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### Impact of spray-drying conditions on flow properties of skim dromedary and cow's milk powders using the FT4 powder rheometer

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### **13 ABSTRACT**

Two sets of spray-drying conditions were applied on skim dromedary and cow's milks to determine the effect of air outlet temperature (75 and 85 °C) and milk type on proximate composition, particle size distribution, and flow properties of spray-dried powders. The particle size distribution was more influenced by the chemical composition than the spray-drying conditions. The air outlet temperature impacted on dromedary milk powder flowability, as the basic flowability energy was significantly increased from 620 to 1018 mJ when the outlet air temperature was increased from 75 to 85 °C, respectively. Specific energy, associated with the flow performance of a powder in a low stress environment, were significantly higher for dromedary milk powder at the higher air outlet temperature. However, spray-drying conditions had no significant effect on specific energy values of cow's milk powder. Dromedary milk powders had better flow behavior than cow's milk powders. Shear tests confirmed that powders spray-dried at 75 °C had better flowability than powders spray-dried at 85 °C. 

### 26 NOVELTY IMPACT STATEMENT

Powdering is particularly interesting for dromedary milk industrialization, as a way to
overcome problems related to production seasonality and commercialization in low production
regions. Using a lower air outlet temperature (75 °C) led to better flowability and lower
compressibility. Shear tests showed that cow's milk powders were more cohesive than
dromedary milk powders.

32 Keywords: spray drying; flowability; dromedary milk powder; basic flowability energy;
33 specific energy; shear test.

### 1. INTRODUCTION

Milk transformation into a powdered product by means of a drying process is widely used in the dairy industry (Hammes, Englert, Noreña, & Cardozo, 2015). Milk powder can be preserved for longer times and its production reduces the cost of certain operations, such as packing, storage, and transportation (Kitamura, Itoh, & Echizen, 2009). Powdering processes allow the almost complete preservation of nutrients (Gaiani et al., 2010). In the case of dromedary milk, powdering is particularly interesting for product industrialization, as a way to overcome problems related to production seasonality and commercialization in low production regions. Indeed, it is possible to store powdered milk in areas and periods of high production, in order to fulfill the demand where and when dromedary milk is not produced enough. 

The drying step determines the quality of resulting powder; the choice of drying method and conditions is dictated by the nature of the material to be dried, the desired powder functionalities, and economic factors (Jinapong, Suphantharika, & Jamnong, 2008; Esquivel & Jiménez, 2012). Spray-drying is the most commonly used technique for industrial production of milk powder, because it generates a product of good quality with better organoleptic and nutritional features than drum-drying, while being faster and cheaper than freeze-drying (Ceballos, Giraldo, & Orrego, 2012; Reddy et al., 2014).

To the best of our knowledge, only a few studies in the literature have been dedicated to spray-dried dromedary milk powder and they were restricted to the determination of the influence of the drying process on the nutritional, physicochemical, and organoleptic properties of powders, as well as their storage stability. For instance, Sulieman, Elamin, Elkhalifa, and Laleve (2014) reported that the physicochemical and functional properties of spray-dried dromedary milk powder (water activity, powder color, solubility, flowability) were greatly affected by the velocity difference (both in terms of intensity and direction) between feed and air and they described thermal properties of freeze-dried whole and skim dromedary milks on 

the basis of glass transition and melting temperatures of their major components (lipids, caseins, whey proteins, and lactose). Al-Juboori, Mohammed, Rashid, Kurian, and El Refaev (2013) revealed the dependence of physicochemical properties (water activity, glass transition temperature, bulk density, free fat content, and whey protein nitrogen index) of spray-dried dromedary milk powders on fat content of feeding concentrate and spray-drying temperature. Zouari et al. (2019) investigated the effect of spray-drying parameters on the production of dromedary milk powder (water activity, glass transition temperature, bulk density, and free fat content). Ho et al. (2019) evaluated the stability of spray-dried dromedary milk powder and denoted modifications of several physicochemical properties upon accelerated aging. To the authors' knowledge, there has been no study on the effect of spray-drying conditions on flow properties of dromedary skim milk powder. Good powder flow properties enable high production efficiency as well as improved powder product quality (Fitzpatrick et al., 2007; Krantz, Zhang, & Zhu, 2009). Indeed, in manufacturing processes involving powders as ingredients or products, many unit operations, such as transfer from a silo or hopper, fluidization, pneumatic transportation, and mixing, require good flow properties of handled powders. Insufficient powder flow may be detrimental for the monitoring of some unit operations, resulting in poor quality of the end product (for instance, cohesive powders generally are more susceptible to agglomerate, resulting in particle sizes higher than specifications) and/or modifications of process conditions (for example, cohesive powders are more hardly fluidized, thus necessitating the use of higher air velocities for fluidization purposes, leading to more intense interparticular and particle-wall shocks that are likely to break particles and affect their size and shape). Moreover, good flow is required in powder mixing processes to ensure even distribution: it is more difficult to form a homogeneous mixture with cohesive powders or agglomerates than with powders composed of non-cohesive individual particles (Krantz, Zhang, & Zhu, 2009). The characterization of powder flowability can be 

achieved with the FT4 powder rheometer (Freeman Technology). Flow properties of various
food products have been determined using the FT4 powder rheometer: for instance, powdered
dairy products (Lapčík et al., 2015), fermented milk-cereal mixtures (Salameh et al., 2016), and
plantain-based formulated powders (Gnagne, Petit, Gaiani, Scher, & Amani, 2017).

Two sets of spray-drying process conditions were applied on skim dromedary and cow's milks in order to evaluate the impact of drying air temperature and milk type on the flow properties of resulting milk powders. The proximal composition and the particle size distribution were also evaluated in order to better understand the links between physicochemical properties and flowability of studied powders.

### 93 2. MATERIAL AND METHODS

#### 94 2.1. Dromedary and cow's milk samples

Dromedary (Camelus dromedarius) milk was obtained from a local breeding located in the south of Tunisia (Gabès governorate, Tunisia). Dromedary milk was constituted of pooled milk samples assembled from twenty different healthy dromedaries (Camelus dromedarius), whose lactation time ranged between 2 and 8 months. Cow's milk, collected from healthy Holstein cows, was provided by a farm located in the north-east of France (experimental domain of La Bouzule, ENSAIA, Laneuvelotte, France). Both milks were collected in sterile milking cans, in which sodium azide was promptly added up to 0.02 % (w/w) to ensure microbiological stability; after that, they were conveyed into a cool box filled with ice to the laboratory and then stored at 4 °C until skimming. Both milks were skimmed at 3 000  $\times$  g during 20 min at 4 °C (Gyrozen 1580MGR, Multi-purpose Centrifuge, Daejeon, Korea). Then, both skim milks were stored at -20 °C until use for spray-drying. 

#### 106 2.2. Spray-drying of skim dromedary and cow's milks

Skim dromedary and cow's milks were sieved at 50 μm mesh size (test sieve, Mesh SSteel, 200 mm diameter, 50 mm height, Retsch GmbH, Germany) in order to remove the

potential residual coalesced fat globules. Spray-drying of skim milks was performed in a MicraSpray MS 150 simple effect pilot plant spray-dryer (Anhydro, Soeborg, Denmark) equipped with a bi-fluid nozzle (Fluid Cap 60 100 + Air Cap 120, Spraying System, Wheaton, Illinois, USA). Aspiration rate was set at 70 % for all performed assays. In the drying chamber, the drying air cocurrently flowed with milk droplets sprayed from the bi-fluid nozzle. The spray-dryer was fed at 27 °C with a FAST Load peristaltic pump (VWR, Leuven, Belgium, 48 mm internal pipe diameter). Dromedary and cow's milks were first skimmed, then stored at -20 °C. For spray-drying experiments, both skim milks were thawed out at 4 °C overnight. Last, both milks were prewarmed at 27 °C in a water bath to improve spray-drying production efficiency (mainly, pumping of the concentrate through the bi-fluid nozzle). 

The spray-drying conditions such as air inlet and outlet temperatures as well as drying air flow rate suitable for the production of skim milk powder were selected not only based on literature (Schuck, Jeantet, & Dolivet, 2012) and but also after preliminary experimentations in order to obtain skim milk powders suitable for storage, i.e. having a moisture content not exceeding 5 % (w/w) (Schuck, Jeantet, & Dolivet, 2012; Felfoul et al., 2020). Two assays differing in spray-drying conditions were carried out with a view to evaluate the influence of air outlet temperature (Table 1). It is worth noting that C1 and C2 spray-drying conditions differed in outlet air temperature (circa 74.5 and 84.5 °C, respectively) and feed flow-rate (about 75 and 67 mL.min<sup>-1</sup>, respectively). 

With this experimental setup, the process parameter having the most influence on powder physicochemical and functional properties should be the air outlet temperature rather than the feed flow-rate. The feed flow rate was a little higher in C1 conditions (Table 1), meaning that liquid velocity at the nozzle outlet was a little higher and resulting in slightly lower air–liquid velocity difference at the nozzle outlet; this should result in slightly larger droplets (Petit et al., 2015; Mandato et al., 2012) and thus slightly larger spray-dried particles in C1 conditions. However, the effect of feed flow-rate was likely negligible compared to the
one of drying air temperature, because the air–liquid velocity difference at the nozzle outlet was
not markedly affected by changing the feed flow-rate, as the air velocity is much greater than
the liquid velocity at the outlet of a bi-fluid nozzle (Petit et al., 2015; Mandato et al., 2012).

The powders were then collected, immediately packed in sealed polyethylene terephthalate bags, and stored at 10 °C until analysis. All analyses were carried out within a week after powder production. Spray-drying yield was calculated as the ratio between the dry mass of powder and the dry mass of feed skim milk.

## 2.3. Proximate composition of skim dromedary and cow's milks and powders

Moisture, protein, and ash contents of skim milks and powders were determined according to the AOAC standard methods (A.O.A.C., 2016). Moisture content was determined by mass loss after drying 10 mL milk or 3 g powder in an air oven at 105 °C for 7 h. Ash content was obtained by mass loss after incinerating 10 mL milk sample or 3 g powder in a furnace at 550 °C during 6 h. Proteic nitrogen content was determined by the Kjeldahl method (Vapodest, Gerhardt GmbH & Co. KG, Königswinter, Germany) and protein content was deduced from nitrogen content using a conversion factor of 6.38 (Felfoul et al. 2020).

Fat content of milks (using 11 mL milk sample) was determined according to the Gerber
acid-butyrometric method (ISO 11870:2009) using 10 mL of sulfuric acid in order to dissolve
milk proteins. The fat separation was carried out by centrifugation in a butyrometer at 1100
tr/min for 5 min at 65 °C in the presence of 1 mL of isoamyl alcohol. Fat content of powders
was determined using the Folch method (1957) by performing solvent extraction of 2 g powder
using a mix of 2:1 (v/v) chloroform/methanol.

156 Carbohydrates content was deduced by difference with other components assuming that157 vitamins were weakly represented (Felfoul et al., 2020).

## **2.4. Particle size distribution**

Particle size distributions of powders were determined with a laser granulometer (Mastersizer 2000 Malvern Instruments, UK), equipped with He-Ne laser (632.8 nm wavelength), using the Aero S module for dry dispersion in the following conditions: 2 bar; 100 % air pressure; 100 % feed rate; 3 mm hopper gap. The mean diameter in volume was chosen as particle size estimator. Classical granulometric parameters D<sub>10</sub>, D<sub>50</sub>, and D<sub>90</sub> were recorded (D<sub>X</sub> corresponds to the size for which X % of the particle population has smaller size). Span was calculated to evaluate the width of the particle size distribution (equation 1) (Felfoul et al., 2020): 

 $Span = (D_{90} - D_{10})/D_{50}$  Equation (1)

## 2.5. Flow properties of skim dromedary and cow's milk powders

Powder flowability was evaluated using the FT4 powder rheometer (Freeman Technology, Worcestershire, UK) as previously reported in the literature (Freeman & Fu, 2008; Freeman, 2011). Stability, compressibility, and rotational shear tests were performed according to the FT4 standard methods. For each test, a powder sample was poured into a FT4 cylindrical vessel of 50 mm diameter. To ensure analytical reproducibility, FT4 analyses include a preliminary step of powder conditioning, performed by a rotational and vertical moving (both downwards and upwards) through the powder sample of a blade (5° helix angle and 48.0 mm diameter) at constant tip speed of 60 mm.s<sup>-1</sup>. This operation removes any packing history such as pre-consolidation or excess entrapped air and generates a uniform powder packing in order to ensure repeatable and comparable data (Freeman, Cooke, & Schneider, 2009). The mass of skim milk powders was automatically recorded by the FT4 apparatus. The different tests carried out with the FT4 powder rheometer are described hereafter. 

2.5.1. Stability test

The stability test is designed to assess the flow energy changes upon repeated testing, it consists in seven successive conditioning and test cycles. Stability test cycles were conducted by the rotating movement of the blade into and through a powder bed contained in the measurement cell (85 mL glass cylinder of 50 mm diameter) at 100 mm.s<sup>-1</sup> tip speed and 5° helix angle. During both downwards and upwards rotational movement of the blade, the torque (related to shear stress  $\tau$ ) and axial force (related to normal stress  $\sigma$ ) required to move the blade though the powder were recorded and used to calculate the total energy input required to make the powder bed flow. The basic flowability energy (BFE), corresponding to the stabilized flow energy, was evaluated as the flow energy required to displace a conditioned powder sample during downwards movement of the blade in the seventh test cycle (Freeman, 2007). The bulk density (BD) was also obtained from the stability test and was calculated as the ratio of sample mass to vessel volume (85 mL). 

The specific energy (SE) is the energy needed to displace the conditioned powder bed
during upwards testing divided by the mass of analyzed powder sample (Equation 2). This gives
an indication of powder flow properties in a loosely packed and unconfined state (Mitra et al.,
200 2017). SE is mostly related to intrinsic particle properties, such as cohesion, particle size and
shape distributions, and surface roughness.

SE = (upwards flow energy of cycle 6 + upwards flow energy of cycle 7)/(2 × split mass)203 (Equation 2)

**2.5.2.** Compressibility

The compressibility test evaluates the change in powder density according to applied normal stress. A powder sample was first placed in the measurement cell (i.e. two overlaid  $50 \text{ mm} \times 85 \text{ mL}$  glass cylinders) and submitted to three conditioning cycles performed as previously described. After that, the vessel was split to remove any excess powder and the dynamic blade was replaced by the 48 mm vented piston. Then, powder was slowly compressed

 by the vented piston vertically moving at 0.05 mm.s<sup>-1</sup> and applying normal stresses  $\sigma$  from 0.5 to 15 kPa (0.5, 1, 2, 4, 6, 8, 10, 12, and 15 kPa) while measuring its volume change. After reaching equilibrium at the target stress, the distance traveled by the vented piston was measured and the compressibility was calculated as a percentage change in volume after compression (C<sub>p</sub>). Compressibility is indicative of the level of cohesion between particles (Freeman, 2007; Bian, Ambrose, & Subramanyam, 2015). Compressed bulk density (CBD) is calculated from bulk density (BD) and compressibility (Equation 3).

217 
$$CBD = \frac{BD}{1 - \frac{C_p}{100}} \quad (Equation 3)$$

# **2.5.3. Shear cell test**

Shear cell measurements are widely used to evaluate the flowability of powders in highshear applications. The rotational shear cell of the FT4 powder rheometer consists of a vessel containing the powder samples and a FT4 shear head applying both normal and rotational shear stresses to the powder bed. Powder sample was first conditioned using the helical blade as previously described (Quintanilla, Castellanos, & Valverde, 2001). After the conditioning step, powder sample was slowly subjected for 60 s to a normal stress of 9 kPa carried out with a vented piston vertically moving at a maximum speed of  $0.08 \text{ mm.s}^{-1}$ . Then, the vessel containing the powder sample was split, the vented piston was changed for the shear cell head, and the powder sample was recompressed at 9 kPa to remove any disturbances caused by the split and ensure that the powder bed surface was properly consolidated. The preconsolidated powder sample was then submitted to a preshearing stage (60 s at 9 kPa normal stress) carried out with the rotational shear cell accessory (48 mm diameter), to achieve a critically consolidated state. The normal stress was then reduced lowered and the incipient shear stress necessary to cause powder bed failure and initiate flow was measured. The preshear/shear sequence was repeated five times at decreasing normal stresses  $\sigma$  from 7 to 3 kPa by 1 kPa steps 

at 18°.min<sup>-1</sup> rotational speed. The curve representing the evolution of incipient shear stress with
applied normal stress is thus the so-called yield locus (Gnagne, Petit, Gaiani, Scher, Amani,
2017). Shear cell parameters, major principal stress (MPS), unconfined yield stress (UYS),
cohesion, and flow function (FF), were then determined with the FT4 software by the Mohr
circle approach using following equations 4-6 (ASTM, 2004, 2005).

239 
$$MPS = \frac{(\sigma_x + \sigma_y)}{2} + \sqrt{\left(\frac{\sigma_x - \sigma_y}{2}\right)^2 + \tau_{xy}^2} \quad (Equation 4)$$

240 Where:

 $\tau_{xy}$ : incipient shear stress (Pa),  $\sigma_x$ : stress at horizontal axis (Pa),  $\sigma_y$ : stress at vertical axis (Pa) 242  $UYS = \frac{P}{A}$  (Equation 5)

244 
$$A = \frac{A_0}{1-\varepsilon} = \text{corrected area (m2)}$$

245 With:  $A_0$  is the initial horizontal area of the powder bed (m<sup>2</sup>) and  $\varepsilon$  designates the axial strain 246 of powder sample (s<sup>-1</sup>).

The cohesion was evaluated as the intercept of the linear regression of the yield locus curve. The flow factor (FF) is the most commonly used parameter for the description of powder flowability (Bian, Ambrose, & Subramanyam, 2015). (Equation 6):

$$FF = \frac{MPS}{UYS} (Equation 6)$$

251 The higher the flow factor, the better the powder flows. Powders were classified by Jenike252 (1980) according to FF values as follows:

- FF < 1: not flowing,

- 254 1 < FF < 2: very cohesive,
- 255 2 < FF < 4: cohesive,
- 256 4 < FF < 10: easy-flowing,

10 < FF: free-flowing.

#### **2.6. Statistical analysis**

259 All analyses were carried out in triplicates and reported values were means  $\pm$  standard 260 deviations. Statistical analysis was performed using the DSAASTAT add-on for Excel 2010 261 (Microsoft, Redmond, USA). The presence of significant differences between sample results 262 was investigated by one-way ANOVA and the means were separated by Tukey's HSD test at p 263  $\leq$  0.05. Pearson's correlation coefficients (r) were calculated to evaluate the degree of 264 correlation between investigated powder physicochemical and functional properties.

Proximate composition of dromedary and cow's skim milks and milk

#### 265 3. RESULTS AND DISCUSSION

# powders

3.1.

The proximate composition of spray-dried powders is displayed in Table 2. The total solids, as well as fat, ash, and carbohydrate contents were significantly different between produced powders. Moisture contents ranged between 2.98 and 7.04 % (w/w) on wet basis for skim dromedary milk powders and between 3.37 and 5.56 % (w/w) on wet basis for skim cow's milk powders. Generally, moisture content lower than 10 % (w/w) ensures powder stability during storage (Kaur, Kaushal, & Sandhu, 2011). When increasing the air outlet temperature during spray-drying of both milk powders, the moisture content significantly decreased whatever the milk type (cf. Table 2). This could be explained by the higher rate of evaporated water and the lower feed rate in C2 conditions (85 °C air outlet temperature and 65 mL.min<sup>-1</sup> feed flow-rate) compared to C1 conditions (75 °C air outlet temperature and 75 mL.min<sup>-1</sup> feed flow-rate). The protein content of the dromedary and cow's milk powders did not exhibit any significant difference. As the outlet air temperature was increased from 75 to 85 °C, the carbohydrates content increased from 53.74 to 55.66 % (w/w) on dry basis for skim dromedary milk powder, while it decreased from 51.31 to 50.71 % (w/w) on dry basis for skim cow's milk 

powder. Also, upon air outlet temperature increase, the ash content decreased from 10.74 to 8.90 % (w/w) on dry basis for dromedary milk powders and from 6.63 to 6.41 % (w/w) on dry basis for cow's milk powders. This reduction of ash content could result from calcium phosphate precipitation during the manufacture of skim milk powder (Lin, Kelly, O'Mahony, & Guinee, 2018). Fat contents ranged between 12 and 16 % (w/w) on dry basis for skim dromedary and cow's milk powders, respectively. These rather high fat contents (> 2% on dry basis) suggested that studied powders should be considered as partially skim milk powders. These results could be justified by the fact that the conditions used for milk skimming by centrifugation in this study were soft compared to what is commonly realized in the literature using a cream separator (Heymann, 2016). Spray-drying conditions did not have a significant impact on the proximate composition of studied milk powders. As it can be seen from Table 2, the proximate compositions of skim milk powders were comparable to those of their corresponding milks. Table 2 shows the water activity values of partially skim dromedary and cow's spray-

dried milks. Water activity was significantly influenced by spray-drying conditions whatever the milk type: it was lower when milks were spray-dried in C2 conditions at 85 °C air outlet temperature (0.203 and 0.274 for partially skim dromedary and cow's milk powders, respectively) than in C1 conditions at 75 °C air outlet temperature (0.391 and 0.385 for partially skim dromedary and cow's milk powders, respectively). These results agreed with Zouari et al. (2019) who reported that water activity of skim dromedary milk powder was lower when spray-dried at 200 °C inlet air temperature compared to 160 and 180 °C air inlet temperatures. Otherwise, Sulieman, Elamin, Elkhalifa, and Laleye (2014) showed that the concentration, temperature, direction of the feed, and milk type did not affect water activity of dromedary milk powders spray-dried at 200 – 220 °C air inlet temperature and 98 - 105 °C air outlet temperature. but it gave different results for cow's milk powders spray-dried in the same conditions. Indeed, 

306 the ideal temperature range for spray-drying cow's milk are 175 - 240 °C for air inlet
307 temperature and 70 - 90 °C for air outlet temperature (Schuck, Jeantet, Dolivet, 2012).

## **3.2.** Particle size distribution of dromedary and cow's milk powders

The granulometric characteristics of powders are shown in Table 3. All powders exhibited monomodal particle size distributions centered around 14 - 20 µm (Figure 1), consistently with previous results obtained with the same combination of pilot spray-dryer and bi-fluid nozzle (Guérin et al., 2017; Khanji et al., 2018). It should be mentioned that a significant effect of both milk type and spray-drying conditions was observed on the particle size distribution of spray-dried milk powders. The median particle sizes  $(D_{50})$  as well as the particle size distributions of spray-dried milk powders significantly differed according to spray-drying conditions and milk type. As for the milk type, significantly higher median particle sizes  $(D_{50})$ were obtained for partially skim cow's milk powders whatever the spray-drying conditions. This result could be explained by the difference in fat content between milk samples (Table 2): indeed, partially skim cow's milk being richer in lipids, obtained powder was expected to be stickier and then have a higher tendency to form agglomerates, leading to increased median particle size. This hypothesis was confirmed by the significantly higher fines proportion of partially skim cow's milk powders whatever the spray-drying conditions. As regards the influence of spray-drying conditions, when increasing the air outlet temperature, median particle size of partially skim dromedary and cow's milk powder was significantly higher but no significant effect was observed for partially skim dromedary milk powders (Table 3). The results obtained for skim cow's milk powders could be attributed to the fact that a higher drying temperature and a lower feed-rate accelerates the drying rate of droplets, promoting the fast formation of a crust at their surface and therefore limit shrinkage, which leads to larger particles (Kim, Chen, & Pearce, 2002). The fact that the particle size distribution of skim dromedary milk was unaffected by spray-drying conditions may be an indication that crust formation did 

not occur upon drying of sprayed droplets, presumably owing to the protein content and casein
proportions differing from cow's milk (Felfoul et al., 2017 ; Lajnaf et al., 2017). Moreover,
these results confirm that feed flow-rate may have a minor influence on particle size distribution
of spray-dried skim milk powders, as particle size distributions obtained in all spray-drying
conditions remained close.

The span was rather close for all spray-dried milk powders, which showed that milk type or spray-drying conditions did not have a great influence on the range of obtained particle sizes. However, there was no marked effect of either the milk type or the spray-drying conditions on the span of spray-dried milk powders.

## **3.3. Basic flowability energy, bulk density, and specific energy**

Mean BFE values obtained from the stability test are presented in Table 4. BFE is known to be a key parameter that expresses powder flowability in low-stress environments (Freeman, 2007). It is generally observed that powders exhibiting low BFE values have good flow properties and inversely, high BFE is often obtained for powders that poorly flow. The BFE of partially skim dromedary and cow's milk powders ranged from 620 to 1018 mJ. The air outlet temperature during spray-drying as well as the milk type exhibited noticeable effects on BFE values. On one hand, partially skim dromedary milk powders had significantly lower BFE values than partially skim cow's milk powders, denoting a better flowability of the former. On the other hand, BFE of partially skim cow's milk powders was about 900 mJ whatever the spray-drying conditions, whereas BFE of partially skim dromedary milk powders was well higher in C2 conditions (1 018 mJ) than in C1 conditions (620 mJ).

Besides, milk type had significant effects on the BD values of spray-dried milk powders, contrarily to spray-drying temperature that showed no significant impact on BD. Whatever the spray-drying conditions; partially skim dromedary milk powders had significantly higher BD values than partially skim cow's milk powders, i.e. 0.31 *vs.* 0.24 g.mL<sup>-1</sup>, respectively (Table 4).

These differences could be attributed to milk powder proximate composition, particle size or shape (Jan, Ghoroi, & Saxena, 2017). Indeed, higher contents in sticky material, mainly lipids, were observed for partially skim cow's milk powders. The presence of surface lipids is expected to make particles stickier, thus increasing powder cohesion and decreasing bulk density. Overall, BFE and BD of spray-dried milk powders, and thus flowability in low stress conditions, was essentially influenced by particle size along with fat content.

Specific energy indicates how easily powders flow in an unconfined environment and it gives a good indication of the level of powder cohesion (Freeman, Cooke, & Schneider, 2009). SE of partially skim dromedary and cow's milk powders ranged from 8.49 to 15.59 mJ/g, which corresponds to moderate-to-high cohesion (Bharadwaj, Ketterhagen, & Hancock, 2010). SE values were significantly affected by spray-drying conditions and milk type, as shown in Table 4. As for milk type, significantly higher SE values, indicating a poorer flowability, were obtained for partially skim cow's milk powders whatever the spray-drying conditions. As regards the influence of spray-drying conditions, SE of partially skim dromedary's milk powders significantly increased in C2 conditions, indicating poorer flow properties. On the contrary, spray-drying conditions had no significant effect on the SE of partially skim cow's milk powders, indicating that they did not affect the flowability of cow's milk powder. The lower SE of partially skim dromedary's milk powder spray-dried in C1 conditions (8.49 mJ/g) could be explained by differences in surface composition (Felfoul et al., 2020). Surface composition of powder particles plays an important role in its flow behavior because flowability involves overcoming surface interactions between powder particles (Fitzpatrick, Barringer, & Iqbal, 2004). Kim, Chen & Pearce (2002) reported that a higher surface fat content might explain the lower flow properties of industrial spray-dried milk powders. Thereby, studied partially skim cow's milk powders, having significantly higher fat contents than partially skim dromedary milk powders, had poorer flow powders whatever the spray-drying conditions. 

Based results obtained from the stability test (BD, BFE, and SE), the flow behavior of studied milk powders could be sorted as follows, from poorer to better flowability: Cow\_C1  $\approx$ Cow C2 < Dromedary C2 < Dromedary C1.

**3.4 Compressibility** 

Compressibility logically increased with applied normal stress for all investigated powders (cf. Figure 2). This behavior is expected to be due to the closer particle packing and the increase in interparticle surface contact at increased applied normal stress (Bian, Ambrose, & Subramanyam, 2015; Jan, Ghoroi, & Saxena, 2017). Compressibilities at 15 kPa ranged between 34.24 and 49.12 %, showing that studied powders were highly compressible, which was in line with the poor flowing properties of spray-dried powders that has already been evidenced with BFE and SE values. The outlet air temperature during spray-drying as well as the The milk type had noticeable effects on powder compressibility at 15 kPa, as displayed by Table 4. In addition, the The higher the air outlet temperature applied during spray-drying of dromedary milk, the higher the compressibility at 15 kPa normal stress. On the contrary, for For partially skim cow's milk powders, no significant difference of compressibility and CBD could be attributed to spray-drying conditions. Moreover, partially skim dromedary milk powders were less compressible than partially skim cow's milk powder whatever the spray-drying conditions; this could be related to the better flow properties of partially skim dromedary milk powder and to the higher fat content of partially skim cow's milk powder (Table 2). Compressibility of studied milk powders increased in the following order: Dromedary C1 <Dromedary  $C2 < Cow C1 \approx Cow C2$ , confirming the classification established on the basis of BFE and SE values. Compressibility at 15 kPa was significantly positively correlated to BFE and SE (Pearson's correlation coefficients r equaled 0.74 and 0.99, respectively, cf. Table 5). Compressibility at 15 kPa was also positively correlated to fat content (Pearson's 

405 correlation coefficient r of 0.80, cf. Table 5). Then, the higher the fat content, the more

compressible the studied milk powder. Fat could act as lubricating agent at the surface of large particle but not at the surface of small particles as reducing particle size increases the contact area between particles allowing greater interaction between particles and thus higher cohesive forces (Fitzpatrick, Barringer, & Igbal, 2004). In addition, fine particles exhibit higher compressibility because of their greater specific surface area and the fewer interparticular voids (Jan, Ghoroi, & Saxena, 2017). Gnagne, Petit, Gaiani, Scher, & Amani (2017) reported that powders with a higher fine particle content and irregular morphology are likely to be more susceptible to compression than more spherical, larger particles. These findings are in disagreement with the results obtained in the present study, where it was observed that cow's milk particles, of greater median size and having a higher fat content, were more compressible. In the case of the present study, the usual negative impact of particle size on powder compressibility is overcame by the high influence of fat content on powder cohesion, and thus on its sensitivity to compression. However, it is worth noting that compressibility was positively correlated with median particle size and BFE (r = 0.80 and 0.74, respectively, Table 5). It has been reported that particle size and milk powder composition, mainly fat content, were the key factors influencing powder flowability. This confirms that the particle shape as well as the milk powder composition, mainly fat content, are the main factors determining the flow behavior of studied milk powders.

#### **3.5.** Shear properties

The yield locus of partially skim dromedary and cow's milk powders are presented in Figure 3. Incipient shear stress logically increased with applied normal stress. Spray-drying conditions and milk type significantly influenced the shear flow properties of spray-dried powders (Table 4). Partially skim cow's milk powders were more cohesive (around 5 kPa cohesion) than partially skim dromedary milk powders (around 4 kPa cohesion) whatever the spray-drying conditions, indicating a poorer flowability of partially skim cow's milk powders,

in agreement with previously evoked flow properties and the higher fat content of partially skim cow's milk. No significant effect of the air outlet temperature was denoted on the cohesion and FF values of the investigated powders. Similarly, no significant effect of the outlet air temperature on the FF values was also observed, showing that both milk powders were extremely cohesive regardless of the applied spray-drying conditions. According to Jenike's classification all produced powders came under the category of very cohesive material and thus exhibited very poor flow properties. Shear and compressibility test results were in good agreement, i.e. the higher the powder cohesion, the lower the FF values, the more compressible the powder bed. Based on stability, compressibility, and shear tests, the flow behavior of all studied powders could be sorted as follows, from poorer to better flowability: Cow C1  $\approx$ Cow C2 < Dromedary C2 < Dromedary C1. Possible reasons for this classification could be the differences in milk composition (mainly fat content), particle size, particle interlocking, packing of smaller particles in void spaces, and surface irregularity, enabling higher cohesive interactions (Fitzpatrick, Barringer, & Iqbal, 2004; Siliveru, Kwek, Lau, & Ambrose, 2016; Siliveru, Jange, Kwek, Ambrose, 2017; Jan, Ghoroi, & Saxena, 2017). In the case of the present study, fat content could justify observed differences in flowability, as it was higher for partially skim cow's milk powder than for partially skim dromedary's milk powder (about 16 vs. 12 % (w/w) on dry basis, respectively, cf. Table 2). Significant correlations of cohesion and FF to fat content (Pearson's correlation coefficients r of 0.99 and 0.97, respectively, cf. Table 5) were obtained, indicating that the presence of fat was surely the main reason of flowability impairment of studied powders. Significantly positive correlations of cohesion and FF to particle size (Pearson's correlation coefficients r of 0.88 and 0.77, respectively, cf. Table 5) were also evidenced. This suggests that particle morphology (more specifically particle size distribution) significantly impacted flowability of studied powders, consistently with Babu, Siliveru, Amamcharla, Vadlani, and Ambrose (2018). Teunou, Fitzpatrick, and Synnott (1999) 

456 also studied the flowability of milk powders differing in particle size and composition (skim 457 milk and whey powders), concluding that the particle size significantly affects flowability: 458 smaller particles possess more contact points with the neighboring particles, thus making 459 rearrangements of the powder bed more difficult and impairing its flowability. The poor 460 flowability of milk powders was also probably due to their irregular surface particles, leading 461 to increased interparticular interactions and particle interlocking (Hart, 2015). Similar 462 observations were reported for different pharmaceutical powders by Lindberg et al. (2004).

#### 463 4. CONCLUSION

The spray-drying process has a significant impact on milk powder flow properties. In this study, partially skim dromedary and cow's milks were spray-dried in different conditions (of air outlet temperature and feed flow-rate) without prior concentration to avoid introducing another source of process variability. Partially skim dromedary and cow's milk powders were very cohesive. The spray-drying conditions, the milk type, the particle size distribution as well as the residual fat content showed a great influence on flow properties of partially skim dromedary and cow's milk powders. Using a higher air outlet temperature (85 °C) led to poorer flowability and higher compressibility whatever the milk type. Shear tests showed that partially skim cow's milk powders were more cohesive than partially skim dromedary milk powders. The shear flow properties of milk powders are expected to be influenced by fat content, particle size distribution, particle morphology, particle interlocking, packing of smaller particles in void spaces, and surface irregularity. Furthermore, in order to help industrializing dromedary milk powder production, further investigations should focus on biochemical and technological (gelation, coagulation, etc.) properties of dromedary milk powders. 

#### 478 CONFLICT OF INTEREST

479 The authors declare no conflict of interest.

#### 480 ACKNOWLEDGMENTS

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# 488 ETHICAL STATEMENT

489 This research study does not include animal and human testing.

#### 490 DATA AVAILABILITY STATEMENT

491 We declare that the data that support the findings of this study are openly available for all useful492 purposes.

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## 632 Figure captions

633 Fig. 1. Particle size distributions of skim dromedary and cow's milk powders spray-dried at 75
634 (C1) and 85 °C (C2) air outlet temperatures.

Fig. 2. Compressibility of partially skim dromedary and cow's milk powders. *Error barsrepresent standard deviations*.

Fig. 3. Evolution of shear stress with applied normal stress for partially skim dromedary andcow's milk powders. *Error bars correspond to standard deviations*.

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 Table 1. Spray-drying conditions used for the production of skim dromedary and cow's milk

 powders.

_	Skim milk	Powder sample	Spray-drying	Air inlet	Air outlet	Feed flow
	sample	acronym	conditions	temperature	temperature	rate
				(°C)	(°C)	(mL.min <sup>-1</sup> )
-	Dromedary	Dromedary_C1	C1	230.0 +/- 1.3	74.5 +/- 0.7	74.7 +/- 2.1
	Cow	Cow_C1	C1	230.4 +/- 1.6	74.4 +/- 0.9	76.5 +/- 2.2
	Dromedary	Dromedary_C2	C2	230.3 +/- 1.8	84.3 +/- 1.1	65.4 +/- 3.7
	Cow	Cow_C2	C2	230.6 +/- 1.3	84.8 +/- 0.8	69.5 +/- 2.9
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## Table 2. Proximate composition of dromedary and cow's skim milks and powders produced in

	SDM	SCM	Dromedary_C1	Dromedary_C2	Cow_C1	Cow_C2
Fotal solids (%(w/w)	$9.52 \pm 0.51^{x}$	$12.05 \pm 0.29^{x}$	$92.96 \pm 0.33^{\circ}$	$97.01 \pm 0.16^{a}$	$94.44 \pm 0.12^{b}$	$96.63 \pm 0.08^{a}$
on wet basis)						
Proteins (%(w/w) on	$32.88 \pm 1.97^{x}$	$29.46 \pm 2.70^{\mathrm{y}}$	$22.50\pm2.48^{\text{a}}$	$23.71\pm0.90^{\text{a}}$	$25.73\pm2.24^{\mathrm{a}}$	$27.00\pm2.38^{\mathrm{a}}$
lry basis)						
Fat (%(w/w) on dry	$7.35 \pm 0.09^{\mathrm{y}}$	$10.04 \pm 0.13^{\rm x}$	$12.20\pm0.47^{b}$	$12.33 \pm 1.89^{b}$	$16.35\pm1.00^{\text{a}}$	$15.89 \pm 0.68^{a}$
oasis)						
Ash (%(w/w) on dry	$8.93\pm0.04^{\rm x}$	$6.64 \pm 0.09^{y}$	$10.74\pm0.38^{\text{a}}$	$8.90\pm0.18^{\text{b}}$	$6.63 \pm 0.23^{\circ}$	$6.41 \pm 0.34^{\circ}$
pasis)						
Carbohydrates	$50.84 \pm 0.02^{\mathrm{y}}$	$53.86 \pm 0.05^{x}$	$53.74\pm2.07^{ab}$	$55.66 \pm 0.21^{a}$	$51.31 \pm 2.62^{b}$	$50.71 \pm 0.15^{b}$
%(w/w) on dry basis)						
Water activity (-)	-	-	$0.391 \pm 0.002^{a}$	$0.203\pm0.003^{d}$	$0.385\pm0.002^{b}$	$0.274 \pm 0.002$
For milks an	d powders separ	rately, means w	vith different supe	erscripted letters i	n the same row	were
significantly	different accordi	ng to Tukey's H	ISD test ( $p < 0.05$	).		
SDM <sup>.</sup> skim d	romedary milk.	SCM <sup>.</sup> skim cow	<i>i</i> 's milk			
	, , , , , , , , , , , , , , , , , , ,					

Page 45 of 47

	Dromedary_C1	Dromedary_C2	Cow_C1	Cow_C2
D <sub>10</sub> (μm)	$6.41 \pm 0.06^{d}$	$6.55 \pm 0.10^{\circ}$	$7.54 \pm 0.06^{b}$	$8.28 \pm 0.03^{a}$
D <sub>50</sub> (µm)	$14.83\pm0.08^{\circ}$	$14.84 \pm 0.21^{\circ}$	$17.09\pm0.18^{\rm b}$	$19.19\pm0.10^{a}$
D <sub>90</sub> (µm)	$35.09\pm0.36^{\circ}$	$32.68\pm0.34^{d}$	$37.43\pm0.50^{b}$	$43.04\pm0.33^a$
Span (-)	$1.93 \pm 0.02^{a}$	$1.76 \pm 0.01^{\circ}$	$1.75\pm0.01^{\circ}$	$1.81 \pm 0.01^{b}$

Table 3. Granulometric characteristics of skim dromedary and cow's milk powders.

Means with different superscripted letters in the same row were significantly different according to

Tukey's HSD test (p < 0.05).

**Table 4.** Flow properties issued from stability, compressibility, and shear cell tests of skim dromedary and cow's milk powders deduced from the FT4 stability test.

	Dromedary_C1	Dromedary_C2	Cow_C1	Cow_C2
BFE (mJ)	$620\pm27^{b}$	$1\ 018\pm 65^{a}$	$917\pm81^{a}$	$907 \pm 68^{a}$
SE (mJ.g <sup>-1</sup> )	$8.49 \pm 0.46^{\circ}$	$12.73 \pm 0.12^{b}$	$15.52 \pm 0.14^{a}$	$15.59 \pm 0.30^{a}$
BD (g.mL <sup>-1</sup> )	$0.31 \pm 0.00^{a}$	$0.31\pm0.00^{\text{b}}$	$0.24\pm0.00^{d}$	$0.25\pm0.00^{\rm c}$
CBD at 15 kPa	$0.47 \pm 0.00^{b}$	$0.53\pm0.00^{a}$	$0.48\pm0.01^{\rm b}$	$0.49\pm0.01^{\text{b}}$
(g.ml <sup>-</sup> ) Compressibility at	$34.2 \pm 1.1^{\circ}$	$42.6 \pm 0.7^{b}$	$48.8 \pm 1.2^{a}$	$49.1 \pm 1.2^{a}$
15 kPa (%)				
Cohesion (kPa)	$4.07\pm0.13^{b}$	$4.01\pm0.35^{\rm b}$	$5.16\pm0.05^{a}$	$5.06\pm0.36^a$
FF (-)	$1.21\pm0.04^{a}$	$1.24 \pm 0.11^{a}$	$1.02\pm0.02^{b}$	$1.09\pm0.06^{ab}$

Means with different superscripted letters in the same row were significantly different according to

Tukey's HSD test (p < 0.05).

	Compressibility	Cohesion	FF (-)	BFE (mJ)	SE (mJ.g <sup>-1</sup> )	Fat content	D <sub>50</sub> (μm)
	at 15 kPa (%)	(kPa)				(%(w/w) on	
						dry basis)	
Compressibility at 15		0.95	0.77	0.74	0.00	0.90	0.90
kPa (%)	-	0.85	-0.//	0.74	0.99	0.80	0.80
Cohesion (kPa)	0.85	-	-0.97	0.28	0.83	0.99	0.88
<b>FF</b> (-)	-0.77	-0.97	-	-0.19	-0.75	-0.97	-0.77
BFE (mJ)	0.74	0.28	-0.19	-	0.76	0.34	0.28
SE (mJ.g <sup>-1</sup> )	0.99	0.83	-0.75	0.76	-	0.86	0.78
Fat content (%(w/w)	0.00		0.05	0.04	0.07		0.05
on dry basis)	0.88	0.99	-0.97	0.34	0.86	-	0.87
<b>D</b> <sub>50</sub> (μm)	0.80	0.88	-0.76	0.28	0.78	0.87	-