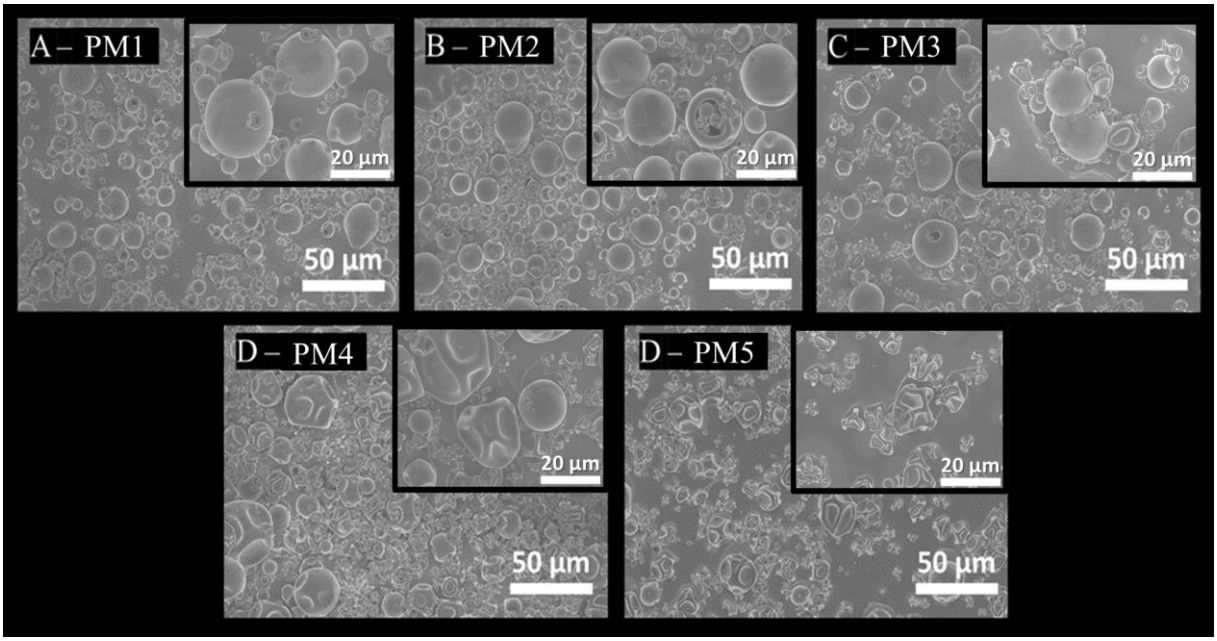


Fig. 2. SEM micrographs of spray-dried protein powders varying in protein composition (in the increasing order of casein proportion: PM 1 (A); PM 2 (B); PM3 (C); PM 4 (D); PM 5 (E)) acquired at × 500 and × 2000 magnifications.

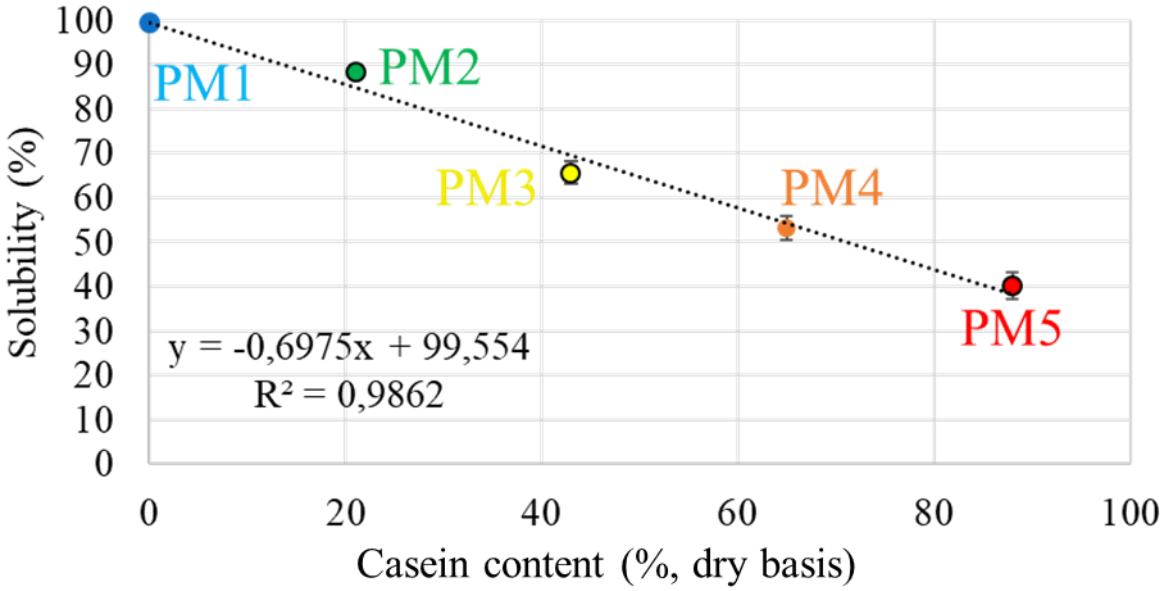
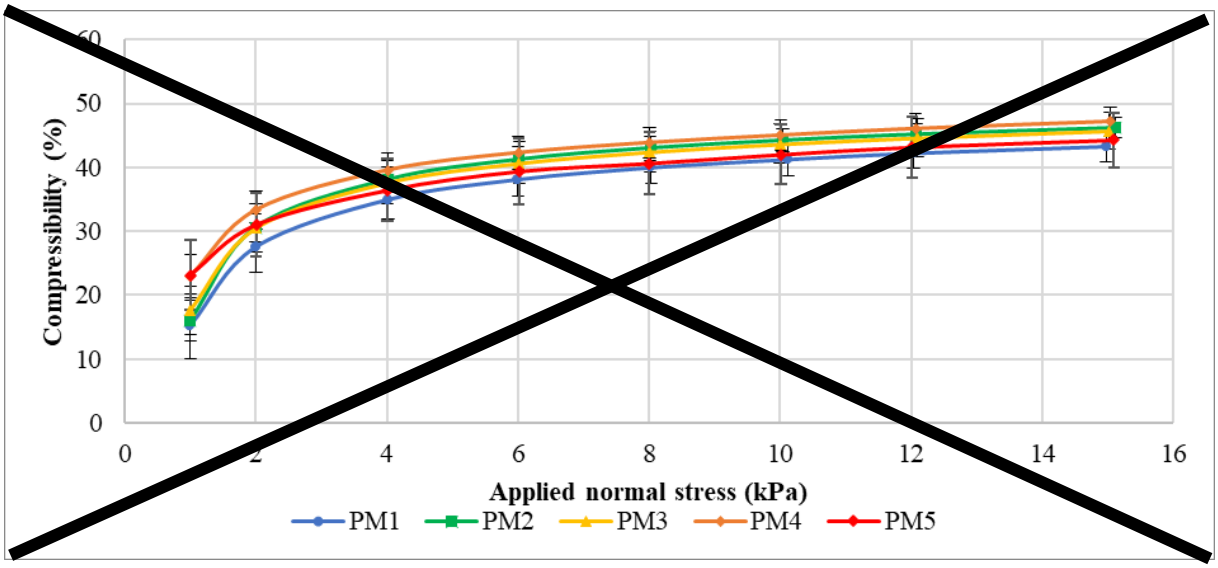


Figure 3. Evolution of powder solubility with casein content for spray-dried dairy protein powders.



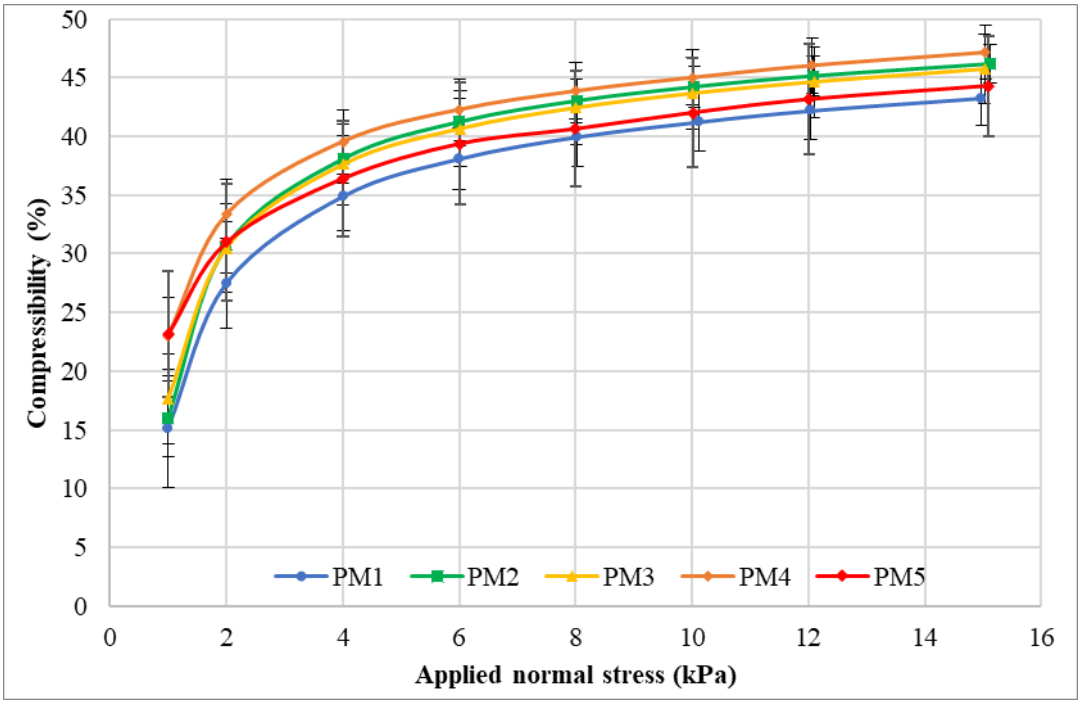
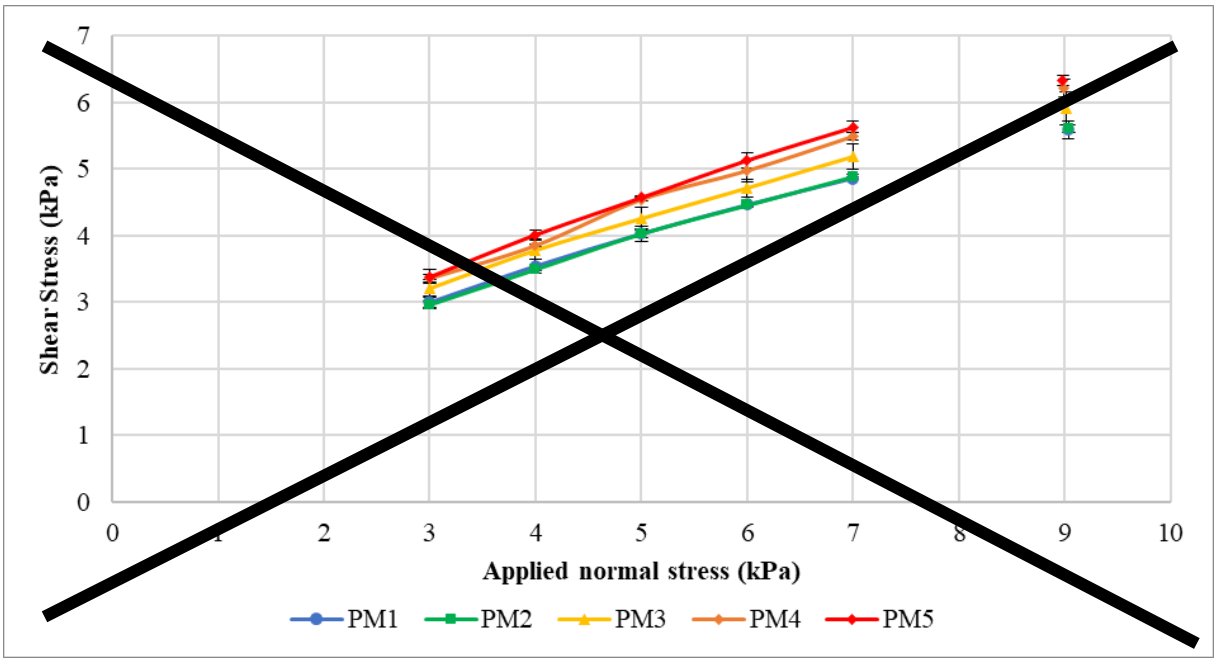


Fig. 4: Compressibility results for PM 1, PM 2, PM 3, PM 4, and PM 5.



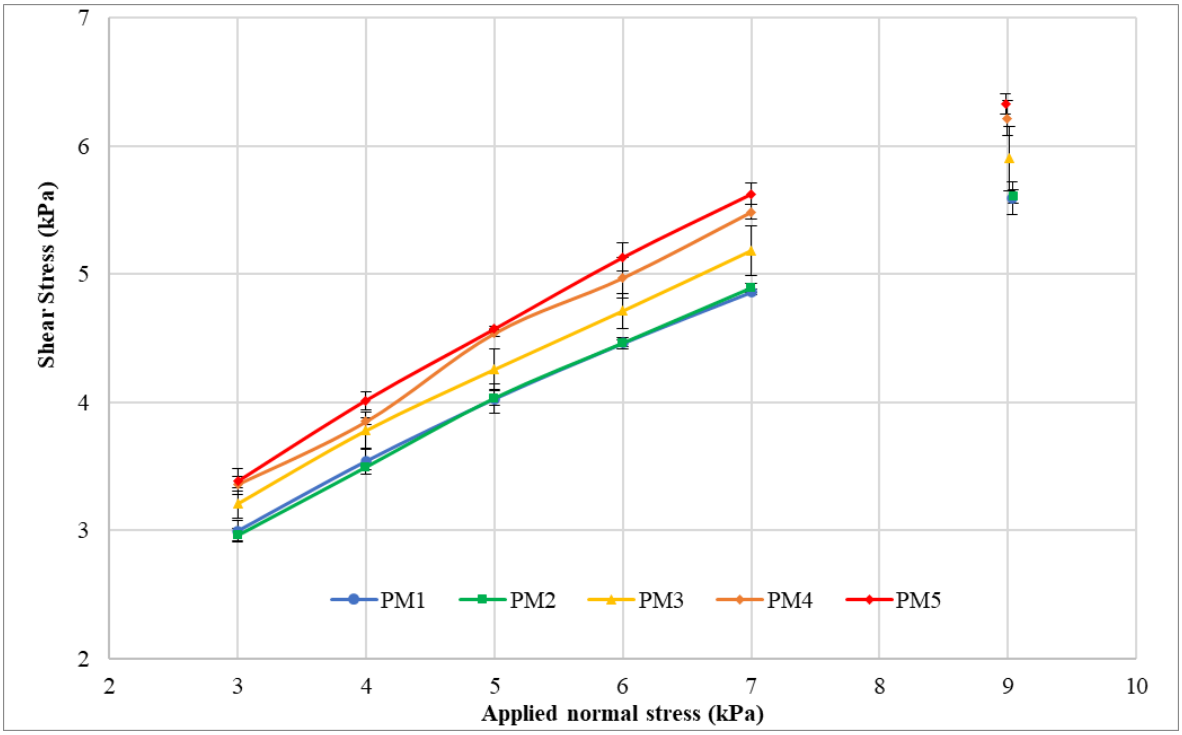


Fig. 5 : Shear test results for PM 1, PM 2, PM 3, PM 4, and PM 5.

Table 1. Proximate composition expressed on wet basis of whey protein isolate and phosphocaseinate powders employed for the preparation of dairy concentrates to be spray-dried.

Powder	Moisture (%)	Proteins (%)	Whey proteins (%)	Caseins (%)	Lipids (%)	Lactose (%)	Minerals (%)
Whey proteins isolate	5.5	85.5	85.5	0	1.0	5.5	2.5
Casein powder	5.0	83.0	10.8	72.2	1.5	2.0	8.5

Table 2. Proximate composition on dry basis of spray-dried concentrates.

Concentrates	Proteins (%)	Whey proteins (%)	Caseins (%)	Lipids (%)	Lactose (%)	Minerals (%)
PM 1	90.5	90.5	0.0	1.1	5.8	2.6
PM 2	89.7	70.6	19.1	1.2	4.9	4.2
PM 3	88.9	50.7	38.3	1.3	4.0	5.8
PM 4	88.1	30.7	57.4	1.5	3.0	7.4
PM 5	87.4	10.8	76.6	1.6	2.1	9.0

Table 3. Granulometric parameters of PM 1, PM 2, PM 3, PM 4, and PM 5 powders.

Powders	D_{10} (µm)	D_{50} (µm)	D_{90} (µm)	Span (-)
PM 1	$3.79^{e} \pm 0.01$	$11.82^{d} \pm 0.11$	$31.02^{d} \pm 0.53$	$2.31^{b} \pm 0.05$
PM 2	$3.92^{d} \pm 0.04$	$11.53^{d} \pm 0.04$	$28.51^{e} \pm 0.34$	$2.15^{c} \pm 0.02$
PM 3	$4.34^{c} \pm 0.03$	$12.80^{c} \pm 0.23$	$33.81^{c} \pm 0.43$	$2.29^{b} \pm 0.02$
PM 4	$5.20^{b} \pm 0.00$	$16.93^{b} \pm 0.14$	$47.04^{b} \pm 0.22$	$2.47^{a} \pm 0.01$
PM 5	$5.61^{a} \pm 0.03$	$18.92^{a} \pm 0.13$	$51.33^{a} \pm 0.15$	$2.42^{a} \pm 0.00$

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. Water content, water activity, wettability, dispersibility, and solubility of spray-dried dairy protein powders.

Dairy protein powder sample	Water content (% (w/w) on wet basis)	Water activity (-)	Wettability (min)	Dispersibility (%)	Solubility (%)
PM 1	$4.82^{a} \pm 0.14$	$0.21^{c} \pm 0.00$	> 90	$5.38^{b} \pm 0.08$	$99.26^{a} \pm 0.00$
PM 2	$3.82^{c} \pm 0.12$	$0.20^{d} \pm 0.00$	> 90	$4.83^{c} \pm 0.09$	$88.35^{b} \pm 0.21$
PM 3	$3.78^{c} \pm 0.04$	$0.23^{b} \pm 0.00$	> 90	$5.06^{c} \pm 0.14$	$65.58^{c} \pm 2.58$
PM 4	$3.78^{c} \pm 0.15$	$0.16^{e} \pm 0.00$	> 90	$8.91^{a} \pm 0.13$	$53.11^{d} \pm 2.80$
PM 5	$4.38^{b} \pm 0.17$	$0.25^{a} \pm 0.00$	> 90	$3.26^{d} \pm 0.08$	$40.12^{e} \pm 2.90$

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

Tableau 5 : Flowability characteristics (from stability, compressibility, and shear cell tests) of PM 1, PM 2, PM 3, PM 4, and PM 5.

	Basic flowability energy BFE (mJ)	Specific flow energy SE (mJ/g)	Compressed bulk density CBD (g/mL)	Bulk density BD (g/mL)	Compressibility at 15 kPa (%)	Cohesion (kPa)	Flow factor *ff* (-)
PM1	$38.72^{b} \pm 3.87$	$8.85^{bc} \pm 0.62$	$0.34^{b} \pm 0.03$	$0.19^{b} \pm 0.01$	$43.28^{a} \pm 2.33$	$1.65^{a} \pm 0.16$	$3.01^{a} \pm 0.16$
PM2	$50.87^{a} \pm 5.19$	$10.55^{ab} \pm 0.39$	$0.36^{ab} \pm 0.01$	$0.20^{b} \pm 0.00$	$46.16^{a} \pm 1.62$	$1.55^{a} \pm 0.09$	$3.12^{a} \pm 0.10$
PM3	$56.45^{a} \pm 6.01$	$11.12^{a} \pm 0.77$	$0.37^{ab} \pm 0.00$	$0.20^{ab} \pm 0.01$	$45.73^{a} \pm 2.96$	$1.79^{a} \pm 0.24$	$2.78^{a} \pm 0.25$
PM4	$59.43^{a} \pm 3.52$	$11.74^{a} \pm 0.98$	$0.41^{a} \pm 0.01$	$0.22^{a} \pm 0.01$	$47.15^{a} \pm 2.27$	$1.75^{a} \pm 0.24$	$2.79^{a} \pm 0.25$
PM5	$50.28^{ab} \pm 0.90$	$8.35^{c} \pm 0.99$	$0.39^{a} \pm 0.03$	$0.22^{a} \pm 0.00$	$44.31^{a} \pm 4.27$	$1.72^{a} \pm 0.24$	$2.80^{a} \pm 0.25$

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

Highlights

- Whey protein/casein ratio impacts on dairy protein powder characteristics
- Dairy protein powder solubility linearly varied with the whey protein/casein ratio
- Standard reconstitution tests were irrelevant for powders spray-dried at pilot scale
- Particle size eclipses the impact of the whey protein/casein ratio on flowability

Credit author statement

Tristan FOURNAISE: Conceptualization, Investigation, Methodology, Validation, Writing, Review & Editing.

Jennifer BURGAIN: Project administration, Conceptualization, Supervision, Review & Editing, Funding acquisition.

Carole PERROUD-THOMASSIN: Investigation, Review & Editing.

Jérémy PETIT: Project administration, Conceptualization, Supervision, Methodology, Validation, Writing, Review & Editing

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Credit author statement

Tristan FOURNAISE: Conceptualization, Investigation, Methodology, Validation, Writing, Review & Editing.

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