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Impact of wet-mix total solids content and heat treatment on physicochemical and techno-functional properties of infant milk formula powders

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ABSTRACT

This paper investigated the effects of heat treatment (75 °C × 18 s and 100 °C × 18 s) and wet-mix total solids level (TS: 50 and 60%, *w*/w) on the physicochemical and techno-functional properties of model infant milk formula (IMF) powders. IMF produced from wet-mixes with 50% TS preheated at 75 °C (50%-75°C) exhibited the longest wettability time (55 ± 2 s) and the poorest flowability, explained by the small particle size (D [4;3]= 16.5 ± 2.29 μm) and low poured bulk density (0.27 ± 0.02 g/cm^3). Larger particles were obtained by increasing both pasteurization temperature and TS. Further, powders from 60% TS wet-mixes showed less particle size uniformity, leading to better packing and higher bulk densities. 50%-75°C powders also showed the lowest onset glass transition temperature, which may affect its storage stability. Wettability time was reduced by increasing TS from 50 to 60% or by increasing pre-heating temperature from 75 to 100 °C. However, as observed by low-field nuclear magnetic resonance, the increase in the pasteurization temperature slowed down the global rehydration process. The flowing properties of the powders improved by increasing TS level of the wet-mix. In conclusion, the pre-spray drying wet-mix processing variables, pasteurization temperature and TS level, had a major effect on the physicochemical and functional properties of the IMF powders. It is crucial to understand how variations in the process parameters affect these powder characteristics, due to their functional, technological and economic importance.

1. Introduction

Infant milk formulae (IMF) are substitutes of human milk designed to provide all the nutritional requirements of infants during the first months of life [1]. The global infant nutrition market represented USD 34.2 billion in 2020 and is projected to continue growing. Reasons behind the market growth include a rise in the number of working mothers and the expansion of the middle class in the emerging and developing countries [2].

Commercial IMF are usually in spray-dried powder form, due to their long shelf life and convenient transport and storage. IMF powders are commonly produced by wet-mix processing. Briefly, the wet-mix is prepared by dissolution of powder ingredients in water or skim milk and addition of oils. Subsequently, the mix is heat treated, homogenized and concentrated by evaporation prior to spray-drying [3]. Due to their complex composition, IMF are susceptible to nutrient interaction during processing and storage, which impacts directly the quality of the final powder [4]. Glass transition is an important property for quality and storage stability. Powders with low glass transition temperatures (Tg) are prone to stickiness, caking and crystallization [5]. IMF are rich in lactose, which, below Tg, is in amorphous glass form, with high internal viscosity and limited molecular mobility. During heating, lactose changes from a glassy state to a viscoelastic state with greater molecular mobility and less stability. Tg depends on IMF compositional variables, such as presence of maltodextrin, galacto-oligosaccharides (GOS) and fructo-oligosaccharides (FOS) or protein concentration [6–9]. Furthermore, Masum et al. [10] reported that increasing the spray-drying inlet air temperature from 180 °C to 200 °C significantly ($P < 0.05$) decreased Tg of IMF powders.

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Powder flowability is a technological challenge for the industry as free flowing powders are required during handling and transport in pipes and hoppers. Flowing properties of powders depend on a number of factors, such as particle cohesion, which in turn is affected by environmental conditions (temperature, relative humidity), type of stress applied and physicochemical characteristics of the powder (particle size and shape, density, fat and water surface content, Tg) [11,12]. At high temperature or relative humidity, glass transition occurs leading to stickiness and caking and thus impairing flowability [5]. Surface free fat also has a detrimental effect on flowability by rendering the particles sticky creating bridges between them [13]. Likewise, particle size affects the flowing properties, since smaller particles are more susceptible to sticking due to the larger surface areas which promote interaction between particles [14,15].

Ability to rehydrate is an essential functional property of IMF powders. Formulae should be easily reconstituted to avoid organoleptic rejection and reduced nutritional contribution related to incomplete solubilization. The powder rehydration process is often described through a number of consecutive, and sometimes overlapped, stages. First, the powder particles get in contact with the liquid and surrounded by it, leading to the sinking of the particles below the surface. Then, the powder agglomerates are disaggregated throughout the bulk liquid and, finally, the individual particles are dissolved forming a solution [16]. The chemical and surface composition of the food powders, as well as their physical characteristics (particle size and shape, density, porosity), affect the rehydration properties [17–21].

Physicochemical and techno-functional properties of dairy powders depend on both compositional and processing variables [22]. Previous studies have mainly focused on the effect of the IMF chemical composition [7–9,22–24]. In addition, the impacts of the spray-drying conditions on the properties of IMF and other dairy powders have been reported [10,19,21,25]. By contrast, less attention has been paid to the impact of pre-spray drying processing conditions.

Rodríguez Arzuaga et al. [26] applied two thermal treatments (75 °C and 100 °C for 18 s) to IMF wet-mixes with two different total solids (TS) levels (50 and 60%), and observed significant differences on the level of whey protein denaturation, viscosity and oil droplet size distribution of the wet-mix at the feed of the spray dryer. These physicochemical changes on the wet-mix are expected to impact the properties of IMF powders.

Thus, the objective of the present work was to determine the effect of two wet-mix processing variables: TS level (50 and 60%) and heat treatment (75 °C – 18 s and 100 °C – 18 s), on the physicochemical (particle size and morphology, color, density, Tg) and techno-functional (rehydration, flowability) properties of spray-dried model IMF.

2. Materials and methods

2.1. Materials and preparation

IMF powders (whey protein-to-casein ratio = 60:40) were produced by the wet-mix method at a pilot scale (15 kg batch), from low-heat skim milk powder, whey protein isolate, lactose, sunflower oil, GOS and FOS, as detailed elsewhere [26]. The variable processing conditions were: TS in the wet mix: 50 and 60% (*w*/w) and thermal treatment: 75 °C - 18 s and 100 °C - 18 s. Each powder was produced in duplicate on two separate days (Table 1).

Table 1
Experimental design.

Processing condition	Total solids in wet mix (%, *w*/w)	Heat treatment
50%-75°C	50	75 °C – 18 s
50%-100°C	50	100 °C – 18 s
60%-75°C	60	75 °C – 18 s
60%-100°C	60	100 °C – 18 s

2.2. Composition, surface free fat and water activity

Moisture content was determined by oven drying at 102 °C until constant weight [27]. Protein content was determined by Kjeldahl [28] with a nitrogen conversion factor of 6.25, as suggested by the European regulation [29]. Fat content was determined by Röse Gottlieb method [30] and lactose content according to AOAC International [31]. Ash content was determined gravimetrically after overnight incineration at 525 °C [32]. Oligosaccharides content (GOS + FOS) was estimated by difference. Surface free fat was quantified by GEA Niro Method No. A10a [33] and expressed as a percentage of powder weight.

Water activity (a_w) was measured at 20 °C using an Aqualab Series 3 TE (Decagon Devices Inc., USA). All determinations were performed in triplicates except for surface free fat that was determined in duplicate.

2.3. Color

Color, as indicated by CIELAB values (L*: black/white, a*: red/green and b*: yellow/blue), was measured using a BYK-Gardner Color Guide 45/0 (BYK-Gardner CB-6692, Gerestried, Germany), calibrated with the standard white tile. Three portions of sample were taken and six measurements were made in different areas of each portion.

2.4. Particle size distribution

Particle size distribution of the powders was measured by laser light diffraction using a Mastersizer 3000 (Malvern Instruments Ltd., Worcestershire, UK) with the dry powder disperser cell (Aero S). Mastersizer 3000 is equipped with a He–Ne laser ($\lambda = 632.8$ nm), a LED light ($\lambda = 470$ nm) and a reverse Fourier lens arrangement. The refractive and absorption indexes were 1.46 and 0.01, respectively [34]. Measurements were made in triplicates.

2.5. Scanning electron microscopy

The surface morphology of the powders was evaluated by scanning electron microscopy (SEM). Powder samples were set on double-sided adhesive carbon tabs, fixed to SEM stubs and coated with gold/palladium for 60 s prior to analysis in a Quanta 200 scanning electron microscope (FEI Company, Hillsboro, OR, USA), equipped with a backscattered electron detector.

2.6. Particle density

Particle density of the samples was determined in duplicates according to GEA Niro Method No. A11a [35] using the AccuPyc 1330 gas pycnometer (Micromeritics Instruments Corporation, Norcross, GA, USA).

2.7. Glass transition temperature

One gram of each sample was weighed in a dish and placed uncovered in a vacuum desiccator containing a saturated solution of CH_3COOK in a room at 20 °C for 7 days (HR approx. 23%). The glass transition temperature (Tg) was determined in triplicates using a differential scanning calorimetery (DSC 1, Star^e System, Mettler Toledo, Switzerland), equipped with a FRS5 ceramic sensor. The DSC was calibrated with indium. After equilibration, samples (10 ± 1 mg) were scanned in hermetically sealed 40-μL aluminum pans using an empty pan as reference. Samples were first heated from 0 to 100 °C at a rate of 5 °C min^{-1}, followed by cooling to 0 °C at a rate of 10 °C min^{-1}, and then heated until 140 °C at a 5 °C min^{-1} rate The Tg was determined in the final heating step [23].

2.8. Rehydration properties

For all the determinations, 12.5 g of powder and 100 mL of deionized water at 40 °C were used, to resemble domestic reconstitution conditions. Wettability [36], dispersibility [37] and solubility index [38] were determined by GEA Niro Analytical methods. Solubility index was calculated as 100 – insolubility index. Determinations were carried out at least in triplicate.

2.9. Rehydration kinetics

The rehydration kinetics of the samples were monitored by 1H low-field nuclear magnetic resonance (LF-NMR) spectroscopy using MQR Spectro-P spectrometer (Oxford Instruments, Oxfordshire, UK) operating at 20 MHz for 1 h. Each sample was reconstituted with distilled water (12.5% *w*/w) into an NMR tube (18 mm o.d.) and data were recorded each minute during mixing (30 rpm) for 40 min. Data were analyzed using the Carr-Purcell-Meiboom-Gill (CPMG) sequence at 25 ± 1.0 °C. The parameters of CPMG were set as follow: recycle delay of 8 s; τ-delay of 400 μs and 1 scan. Data from 8000 echoes were acquired with a receiver gain of 5.0. Transverse relaxation times (T_{2n}) and relative populations (Mn) of different relaxation components were obtained using an in-house MATLAB (version R2019a, The Math-Works, Natick, MA, USA) script designed for fitting the relaxation curves to a sum of exponential decays according to Eq. 1.

$$I(t) = \sum_{n=1}^{N} Mn * e^{-t/T2n} \quad (1)$$

Where: I(t) corresponds to the echo intensity as a function of time, N is the number of relaxation components, the transverse relaxation time for site n is T_{2n}, and the corresponding abundance of site n is Mn. Measurements were conducted in triplicate.

2.10. Flowing properties

The flow properties of the IMF powders were determined with a powder flow tester (PFT) from Brookfield Engineering Laboratories Inc. (Middleboro, MA, USA). PFT complies with the test procedure ASTM D6128 using the annular and Jenike's shear test techniques [39]. The powder flow function is a plot of the unconfined failure strength versus the major principal consolidation stress. A vane lid was used in the PFT measurements of the flow functions of the powders. The flow function tests were undertaken using the 38 cm^3 volume shear cell and running the standard flow function test program. Each IMF powder sample was carefully loaded by sieving procedure in the shear cell of the equipment aiming to the minimum compaction during sample preparation. The program measures the flow properties over the range of five major principal consolidation stresses in a geometric progression that generates values of circa 1.6, 3.3, 6.9, 14.0, and 28.2 kPa. PFT was connected to a computer provided with Powder Flow Pro V1.2 software. A flat wall friction lid was used in the measurement of the bulk density of the powders. The bulk density tests were undertaken using the 38 cm^3 volume shear cell and running the standard bulk density test program. This program measures the densities over the range of five major principal consolidation stresses in a geometric progression that generates values similar with the flow function tests. Flow function and bulk densities represent the average values of three independent measurements. The ratio of the major principal consolidation stress σ_1 and unconfined yield strength σ_c defines the flow factor index (ff_c), according to Eq. 2. The flowability of the powder was classified according to ff_c: non-flowing, $ff_c < 1$; very cohesive, $1 < ff_c < 2$; cohesive, $2 < ff_c < 4$; easy-flowing, $4 < ff_c < 10$; and free flowing, $10 < ff_c$ [39,40]. All measurements were carried out under ambient conditions (temperature 21 ± 1 °C, relative humidity 48.8 ± 0.3%).

$$ff_c = \frac{\sigma_1}{\sigma_c} \quad (2)$$

2.11. Statistical analysis

Analysis of variance (ANOVA) was performed by means of JMP® 12.1.0 software (SAS Institute Inc., NC, USA). Tukey's test was employed with a level of confidence of $P < 0.05$.

3. Results

3.1. Physicochemical properties of IMF powders

The proximate composition and water activity (a_w) of the IMF powders did not vary ($P > 0.05$) among samples produced under different processing conditions (Table 2). Protein, fat, lactose and oligosaccharides contents were in compliance with the European legislation [29]. Moisture content was below 3% and a_w below 0.2 for all samples, as recommended to maximize stability [41,42].

Surface free fat ranged between 0.42 and 1.36 g free fat 100 g^{-1} powder, in agreement with values reported for commercial IMF [18,43]. Despite powders produced from a wet-mix with 50% TS heated at 75 °C seemed to have the highest surface free fat content, the difference was not statistically significant ($P > 0.05$) (Table 2).

Particle size of the IMF powders was affected by both heat treatment temperature and TS in the wet-mix. 50%-75°C sample had the smallest mean particle size, followed by 50%-100°C and 60%-75°C, whereas the largest mean particle size was obtained for 60%-100°C IMF (Table 3). Powders produced from 50% TS wet-mixes had the narrowest particle size distributions (Fig. 1). Moreover, while samples preheated at 75 °C had monomodal profiles, a second population at values >100 μm was observed for powders preheated at 100 °C, which was more evident for IMF 50%-100°C due to the higher volume of large particles obtained for 60%-100°C. No significant differences ($P > 0.05$) were found among the color values (i.e., L*, a* and b*) of the samples (Table 3).

Scanning electron microscopy (SEM) technique was used to study the surface morphology of the powders. In general, all IMF powders presented agglomerated particles with spherical regular shape and smooth surface with smaller particles attached to them (Fig. 2). Particle size differences among samples obtained by laser diffraction (Table 3) were evident at the microscope. Although a range of particle sizes can be observed in the micrographs, 60%-100°C presented the largest particles, followed by 60%-75°C and 50%-100°C, while particles of 50%-75°C were in general the smallest. Besides the increase of primary particle size with TS and temperature, the extent of spontaneous particle agglomeration, observed by small particles adhering to larger particles, also seemed to increase with the mean particle size (60%-100°C > 60%-75°C > 50%-100°C > 50%-75°C). Presence of broken particles was observed in 60%-100°C and, to a lesser extent, in 60%-75°C particles, exposing the porous interior, as indicated in Fig. 2. No sharp lactose crystals were observed for any of the samples.

Particle density did not vary ($P > 0.05$) among samples and ranged 1.24–1.27 g/cm^3 (Table 4), in agreement with values previously reported for IMF [44]. Poured bulk density significantly ($P < 0.05$) varied with TS: powders produced from wet-mixes with 60% TS had higher bulk density than those produced from 50% TS wet-mixes (Table 4). On the other hand, the pasteurization temperature did not have a significant effect on the poured bulk density ($P > 0.05$).

Tg of the IMF powders were within the range of Tg values previously reported for infant formula at similar a_w conditions (Table 4) [9,23]. Sample 50%-75°C had the lowest Tg onset, while there were no significant differences (P > 0.05) in Tg onset between 50%-100°C, 60%-75°C and 60%-100°C powders, which ranged 50.2–51.0 °C. Further, Tg midpoint was significantly lower for 50%-75°C than 50%-100°C (Table 4).

Table 2
Composition, surface free fat and water activity of infant milk formula powders.[a]

Processing condition	Moisture (%, w/w)	Protein (%, w/w)	Fat (%, w/w)	Lactose (%, w/w)	Oligosaccharides (%, w/w)	Ash (%, w/w)	Surface free fat (%, w/w of total powder)	Water activity
50%-75°C	0.85 ± 0.14	11.0 ± 0.19	25.8 ± 0.49	56.8 ± 1.34	3.85 ± 0.90	1.65 ± 0.01	1.36 ± 0.51	0.09 ± 0.00
50%-100°C	1.31 ± 0.66	11.3 ± 0.30	24.7 ± 3.11	57.0 ± 2.55	4.02 ± 0.24	1.65 ± 0.03	0.71 ± 0.02	0.11 ± 0.06
60%-75°C	0.88 ± 0.14	11.0 ± 0.01	28.5 ± 1.39	53.4 ± 1.34	4.56 ± 0.04	1.73 ± 0.06	0.62 ± 0.07	0.10 ± 0.03
60%-100°C	1.20 ± 0.16	11.4 ± 0.33	27.7 ± 0.42	54.0 ± 0.42	4.16 ± 0.49	1.61 ± 0.00	0.42 ± 0.19	0.13 ± 0.03

[a] Values are means ± standard deviation from replicate trials. There were no significant differences ($P > 0.05$) between processing conditions in any of the parameters.

3.2. Rehydration properties of IMF powders

All powders presented high wettability (< 30 s) except for 50%-75°C, which had a significantly longer wetting time ($P < 0.05$) and was wettable (wettability <60 s) (Fig. 3.A) [32]. On the other hand, this powder was more dispersible than powders produced from wet-mixes with 60% TS. No significant differences ($P > 0.05$) between the solubility indexes of the powders (Fig. 3.B) were observed.

IMF powders were studied by LF-NMR based on the strength of water binding during rehydration. Transverse relaxation times (T_{21}, T_{22}) after bi-exponential fitting of the CPMG curves are shown in Fig. 4. Two proton populations were observed during rehydration, the water fraction bound to proteins was characterized by transverse relaxation time (T_{21}), whereas the second relaxation component (T_{22}) corresponded to the free water. An increase in the T_{21} was observed for all samples upon rehydration. On the other hand, the amount of free water decreased over time indicating interactions with the protein demonstrating the rehydration kinetics. The increase in pasteurization temperature (75 to 100 °C) led to a slower rehydration as showed by a slower decay on T_{22}. Likewise, the dry matter content affected the rehydration kinetic when pasteurized at 75 °C, i.e., the increase in TS from 50 to 60% led to a slower rehydration. However, no difference was not found for samples pasteurized at 100 °C.

3.3. Flowing properties of IMF powders

Fig. 5 gives the flow function plots of the IMF powders at different levels of consolidating stress. The flow function gives the unconfined yield strength of the powder as a function of major principal consolidation stress and represents the strength developed within a powder when consolidated, which must be overcome to make the powder flow [45,46]. A flat curve indicates an easily flowing material, while a steep curve indicates higher flowing difficulties [47]. Both IMF powders produced from mixes with 50% TS presented flow functions in the very cohesive area (Fig. 5). Although there were small differences between the 50% TS powders heated at 75 and 100 °C, both lied in the very cohesive area under the different applied major principal consolidation stresses. At low levels of major principal consolidation stress all the IMF powders behaved as very cohesive while over the stress threshold of 4 kPa the 60% TS samples passed to the cohesive zone of the plot revealing better flow properties than the 50% TS samples. All the powders showed loss of strength at the highest consolidation level, therefore the maximum test stress had to be lowered.

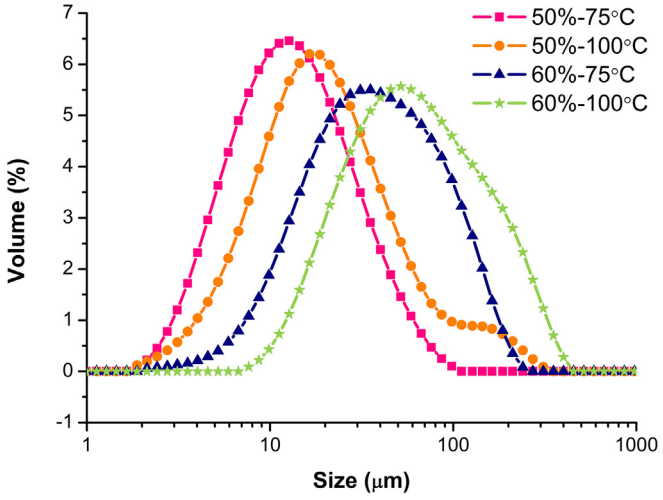

Fig. 1. Particle size distributions of infant milk formula powders obtained under the different processing conditions.

4. Discussion

The effects of the TS level and heat treatment of the wet-mix on the physicochemical and techno-functional properties of IMF powders were assessed. Table 5 summarizes the main significant changes observed on the powder properties.

Bulk composition, surface free fat, a_w, particle density and color of the samples were not significantly ($P > 0.05$) affected by the studied processing conditions. On the other hand, particle size, morphology, Tg and bulk density of IMF powders were significantly affected by the pasteurization temperature and the TS level of the wet-mix.

Surface free fat is an important parameter for dairy powders, negatively affecting their wettability, flowability and organoleptic properties [13,19,43]. Emulsion stability of the wet-mix, spray-drying temperature and lactose state have been shown to affect the surface free fat content [19,48,49]. Although we previously reported improved emulsion stability in the wet-mixes heated at 100 °C [26], the differences in the surface free fat contents were found to be not significant ($P > 0.05$), due to large sample variability.

Powder particle size is correlated with the size of the droplets created during atomization at the spray dryer, which is in turn directly

Table 3
Instrumental color parameters and particle size distribution of infant milk formula powders.[1]

Processing condition	Instrumental color parameters			Particle size distribution			
	L*	a*	b*	D[4;3] (μm)	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)
50%-75°C	96.1 ± 1.72[a]	−0.41 ± 0.43[a]	4.68 ± 0.71[a]	16.5 ± 2.29[a]	4.90 ± 0.60[a]	12.4 ± 1.81[a]	33.6 ± 4.83[a]
50%-100°C	96.1 ± 0.50[a]	−0.78 ± 0.22[a]	5.80 ± 0.70[a]	29.5 ± 3.49[b]	8.85 ± 2.67[ab]	21.2 ± 2.24[b]	57.7 ± 13.2[a]
60%-75°C	95.80 ± 0.44[a]	−0.53 ± 0.06[a]	7.05 ± 0.19[a]	55.5 ± 0.45[c]	13.5 ± 0.57[c]	38.9 ± 0.97[c]	120 ± 0.47[b]
60%-100°C	94.23 ± 3.21[a]	−1.14 ± 0.63[a]	7.86 ± 1.74[a]	93.7 ± 2.72[d]	22.1 ± 0.16[d]	63.2 ± 0.17[d]	214 ± 9.61[c]

[1] Values are means ± standard deviation from replicate trials. Values within a column not sharing a common superscript letter differ significantly ($P < 0.05$).

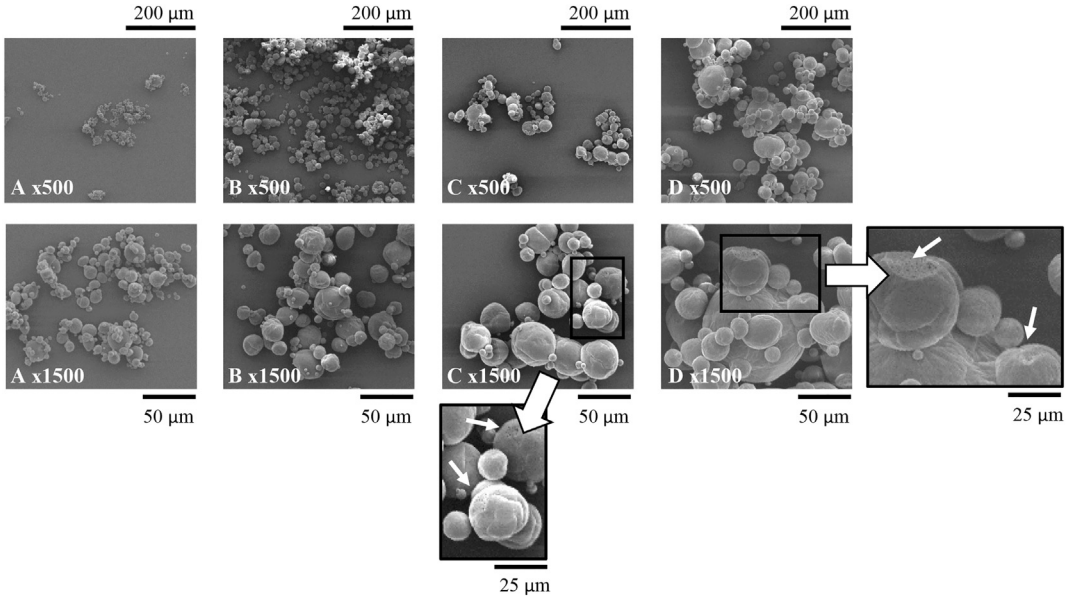

Fig. 2. Scanning electron micrographs of infant milk formula powder powders obtained under the different processing conditions (A: 50%-75°C; B: 50%-100°C; C: 60%-75°C; D: 60%-100°C) at 500× and 1500× magnification. Extract of micrographs C × 1500 and D × 1500 were enlarged and white arrows were used to indicate the presence of pores.

Table 4
Glass transition temperature (Tg), particle density and poured bulk density of infant milk formula powders.

Processing condition	Tg (°C)[1]		Particle density (g/cm^3)	Poured bulk density (g/cm^3)
	Onset	Mid-point		
50%-75°C	46.4 ± 1.36^a	51.0 ± 1.45^a	1.24 ± 0.00^a	0.27 ± 0.02^a
50%-100°C	50.9 ± 1.44^b	55.6 ± 1.05^b	1.27 ± 0.02^a	0.28 ± 0.01^a
60%-75°C	50.2 ± 1.12^b	54.4 ± 0.82^{ab}	1.24 ± 0.01^a	0.35 ± 0.03^b
60%-100°C	51.0 ± 0.16^b	55.0 ± 0.31^{ab}	1.25 ± 0.01^a	0.38 ± 0.01^b

Values are means ± standard deviation from replicate trials. Values within a column not sharing a common superscript letter differ significantly ($P < 0.05$).
[1] Glass transition (Tg) as determined for powders with $a_w = 0.241 \pm 0.00$.

related to the viscosity of the feed concentrate, at constant feed rate and atomization design [50]. Apparent viscosities of the wet-mixes at the feed of the spray dryer were 7.7 ± 0.2 mPa.s, 17.6 ± 0.5 mPa.s, 49.5 ± 1.9 mPa.s, and 95.9 ± 1.0 mPa.s, respectively for 50%-75°C, 50%-100°C, 60%-75°C and 60%-100°C, as reported in our previous study [26]. Indeed, in the current study a positive correlation between feed viscosity and mean particle size was obtained. Heat treatment and TS level of the wet-mix affected its viscosity and, thus, the powder particle size. Increasing pasteurization temperature from 75 to 100 °C and/or TS level from 50 to 60%, produced an increase in the mean particle size as determined by laser light diffraction (Table 3) and observed by SEM (Fig. 2). Moreover, a second population of particles with a mean particle size of

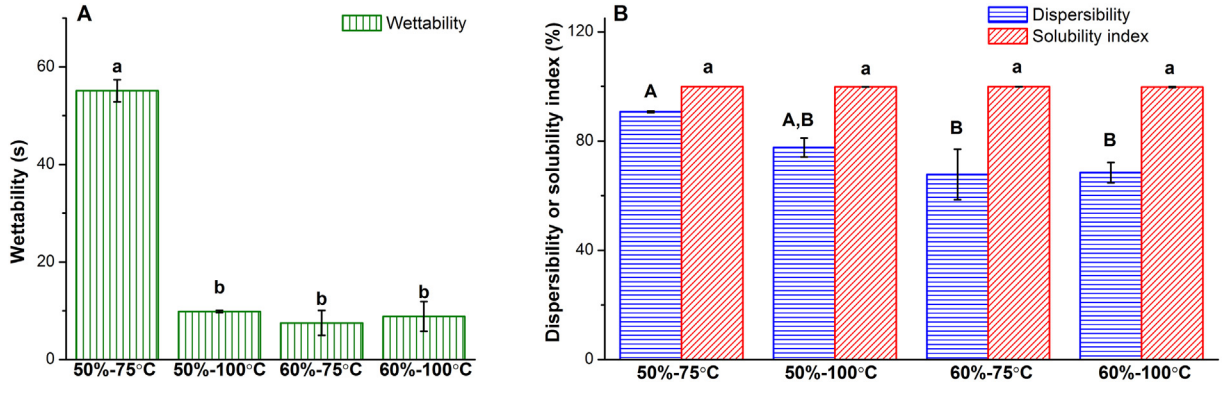

Fig. 3. Wettability (A), dispersibility and solubility index (B) of infant milk formula powders obtained under different processing conditions.
Bars represent standard deviation.
A: Columns not sharing lower-case letters have significantly different ($P < 0.05$) wettabilities.
B: Columns not sharing upper-case letters have significantly different ($P < 0.05$) dispersibilities. Columns not sharing lower-case letters have significantly different ($P < 0.05$) solubility indexes.

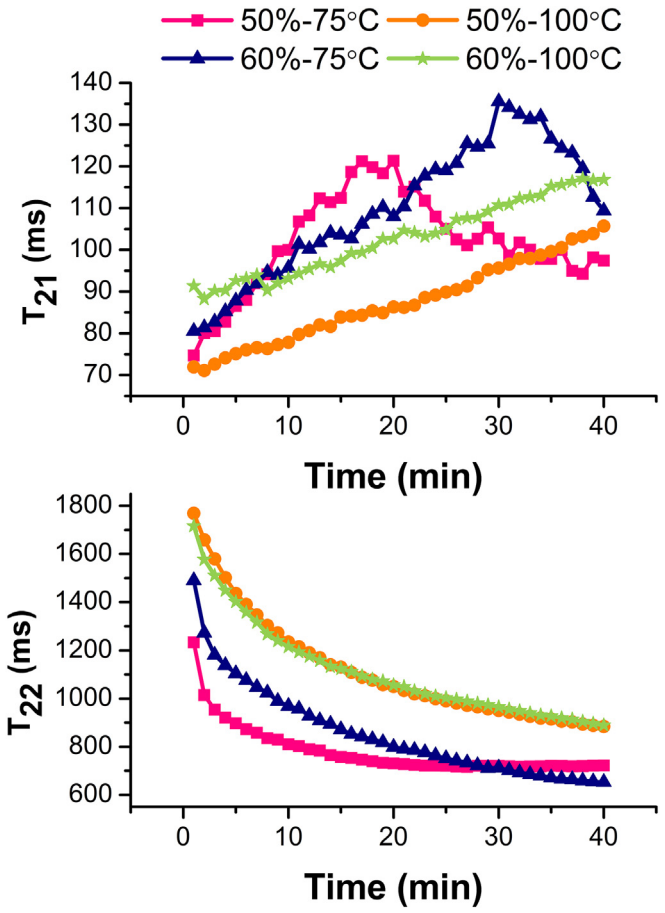

Fig. 4. Relaxation time (ms) of bound (T_{21}) and free (T_{22}) water fractions obtained by LF-NR during rehydration of infant milk formula powders obtained under different processing conditions.

ca. 200 μm was detected in powders preheated at 100 °C (Fig. 1), which is likely due to protein aggregation [51]. The increase in viscosity also enhanced the cohesive interactions between particles during spray-drying, increasing the extent of particle agglomeration, as observed in the SEM micrographs (Fig. 2) [34].

As the particle size decreases the surface area increases allowing more reflection of light, which may affect the color of powder [52].

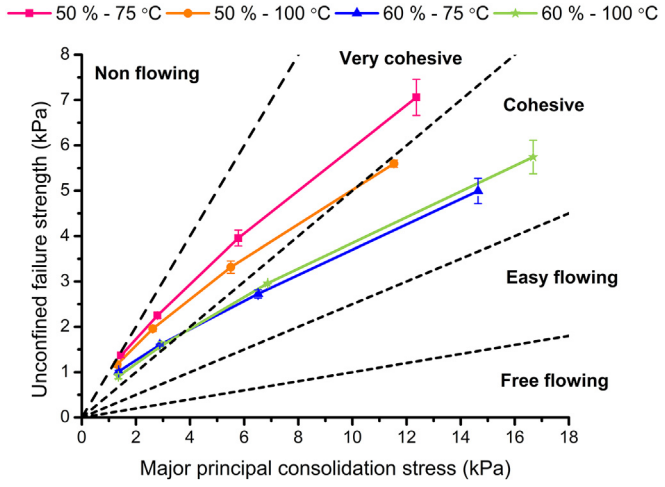

Fig. 5. Flow function plots of the infant milk formula powders at different levels of major principal consolidating stress.

Table 5
Summary of main effects of different processing conditions on physicochemical and techno-functional properties of infant milk formula powders.

Property	Processing condition			
	50%-75°C	50%-100°C	60%-75°C	60%-100°C
Surface free fat	−	−	−	−
Mean particle size	−	+	++	+++
Color (L*, a*, b*)	−	−	−	−
Particle density	−	−	−	−
Poured bulk density	−	−	+	+
Tg onset	−	+	+	+
Tg mid-point	−	+	−/+	−/+
Wettability time	+	−	−	−
Dispersibility	+	−/+	−	−
Solubility	−	−	−	−
Rehydration up to 20 min	++	−	+	−
Flowability (> 4 kPa)	−	−	+	+

+++ > ++ > + > −. −/+ means no increase respect to – and + .

However, no significant differences ($P > 0.05$) in the instrumental color parameters were found among samples (Table 3). Hence, it could be reasoned that the apparent color differences generated by particle size were contrasted by other changes in color, such as browning. In a previous work, we reported that powders preheated at 100 °C had a significant lower content of available lysine than those preheated at 75 °C, indicating development of Maillard reaction at early stages [26]. At advanced stages of Maillard reaction formation of colored melanoidins may affect the color of the powder [53]. Nevertheless, sample 60%-100°C, which was preheated at 100 °C and had the largest particle size (Table 3), did not show a significantly darker color than the rest of the powders, indicating that advanced stages of Maillard were not reached during processing. Even though darkening was not detected in the fresh powders, samples preheated at 100 °C, which had a significantly lower content of available lysine, may develop melanoidins during storage earlier than samples preheated at 75 °C.

Bulk density is a key property from an economical, technological and commercial point of view. High densities influence mass packaging and transport and storage costs, and improve rehydration properties [32]. Moreover, they play a particularly important role in IMF since dosage of the powder is made volumetrically in domestic conditions. Samples produced from 50% TS wet-mixes exhibited a significantly ($P < 0.05$) lower poured bulk density than samples produced from 60% TS wet-mixes (Table 4). These differences are not related to the particle density that was shown to be the same for all the powders (Table 4). It has been reported that increasing the TS level of the concentrate increases the bulk density of the powder [32]. In addition, as shown in Fig. 1, powders produced from 60% TS wet-mixes resulted in wider particle size distributions. The heterogeneity in the particle sizes favored a closer packing which increased the bulk density [32]. The pasteurization temperature did not affect ($P > 0.05$) the density of the powders. The denaturation of whey proteins has been shown to enhance their foaming properties, which could have led to an increased level of air incorporation and a reduced density. However, at high levels of denaturation the increase in the viscosity of the continuous phase makes difficult the incorporation of air bubbles [54].

Transition temperatures of dairy solids and their associated physicochemical changes are usually influenced by low molecular weight amorphous carbohydrates rather than polysaccharides and proteins, that have higher Tg values and are more stable [5,55]. In the present study, Tg is mainly related to lactose transition (concentration > 50% (*w*/w), Table 2). When powders containing amorphous components, such as lactose, are exposed to temperatures above their Tg, molecular mobility increases resulting in stickiness and caking [56]. Hence, high Tg are desirable in order to reduce risks during transportation or storage, where elevated temperatures may occur. Sample 50%-75°C had the lowest Tg onset (Table 4). Consequently, a significant increment in

the Tg onset (or delay in the start of the glass transition) was obtained by increasing the TS level of the wet-mix from 50 to 60% and/or increasing the pasteurization temperature from 75 to 100 °C. The higher Tg obtained for the 50%-100°C, 60%-75°C and 60%-100°C compared to 50%-75°C, can be explained by interactions between large protein molecules and lactose [57]. Those interactions could have been promoted by protein unfolding at high heat treatment temperature (i.e. 100 °C) as demonstrated early [26] and/or by higher proximity between molecules due to higher total solids level (i.e. 60%).

The 50% TS wet-mix pasteurized at 75 °C resulted in IMF powders (50%-75°C) with the smallest particle size, the lowest bulk density and a slightly higher, though not significant, surface free fat content. As a result, 50%-75°C showed the poorest wettability (Fig. 3.A). Powders with high concentration of hydrophobic components, such as lipids, at the particles' surface have poor wetting properties [19]. Similarly, powders with small particle size usually have poorer wettability than powders with larger particle diameter where the surface-to-volume ratio is lower [58]. Finally, a lower bulk density also contributes to impair the wetting properties. At higher densities, the tendency for particles to sink increases, thus favoring rehydration [59]. As reported earlier, the degree of whey protein denaturation in IMF powders pasteurized at 75 °C was in average ~ 7%, regardless of the TS level, while for samples preheated at 100 °C a level of denaturation of ~ 82% was found [26]. Denatured proteins can reduce wettability, however, and in agreement with Gaiani et al. [19], in the present study the powders with the lowest wetting times also presented the highest level of denaturation, suggesting that other physicochemical properties exert a more important effect on wettability. On the other hand, wettability of 50%-100°C, 60%-75°C and 60%-100°C samples ranged 7–10 s (Fig. 3.A). As mentioned above, those samples had larger particle sizes. In addition, 60%-100°C particles were more agglomerated and presence of pores was observed on the particles of both 60%-100°C and 60%-75°C powders (Fig. 2). Agglomeration and porosity improve wettability of dairy powders [60,61]. By contrast, 50%-75°C sample showed better dispersibility than powders produced from 60% TS wet-mixes (Fig. 3.B), indicating that the small particle size did not impair the dispersibility. Ji et al. [60] explained that while wettability of dairy powders can be improved by increasing particle size through agglomeration, that is not the case for dispersibility, which is mainly controlled by the nature of the primary particles. Moreover, samples 60%-75°C and 60%-100°C, which had lower dispersibility than 50%-75°C, also had higher bulk density (Table 4). Chever et al. [58] found a negative correlation between wettability and dispersibility as well as for dispersibility and bulk density in whole milk/sugar mixture powders. Nevertheless, the solubility index did not vary significantly ($P > 0.05$) among samples and was >99.7% for all conditions, indicating that global rehydration was good for all IMF powders.

LF-NMR provided deeper insight of the effect of the heat treatment on the rehydration process. For IMF produced from 50% TS wet-mixes, increasing heat treatment temperature from 75 to 100 °C slowed the powder rehydration, as indicated by a slower increase in the bound water (T_{21}) and a slower decay in the free water (T_{22}) (Fig. 4). Such effect was not detected by the traditional methods (Fig. 3). It can be hypothesized that while the increased particle size in 50%-100°C compared to 50%-75°C (Fig. 1, Table 3) improved the wettability (Fig. 3.A), the presence of denatured and aggregated proteins in 50%-100°C slowed down the global rehydration process. In addition, for samples pasteurized at 75 °C the increase of TS from 50 to 60% slowed rehydration, while for samples pasteurized at 100 °C that effect was not evident. This may be due to the predominant effect of the protein denaturation and aggregation obtained for the samples heated at 100 °C, that prevented detecting differences related to the TS level. Fig. 4 shows a decay in the level of bound water (T_{21}) after approx. 20 and 30 min for samples 50%-75°C and 60%-75°C, respectively. Such effect was not detected for powders preheated at 100 °C. This result might also be related to the state of the proteins. Whey proteins in samples pasteurized at 75 °C, which were mainly in a native state, bind water rapidly (high slope in T_{21} curves), but, after a given time, the structure loses the bound water (Fig. 4). On the other hand, such behavior was not observed for samples preheated at 100 °C, which presented extensively denatured whey proteins, at least for the test duration (40 min).

Although flow function plots showed that powders also differed in their flowability (Fig. 5), all IMF behaved as cohesive or very cohesive indicating poor flowability, which can cause handling problems. Cohesive powders can create structures during storage in hoppers, that may limit or inhibit the flow and discharge from silos and may also affect transport through pipes by pneumatic conveying [62]. IMF samples produced from 50% TS were very cohesive while increasing TS to 60% produced a limited improvement on the flowability of the samples which behaved as cohesive at stresses >4 kPa. 60% TS had larger particle size (Fig. 1 and Table 3) and higher bulk density (Table 4) than 50% TS samples. In general, particle size and flow properties are correlated: the finer the powder is, the more cohesive behavior it has [45]. This correlation seemed to be in accordance with the results acquired for the flow properties of 50% TS and 60% TS. In order to flow, powder particles need to overcome the surface interactions between them. As the particle size decreases, the contact surface area per unit of mass of powder increases and, thus, the van der Waals forces attracting the particles are greater [63]. Furthermore, bulk density of powders can be an indicator of the flow properties since more cohesive powders have greater surface attractive forces that help them overcome gravity, so that particles can support themselves around void spaces. This creates greater voids within the bulk when first poured producing a greater poured volume (or lower poured bulk density) [63]. Further, the particle size of a powder has been also correlated with liquid bridges which can be built between particles and develop adhesive forces [39]. Those adhesive forces are higher for smaller particle size powders [39]. Therefore, the higher cohesivity in the 50% TS powders might also be related with the greater adhesive impact of the free surface fat liquid bridges due to their smaller particle size. Heat treatment temperature of 60% TS samples did not affect the flowability of the powders, while small differences in the flow plots were found between 50%-75°C and 50%-100°C. Likewise, 50%-75°C had a significantly lower Tg (Table 4) and, although no statistically significant, higher surface free fat content (Table 2). Increased presence of lipids at the particle surface impairs flowability by rendering the particles stickier [12]. In addition, powders with lower Tg are more susceptible to temperature and relative humidity changes, which lead to stickiness when Tg is overcome, and lactose goes from an amorphous to a crystalline state.

In summary, TS and pasteurization temperature affect not only energy consumption and wet-mix rheological and emulsifying properties as demonstrated early [26], but also physicochemical, rehydration and flowing properties of the infant formula resulting powders, which has relevance for the practical applications of these powders.

5. Conclusions

This study gave insight of the effects that pre spray-drying processing variables, such as TS and pasteurization temperature of the wet-mix, exert on the physicochemical and, thus, functional properties of infant milk formula powders.

Increasing the TS level of the wet-mix from 50 to 60% (*w*/w) resulted in powders with less uniform particle size and higher mean particle size, bulk density and Tg onset. Functionality of the powders was enhanced by increasing TS: IMF from 60% TS wet-mixes showed improved wettability and flowability. Additionally, increasing the pre-heating temperature of the wet-mix led to powders with higher mean particle size and Tg onset and reduced wettability time. However, the rehydration kinetics obtained by LF-NMR showed that increasing pasteurization temperature from 75 to 100 °C slowed down the global rehydration process.

Declaration of Competing Interest

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

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