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Characterization of the superconcentration and granulation steps of a disruptive spray-drying free process for the manufacture of dairy powders

Maheshchandra H. Patil [a,b], Eoin G. Murphy [b], Gaëlle Tanguy [a], Cécile Le Floch-Fouéré [a], Romain Jeantet [a,*]

[a] STLO, INRAE, Institut Agro, 35042, RENNES, France
[b] Food Chemistry and Technology Department, Teagasc Food Research Centre, Moorepark, Fermoy, County Cork, Ireland

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ABSTRACT

Breakthrough energy savings were recently projected by a spray-dryer free dairy powder manufacturing process based on superconcentration and granulation. The extent of superconcentration and the recirculation rate of powder for granulation, determines process performance. In this study, the evolution of physical properties during superconcentration and granulation was investigated for a model system (skim milk microfiltrate) using rheological and shear-cell based techniques. Results demonstrated that superconcentration leads to the development of a cohesive non-flowing state. The transition from concentrate to cohesive wet powder regime, manifested as a sharp increase in cohesiveness, occurring at around 82% w/w dry matter (DM). Powder addition for successful granulation was related to DM at the end of the cohesive phase (~89% w/w DM). Interestingly, laboratory-based rheological and shear cell measurements were well correlated with the amperage of the mixer used; similar measurements could be applied in-process providing a better understanding and control of the product behavior within the system.

1. Introduction

Dehydration of perishable liquid dairy streams into stable powder formats is a widely practiced means of increasing ambient shelf life by up to 3 years. Conventional dairy powder manufacturing practice includes production of a viscous concentrate (25–60% w/w dry matter (DM) content depending upon composition, especially protein content) using membrane concentration and/or falling film evaporation. Subsequently, the concentrate is transformed into powder using spray drying (SD), wherein atomization of fine droplets in a hot air stream results in rapid dehydration. SD accounts for almost 50% of total energy in dairy powder plants, while the specific energy for water removal is 10–20 times higher than falling film evaporators (Schuck et al., 2015). Furthermore, in some cases the high operational costs associated with SD can mean the process returns little or no profit when applied to low value streams such as permeate. Thus, replacing energy intensive SD with alternative, energy efficient operations can result in greatly improve the environmental footprint and profitability of dairy powder manufacturing plants (Patil et al., 2021a,b; Schuck et al., 2016; Tanguy et al., 2017).

Recent investigation of a superconcentration-granulation based process (*poudre sans tour [PST] process;* Fig. 1) estimated energy savings at more than 30% compared to SD during production of permeate powders at a pilot scale (100 kg h^{-1} feed rate). The improved performance was mainly attributed to superconcentration to 80% w/w DM (vs 60% w/w DM in conventional process) using high-shear, thin-film horizontal rotary evaporators (Tanguy et al., 2017). In another investigation, a similar agitated thin film drying technology (ATFD) was proposed for energy efficient production of dairy and food powders at a lab scale (~0.5 kg h^{-1} feed rate). However, evolution of a sticky rubbery mass significantly impaired agitation while drying products consisting of lower molecular weight sugars (Qiu et al., 2019). In contrast to the ATFD process, the PST process uses an innovative 'granulation' feature, which involves addition (recycling) of dry powder (>97% w/w DM) into superconcentrated paste (up to 80% w/w DM) in order to produce large discrete powder particles which are subsequently dried (Tanguy et al., 2017).

While both PST and ATFD processes differ in final powder production step, superconcentration in high shear conditions is a common feature for both technologies. Achieving maximum product

* Corresponding author.
E-mail address: romain.jeantet@agrocampus-ouest.fr (R. Jeantet).

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concentration in contact evaporators (falling film evaporators, thin film high shear evaporators) is a key to sustainable production of dairy and food powders (Patil et al., 2021a,b; Schuck et al., 2015, 2016; Walmsley et al., 2018). However, little is known about the nature and physical behavior of products during superconcentration, particularly, the evolution of rheological properties that restrict the superconcentration step beyond specific DM content. The end-point of superconcentration directly influences the process performance with respect to energy savings and productivity. For instance, reduction in permeate superconcentration from 80% w/w DM to 75% w/w DM, was estimated to increase the recirculation rate by a factor of two-fold for permeate powder (Tanguy et al., 2017). Additionally, granulation of wet pastes by addition of dry powders is a relatively new process which has not been studied for dairy systems.

The primary objective of the present investigation was to increase the understanding of dairy superconcentration and granulation processes through application of a variety of rheological (flow curves, flow stress - oscillation amplitude test, cohesion – probe-tack test) and wet mass characterization techniques. A variety of techniques were applied to gain insights into the complete range of DM contents that will be encountered during both superconcentration and granulation, as some techniques may not be practical to handle higher DM content superconcentrated pastes.

2. Materials and methods

2.1. Materials

Skim-milk microfiltration permeate (MFP) at approximately 6% w/w DM was obtained by membrane filtration (0.1 μm cut-off ceramic membrane, PALL, UK) of fresh skim milk at 50 °C in the Dairy Platform (STLO, Rennes, France). In order to obtain sufficient quantity of superconcentrate at the end of the concentration step in rotary evaporator, MFP was preconcentrated to approximately 30% w/w DM using a pilot scale falling film evaporator *(GEA Process Engineering, France)* at a feed flow rate of 70 kg h^{-1} and an evaporation temperature of 60 °C. Lastly, MFP concentrate was stored in a cold room (4 °C) for subsequent use. No solidification of the MFP concentrate occurred during storage. Only a small quantity (less than 2%) deposited in the bottom of the bottles. The pH of the MFP concentrate was checked before each experiment and was equal to 6.08 ± 0.02.

2.2. Preparation of superconcentrates

2.2.1. Concentration and crystallization

The simplified experimental set-up used to approximate the superconcentration-granulation process is described in Fig. 2. A lab scale

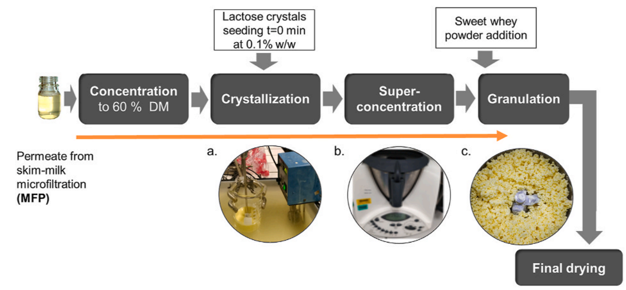

Fig. 2. Lab-scale experimental set-up to simulate the superconcentration-granulation process. a - crystallization set-up; b - temperature-controlled mixer for superconcentration; c - granulated product.

rotary evaporator *(Hei-VAP Value Digital, Heidolph Instruments, Schwabach, Germany)* was used to continue the MFP concentration from 30 to 60% w/w DM at 60 °C under ~100 mbar vacuum. Batch crystallization was then performed in a 1-L glass beaker (diameter - 11 cm), which was immersed in a temperature-controlled water bath at 30 °C and over which a paddle agitator (diameter - 4.5 cm) was installed for controlled stirring using an electric motor *(Eurostar digital, IKA-Werke, Germany)*. Around 600 ml of concentrated MFP was poured in the glass beaker and agitated at 600 ± 50 rpm. Lactose crystals (100 mesh; *Lactalis Ingredients, France)* were seeded at time t = 0 min at 0.1% w/w. The crystallization procedure was based on the previous studies of Gernigon et al. (2013) and Mimouni et al. (2005). A handheld refractometer *(Atago, Tokyo, Japan)* was used to measure the refractive index (expressed in°Brix) as a function of time and monitor the lactose crystallization. Measurements were made every 30 min during the first 6 h, and then at the end of crystallization (overnight around 16–18 h). Particle size of the crystals was measured using laser light diffraction *(Mastersizer, 2000; Malvern Instruments Ltd., Malvern, UK);* saturated lactose solution (29 g.100 g^{-1} water) was used as a dispersing medium to avoid solubilization of lactose crystals (Mimouni et al., 2005). Crystal morphology was observed under light microscopy *(model BX51*TF, *Olympus, Japan)*. Lactose crystallization kinetics were derived using the method and equations described by Mimouni et al. (2005).

2.2.2. Superconcentration

Crystallized MFP was further concentrated up to about 85% w/w DM in a temperature-controlled mixer *(Thermomix, TM5, Vorwerk, Germany)* under high shear conditions at 60 ± 2 °C. The mixer bowl had an internal diameter of ~15 cm and the 4-blade knife agitator had a diameter of 13.5 cm. High shear was achieved by operating at a high agitation speed (500 rpm). The temperature of 60 ± 2 °C is inline with temperatures used during concentration at industrial scale while the speed of

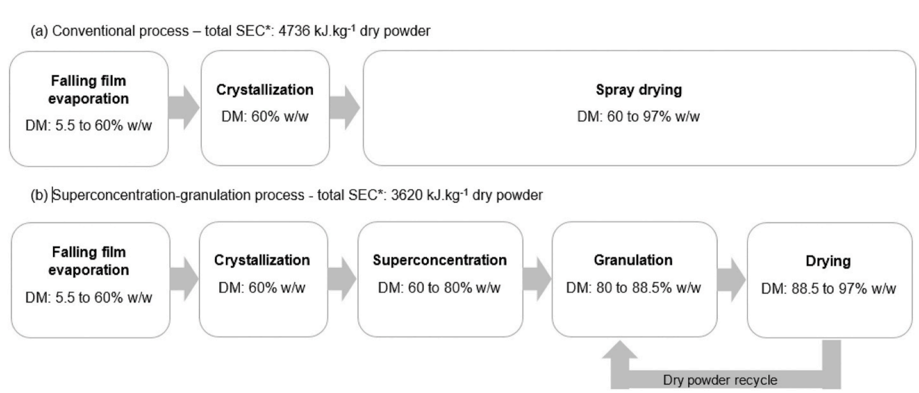

Fig. 1. Schematic representation for the manufacture of whey permeate powder (a) conventional spray drying and (b) novel superconcentration-granulation process for manufacture of whey permeate powder (WPP). DM, Dry matter; SEC, Specific energy consumption for dehydration (kJ.kg^{-1} dry powder); *Energy estimates from the study of Tanguy et al. (2017).

500 rpm was the highest speed that could be employed while avoiding product splashing. During concentration, samples were taken at regular intervals for further characterization. MFP samples with DM higher than 85% w/w, that could no longer be processed in mixer, were obtained by drying superconcentrate (~85% w/w) at 60 °C in a hot air oven for 30, 60 and 90 min. Drying time was varied to produce a range of samples with increasing DM content. The resulting MFP samples were used for characterization of wet mass flow properties using shear cell measurement as described in following section 2.3.4.

2.3. Characterization of superconcentrates

2.3.1. Dry matter content

The DM contents of superconcentrates were measured by oven method (105 °C, 7 h) according to Schuck et al. (2012). DM content was determined by weight loss using 3–5 g product samples carefully mixed with sand in a capsule. Measurements were performed in duplicates.

2.3.2. Rheological properties

Flow curves, flow stress and cohesion were used to characterize the rheological behavior during superconcentration. All measurements were performed in a stress-controlled rheometer *(DHR2, TA Instruments, France)* using a flat parallel plate (50 mm diameter) geometry with a constant gap of 1 mm.

For flow curves, shear rate ranged from 0.01 to 1000 s^{-1} to obtain 5 measuring points at a constant temperature (25 °C). Product discharge and slip were more pronounced at higher temperature and higher shear rates depending on the geometry and surface roughness (Sharma et al., 2015), therefore flow curves were characterized at 25 °C. Serrated (hatch-plate) plate (40 mm diameter) was used for higher DM samples to avoid the product slipping. Apparent viscosity at a shear rate of 100 s^{-1} was presented as a function of DM content. Oscillation amplitude sweep was performed by varying the stress from 0.1 to 1000 Pa logarithmically, at a constant frequency and temperature of 1 Hz and 60 °C respectively. Paraffin oil was used to avoid evaporation from sample while performing measurements. Stress corresponding to cross over point of the storage modulus (G′) and loss modulus (G″) curves was determined, which is described as flow point or flow stress (Mezger, 2011; Zhu et al., 2019), using the rheometer software *(TA Instruments Trios V5.0.0.44616)*. Cohesion was determined by measuring axial force required to separate the top plate from the bottom plate of the rheometer (similar to Probe Tack test described by Malvern Instruments limited, 2021). Sample was loaded on the bottom plate and the top plate was lowered to a gap of 1 mm after which excess product was carefully trimmed. Prior to analysis the sample was pre-sheared at 50 s^{-1} for 1 min followed by equilibration at rest for 1 min. During analysis, the top plate was lifted at a constant velocity of 1 mm s^{-1}. The axial force experienced by the top plate is recorded over time until complete separation of both plates. Cohesion is indicated by maximum force (N) required while lifting the top plate. Measurements were performed at 25 °C in duplicates. Higher temperature was avoided due to possibility of evaporation modifying product properties, especially at higher DM. Application of paraffin oil to avoid evaporation was not possible for this technique.

2.3.3. Specific power consumption during superconcentration

The electric current amperage (A), reflecting the electric power consumption of the temperature-controlled mixer, was used for gaining more information about the evolution of the product during superconcentration. Power consumption was measured at 500 rpm using an energy meter (*Energy Logger* 4000 FR*, Voltcraft, Germany*). In parallel, the mass of superconcentrate in the mixer was weighed before each sampling for rheology characterization. From both measurements, the specific power consumption, i.e. electrical power consumption per kg of product, was determined at all stages of superconcentration.

Additionally, a disruption (breakdown) in continuous processing of superconcentrated paste was simulated in order to understand the magnitude of potential changes in paste rheological properties arising from said disruption. This information could be crucial in designing the torque requirements of the equipment to handle breakdowns in operations at pilot-scale/industrial scale. Superconcentrated pastes were stored in a closed container under ambient conditions (25 °C, 40% RH) for 1 h. Flow curves, flow stress and power consumption measurements were again performed to evaluate changes in the superconcentrate behavior.

2.3.4. Flow properties of superconcentrated paste using a shear cell measurement

Flow properties of superconcentrated pastes were described by cohesiveness and flowability function (ffc) using shear cell tests. A ring-shear tester *(RST-XS, Dr. Dietmar Schulze Schüttgutmesstechnik, Wolfenbüttel, Germany)* was used to characterize flow properties of superconcentrated pastes at various DM contents at 25 °C. Superconcentrated paste was filled in the shear cell (24 cm^3) and excess material was scraped off. Shear test measured yield locus of a pre-consolidated bulk solid sample, by applying shear stress until failure. Cohesiveness and ffc were determined by analyzing the yield locus measurements using RST control software *(RSV 95)*. All yield locus measurements were performed at a normal pre-shear stress of 2000 Pa using four shear points (400, 1000, 1600 and 400 Pa). Subsequently, the cohesiveness and ffc values were derived. Detailed description of the ring-shear tester equipment and its operation can be found in recent works (Schulze, 2014; Tobin et al., 2017).

2.4. Granulation and characterization of granules

Granulation was carried out by fragmenting the superconcentrated paste (82% w/w DM) with addition of sweet whey powder at 97% w/w DM (*Lactalis Ingredients, France)* in the temperature-controlled mixer. The powder was added gradually in the superconcentrated paste at 60 °C under constant agitation of 500 rpm, according to Patil et al. (2021a,b). The end point of powder addition was determined by following the power consumption and the formation of visible granules in the mixer. The recirculation rate expresses the ratio between the mass of dry powder added and the mass of the superconcentrated paste. The DM content of granules was determined according to the same method described for superconcentrates.

3. Results and discussion

3.1. Characterization of MFP superconcentrates

3.1.1. Lactose crystallization

Controlled lactose crystallization of MFP concentrates at around 60% w/w DM was performed with initial seeding of 0.1% lactose. Close to 70% lactose was crystallized at the end of crystallization (~16 h), while more than 60% lactose was crystallized within first 4 h of crystallization (Fig. 3a). The typical tomahawk shape was observed for crystallized lactose with an average size around 60 µm and monomodal particle size distribution (Fig. 3b). The results with respect to the extent of lactose crystallization, the shape and size of crystals were in agreement with previous published studies (Mimouni et al., 2005; Simeão et al., 2017).

3.1.2. Rheological properties of superconcentrates

The flow behavior of the superconcentrates (DM>60% w/w) was analyzed using the rheometer software *(TA Instruments Trios V5.0.0.44616)*. The Herschell-Bulkley model well described MFP superconcentrate flow behavior ($R^2 = 0.99$). Table 1 presents the yield stress, consistency coefficient and flow behavior index for MFP superconcentrates (60%<DM<78% w/w). Modelling of flow curves could not be performed for DM higher than 78% w/w due to wall slippage and product discharge. Similar challenges, i.e. wall slippage at higher shear

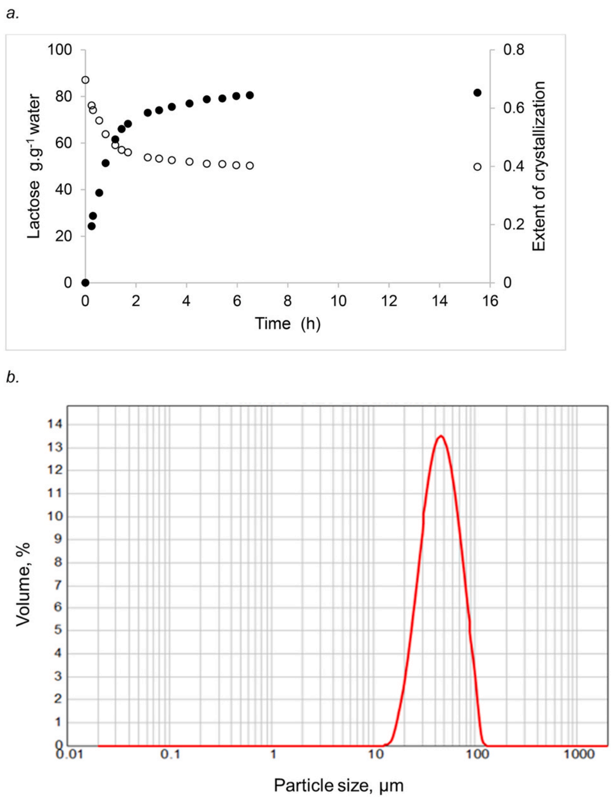

Fig. 3. a. Desupersaturation curve of MFP during crystallization at 60% w/w 30 °C. (Lactose g.g^{-1} water – open circles; Extent of crystallization – dark circles); b. Particle size of the lactose crystals measured using laser light diffraction.

Table 1
Flow behavior of skim milk microfiltrate (MFP) superconcentrates at different DM (% w/w) represented by Herschel-Bulkley model (R^2 = 0.99).[11] Measurements at 25 °C.

Dry matter % (w/w)	Yield stress σ_y (Pa)	Consistency coefficient K (Pa s^n)	Flow behavior index n (–)
61.1	13.1 ± 1.0	0.1 ± 0.01	1.0 ± 0.01
64.7	5.9 ± 0.4	1.5 ± 0.3	0.7 ± 0.03
70.0	10.1 ± 0.2	11.8 ± 0.6	0.6 ± 0.01
72.9	17.2 ± 10.0	12.1 ± 5.5	0.6 ± 0.08
77.8	128 ± 17.0	22.6 ± 11.0	0.6 ± 0.07

rates, were reported by Sharma et al. (2015) during characterisation of a different product (cheese) using flow curves. As DM content increased from 61,1–70% w/w DM, MFP superconcentrates showed shear thinning behavior at higher DM i.e. flow behavior index decreased from 1.0 at 61.1% to <0.7 at DM > 64.7% w/w. The apparent viscosity was strongly influenced by concentration (Fig. 4). Apparent viscosity as a function of DM was correlated by an exponential model (R^2 = 0.98). Additionally, the agitator power consumption, expressed in current (A), demonstrated a robust linear correlation (R^2 = 0.99) with apparent viscosity (Fig. 5). A similar trend (R^2 = 0.93) was observed between power consumption and viscosity during pumping of a dairy concentrate (Schuck et al., 2005). These results are consistent with previous studies on dairy products which reported increased shear-thinning behavior at increasing concentrations (Morison et al., 2013), exponential rise in apparent viscosity with increasing DM contents, and a substantial rise in viscosity corresponding to a small increase in DM at higher concentration levels (Snoeren et al., 1981; Vélez-Ruiz and Barbosa-Cánovas, 2000). The typical shear thinning behavior confirms the advantage of high shear processes (e.g. PST) to produce superconcentrated dairy

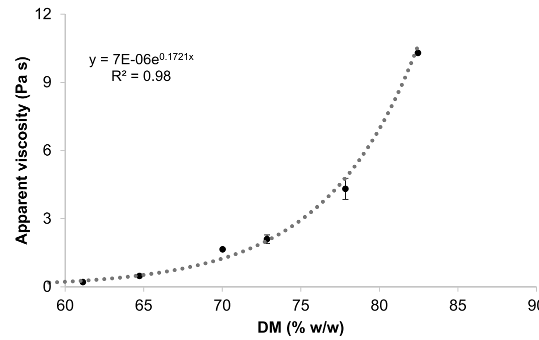

Fig. 4. Apparent viscosity of superconcentrates as a function of dry matter (DM). Measurement at 25 °C and 100 s^{-1}. Error bars represent standard deviation of measurements performed in duplicates.

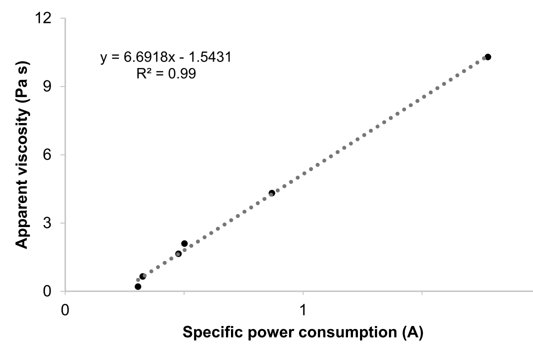

Fig. 5. Correlation of apparent viscosity of superconcentrates (Pa s) and specific power consumption of mixer expressed as current (A). Viscosity measurement made at 25 °C and 100 s^{-1}. Agitation speed of 500 rpm. Correlation is based on average values of apparent viscosity (n = 2).

streams, where viscosity increase is limited by the rigorous mechanical shear. This enables higher water removal in energy efficient contact evaporators, which is a challenge for conventional falling film evaporators (Patil et al., 2021a,b; Tanguy et al., 2017).

Oscillation amplitude sweeps were performed to determine the stress required to initiate flow in the superconcentrates. Across all the DM contents studied (i.e. DM > 60% w/w), G′ was higher than G″ at lower stresses. Concentration highly influenced the stress required to initiate flow, i.e. cross over point of G′ and G’’ (Fig. 6). This reflects another limitation while processing superconcentrates, namely higher resistance for startups after sudden breakdown during operations, which is substantially amplified at higher DM contents. A steep increase (4.5X) in stress to initiate flow was observed for a small change in DM content from 78.3% to 81.7% w/w DM. This dramatic increase in flow stress indicating marked evolution in rheological properties points to transformation of suspensions (fluid-like state) into capillary state (strong

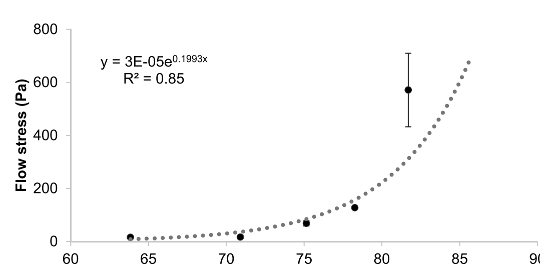

Fig. 6. Flow stress (Pa) using oscillation amplitude sweep as a function of dry matter of superconcentrates (% w/w). Measurement made at 60 °C and constant frequency of 1 Hz. Error bars represent standard deviation (n = 2).

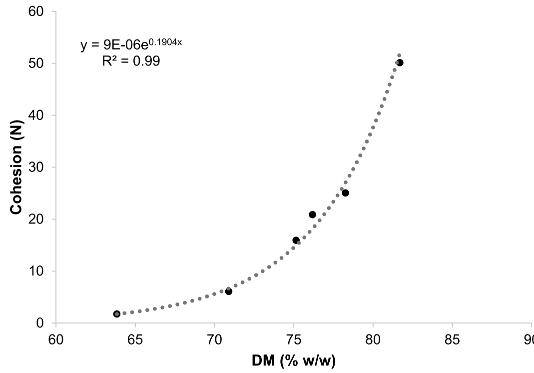

Fig. 7. Cohesion (in N) as a function of DM of superconcentrates (% w/w). Measurements at 25 °C. Standard deviation less than 3% (n = 2), error bars are within the symbols.

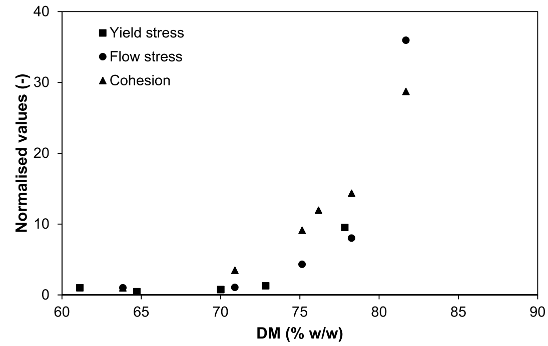

Fig. 8. Comparison of the three rheological techniques based on normalized results of yield stress (Herschell-Bulkley model), flow stress (oscillation amplitude sweep) and cohesion (probe track test) as a function of DM content of superconcentrates (% w/w). Values were normalized on Y-axis with respect to the value corresponding to DM on x-axis.

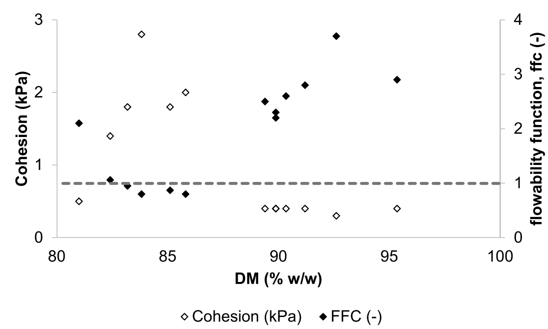

Fig. 9. Cohesiveness and flowability function as a function of dry matter (DM) of superconcentrates, dashed line represents threshold for non-flowing state (ffc <1).

gel-like state), as observed by Koos (2014).

Furthermore, the impact of increasing DM on cohesion during superconcentration was demonstrated using a rheometer (probe-tack test). A robust exponential correlation ($R^2 = 0.99$) was observed between cohesion and DM content. A two-fold increase in cohesion was noticed for a small increase in DM content from 78.3% to 81.7% w/w DM (Fig. 7).

Three different rheological techniques were applied in this study to understand the evolving product behavior during superconcentration. In order to compare the results obtained, normalized results of yield stress (Herschel-Bulkley model), flow stress (oscillation amplitude sweep) and cohesion (probe-tack test) are presented in Fig. 8. Values were normalized on Y-axis with respect to the value corresponding to DM on X-axis. Interestingly, a similar trend was observed for all the normalized values, particularly marked increase in flow stress and cohesion for a small change in DM closer to 81.7% w/w DM. PST process performance (energy savings and production capacity) can be optimized by achieving the highest possible superconcentrate DM content which does not exhibit excessive cohesion. Therefore, techniques which highlight onset of marked evolution in product properties (flow stress, cohesion) as a function of DM, can guide optimization of superconcentration process.

Furthermore, the effect of holding the superconcentrated paste under static conditions was evaluated. The product at around 78% DM and 60 °C was left unagitated for 60 min. Initial product behavior was like a smooth flowing paste with 0.32 A power consumption. At the end of 60 min, the product appeared like a very thick paste, with an immense resistance to initial flow, increases the amperage requirement from 0.32 to 0.85 A. However, within short time (1–2 min) of continuous shearing, the thick paste transformed into easy flowing paste resembling the consistency before stand-by, with slight increase in power consumption close to 0.40 A, which can also be due to lower temperature or slightly increased DM content. Substantial increase (up to 4X) in the yield stress and flow stress was observed due to aging. This information can be critical in designing the equipment, as an abrupt breakdown can lead to substantially higher torque requirements to re-initiate the production.

3.2. Shear cell measurements

The behavior of superconcentrated MFP pastes were typified by cohesiveness and ffc using a ring shear tester. Cohesiveness represents internal binding strength of the material, whereas ffc indicates if the material has a good or poor flow behavior. The ffc is a function of consolidation stress and unconfined yield stress. A non-flowing (hardened) state is attributed to ffc <1, whereas higher ffc value indicate greater flowability (Schulze, 2014). Flow properties of superconcentrated MFP pastes were highly dependent on DM (Fig. 9). As the DM content increased from 81% to 93% w/w DM, cohesiveness and flowability values evolved virtually in opposite direction, e.g. when cohesiveness increases, ffc decreases and vice versa. While such concentrations would not be possible in an industrial setup, due to extreme cohesivity, these lab-scale results provide useful insights, validating the limits of superconcentration determined by rheological methods as well as giving insight minimum rate of dry powder addition for granulation. Markedly lower cohesiveness was observed at 81% and 89% w/w DM, in contrast to 82%–86% w/w DM, wherein excessive cohesiveness (3X to 6X) was recorded. Similarly, within the DM range of 82%–86% w/w DM, ffc values were less than 1, while higher ffc values were noted for 81% and 89% w/w DM. Therefore, a highly cohesive non-flowing hardened state was located within 82%–86% w/w DM. The clear distinction with respect to the product flowability, indicated by ffc, marked the limiting DM for the superconcentration of MFP.

Inversion of ffc and cohesiveness values has previously been reported to represent the transition from cohesive wet powder regime to a concentrated suspension as DM content was progressively reduced (Althaus and Windhab, 2012; Tobin et al., 2017). In contrast this study progressively increased DM during superconcentration, however, a similar trend was observed at around 81% w/w DM, which can be indicative of a transition from concentrated suspension to cohesive wet powder regime. Moreover, while cohesiveness demonstrated more or less constant values within 89%<DM<96% w/w, an abrupt increase was observed on transition from 81% to 82% w/w. This may indicate a transition from an overwet state (i.e.>80% saturation corresponding to capillary state) as was observed by Althaus and Windhab (2012) albeit

[1] Standard deviation (n = 2).

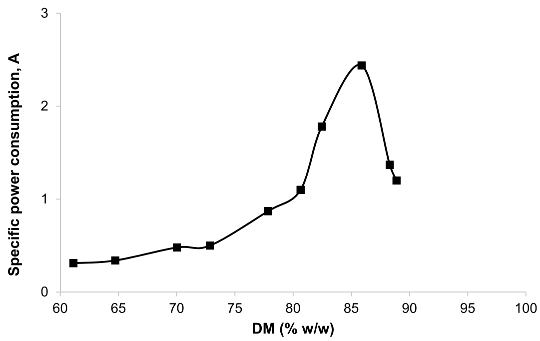

Fig. 10. Specific power consumption of mixer expressed in ampere (A) as a function of dry matter (DM) of superconcentrates. Experiments carried out at 500 rpm and 60 °C.

for a different composition. Also, the yield locus of MFP as it approached 81% w/w DM was not stress-dependent, which is encountered in a state very close to that of slurry, wherein the applied normal stress does not have an influence on the forces between particles (Schulze, 2021). Overall, multiple indications of transition into capillary state were observed that can be related to onset of highly cohesive phase, based on rheology and shear cell data, which consistently pointed to 81% w/w DM in the case of MFP.

3.3. Effect of product behavior as a function of DM on in-process parameter (motor amperage)

Fig. 10 depicts variation in power consumption as a function of DM. Initially, a gradual rise in power consumption was observed as the concentration increased. A dramatic rise can be seen around 81% w/w DM, with a peak at 86% w/w DM and a subsequent sharp fall at a DM greater than 88% w/w.

Remarkably, the overall trend in power consumption was closely related to the product properties determined using rheometer as well as the flow properties measured by ring-shear tester. A close relationship was evidenced between the product characteristics and process parameter (power consumption). Thus, the monitoring of power consumption can be a reliable tool to effectively control the process and determine the highly cohesive regime zone, in addition to the laboratory tools such as rheological and shear cell measurements to characterize the superconcentrated product. In the case of MFP, the highly cohesive regime was observed within the range 81% $<$ DM $<$ 88%, which needs to be overcome for efficient operation. The different techniques used in this study, consistently point out a dramatic evolution as the DM exceeds 81% w/w for MFP; this is likely related to transition from over-wet slurry into a capillary state, as discussed earlier in section 3.1.2 and 3.2. In order to avoid the risk of equipment damage due to excessive resistance in highly cohesive phase, superconcentration to 81% w/w can be considered optimum for MFP. This will be further validated at a pilot scale (100 kg h^{-1}). The highly cohesive phase is overcome in the PST process by recirculation of dry powders in the granulation step.

3.4. Granulation and characterization of granules

Granulation of the superconcentrated paste (82% w/w DM) by addition of dry powder was evaluated. The recirculation rate of dry powder for effective granulation was 1.0 ± 0.1 fraction (w/w) of paste. While granulation was successful there was nonetheless elongated and irregular shaped ("dough-like") pieces present (~3% of material). DM content of the dough like pieces was found to be <86.5% w/w, whereas for discrete granules it was >88.5% w/w. This illustrates that even within a successfully granulated powder a critical DM content must be reached (>88% w/w DM for MFP), to obtain granules. This is consistent with both the shear cell findings of the current study (see Fig. 9) and a previous study that suggested >88% w/w DM for granulation of permeate (Tanguy et al., 2017).

Energy saving potential of the superconcentration-granulation based powder manufacturing process is based on the extent of superconcentration, which in turn sets the required dry powder recirculation rate. Extent of superconcentration is limited by onset of cohesive phase, while quantity of dry powder recirculation is defined by DM content at the end of cohesive phase. The information with respect to onset as well as extent of cohesive phase (DM contents at onset and end), is crucial for screening suitability of products for the new approach. For example, a product with earlier onset and longer cohesive phase, will have lower superconcentration potential and require higher dry powder recirculation rate, which can result in poor process performance.

4. Conclusion

This study characterised dairy superconcentrates using rheological and shear-cell based techniques to gain relevant insights which can be used to control superconcentration and granulation processes. Flowability function (ffc) and cohesiveness effectively demarcated a highly cohesive non-flowing state (ffc <1) of the superconcentrated paste. Interestingly, the rheological techniques used (flow curves – Herschel Bulkley model, oscillation amplitude sweep and cohesion – probe-tack test) demonstrated a similar evolution of the product behavior as a function of DM. A dramatic increase was observed in flow stress and cohesion as the DM approached onset of the highly cohesive non-flowing state, ~82% w/w DM for MFP.

Overall, this study demonstrated that there is a critical DM content that imposes practical limitations on superconcentration as well as achieving discrete granules formation. Efficient process performance is dependent on achieving maximum superconcentration with minimum recirculation rate for granulation. The techniques presented in this study provide useful insights into the evolution of product behavior, particularly, identification of a cohesive non-flowing state, with increasing DM content, while reliably demarcating the boundaries for superconcentration and granulation. In the case of MFP, the superconcentration limit was ~82% w/w DM; the target for dry powder addition required to overcome the cohesive phase and achieve effective granulation was greater than 89% w/w DM. Additionally, the protocol of monitoring the power consumption can be a useful inline tool to control the new process. The techniques can be utilised for characterisation of other dairy and food products to understand the feasibility of applying the PST process. Future work will focus on validating the findings at pilot and industrial scale for a range of dairy compositions.

Author statement

Maheshchandra H. Patil - Conceptualisation; Investigation; Methodology; Writing – original draft, Gaëlle Tanguy - Supervision; Writing – review & editing, Cécile Le Floch-Fouéré; - Supervision; Writing – review & editing, Eoin G. Murphy – Conceptualisation; Supervision; Writing – review & editing; Project administration, Romain Jeantet – Conceptualisation; Supervision; Writing – review & editing; Project administration.

Declaration of competing interest

We confirm that there are no conflicting interests.

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