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Influence of mechanical integrity during pneumatic conveying on the bulk handling and rehydration properties of agglomerated dairy powders

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ABSTRACT

Agglomerated powders are susceptible to breakdown on handling, most notably, during powder conveying. In this study, three agglomerated dairy powders (whey protein concentrate powder, WPC; fat-filled milk powder, FFMP and infant formula powder, IF) were conveyed through a custom-fabricated dispersion rig to understand the effects of agglomerate breakdown on dairy powder handling and application. All samples displayed significant breakdown on dispersion, evidenced by reducing particle size and increasing bulk density. The resulting flowability of these powders was impaired (flow index: WPC: 9.3 to 5.1, FFMP: 5.7 to 4.9 and IF: 16 to 10) *via* increased particle-particle interactions. The initial stages of rehydration were impeded by agglomerate breakage (42.9–47.0% wettability reduction and 7.22–16.4% dispersibility reduction), while powder solubility remained relatively unchanged. This study provides insights into the alterations of agglomerated dairy powder properties on agglomerate breakdown, while identifying the effects these alterations have on the functional properties of these powders.

1. Introduction

The production of dairy powders, *via* spray drying, concentrates the numerous nutritional and functional properties present in a dairy system in a solid form that allows for more economical storage and transport (Schuck, 2013). In order to fully utilise the functional and nutritional properties of these ingredients on further application, these powders need to be both easy to handle (i.e., possess good flowability) and to rehydrate. Certain dairy powder types display challenges with handling and rehydration, attributable mainly to their bulk composition and/or physical properties (Fitzpatrick et al., 2016, 2007) and for such powders, agglomeration may be utilised to overcome these challenges.

Powder agglomeration is a process of particle size enlargement that is routinely utilised in the production of spray dried dairy powders, whereby numerous individual primary particles are combined together into cluster-like structures where individual primary particles may still be distinguished (Cuq et al., 2013; Iveson et al., 2001). Agglomeration has been shown to alter the powder physical and bulk-handling properties, ultimately increasing their flowability, by reducing the extent of interparticle interactions (e.g., van der Waals forces) occurring between individual powder particles (Barkouti et al., 2013; Szulc and Lenart, 2013). The improvement of powder rehydration in the initial stages (i.e., wettability) on agglomeration is another, well established, beneficial effect of agglomerating dairy powders and is achieved mainly through increasing the volumes of interstitial air within the resulting powder (Gaiani et al., 2007; Ji et al., 2016).

The industry standard agglomeration process utilised commercially during the production of dairy powders occurs in the spray dryer main chamber (Pisecky, 2012); however, on exiting the spray dryer, transport of the agglomerated powder, to other locations in the processing plant *via* conveying lines is required for further handling, storage, packaging or application. During conveying, powder particles have the propensity to collide with other powder particles (i.e., attrition) and solid surfaces (i.e., collision) to an extent that is dependent on the type of conveying system used (i.e., dilute or dense phase). The attrition and collision forces experienced by the agglomerated particles can cause a breakdown in particle structure, which has the ability to negatively affect the bulk and functional properties of the powder.

Numerous studies have been completed in the area of pharmaceutical sciences to study the breakage of granulated powder material, including understanding the causes, mechanisms and means of measurement (Iveson et al., 2001; Reynolds et al., 2005; Subero; Ghadiri,

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2001). Granulation, although another form of powder particle size enlargement, forms particles that are very different to agglomerated dairy powder particles. These particles are usually large (~1 mm), spherical and dense, with high mechanical integrity (Faure et al., 2001). These properties contrast with those of agglomerated dairy powder particles, which generally have low mechanical integrity. Therefore, it proves challenging to extend any correlations between the breakdown of granulated pharmaceutical powders and agglomerated dairy powders.

This study was designed with the aim of identifying both the overarching mechanisms responsible for dairy powder agglomerate breakdown, as well as to assess the effects that breakdown has on selected key quality attributes of the resultant powders. Three representative agglomerated dairy powders were chosen (i.e., whey protein concentrate powder, fat filled milk powder and infant formula powder) which had diverse bulk composition, physical properties and intended applications in order to generate inter-relationships between these properties applicable to many different forms of agglomerated dairy powders. The powders were dispersed through a custom-fabricated dispersion rig with analysis before and after dispersion used to develop a deep understanding of the causes and effects of agglomerate breakdown, ultimately providing new insights to allow for greater control of the functional and physical properties of agglomerated dairy powders during conveying.

2. Material and methods

2.1. Materials and composition

Three commercially significant agglomerated dairy powders were used in this study. Agglomerated whey protein concentrate (WPC) and agglomerated fat-filled milk powder (FFMP) ingredients were kindly donated by Carbery Ingredients (Ballineen, Cork, Ireland) and Lakelands Dairies (Bailieborough, Cavan, Ireland) respectively, while a first-age, whey-dominant, infant formula (IF) powder was sourced from a local commercial outlet. Data for composition was provided by the suppliers of the WPC (protein: 80.0% and fat: 8.50%) and FFMP (protein: 26.4% and fat: 28.7%) ingredients and was calculated from the product packaging for the IF powder (protein: 8.52% and fat: 25.6%).

2.2. Powder dispersion process for agglomerate breakdown

A pressure dispersion rig was custom fabricated by Liam A. Barry Ltd. (Little Island, Cork, Ireland) to achieve controlled breakage of agglomerated dairy powders in a simulated lean phase pneumatic conveying configuration. The rig was composed of a compressed air source, an eductor (Fig. 1), a powder hopper and stainless steel and flexible tubing. Compressed air, at 1 and 3 bar, was passed through the rig, creating a venturi effect as the compressed air accelerated through the narrowing orifice of the eductor. The agglomerated powder was slowly poured into the powder hopper to avoid bridging or arching at the exit of the hopper and the venturi in the eductor component created a vacuum that drew the powder in the hopper downwards, into the compressed air stream. The powders, on exiting the eductor, entered stainless steel tubing (20 mm in length) where particle-particle and particle-wall collisions occurred, causing the agglomerated powder structures to break down further, before sample collection. On collection, the powders were transferred to, and stored in, two (double bagged) zip-lock, low density polyethylene bags (VWR, Belgium) at room temperature (22 °C) and protected from light until analysis. One kilogram of each powder was dispersed at 1 and 3 bar dispersion pressure yielding three samples for each agglomerated powder: a control, which had not been dispersed through the rig (C), and a 1 bar (1b) and 3 bar (3b) sample. Cleaning of the rig was completed by disassembling, washing (warm water) and drying (50 °C) the rig's components between each dispersion run to avoid cross-contamination between samples.

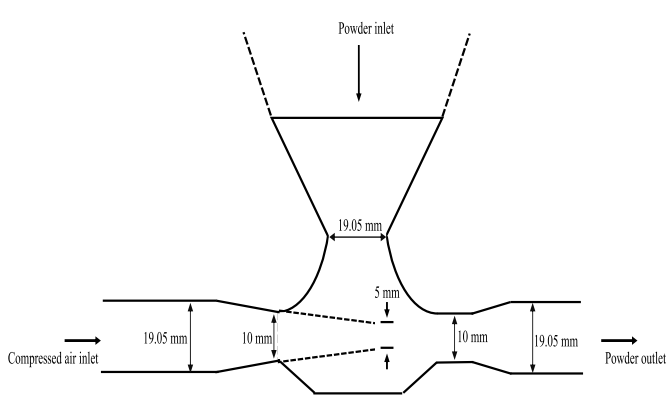

Fig. 1. Schematic representation of the eductor component of the custom-fabricated pressure dispersion unit.

2.3. Powder physical properties

2.3.1. Agglomerate size and morphological analysis

The particle size distribution (PSD) and specific surface area (SSA) analysis of the powders was measured using a Mastersizer 3000 equipped with an automated Aero S dry powder disperser cell (Malvern Instruments, Worcestershire, UK). The lowest possible dispersion pressure of 0.2 bar was used to minimise additional breakage on dispersion during all PSD analyses. Background and measurement durations of 20 s were used, and the particle refractive and absorption indices were set at 1.45 and 0.01, respectively. The particle size of the agglomerated dairy powders was reported as the volume-weighted median particle diameter (i.e., Dv50) and this data was used in later calculations such as agglomerate breakage.

The morphology of the powder samples was analysed *via* scanning electron microscopy (SEM) following a method described previously by Drapala et al. (2017). Magnifications varied from 150–400 X, due to the different particle sizes of the three different agglomerated powders.

2.4. Powder agglomerate breakage

The extent of agglomerate breakage following dispersion at 1 and 3 bar, as described in Section 3.2, was calculated for each sample following the method described by Schuck et al. (2012a), utilising the following equation:

$$\text{Agglomerate breakage } (\%) = \frac{\text{Ctrl d50 } (\mu m) - \text{d50 @ X bar } (\mu m)}{\text{Ctrl d50 } (\mu m)} \cdot 100$$

To quantify the fine material generated on agglomerate breakdown, the Dv10 value (i.e., the particle size value below which 10% of the material volume exists) was used as a threshold and all material smaller than this was quantified by calculating the area (utilising the Trapezoidal rule) under the volume distribution curve (from PSD analysis) for each sample, at each dispersion pressure.

2.5. Distribution of fat in powder particles

The surface free fat content of each powder sample was quantified post conveying using the GEA Niro analytical methods No. 10a (GEA Niro, 2006a) with minor modifications previously described by Schmidmeier et al. (2019). Confocal laser scanning microscopy (CLSM) was used to visualise the distribution of fat throughout the powder particles and to observe any changes occurring as a result of breakdown of agglomerated powders containing high bulk fat levels following a method previously described by Drapala et al. (2017).

2.6. Powder physical and bulk-handling properties

2.6.1. Particle density, bulk density, occluded and interstitial air

The particle density of the samples was measured using a Micromeritics Accupyc II 1340 gas pycnometer as described in GEA analytical methods No. 11a (GEA Niro, 2006b). The bulk and tapped density of the agglomerated powders were measured as per GEA method No. A2a (GEA Niro, 2006c) using a STAV 2003 Stampf-volumeter (J. Engelsmaan Apparatebau, Ludwigshafen, Germany) to assess tapped density. The volumes of interstitial and occluded air of the samples were then calculated using the method described by Schuck et al. (2012b).

2.6.2. Powder flowability

The flow index (i) of each sample was determined using a Brookfield Powder Flow Tester (PFT; Brookfield Engineering Laboratories Inc., Middleboro, MA, USA) using a method described previously by Crowley et al. (2014).

2.7. Rehydration properties

2.7.1. Wettability

Initially, the IDF wettability standard method 87:2014 (IDF, 2014) was used to assess the impact of agglomerate breakdown on the wetting properties of the powder samples. For powders that took longer than 60 min to wet (i.e., WPC and FFMP), a modification to the IDF standard procedure, described previously by Fitzpatrick et al. (2016), was employed. In brief, this modification includes carefully removing any powder particles that have not migrated below the water surface (i.e., have not been wetted) 60 min after powder addition. The un-wetted particles were transferred to a pre-weighed moisture dish before drying at 103 °C overnight, after which the samples were allowed to cool to room temperature in a desiccator before being weighed using an analytical balance. The weight of the un-wetted particles is used to determine the amount of wetted particles by difference from the starting weight. The mass of wetted particles is then used to determine wettability of each sample using the following calculation:

$$Wettability\ (\%) = \frac{mass\ of\ wetted\ particles\ (g)}{mass\ of\ inital\ powder\ (g)}.100$$

2.7.2. Dispersibility

A modified version of the IDF dispersibility method 87:2014 (IDF, 2014) was utilised whereby powder (10 g) was added to the surface of 250 ml of ultrapure water in an 80 mm diameter beaker. The solution was stirred for 30 s using a metal spatula, allowing one motion across the diameter of the beaker per s, after which, the beaker was left to stand for a further 30 s. The contents of the beaker were then passed through a 150 μm analytical sieve into a receiver beaker. The sieved solution (10 ml) was pipetted into pre-weighed moisture dishes and dried at 103 °C for 2 h. Following drying, the dishes were cooled in a desiccator and weighed to calculate total solids (TS) of the sieved material. This value was incorporated in a calculation as described in the IDF method to calculate the percent dispersibility of samples.

2.7.3. Solubility

To assess powder solubility, a 10% (w/w) powder suspension was formed by adding 30 g of sample to 270 g of ultrapure water (~22 °C) in a beaker of 80 mm internal diameter. The suspension was stirred at 400 RPM, using an overhead stirrer (Eurostar 100 Control; IKA-Werke GmbH & Co. KG, Staufen, Germany) equipped with a three-blade impeller (diameter- 50 mm) for 60 min. Following this, a method described previously by McCarthy et al. (2014) was used to quantify the solubility of the suspensions.

2.8. Statistical data analysis

All experimental analyses were conducted in triplicate with the data generated being subjected to one-way analysis of variance (ANOVA) using SPSS version 25 (SPSS Inc., Chicago, IL, USA). A Tukey's paired-comparison post-hoc test was used to determine statistically significant differences ($P < 0.05$) between mean values for different samples, with mean values deemed to be significantly different from each other at a 95% confidence level. Unless otherwise stated, results are expressed as mean ± standard deviation from triplicate analysis, with statistically significant differences identified using superscript letters.

3. Results and discussion

3.1. Quantification and mechanisms of agglomerate breakage

Initially, all three powders had significantly ($P < 0.05$) different particle sizes before pressure dispersion, with the infant formula (IF) sample having the largest Dv50 value of 252 μm, followed by the whey protein concentrate (WPC) and the fat filled milk powder (FFMP) samples with Dv50 values of 209 and 120 μm, respectively (Table 1). Powder particle size analysis, before and after pressure dispersion at 1 and 3 bar, was utilised to quantify the extent of agglomerate breakage occurring in each sample. The results, presented in Table 1, indicate that significant ($P < 0.05$) agglomerate breakage occurred for all samples; with the Dv50 value for each sample being reduced significantly as the samples were conveyed through the dispersion rig at both 1 and 3 bar dispersion pressures. The WPC and IF samples, which had highest initial Dv50 values, displayed the greatest reductions in particle size on dispersion at 1 and 3 bar. The reduction in Dv50 value for the FFMP sample, although significant ($P < 0.05$), resulted in less of a reduction in particle size, attributable to the smaller initial Dv50 value of the sample in comparison to the WPC and IF samples (Table 1). These results indicate that agglomerated dairy powders, of larger initial particle size, are more susceptible to reduction in particle size during powder conveying due to a greater extent of attrition and collisions occurring between other particles and solid surfaces, respectively. This finding is in agreement with results presented by Boiarkina et al. (2016) in a study comparing the extent of agglomerate breakage from different infant formula powder conveying systems (pneumatic and bucket elevator conveying).

Although larger agglomerates are more susceptible to reductions in particle size during powder conveying; the initial size of the powder must be considered when reporting agglomerate breakage. The percentage of agglomerate breakage was calculated for each sample after 1 and 3 bar dispersion and the results (Table 2) show that relatively similar levels of agglomerate breakage were displayed by all samples at each dispersion pressure. At 1 bar dispersion, the WPC and FFMP samples were broken down by 8.12 and 8.56%, respectively, whereas, the IF samples showed a lesser extent of agglomerate breakage at this dispersion pressure, as only 5.29% agglomerate breakage occurred. At 3 bar dispersion, the FFMP sample showed the lowest level of agglomerate breakage (21.7%), followed by the IF (23.1%) and WPC (26.3%) samples. These results indicate that although larger agglomerates are more susceptible to reductions in particle size during conveying, the resulting levels of agglomerate breakage for small (FFMP) and large (WPC and IF) agglomerated powders are relatively similar. The importance of initial particle size of agglomerated dairy powders is highlighted here as a range of powders with very different chemical composition and powder physical properties all yielded similar levels of agglomerate breakage. This suggests that powder particle size, an important influencer of many functional properties for powder handling, is also very influential in determining the extent of breakdown taking place during pneumatic conveying of agglomerated dairy powders.

Table 1
Powder physical properties, including particle size distribution parameters, specific surface area (SSA) and particle density (ρ_p) of whey protein concentrate (WPC), fat filled milk (FFMP) and infant formula (IF) powders before (C) and after dispersion at 1 (1b) and 3 (3b) bar.

		Dv10 μm	Dv50 μm	Dv90 μm	D[4,3] μm	D[3,2] μm	SSA m^2 kg^{-1}	ρ_p g cm^{-3}
WPC	*C*	95.6 ± 0.52[a]	209 ± 1.15[a]	365 ± 4.73[a]	221 ± 1.53[a]	165 ± 0.58[a]	27.3 ± 0.12[a]	1.25 ± 0.01[a]
	1b	87.6 ± 0.06[b]	192 ± 0.58[b]	336 ± 2.52[b]	203 ± 1.00[b]	152 ± 0.58[b]	29.7 ± 0.08[b]	1.24 ± 0.01[b]
	3b	67.1 ± 0.15[c]	154 ± 1.53[c]	269 ± 4.73[c]	162 ± 1.53[c]	118 ± 0.58[c]	38.3 ± 0.15[c]	1.25 ± 0.01[a]
FFMP	*C*	43.7 ± 0.35[a]	120 ± 1.15[a]	231 ± 3.51[a]	130 ± 1.53[a]	83.7 ± 0.71[a]	53.9 ± 0.46[a]	1.23 ± 0.01[a]
	1b	40.0 ± 0.23[b]	110 ± 1.15[b]	213 ± 6.35[b]	120 ± 2.31[b]	76.3 ± 0.59[b]	59.2 ± 0.44[b]	1.22 ± 0.01[b]
	3b	34.5 ± 0.10[c]	94.5 ± 0.45[c]	183 ± 3.00[c]	103 ± 0.88[c]	65.2 ± 0.26[c]	69.2 ± 0.26[c]	1.23 ± 0.01[a]
IF	*C*	109 ± 0.58[a]	252 ± 2.65[a]	492 ± 6.66[a]	280 ± 3.06[a]	193 ± 1.53[a]	23.5 ± 0.17[a]	1.26 ± 0.01[a]
	1b	99.2 ± 0.71[b]	239 ± 3.79[b]	455 ± 9.17[b]	259 ± 0.10[b]	178 ± 2.65[b]	25.4 ± 0.35[b]	1.26 ± 0.01[a]
	3b	76.1 ± 0.15[c]	194 ± 1.53[c]	352 ± 4.16[c]	205 ± 2.08[c]	139 ± 0.58[c]	32.5 ± 0.16[c]	1.26 ± 0.01[b]

Values followed by different superscript letters in the same column, for each sample are significantly different ($P < 0.05$).
[d] D[4,3] volume-weighted mean particle diameter.
[e] D[3,2] surface-weighted mean particle diameter.
[a] *Dv10* Particle size below which 10% of material volume exists.
[b] *Dv50* Particle size below which 50% of material volume exists.
[c] *Dv90* Particle size below which 90% of material volume exists.

3.2. Influence of agglomerate breakage on powder properties

3.2.1. Powder physical and bulk properties

Clear changes to the powder particle properties for all samples were measured on increasing dispersion pressure. A reduction in particle size (Fig. 2) resulted in a subsequent and significant ($P < 0.05$) increase in the SSA of each sample (WPC; 27.3–38.3 kg m^{-2}, FFMP; 53.9–69.2 kg m^{-2} and IF; 23.5–32.5 kg m^{-2}) (Table 1). As powder particles break down, and the mean particle size is reduced, the exposed surfaces of the newly-broken powder particles will be of much greater area, allowing for increased inter-particle interactions to take place at the bulk powder level, which can significantly influence the behaviour of such powders during further handling, storage, packaging and transport (Crowley et al., 2014; Han et al., 2019).

The reduction in particle size, and subsequent increase in the appearance of fine materials in all samples, led to a further significant increase ($P < 0.05$) in the bulk density of each sample, a key quality parameter for further application of powder, e.g., transport costs and dosage control (Schulze, 2008). The WPC sample showed an 8% increase in bulk density at 3 bar dispersion while the bulk density of the FFMP and IF samples increased by 13 and 14% respectively, at the same dispersion pressure (Table 2). The increase in bulk density occurring on agglomerate breakage is a consequence of the broken down agglomerates packing together into closer arrangement due to their decreased size and increased uniformity, therefore taking up a smaller volume in a given space. These results are in agreement with work completed by Hanley et al. (2011a) who also showed an increase in bulk density for a range of IF samples after lab-scale powder conveying. In the present study, breakage-induced increases in the bulk density of these powders may significantly impact their applications, most notably for the IF sample where the bulk density of such powder products directly influences the scoop delivery, which is important in controlling the nutrient composition of a feed serving (Renfrew et al., 2003).

The WPC sample had over twice the amount of occluded air (11.0 ml 100 g^{-1}) than that of the FFMP and IF samples (5.13 and 4.77 ml 100 g^{-1}, respectively). Occluded air is incorporated into spray dried powders when air is introduced and further stabilised in the feed solutions prior to spray drying, with the extent of such air incorporation being dependent on numerous processing practices and parameters, such as agitation of concentrates and choice of atomisation device (O'Sullivan et al., 2019; Pisecky, 2012). The higher bulk protein content of the WPC sample (compared with the FFMP and IF samples) would be expected to facilitate greater levels of air entrapment in the liquid concentrate prior to spray drying, contributing to the more hollow primary powder particles, with large amounts of entrapped air, formed during spray drying (Bouman et al., 2016) (Table 2). A similar finding was reported by Crowley et al. (2014) in a study where the content of occluded air increased on increasing protein content over a range of milk protein concentrate powders. The presence of occluded air is clearly evident in the scanning electron micrographs where the WPC powder particles seem to be agglomerates of hollow primary particle shells with large amounts of entrapped occluded air, also present, but to a lesser extent, in the FFMP sample (Fig. 3). It is hypothesised here that these hollow particle shells, of which the WPC agglomerates (and to some extent the FFMP sample) were composed of, acted as fragile points in the agglomerate structures, allowing for extensive breakage to occur at these points during the impacts experienced during conveying. This hypothesis is reinforced by the significant increase ($P < 0.05$) in the presence of fine material present in the powder bulk of the WPC sample, which was the highest of all three samples, which easily breaks off of the

Table 2
Agglomerate breakage, bulk density (ρ_{bulk}), tapped density (ρ_{tapped}), volume of interstitial air (V_{ia}), volume of occluded air (V_{oa}) and flow index (*i*) of whey protein concentrate (WPC), fat filled milk (FFMP) and infant formula (IF) powder before (C) and after dispersion at 1 (1b) and 3 (3b) bar.

		Agglomerate breakage %	ρ_{bulk} g cm^{-3}	ρ_{tapped} g cm^{-3}	V_{ia} ml 100 g^{-1}	V_{oa} ml 100 g^{-1}	i
WPC	*C*	n.a.	0.25 ± 0.01[a]	0.36 ± 0.01[a]	198 ± 2.17[s]	11.0 ± 0.19[a]	9.32 ± 0.64[a]
	1b	8.12 ± 0.79	0.26 ± 0.01[b]	0.38 ± 0.01[b]	184 ± 4.17[b]	11.6 ± 0.23[b]	7.50 ± 0.29[b]
	3b	26.3 ± 0.76	0.27 ± 0.01[c]	0.42 ± 0.01[c]	156 ± 3.00[c]	11.2 ± 0.23[ab]	5.07 ± 0.67[c]
FFMP	*C*	n.a.	0.39 ± 0.03[a]	0.54 ± 0.01[a]	103 ± 1.98[a]	5.13 ± 0.04[a]	5.58 ± 0.18[a]
	1b	8.56 ± 0.92	0.40 ± 0.03[b]	0.58 ± 0.01[b]	91.0 ± 4.21[b]	5.34 ± 0.06[b]	5.42 ± 0.60[a]
	3b	21.7 ± 0.44	0.44 ± 0.09[c]	0.60 ± 0.01[c]	84.0 ± 1.09[c]	5.03 ± 0.09[a]	4.93 ± 0.28[a]
IF	*C*	n.a.	0.43 ± 0.03[a]	0.56 ± 0.01[a]	99.3 ± 1.13[a]	4.77 ± 0.05[a]	16.1 ± 0.68[a]
	1b	5.29 ± 1.00	0.45 ± 0.03[b]	0.58 ± 0.01[b]	92.6 ± 1.98[b]	4.73 ± 0.04[b]	12.9 ± 1.39[b]
	3b	23.1 ± 0.23	0.49 ± 0.09[c]	0.63 ± 0.01[c]	78.9 ± 2.00[c]	4.52 ± 0.01[ab]	10.2 ± 0.30[c]

Values followed by different superscript letters in the same column, for each sample are significantly different ($P < 0.05$).
n.a. = not applicable.

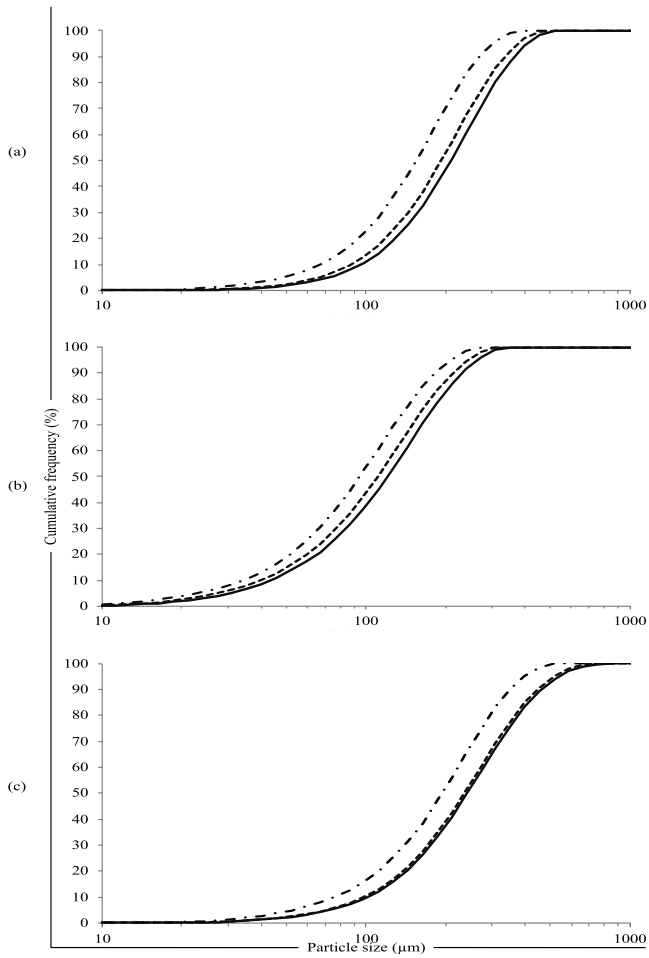

Fig. 2. Cumulative distribution profiles showing particle size of whey protein concentrate (WPC; a), fat filled milk (FFMP; b) and infant formula (IF; c) powder before (—) and after dispersion at 1 (- - -) and 3 (-˙-) bar.

friable, hollow powder particles present in the WPC powder bulk (Fig. 4).

A significant reduction ($P < 0.05$) in the volume of interstitial air was experienced for all samples, with the WPC sample, which had the largest initial volume, again, experiencing the greatest reduction on agglomerate breakdown, reducing from 198 to 156 ml 100 g^{-1} on pressure dispersion at 3 bar. The IF sample had a decrease from 99.3 to 78.9 ml 100 g^{-1} while the volume of interstitial air in the FFMP was reduced from 103 to 84.0 ml 100 g^{-1}, at the same dispersion pressure (Table 2). The presence of interstitial air located between agglomerates and between neighbouring powder particles within an agglomerate structure, is a key quality characteristic desired from the agglomeration process. This interstitial air contributes to the improvement of powder flowability and wettability associated with powder agglomeration, reducing the magnitude of interparticle interactions and encouraging the movement of water through the powder bulk *via* capillary action, respectively (Forny et al., 2011; Shah et al., 2017).

3.2.2. Fat distribution

Initially, all samples had relatively similar concentrations of surface free fat for the agglomerated powder particles (WPC; 0.57 g 100 g^{-1}, FFMP; 0.53 g 100 g^{-1} and IF; 0.66 g 100 g^{-1}) (Fig. 5) even though large variations in the bulk fat concentrations were present between the samples. Both the FFMP and IF samples contained high concentrations of bulk fat (28.7 and 25.6% fat, respectively) while the WPC sample in comparison, contained a much lower concentration (8.50% fat). The similar levels of surface free fat, before pressure dispersion, is attributed to the over-representation of fat at the surface of spray dried dairy powders, caused by the atomisation step of the spray drying process. Here, the spray dryer feed is preferentially dispersed into discrete droplets at the presence of fat globules (lowest point of cohesion in the liquid stream), therefore arranging fat globules at the surface of the droplet where they are stabilised on subsequent spray drying (Foerster et al., 2016a, 2016b).

Although all samples had similar initial surface free fat contents, a significant increase ($P < 0.05$) in the concentration of free fat on the powder surfaces was only distinguishable in the FFMP (0.55–0.93 g 100 g^{-1}) and IF (0.66–0.82 g 100 g^{-1}) samples on increasing dispersion pressure (Fig. 5). As these powders contained much higher concentrations of fat, a homogenisation step is utilised during their production in order to stabilise the fat by reducing fat globule size before spray drying (Drapala et al., 2017; O'Sullivan et al., 2018). On agglomerate breakdown however; new surfaces, once hidden within the interior of the powder particles, become exposed, leading to increases in the concentrations of free fat on the particle surfaces for the FFMP and IF samples. This increase in surface free fat was significant ($P < 0.05$) at both the 1 and 3 bar dispersion pressures for the FFMP sample, whereas, for the IF sample, the increase was only significant after dispersion at 3 bar due to increased breakage at this pressure (Fig. 5). These results are in agreement with previous findings by Hanley et al. (2011b) who also reported an increase in surface free fat on breakdown of an IF sample.

CSLM analysis was conducted on the FFMP sample which showed the greatest increase in the concentration of surface free fat occurring on agglomerate breakdown. The resulting micrographs (Fig. 6) showed a distribution of small fat globules, dispersed through the particle's bulk structure. However, also present in the CSLM micrographs were large pools of coalesced fat, which was primarily located along the pockets of occluded air, within the powder particle. During spray drying, fat migrates through the atomised droplet, towards air/water interfaces, including any pockets of occluded air that form during the spray drying process (Fäldt and Bergenståhl, 1996; Kim and Pearce, 2009). As previously stated, occluded air pockets provide weak points in powder particle structures, therefore, agglomerate breakage at these points caused large pools of coalesced fat to become exposed at the newly altered powder surface. Now exposed, at surface level, fat acts to strongly influence (generally inversely) the resulting bulk handling and rehydration properties of a powder. Increasing surface free fat leads to an increase in the surface hydrophobicity of the bulk powder and contributes to increased interparticle interactions, critical to the wettability and flowability properties, respectively (Fitzpatrick et al., 2017; Kim et al., 2005).

3.3. Effect of agglomerate breakdown on powder flowability

Agglomerate breakage led to a reduction in the flow index (*i*) value for all samples (Table 2). This reduction was significant ($P < 0.05$) for the WPC and IF samples, as these samples experienced the greatest reduction in particle size after dispersion, whereas for the FFMP sample, the reduction was not significant ($P > 0.05$). The WPC sample, which showed the greatest extent of agglomerate breakage, also displayed the greatest reduction in powder flowability, with the *i* value reducing from 9.32 to 5.07 (45.6% reduction) after dispersion at 3 bar. This may be attributed to the significant ($P < 0.05$) increase in powder bulk density and SSA, as well as the significant ($P < 0.05$) decrease in the volume of interstitial air (Tables 1 and 2). In combination, this acts to bring the powder particles into closer arrangement, resulting in a greater number and strength of interparticle interactions (e.g., van der Waals and electrostatic interactions) occurring at bulk powder level, reducing powder flowability through increased cohesive bulk strength (Zafar et al., 2017). A considerable reduction in the flow index also occurred in the IF sample (16.1–10.2 i.e., 36.6% reduction) due to the same resulting effects of a reduction in particle size as described above for the WPC sample.

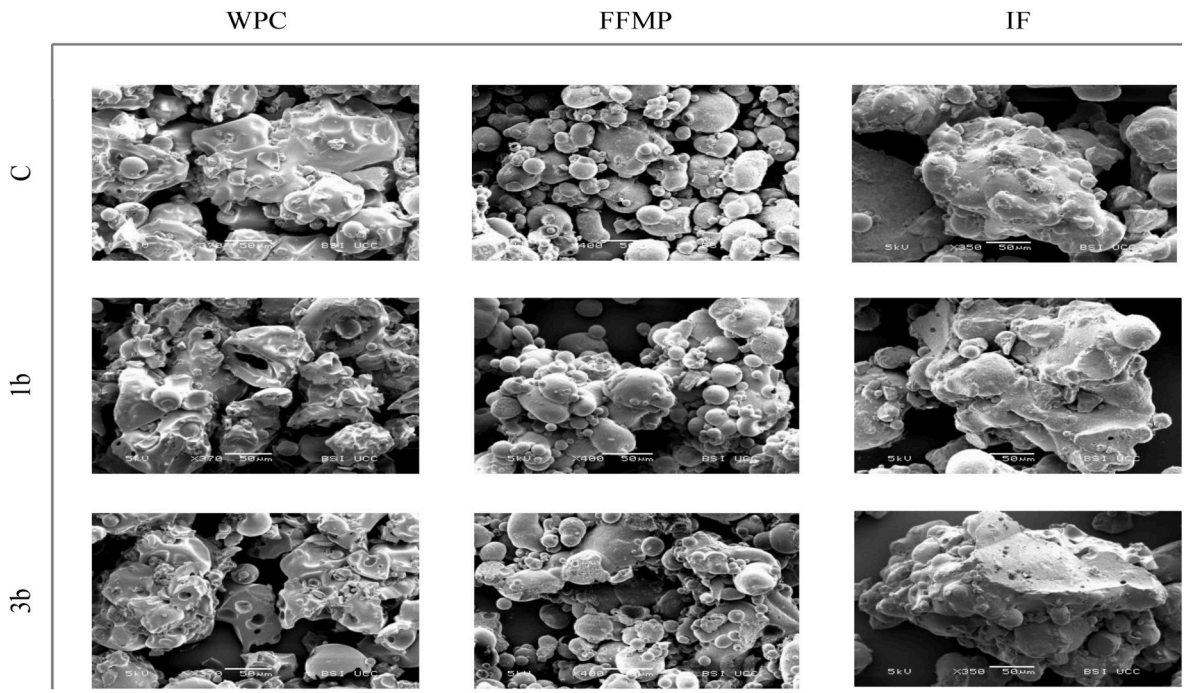

Fig. 3. Scanning electron micrographs of whey protein concentrate (WPC) – 370 X magnification, fat filled milk powder (FFMP) – 400 X magnification and an infant formula (IF) powder sample – 150 X magnification; before (C) and after dispersion at 1 (1b) and 3 (3b) bar.

Additionally, the increase in surface free fat occurring in the FFMP and IF samples on agglomerate breakdown may contribute to the reduction in powder flowability (Figs. 5 and 6). Once exposed on a powder particle surface, fat has the propensity to form liquid bridges between neighbouring particles, causing a further increase in the cohesive forces in the powder bulk; this is particularly problematic in dairy products such as FFMP and IF which contain high concentrations of fat in their bulk composition (Foster et al., 2005). Although the reduction in the flow index of the FFMP sample was not significant ($P > 0.05$) (5.58–4.93 i.e., 11.6% reduction), it must be noted that the flowability of the control FFMP (non-dispersed form) was considerably lower than that of the WPC and IF samples, due to the smaller size of the initial agglomerated powder particles and therefore, even a relatively small reduction in flowability could have considerable effects on storage, handling and further applications (e.g., bridging, rat-holing and impaired silo discharge) (Barbosa-Cánovas, 2005).

3.4. Effect of agglomerate breakdown on powder rehydration

3.4.1. Wettability

For the IF sample, due to its instant nature, the standard IDF method was appropriate for measuring the sample's wettability and the results showed that the control sample wetted within 18.7 s. The time required for wetting increased significantly ($P < 0.05$) after dispersion at 1 and 3 bar pressure with wetting times of 21.3 and 35.0 s, respectively (Table 3).

Analysis of the wettability of both the WPC and FFMP samples required a modification of the IDF method, due to their non-instant nature (Schuck et al., 2012c). The results showed that both samples also displayed a significant ($P < 0.05$) impairment in their wettability on agglomerate breakdown. The WPC sample had a wettability of 68.7% but this was reduced to 59.4 and 39.2% on pressure dispersion at 1 and 3 bar, respectively. Similarly, the FFMP initially had a wettability of 65.9%, with reductions to 46.6 and 34.9% when dispersed at 1 and 3 bar, respectively.

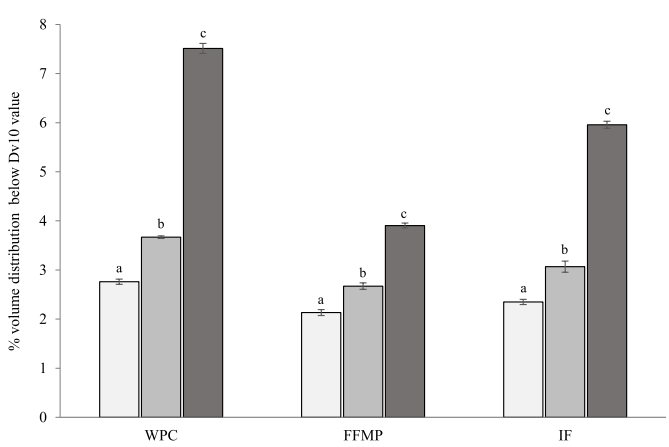

Fig. 4. Proportion of total volume of powder with particle size less than Dv10 value of whey protein concentrate (WPC), fat filled milk (FFMP) and infant formula (IF) powder before (C; □) and after dispersion at 1 (1b; ▨) and 3 (3b; ■) bar.

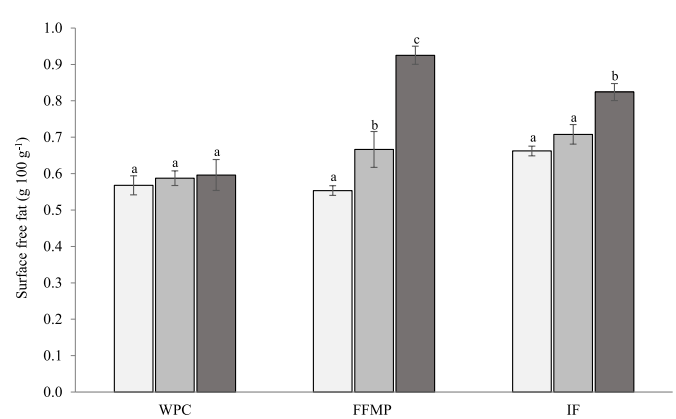

Fig. 5. Concentrations of free fat present on the surface of whey protein concentrate (WPC), fat filled milk (FFMP) and infant formula (IF) powder before (C; □) and after dispersion at 1 (1b; ▨) and 3 (3b; ■) bar.

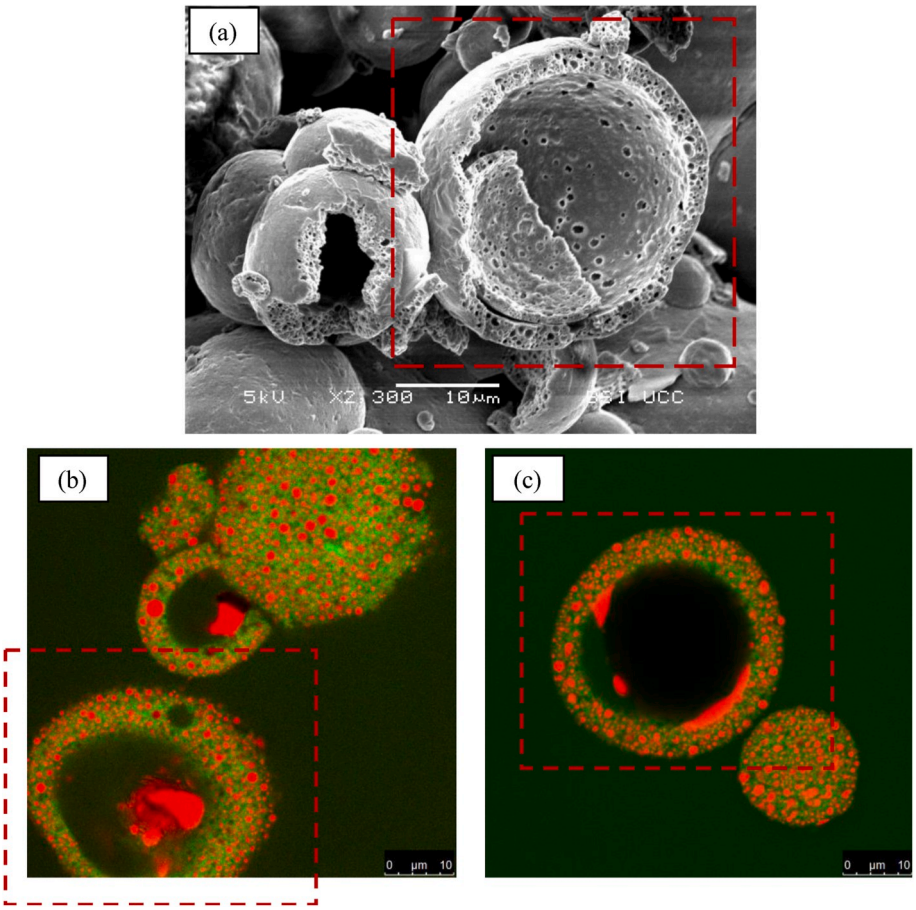

Fig. 6. (a) Scanning electron micrograph of fat filled milk powder sample (2300 X magnification) displaying a fragmented particle containing a large air vacuole and (b and c) confocal scanning laser micrographs of the same sample illustrating large pools of exposed coalesced fat (stained red), now exposed due to agglomerate breakdown post pressure dispersion.

Table 3
Rehydration properties of whey protein concentrate (WPC), fat filled milk (FFMP) and infant formula (IF) powder before (C) and after dispersion at 1 (1b) and 3 (3b) bar.

		Wettability		Dispersibility	Solubility
		s	%	%	%
WPC	*C*	>1 h	68.7 ± 4.41[a]	89.4 ± 0.95[a]	95.8 ± 0.39[a]
	1b	>1 h	59.4 ± 2.71[a]	87.9 ± 0.80[a]	94.4 ± 0.48[a]
	3b	>1 h	39.2 ± 3.88[b]	82.8 ± 1.28[b]	92.8 ± 0.34[a]
FFMP	*C*	>1 h	65.9 ± 3.64[a]	75.2 ± 1.90[a]	73.8 ± 0.73[a]
	1b	>1 h	46.6 ± 4.02[a]	69.8 ± 3.38[b]	72.1 ± 0.17[a]
	3b	>1 h	34.9 ± 3.20[b]	62.9 ± 3.14[c]	71.0 ± 0.63[a]
IF	*C*	18.7 ± 0.61[a]	100	91.4 ± 0.31[a]	99.5 ± 0.27[a]
	1b	21.3 ± 1.24[b]	100	89.4 ± 1.27[b]	99.0 ± 0.38[a]
	3b	35.0 ± 1.02[c]	100	84.8 ± 0.39[c]	98.6 ± 0.34[a]

Values followed by different superscript letters in the same column, for each sample are significantly different ($P < 0.05$).

The impairment to powder wettability, resulting from agglomerate breakdown can be attributed to two major causes, firstly; the reduction in the volume of interstitial air, and secondly, the increase in concentrations of surface free fat. The presence of interstitial air, between agglomerates and between neighbouring powder particles within an agglomerate structure, encourage rapid wetting, as they act as channels, allowing water to more freely penetrate the powder bulk *via* capillary movement. A reduction in the volume of interstitial air will cause the wetting to occur at a much slower pace, such as seen in the WPC sample (Table 2). The increase in concentration of free fat at the surface of powder particles, caused by agglomerate breakdown, also acted to inhibit efficient wettability by causing an increase in the hydrophobicity of the surfaces of the powder particles, which contributed to the impairment of wettability of the FFMP and IF samples (Figs. 5 and 6). For optimal expression of functionality, these powders are mostly required to be efficiently and fully rehydrated, with powder wetting being the first key stage in the rehydration process. Therefore, the impairment in wettability measured in this study, could have significant implications for selected industrial and commercial applications of these powders (Fitzpatrick et al., 2017).

3.4.2. Dispersibility and solubility

Lower dispersibility was also displayed by all samples after pneumatic conveying. This reduction in dispersibility was significant ($P < 0.05$) for both the FFMP and IF samples at each dispersion pressure (1 and 3 bar), with reductions in the levels of dispersibility from 65.9 and 91.4% to 62.9 and 84.8%, respectively (Table 3). The reduction of dispersibility for WPC was not significant ($P > 0.05$) between the control and 1 bar sample (89.4–87.9%), but at the 3 bar dispersion level, where further agglomerate breakdown occurred, this reduction in powder dispersibility (82.8%) was significant ($P < 0.05$) (Table 3). At the final stage of the rehydration process (i.e., powder solubility), enough time and energy had been provided to overcome the negative effects of agglomerate breakdown as there was no significant difference ($P > 0.05$) in the solubility at each dispersion pressure for all samples.

4. Conclusion

In conclusion, the results from this study indicate that the initial size of agglomerated dairy powder particles is a determinant of the extent to which particle size reduction occurs on powder conveying, i.e., larger agglomerate structures experience a greater reduction in particle size. The resulting effects of this breakage were displayed on analysis of the physical and bulk-handling properties of the samples, with significant decreases measured in particle size, volumes of interstitial air and a concomitant increase in bulk density of each sample on pressure dispersion. The significant adverse effects agglomerate breakage can have on both the flowability and rehydration properties of the powder samples were demonstrated through this study. Powder flowability was decreased for all samples, attributed to increased particle-particle interactions, as broken-down agglomerates could pack together into closer conformations. The initial stages of rehydration (i.e., wettability and dispersibility) were significantly impeded by agglomerate breakdown though a combination of increased surface free fat (for the FFMP and IF samples) and a reduction in the volumes of interstitial air, both of which limit the movement of water through the powder bulk on rehydration. Although partial agglomerate breakdown is inevitable on powder conveying, the new insights provided here may be utilised to improve exiting industrial powder handling processes and final powder functionality.

Declarations of competing interest

None.

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.

CRediT authorship contribution statement

R. Hazlett: Conceptualization, Methodology, Investigation, Visualization, Writing - original draft, Software. **C. Schmidmeier:** Conceptualization, Methodology, Writing - review & editing. **J.A. O'Mahony:** Funding acquisition, Conceptualization, Supervision, Writing - review & editing, Project administration.

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