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Determination of limiting factors in a novel superconcentration-granulation based dairy powder manufacturing process

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ABSTRACT

An innovative approach based on superconcentration and granulation was investigated to manufacture dairy ingredients at lab-scale. A wet mass characterization technique, which measured agitator current consumption, was developed to study cohesiveness of super-concentrated products at various dry matter (DM) contents. For all ingredients, a composition-dependent cohesive phase was observed as DM increased, which was typified by a sharp rise and subsequent fall in power consumption. The effect of powder back-mixing on granulation was studied using three superconcentrate:powder (*w/w*) ratios (1:0.8, 1:1 and 1:1.2, respectively). Minimum powder addition rate for successful granulation was related to DM content at the end of the cohesive phase. Granulated powders had larger particle size, higher densities, lower porosities and enhanced flow properties compared to commercial spray-dried powders. The lab-scale model provided useful information on physical properties and limits during superconcentration and granulation, which increases the scientific knowledge relating to this novel powder production approach.

Industrial relevance: Spray drying is the most widely utilized powder manufacturing technology in the dairy industry, especially for producing ingredients and nutritional products. It is, however, extremely energy intense and therefore spray drying of high-volume, low-value dairy streams such as permeate represents a poor use of resources for industry. An alternative spray dryer-free process has been developed for such streams, with significant savings. This process is based on superconcentration of streams to DM content in excess of what is typically seen in a spray-drying process (up to 80% *w/w* DM) followed by granulation achieved by back-mixing of finished product and, finally, drying of granules. However, little information is available on how various dairy ingredients behave in this system. Therefore, a novel lab-scale production model was produced to determine limits of superconcentration and granulation behavior of various ingredients. This work provides vital information and represents the first step in a larger program which will culminate in demonstration of the industrial applicability of the new approach for drying of various dairy streams.

1. Introduction

Dairy powders are produced by dehydration of highly perishable liquid ingredients, which increases the shelf life by up to 2 years and also reduces volume (> 80%) and weight required for transportation. The transformation of liquid to a stable powder form (water activity, $a_w < 0.3$) requires removal of almost all the water and is generally realized through two or three operations in series. Initially, the bulk of the water (>90%) is removed by means of relatively low energy unit operations i.e. membrane filtration and/or vacuum evaporation. Water removal by membrane filtration is typically achieved through reverse osmosis or nanofiltration and can be utilized to concentrate to between 25 and 30% w/w dry matter (DM). Vacuum evaporation, which can be employed as the sole concentration operation or in combination with membrane filtration, is typically achieved through falling film evaporation (FFE) and transforms the liquid into a viscous concentrate (25–60% w/w DM, depending on composition). Spray drying (SD) then removes the remaining moisture, resulting in dry powders (typically >95% w/w DM). The primary step in SD is atomization of the concentrate into fine droplets in a flow of hot air. In some cases (i.e. 2 or 3 stage drying),

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additional fluid bed dryers are used to remove the final quantities of water, thereby increasing energy efficiency (Schuck et al., 2015; Westergaard, 2004).

SD is widely used to manufacture dairy powders because the majority of water is removed rapidly which results in high retention of functionality with minimal nutrient degradation. Furthermore, the technology is mature and is available from a number of equipment suppliers at production capacities ranging from small (kg.h^{-1}) to large (tonnes.h^{-1}). However, SD is an energy intensive operation, which can consume up to 10 times more energy than FFE. Despite removing less than 10% water, SD can account for almost 50% of the total energy consumption in a dairy powder plant (Ladha-Sabur, Bakalis, Fryer, & Lopez-Quiroga, 2019).

High solids drying is a promising prospect for energy efficient operation (Murphy, Tobin, Roos, & Fenelon, 2013; Walmsley, Atkins, Walmsley, Philipp, & Peesel, 2018), which minimizes the quantity of water to be removed by SD, either through maximization of water removal by FFE, or, reconstitution of dried ingredients to high DM, as can sometimes be employed in the manufacture of formulated products such as infant formula. However, technical constraints limit the high solids drying approach, mainly due to exponential rises in viscosity as a function of DM. Firstly, achieving higher DM in the evaporator is limited by poor product distribution, increased fouling and disrupted flow at the evaporator outlet. Secondly, poor atomization of the concentrate at higher total solids (due to higher viscosity) can result in incomplete drying and stickiness issues which can, in turn, impede SD operations and cause poor final product quality (Schuck et al., 2016; Tanguy et al., 2017).

Several investigations have examined approaches for controlling the viscosity issues associated with high solids SD in order to improve plant efficiency (Murphy et al., 2013; Patil, Tanguy, Floch-Fouéré, Jeantet, & Murphy, 2021; Sutariya, Huppertz, & Patel, 2017; Tanguy et al., 2015; Walmsley et al., 2018). However, only a few aspiring attempts have investigated breakthrough technologies which transform viscous concentrates into powders without using SD (Patil et al., 2021). For example, the patented technologies Tixotherm® (Písecký, 2005) and PST (Poudre sans tour or Towerless powder) process (Garreau et al., 2016) are highly compact installations which do not utilize SD and can result in lower capital and operational costs (energy, maintenance, cleaning, wastes, etc). In the superconcentration-granulation based process, the initial water removal is achieved in energy-efficient multiple effect FFEs i.e. similar to conventional SD process. For whey and permeate type streams, this step is usually terminated at ~60% DM, where viscosity limits further FFE concentration and/or spray drying. The subsequent superconcentration step, however, is capable of handling much higher concentrate viscosities. The vigorous mechanical agitation during superconcentration results in efficient water removal, taking DM from 60% to 80% w/w in the case of whey and permeate. Subsequently, the PST process uses a 'back-mixing' operation, where dry powder is recirculated into the superconcentrate, thereby transforming the resulting mixture into discrete granules which are subsequently dried. Back-mixing is widely used to overcome stickiness issues in wastewater sludge drying, where it is used to transform cohesive masses into discrete non-sticky granules (Kudra, 2003; Peeters, Dewil, & Smets, 2014). However, as an operation, it is not widely reported in the dairy industry with the exception of Tanguy et al. (2017) who demonstrated feasibility of the PST process for manufacture of whey permeate powders. Powders produced were comparable to SD powders, however, it was estimated that 32% less energy was used to transform concentrates (60% w/w DM) into powders (97% w/w DM).

As water is removed, transition of products into a highly cohesive (sticky, rubbery) phase with increased resistance to flow eventually becomes a limiting factor in high-shear, thin-film dehydration technologies (Bennamoun, Arlabosse, & Leonard, 2013; Peeters et al., 2014; Qiu, Boom, & Schutyser, 2019). In dairy systems, information regarding physical behavior during superconcentration under high shear conditions is not reported. Furthermore, application of Tixotherm® and PST is focused on only high lactose streams (whey and permeate) with very few studies published. To the best knowledge of the authors, no studies are published in reference to suitability of these technologies to other dairy products – skim milk, fat-filled milk, etc. It is crucial to build understanding of dairy superconcentrate behavior in order to optimize superconcentration-based powder production. Furthermore, it follows that the knowledge of granulation of these systems through recycling of final powders is also not well understood.

The current study examines the superconcentration and granulation approach for production of dairy powders at lab scale. Four dairy powders were selected to cover a range of compositions with respect to lactose, protein and fat content. The objectives of this investigation were : 1. To identify the limits of superconcentration for dairy products by developing a simple and reliable lab protocol, 2. To produce dairy powders utilizing the novel process at lab scale and compare powder properties with standard spray-dried materials. This investigation provides insights into the functional behavior of the different dairy products, as they are transformed from liquid to powders, based on their composition.

2. Material and methods

2.1. Materials

Whey permeate powder (WPP), demineralized (90%) whey powder (DWP), skimmed milk powder (SMP) and fat-filled milk powder (FFMP) were acquired from local dairy ingredient suppliers. Table 1 presents specifications of the dairy powders used in experiments.

2.2. Characterization of dairy superconcentrates as a function of DM

Prior to investigating powder production at lab scale, the flow-related physical properties of superconcentrates at various DM contents were measured. Dairy powders were reconstituted in demineralized water (to produce samples varying from 40 to 80% w/w DM) under high rotational speed (500 rpm) at 50 °C for 30 min in a temperature-controlled mixer *(TM31, 1.5 kW, Thermomix, Vorwerk, Germany)*. Where possible, concentrates were characterized by their resistance to flow at various DM contents. Viscosity measurements were performed for viscous concentrates using a rheometer *(AR G2, TA Instruments, Crawley, UK)* in a flat parallel plate (60 mm diameter) assembly with a gap of 1 mm at 60 °C. A serrated plate was used for higher DM content concentrates. Samples were pre-sheared at 300 s^{-1} for 20 s and then equilibrated at rest for another 30 s. Shear rate was increased in steps from 1 to 1000 s^{-1} and apparent viscosity was reported at a shear rate before sample discharge or slip was observed. The viscosity measurements were limited up to a certain DM content, beyond which it was not practical to perform measurements at high shear due to slip and discharge of the thick pastes from the geometry.

In addition to viscosity measurements of dairy ingredients, agitator power consumption of the mixer was measured to characterize resistance to flow as a function of DM content. An energy consumption meter *(Energy Logger 4000 FR, Voltcraft, Germany)* attached to the power cable

Table 1
Composition of dairy powders used in the study; whey permeate powder (WPP), demineralized whey powder (DWP), skim milk powder (SMP) and fat-filled milk powder (FFMP).

	WPP	DWP	SMP	FFMP
Lactose (% w/w)	85.5	83.5	54.8	38.0
Protein (% w/w)	2.5	12.0	34.0	24.0
Fat (% w/w)	1.2	1.2	1.2	29.0
Moisture (% w/w)	2.8	3.5	4.0	3.0
Ash (% w/w)	8.0	< 1	6.0	6.0

indicated the power consumption, expressed as the current (ampere, A) supplied to operate the mixer at a constant agitation rate of 500 rpm, for various DM contents. Demineralized water was progressively added in to the dry powder to produce samples with varying DM content and corresponding power consumption was measured.

2.3. Novel lab scale superconcentration-granulation process to produce dairy powders

The lab scale process developed and investigated an innovative approach to produce dairy powders, which comsists of superconcentration of dairy concentrates to between 60 and 80% w/w DM (depending on composition) followed by granulation through addition of dry powders to produce dairy powders as described by Tanguy et al. (2017). In that study, superconcentration, granulation and drying were achieved using three horizontal thin-film, rotary evaporators in series at pilot scale (VOMM, Rozzano, Italy). Trials were performed for the production of permeate powder. For superconcentration, the high shear within the unit allowed for effective concentration despite significant viscosity development at high DM. Granulation was achieved through back-mixing of powder on a 1:1 basis. Finally, drying of the subsequent granules was achieved using a similarly configured rotary evaporator/dryer.

Table 2 provides details of the experimental conditions followed. Initially, the dairy powders were reconstituted in demineralized water to produce superconcentrated pastes of desired DM contents as described in section 2.2. The resulting highly viscous paste was fragmented into discrete granules by addition of respective dry dairy powders. The optimum quantity of powder addition rate (back-mix) for successful granulation was studied by addition of dry powders into paste at three superconcentrate:powder w/w ratios (1:0.8, 1:1 and 1:1.2, respectively). The standardized process of powder addition for 30 s at 500 rpm and granulation at 800 rpm for 10 s was followed for all the experiments. The granules obtained were dried immediately as a thin layer (around 5 mm) over a drying pan in a vacuum oven *(OV-12, 1.5 kW, JEIO TECH, Korea)* at 45 °C overnight. The dried granules were ground in the mixer at 500 rpm for 30 s and 800 rpm for 5 s. The powders obtained were immediately stored in airtight plastic containers.

In the process described above, the powder used to granulate had the same dry matter composition as the superconcentrate. However, it is also possible to use powders with different compositions to granulate. Such an approach can result in novel compositions while also potential affecting the granulation process and therefore the overall efficiency of the process. Therefore, to test the effect of different compositions, a common drying aid (Maltodextrin (DE12) and high-water holding capacity (apple fiber) were also used during granulation. Maltodextrin (DE12) and apple fiber were procured from local suppliers. Powders were gradually added into whey permeate (WPP) superconcentrated paste (80% w/w DM), until discrete non-sticky particles were obtained.

2.4. Physical characterization of granulated powders

2.4.1. Moisture and Total Solids

The moisture content of the powder samples was measured in a rapid Halogen moisture analyzer *(Sartorius, Germany)*. The DM of the concentrates were measured by differential weighing using a rapid microwave moisture analyzer *(Smart Trac CEM, Germany)*. For higher DM superconcentrates and granules, samples were first diluted in a known quantity of deionized water to measure the total solids. The measurements were determined in duplicate.

2.4.2. Bulk density, true density and porosity

The loose and tapped bulk densities were measured by a tapped volumeter with a graduated cylinder *(Funke Gerber, Germany)*. The volume occupied by 100 g powder was used to calculate the loose density while the tapped density was calculated using the volume after 100 taps (GEA-Niro, 2006). The true density of powders was measured by Gas Pycnometer *(AccuPyc II 1340, Micrometrics Instrument Corporation, USA)*. Finally, the porosity was calculated using the tapped density and true density.

2.5. Particle size distribution, rehydration properties and flowability

A laser scattering granulometer *(Mastersizer 2000, Malvern Instruments Ltd., Malvern, UK)* was used to determine the particle size distribution. The rehydration properties of experimental granulated and standard spray-dried powders were characterized by dispersibility index and solubility index; which were determined according to Schuck, Dolivet, and Jeantet (2012). Flowability is represented as time in seconds required for sample powder to leave a rotary drum through a slit (GEA-Niro, 2006). Each sample was analyzed in duplicate.

2.6. Light microscopy and scanning electron microscopy

Crystalline structure of powder was observed using polarized light microscopy *(Olympus Corporation, Japan)*. For the scanning electron microscopy (SEM) measurement, the powder samples were mounted on a double-sided carbon tape and fixed to SEM stubs and then sputter coated with Chromium *(Emitech K550X, Ashford, UK)*. Powder samples were examined with a field emission scanning electron microscope *(Zeiss Supra, Carl Zeiss SMT Ltd., Cambridge, UK)* at 5000× magnification.

2.7. Estimation of energy consumption

Energy consumption corresponding to water removal was calculated

Table 2
Experimental conditions for a) generation of superconcentrated paste and b) granulation by addition of dry powder at three levels.

		Superconcentration			Granulation			
S. No	Ingredients	Initial mass of powder (g)	Initial mass of water (g)	Superconcentrated paste (% w/w DM)	Back-mix fraction (−)	Mass of dry powder added (g)	Bulk granules (% DM)	Discrete granules formed?
1	Whey Permeate (WPP)	163	37	79.4 ± 0.7	0.8	160	86.4 ± 0.3	No
2		163	37	80.0 ± 0.7	1.0	200	88.1 ± 0.9	Yes
3		163	37	79.4 ± 0.6	1.2	240	89.1 ± 0.1	Yes
4	Demineralized whey (DWP)	163	37	79.4 ± 0.9	0.8	160	87.5 ± 0.3	Large granules
5		163	37	80.4 ± 0.6	1.0	200	88.6 ± 0.6	Yes
6		163	37	79.9 ± 0.9	1.2	240	89.4 ± 0.3	Yes
7	Skim milk (SMP)	124	75	61.2 ± 0.9	0.8	160	75.3 ± 0.1	No
8		124	75	60.2 ± 0.7	1.0	200	78.3 ± 1.0	Large granules
9		124	75	59.9 ± 1.5	1.2	240	81.8 ± 3.9	Large granules
10	Fat-filled milk (FFMP)	160	75	65.3 ± 2.4	0.8	191	77.4 ± 1.0	Yes
11		160	75	65.1 ± 1.5	1.0	235	80.7 ± 0.3	Yes
12		160	75	66.2 ± 0.1	1.2	273	83.5 ± 1.2	Yes

for the novel and conventional SD processes. The evaluation was based on the previously established methods to estimate energy usage during dehydration steps (Schuck et al., 2015; Tanguy et al., 2017). Energy consumption ratio (ECR, kJ.kg^{-1} water removal) was used to determine the energy required during dehydration steps (Table 3). The energy calculations were based on conventional inlet and outlet DM contents for SD (Schuck et al., 2015; Tanguy et al., 2017), while DM for the superconcentration-granulation process were determined using the data obtained in the present study. The detailed calculations are explained in Tanguy et al. (2017) where the specific energy consumption ratio (ECR) was estimated as described below for permeate:

- The first concentration step from 5.5 to 60% w/w DM, performed by FFE, was considered to involve an ECR ranging from 75 to 400 kJ·kg^{-1} of evaporated water depending on the evaporator configuration.
- The ECR of the superconcentration step i.e. from 60 to 80% w/w DM, was estimated at 2875 kJ·kg^{-1} of evaporated water (manufacturer's data).
- The ECR of the final drying i.e. from 88.5 to 97% w/w DM, was estimated at 5400 kJ·kg^{-1} of evaporated water (manufacturer's data).

3. Results

3.1. Behavior of dairy ingredients at superconcentrated DM content

This study investigated behavior of dairy systems at DM contents higher than those typically encountered in vacuum concentration. The concentrates were characterized by measuring viscosity and power consumption as a function of DM content (Fig. 1). All the dairy ingredients studied, exhibited a strong concentration dependence and an exponential rise (Fig. 1a) in apparent viscosity with increasing DM content, which is consistent with previous studies (Trinh, Trinh, & Haisman, 2007; Vélez-Ruiz & Barbosa-Cánovas, 1998). SMP and FFMP (medium protein content) demonstrated exponential rise close to 50% and 55% w/w DM, whereas DWP and WPP (low protein content) showed exponential rise at higher DM content (~ 65% w/w DM). These observations are in agreement with previous studies that reported apparent viscosity was strongly affected by protein content (Murphy, Fenelon, Roos, & Hogan, 2014; Schuck, Méjean, Dolivet, Beaucher, & Famelart, 2005).

Furthermore, Fig. 1b shows power consumption in the temperature controlled high-shear mixer as a function of DM content. As the DM content progressively increased, all compositions demonstrated a similar trend i.e. a sharp increase to peak power consumption followed by a sharp decrease. The onset of rise in power consumption and subsequent fall was highly influenced by composition. Onset occurred at lower DM content for SMP and FFMP, around 62% and 66% w/w DM respectively, compared to DWP and WPP, where onset was at ~80% w/w DM. Among all the compositions studied, SMP showed the highest peak in power consumption indicating higher resistance to flow (higher cohesiveness).

3.2. Granulated powder production

Granulated powders were produced using the novel superconcentration-granulation process as described in Table 2. Dry powder was added into superconcentrated paste in order to fragment the paste into discrete large particles. Fig. 2 depicts the granulation achieved at different back-mix (dry powder addition) ratios for the dairy products. WPP and DWP granulated at a minimum DM of 88% and 87% w/w, which corresponds to dry powder addition rate of 1:1 and 1:0.8 (superconcentrate:powder w/w ratios), respectively. WPP results are consistent with previously reported findings i.e. whey permeate granulation occurred at DMs greater than 88% w/w (Tanguy et al., 2017).

Table 3
Estimation of the specific energy consumptions (SEC) for dehydration in conventional spray drying (SD) and novel superconcentration-granulation process.[1]

		Energy consumption ratio (ECR)	Skim milk			Fat-filled milk			Demineralised whey			Whey permeate		
			Inlet DM	Outlet DM	SEC	Inlet DM	Outlet DM	SEC	Inlet DM	Outlet DM	SEC	Inlet DM	Outlet DM	SEC
		(kJ.kg^{-1} of evaporated water)	% (w/w)	% (w/w)	(kJ.kg^{-1} dry powder)	% (w/w)	% (w/w)	(kJ.kg^{-1} dry powder)	% (w/w)	% (w/w)	(kJ.kg^{-1} dry powder)	% (w/w)	% (w/w)	(kJ.kg^{-1} dry powder)
Conventional	Two-stage MVR evaporator[a]	*75*	*9*	*50*	*683*	*12.5*	*55*	*464*	*17*	*60*	*316*	*5.5*	*60*	*1239*
	SD[a]	*5500*	*50*	*97*	*5330*	*55*	*97*	*4330*	*60*	*97*	*3497*	*60*	*97*	*3497*
Novel	Two-stage MVR evaporator[a]	*75*	*9*	*50*	*683*	*12.5*	*55*	*464*	*17*	*60*	*316*	*5.5*	*60*	*1239*
	Superconcentration[b]	*2875*	*50*	*62*	*1113*	*55*	*65*	*804*	*60*	*80*	*1198*	*60*	*80*	*1198*
	Post-granulation drying[b]	*5400*	*80*	*97*	*3143*	*80*	*97*	*2741*	*88.5*	*97*	*1183*	*88.5*	*97*	*1183*
% energy savings[c] (global process)					*18*			*16*			*29*			*24*
% energy savings[c] (minus evaporator)					*20*			*18*			*32*			*32*

[1] ECR values from Tanguy et al. (2017); MVR, mechanical vapour recompression.
[a] DM are typical values observed in industrial practice.
[b] DM values taken from current study.
[c] Novel vs. Conventional.

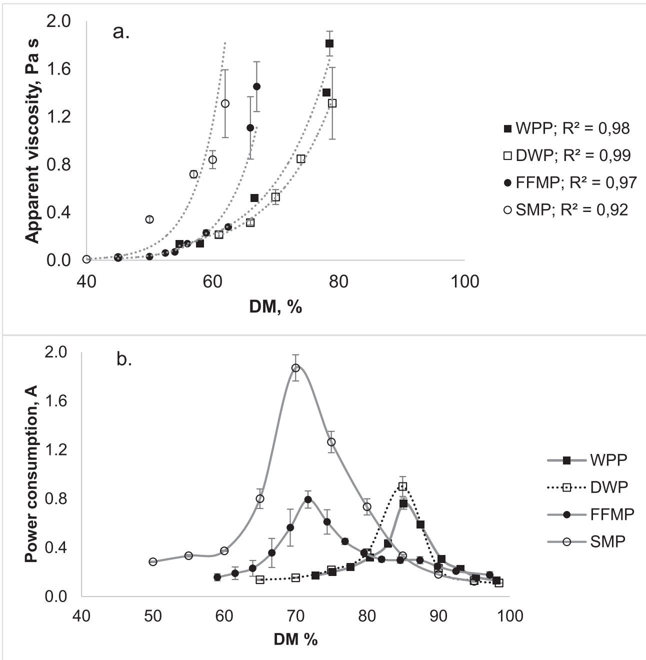

Fig. 1. (a) Apparent viscosity and (b) variation in power consumption as a function of dry matter (DM) content for dairy products – WPP, whey permeate powder, DWP, demineralized whey powder, FFMP, fat-filled milk powder and SMP, skim milk powder. R^2 values are for exponential fit of apparent viscosity as a function of DM using average values of apparent viscosity.

SMP granulated into larger particles at a minimum DM content of 78% corresponding to minimum back-mix of 1:1 ratio, whereas, FFMP granulated at a back-mix ratio of 1:0.8 corresponding to a minimum DM content of 77% w/w DM. For the lower back-mix ratio of 1:0.8, WPP and SMP continued to exist in a highly cohesive and sticky mass with high resistance to flow i.e. no granulation was observed (Fig. 2).

Differences in granulation behavior were observed for different compositions. For SMP and FFMP, granules obtained under constant conditions (1:1 back-mix, powder addition for 30 s at 500 rpm and 10 s at 800 rpm) were manually separated using a combination of 2, 1 and 0.63 mm sieves. SMP had 88% mass fraction over 2 mm sieve size, whereas FFMP had 41%. SMP and FFMP had 2% and 21% mass fractions finer than 0.63 mm, respectively. It is evident that SMP offered higher resistance to fragmentation (granulation) into discrete particles as compared to FFMP.

3.3. Granulated powder physical and rehydration properties

Table 4 provides a comparison of physical and functional properties of the granulated powders. Overall, the density of the granulated powders was higher than standard spray-dried powders. Lower porosity was observed in all the granulated powders, which is consistent with features of high shear granulation, where strong shear forces compact the granules providing comparatively higher density and lower porosities (Ji et al., 2016). Higher particle size was observed for all the granulated powders, which is in agreement with observations made in the previous study on PST-manufactured powders (Tanguy et al., 2017). Granulation improved flowability for all powders. DWP and WPP had comparable rehydration properties, whereas SMP and FFMP exhibited poor rehydration properties (Table 4). Person et al. (2018) reported that rehydration properties of skim milk agglomerates were influenced by the final drying stage and DM content. The solubility of wet FFMP granules at 80% w/w DM was found to be 99.8% indicating that the poor solubility was caused by the drying step.

Granulated SMP and FFMP powders had a higher content of

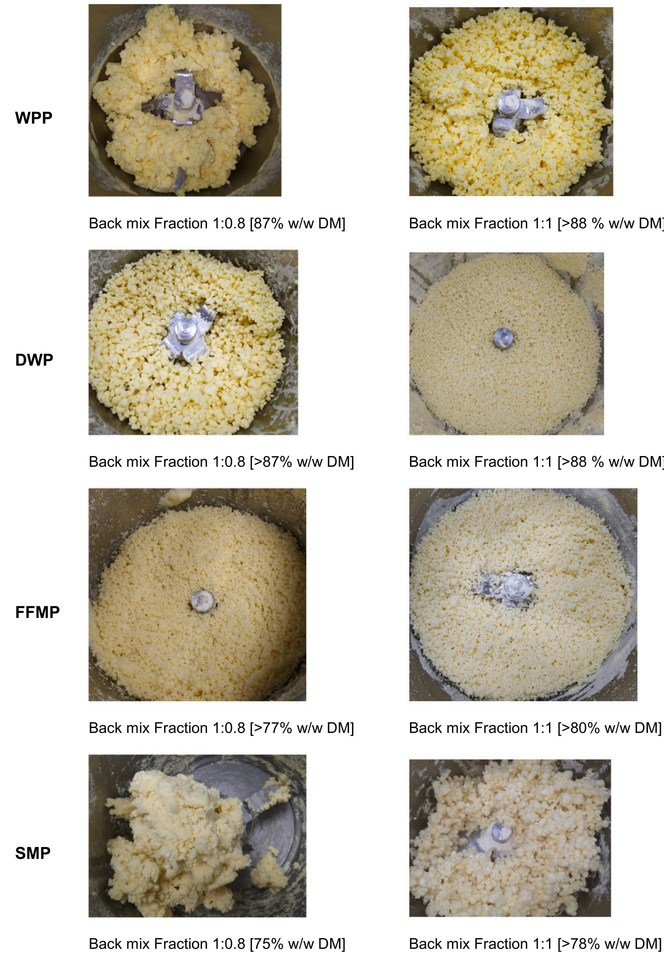

Fig. 2. Granulation obtained for different back-mix fractions for dairy products – WPP, whey permeate powder, DWP, demineralized whey powder, FFMP, fat-filled milk powder and SMP, skim milk powder.

crystallized lactose compared to standard spray-dried powders, which can be seen as bright spots in light microscopy images (Fig. 3). The granulated powders had compact sharp structures with very low porosity, whereas the spray-dried powders demonstrated typical spherical porous structures (Fig. 4). A similar, highly compacted structure with low porosity was observed in the high shear granulation of milk protein powders (Ji, Fitzpatrick, Cronin, Fenelon, & Miao, 2017).

4. Discussion

The primary objective of this study was to investigate limiting factors of the novel superconcentration-granulation approach for producing dairy powders. Four dairy ingredients covering a range of compositions (protein:lactose ratio and fat content) were investigated to understand the influence of composition. A lab scale model was developed to simulate the PST approach using a temperature-controlled mixer; power consumption was measured as a function of DM content to gain insights into the behavior of superconcentrates.

4.1. Limiting factors for production of dairy powders using superconcentration-granulation process

4.1.1. Evolution of a highly cohesive phase restricts superconcentration of dairy ingredients

Fig. 1a clearly shows how viscosity can have a limiting effect on the water removal process. The exponential rise in viscosity with increasing

Table 4
Physical properties, rehydration properties and flowability of dairy powders: lab scale granulated powders (G) and conventional spray-dried powders (SD).[1]

		WPP		DWP		SMP		FFMP	
		G	SD	G	SD	G	SD	G	SD
Dry matter	(g.kg^{-1})	990	977	993	982	990	975	993	975
Bulk density	(kg.m^{-3})	717 ± 3	568 ± 2	730 ± 5	532 ± 4	800 ± 6	550 ± 7	606 ± 4	405 ± 5
Tapped density	(kg.m^{-3})	787 ± 6	686 ± 4	877 ± 8	657 ± 5	862 ± 7	660 ± 3	694 ± 0	508 ± 3
Porosity	(%)	47	55	39	57	38	41	35	60
d(0.1)	(μm)	139 ± 4	20 ± 0	33 ± 0	16 ± 0	289 ± 35	37 ± 0	231 ± 9	62 ± 2
d(0.5)	(μm)	615 ± 14	148 ± 1	582 ± 9	73 ± 1	711 ± 37	105 ± 3	681 ± 7	169 ± 6
d(0.9)	(μm)	1441 ± 9	324 ± 1	1380 ± 9	188 ± 13	1384 ± 28	224 ± 23	1428 ± 4	428 ± 40
Span	(–)	2.1	2.0	2.3	2.4	1.5	1.8	1.8	2.2
Solubility	(%)	96.8 ± 0.4	98.8 ± 0.0	99.8 ± 0.0	99.5 ± 0.0	76 ± 0.0	99.8 ± 0.0	76 ± 0.0	99.8 ± 0.0
Dispersibility	(%)	85.4 ± 1.2	93.6 ± 0.5	92.8 ± 0.7	93.5 ± 0.4	20.0 ± 3.0	94.3 ± 0.9	18.0 ± 3.6	86.4 ± 0.8
Flowability	(g.min^{-1})	47.8 ± 1.0	46.2 ± 1.8	39.9 ± 1.2	12.2 ± 0.3	48.9 ± 0.9	14.2 ± 0.4	26.1 ± 0.7	9.1 ± 0.2

[1] WPP, whey permeate powder; DWP, demineralized whey powder; SMP, skim milk powder and FFMP, fat-filled milk powder; d(0.1), d(0.5) and d(0.9) represent the particle sizes below which 10%, 50% and 90% of the powder volume exists, respectively.

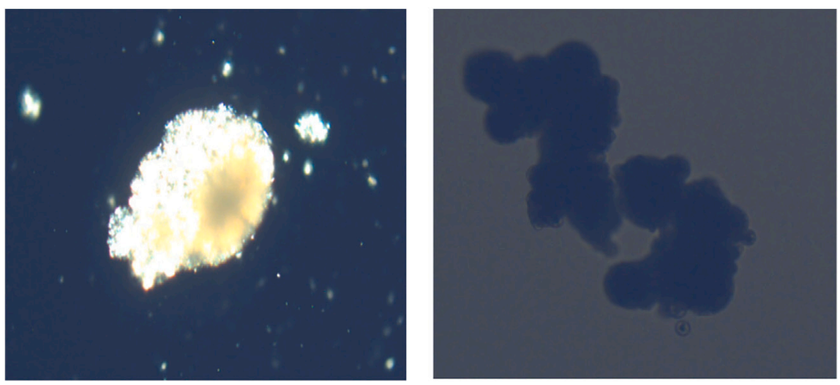

Fig. 3. Polarised light microscopy images of fat-filled milk powder (FFMP): Granulated (left) and standard spray-dried (right).

total solids imposes practical limits on the concentration levels achieved and is strongly composition dependent. It is well known increased viscosity negatively influences flow distribution and heat transfer in FFEs and subsequent atomization during SD (Schuck et al., 2016; Tanguy et al., 2017). This can result in product burn-on, increased fouling in the evaporators, incomplete drying and stickiness in the drying tower, all of which negatively impact powder properties and functionality. Therefore, increased viscosity is a key limiting factor in the conventional dairy powder manufacturing process. From the data presented here it would appear that the viscosity becomes a limiting factor for in SMP production at lower DM content compared to WPP and DWP. This is broadly correlated with protein content (Table 1) and is also in agreement with the typical DM contents achieved for these products during FFE, with typical post-evaporation DM contents for skim milk being ~50% w/w DM compared to ~60% w/w DM for whey and permeate. This is an important point because the maximum levels of concentration, in turn, define the conditions for superconcentration. Therefore, in this study, superconcentration is a composition dependent term which is >50% w/w DM for skim milk, >55% w/w DM for fat-filled milk and > 60% w/w DM for whey and permeate.

In processes which are capable of handling superconcentrated materials, such as PST, the viscosity increase, as seen in Fig. 1a, is not a significant factor due to the inherent high shear and the lack of a requirement for atomization. However, the limiting factor for such systems appears to be the transition of the product into a highly cohesive mass at a composition dependent DM content (Fig. 5). In addition, the present investigation demonstrates that this highly cohesive phase exists over a composition dependent DM content range (Fig. 1b). The details of the cohesive phase were found to shed light not only on the limits of superconcentration but also on subsequent granulation operations. While the onset of the cohesive phase resulted in extreme resistance to flow, as indicated by a sharp rise in the power consumption, which designated the maximum extent of superconcentration, the end of the cohesive phase also gave information on the minimum DM content at which the wet mass transformed into non-sticky, discrete granules. A cohesive phase was observed in all the dairy compositions studied, however the onset was highly influenced by the composition (Fig. 1). In particular higher protein content resulted in earlier onset (SMP 62% w/w DM, FFMP 65% w/w DM), whereas lower protein and higher lactose contents resulted in delayed onset (DWP and WPP at 80% w/w DM). Thus, lower protein streams can be superconcentrated to higher DM contents compared to higher protein dairy streams.

The onset of the cohesive phase is a significant technical challenge, which defines the superconcentration limits for the superconcentration-granulation process. The cohesive phase offers huge resistance to flow and can result in uncontrolled agglomeration which drastically reduces heat and mass transfer rates leading to highly inefficient drying conditions (up to 60% lower heat transfer rates) (Kudra, 2003). Furthermore, the highly cohesive mass has the potential to damage the mechanical integrity of rotating parts of the equipment (Kudra, 2003; Peeters et al., 2014). Therefore, the main requirement of an efficient superconcentration and granulation process should be to avoid the cohesive phase through termination of the first stage at an appropriate concentration, followed by application of an adequate back-mixing ratio. Table 5 presents DM content limits for the three distinct regions of behavior observed in the superconcentrated ingredients studied.

The approach applied here, while novel for dairy ingredients, is analogous to approaches applied in drying of sludge. Technologies similar to PST, i.e. utilizing high shear thin film conductive dehydration process, have reported a similar evolution of three distinct phases while drying wastewater sludge, as reviewed by Bennamoun et al. (2013). The waste water sludge passes through pasty, lumpy and granular phases

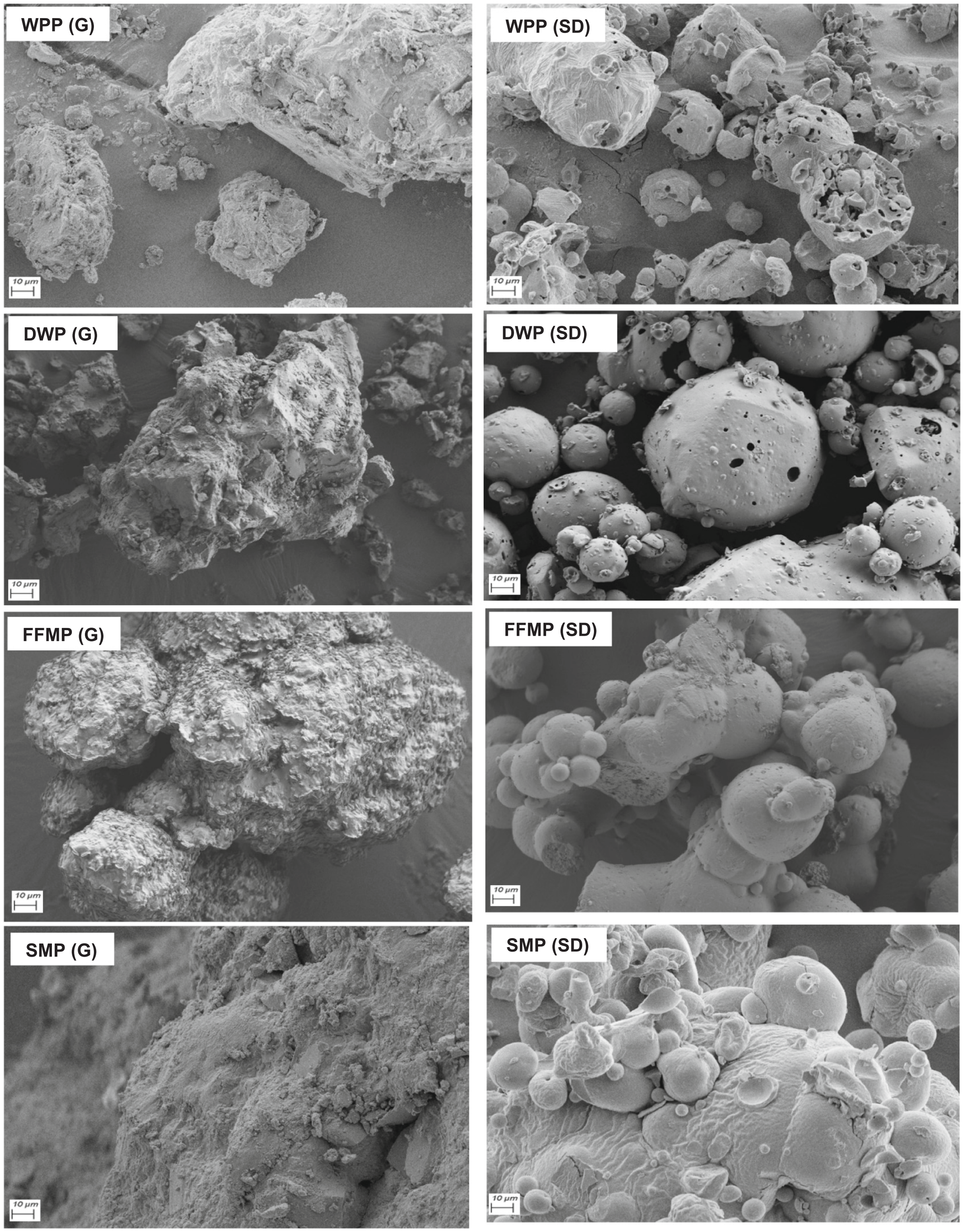

Fig. 4. Scanning electron microscopy images (5000× magnification, scale bar – 10 μm) of dairy powders: WPP, whey permeate powder; DWP, demineralized whey powder; FFMP, fat-filled milk powde; and SMP, skim milk powder; G, granulated powders; SD, standard spray-dried powders.

which are detected by following variations in torque during concentration (Bennamoun et al., 2013; Ferrasse, Arlabosse, & Lecomte, 2002; Kudra, 2003). Another study utilized a shear-based lab protocol to map the sticky phase (highly cohesive-rubbery phase) encountered at intermediate moisture content ranges during drying of activated sludge, consequently providing valuable information to circumvent or manage the stickiness phenomenon in industrial sludge dryers (horizontal rotary dryers similar to PST process) (Peeters, Dewil, Van Impe, Vernimmen, & Smets, 2010).

Across all dairy compositions in this study, the evolution of cohesive phase was observed at DM approximately between 10 and 20% w/w greater than the concentration at which exponential viscosity increase occurred. The exponential viscosity rise indicates dramatic change from liquid to solid as the system approaches a jamming state (Hogan, O'Loughlin, & Kelly, 2016; Ovarlez & Coussot, 2007). Due to the higher water holding capacity of proteins (casein - ~4 g/g, whey - ~1.5 to 3.5 g/g), the higher protein materials, SMP and FFMP, have less "free" water at a given DM (Liu et al., 2018), which manifests itself as an increase in cohesion at lower DM. Similarly, as DM is further increased, the higher water binding capacity in SMP and FFMP likely results in less available water for interparticle liquid bridging, resulting in a drop in cohesion and granule formation at lower DM (Iveson et al., 2001).

4.1.2. *Recirculation rate and process efficiency*

Achieving maximum possible superconcentration, while avoiding the cohesive phase, is key to the efficient operation of the novel process.

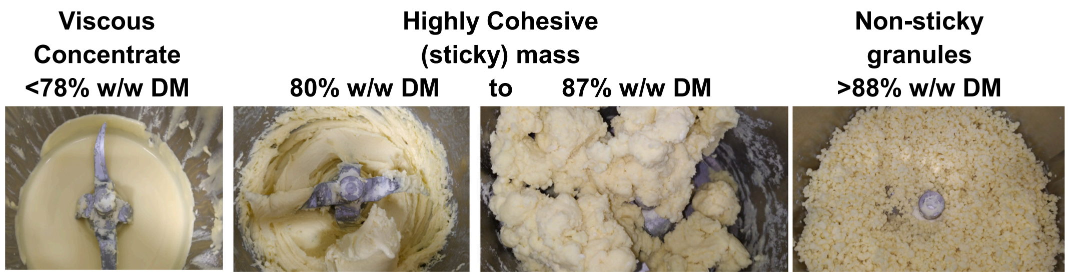

Fig. 5. Transition of whey permeate through three different phases – viscous, highly cohesive (sticky) mass and non-sticky discrete granules as a function of dry matter (DM) content.

Table 5
Dry matter contents (w/w) for viscous phase (maximum superconcentration), cohesive phase and discrete non-sticky granules for different dairy compositions - whey permeate powder (WPP), demineralized whey powder (DWP), skim milk powder (SMP) and fat-filled milk (FFMP).

	WPP	DWP	SMP	FFMP
Viscous phase	< 80%	< 80%	< 62%	< 65%
Cohesive phase	80–88%	80–87%	62–78%	66–77%
Discrete phase	>88%	>87%	>78%	>77%

It is evident that there exists a critical DM content for successful granulation in each of the compositions which is well related to the cohesive phase mapping (variation in power consumption) represented in Fig. 6. Table 5 highlights the maximum DM content during superconcentration and minimum DM required for granulation of different dairy streams. The cohesive phase for SMP extended over 16% DM (from 62% to 78% w/w DM), compared to ~8% for DWP and WPP (from 80 to 88% w/w DM). Therefore, the required quantity of dry powder to achieve granulation was markedly higher for SMP which ultimately affects the process performance for higher protein compositions.

Recirculation rate increases if the DM content of the superconcentrated paste falls below the maximum possible, as successful granulation is linked to DM content at the end of cohesive phase. Recirculation rate is doubled for a 5% reduction in concentration of the paste from an initial 80% w/w DM (Tanguy et al., 2017), which drastically reduces the powder outlet flow and can nullify benefits in energy consumption vis-à-vis SD.

Furthermore, compositions like FFMP and SMP, which need removal of more than 20% moisture in final convective drying, could potentially end up consuming similar energy consumptions as that of SD, if not more, as specific energy consumption in final convective drying steps is highest among all drying steps in conventional process (Schuck et al., 2015; Westergaard, 2004).

Although no energy consumption measurements can be performed at this stage, an estimate based on key principles and procedures presented

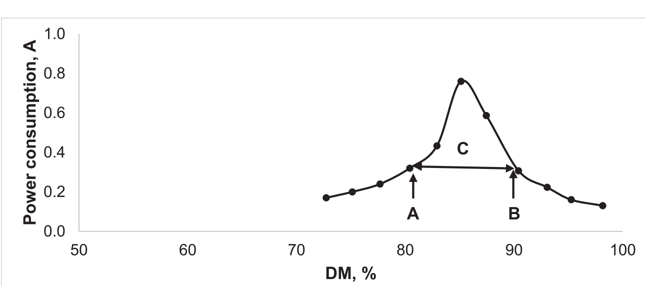

Fig. 6. Cohesive phase of whey permeate (WPP), A – Maximum possible superconcentration; B – Minimum dry matter (re-circulation rate) for successful granulation; C – Highly cohesive (so-called sticky) phase which needs to be avoided by recirculation of dry powder for efficient and safe operation.

in previous studies (Fig. 7) was undertaken to guide future scoping (Schuck et al., 2015; Tanguy et al., 2017). Table 3 presents the energy estimates based on water removal, in alignment with the previous studies. Theoretically, all the dairy powders can be produced at lower operational costs as compared to SD, largely due to the lower energy costs associated with superconcentration as compared to drying (Tanguy et al., 2017). Furthermore, the new process may have additional benefits: very compact installation that should come with lower cleaning solution and effluents volumes; lower capital (up to 40% based on estimates); and simpler operations (Tanguy et al., 2017). These factors in combination with energy estimates in this study, can encourage industries to test the robustness of the new process at a pilot scale. However, it must be stated that the lower DM content of SMP and FFMP superconcentrates results in a less favorable drying energy reduction as compared to DWP and WPP (18–20% reduction for SMP and FFMP; ~32% reduction for DWP and WPP). This finding, in combination with the relatively poor functional attributes associated with the lab-scale SMP and FFMP powders produced, indicate that such a process may have limitations in handling high protein/fat materials.

4.1.3. Influence of final drying stage on phase transitions and final powder structure and functional properties

Producing specific final powder structures (particle size, density etc.) is key to delivering the required functional properties (rehydration, flow properties, stability) in dairy powders (Palzer, Dubois, & Gianfrancesco, 2012). Stability and functional properties of granules is highly influenced by the drying stage (Person et al., 2018). The rapid dehydration associated with SD ensures lactose remains in a stable glassy state for SMP and FFMP. The PST process encounters longer residence time (~30 mins) in oversaturated zone, conditions which can initiate spontaneous crystallization of lactose (Vuataz, 2002). Crystallized lactose is clearly visible in SMP and FFMP powders produced in lab scale; longer residence times during dehydration of agglomerated skim milk powders have been shown to result in increased amount of crystallized lactose and influenced rehydration properties (Person et al., 2018). Moreover, high shear granulation produces powders with dense structures and lower porosity as compared to SD (Ji et al., 2017). Therefore, it is to be expected that powder properties such as rehydration and flowability will differ to standard SD powders, as observed in this study.

Conversely, the crystallization observed in the PST process may be a positive in the case of DWP and WPP production, having the potential to partially replace or enhance the batch crystallization process, bringing associated benefits in process efficiencies. The application of this feature is evidenced in patented Tixotherm® process (Písecký, 2005), where the batch crystallization step is completely avoided. Further investigation is necessary to understand impact of superconcentration on lactose crystallization and final powder stability and functionality. For example, the relatively long residence time of lactose crystals in a highly supersaturated state will likely result in the production of a large number of small crystals (Parimaladevi & Srinivasan, 2014), which may increase the ease of lactose crystal solubilization upon rehydration.

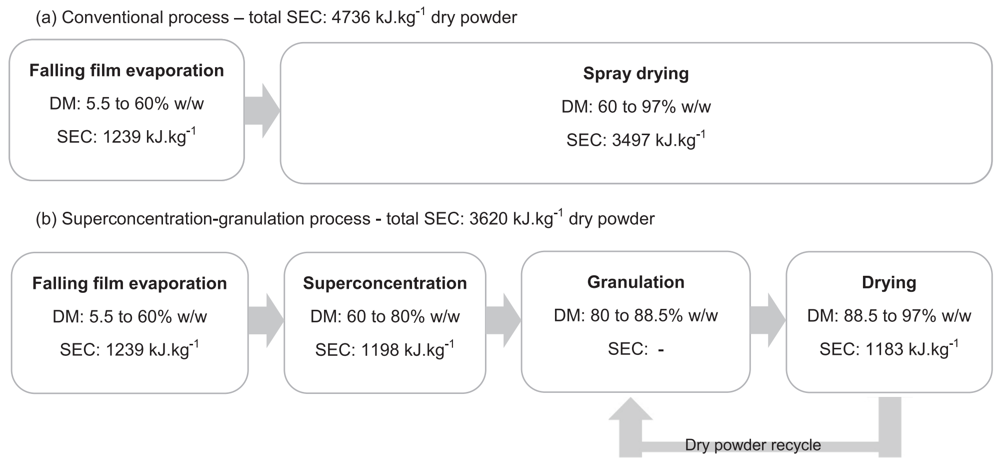

Fig. 7. Block diagram with estimation of specific energy consumptions for dehydration steps in (a) conventional spray drying and (b) novel superconcentration-granulation process for manufacture of whey permeate powder (WPP). DM, Dry matter; % (w/w); SEC, Specific energy consumption (kJ.kg^{-1} dry powder).

4.2. *Prospects and perspective*

The investigated process demonstrated, at lab scale, an interesting low-energy, compact method to produce dairy powders. A key feature of the process is the recycling step, whereby powder from the end of the process is used to granulate. However, it is also possible to replace, or partially replace the final product with different powders, thereby effecting the granulation. Utilization of other powders – such as powders with higher water holding capacity or drying aids can be evaluated to overcome recirculation challenges. Two powders were tested at lab scale for their feasibility, maltodextrin (typical drying aid) and a fruit fiber (high water holding capacity, high fiber ingredient) a coproduct from fruit juice industry. Maltodextrin addition reduced the powder addition rate by 40%, whereas fruit fiber addition reduced by 80%. The addition of such powders thus reduces the recirculation requirement and tremendously improves the process efficiency and capacity of new approach. The low-cost technology can be a platform for further investigation of new opportunities with respect to low value dairy streams and food industry co-products such as fibers i.e. mixing streams of different compositions to generate novel ingredients which contribute to the circular economy.

The lab scale model and the protocol to measure cohesive phase development can be a valuable tool for scale up, optimisation and assessing the feasibility of applying the novel process to other dairy and food ingredients. The effectiveness of this tool will be further investigated at a pilot scale.

This study was performed using reconstituted powders which was the most viable option from a logistical point of view. The results are encouraging because they tracked the previous study (Tanguy et al., 2017), providing new insights into superconcentration and granulation at a lab scale. Therefore, further studies are being undertaken to test the lab scale protocol using fresh liquid ingredients as a starting material. It should also be stated the drying process utilised here (vacuum oven drying) differed significantly to the pilot/ industrial scale process (as described in section 2.3). The effect of drying on the final product properties will be assessed during large scale (100 kg.h^{-1}) pilot plant trials, incorporating the findings achieved using the lab scale protocol.

5. Conclusion

A reliable protocol was developed to characterize the behavior of superconcentrates of dairy ingredients. It was shown that measuring power consumption as a function of DM content can be used to identify a cohesive phase, which appeared for each of the ingredient studied. The exact onset and extent of the cohesive phase was, however, composition dependent and points to different behavior of ingredients as a function of composition.

Mapping of the cohesive phase is particularly useful in superconcentration-granulation based processes. In the first instance it provides information on the limits of superconcentration achievable for a given composition. This data may be inferred from flow rheology; however, viscosity increases exponentially in such analyses at DM contents between 5 and 20% lower than onset of the cohesive phase, which in this study was deemed to be the upper limit of superconcentration. Furthermore, the width and the endpoint of cohesive region gives useable information on the extent of powder mixing required to form discrete powder granules, which is the ultimate goal of the process. The data generated can be used to give an indication of the potential energy reduction associated with this innovative process.

Furthermore, granulated powders produced at a lab scale model were characterized and compared with standard spray-dried powders. While the powders were dried in a different manner (vacuum oven drying) to the industrial protocol, the results point to some interesting conclusions. Firstly, similar to observations by Tanguy et al. (2017), DWP and WPP produced powders with excellent rehydration and flow properties, which was in contrast to SMP and FFMP powders which both behaved poorly upon rehydration.

The identification of limits of superconcentration and granulation as a function of DM content and composition give strong indications on the potential in-process behavior of different dairy streams. In particular, the relatively low drying energy reduction associated with SMP and FFMP in comparison to DWP and WPP, coupled with poor rehydration properties, point to potential limitations of the process to handle higher protein and/or fat containing powders. While the results presented here provide useful indications of behavior and, in the case of WPP align with other pilot-scale studies, future studies are planned to determine the transferability of the lab-based protocol to an industrial scale.

Declaration of Competing Interest

No conflicts of interest.

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