

Title	Influence of mechanical integrity during pneumatic conveying on the bulk handling and rehydration properties of agglomerated dairy powders
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Publication date	2020-04-30
Original Citation	Hazlett, R., Schmidmeir, C. and O'Mahony, J. A. (2020) 'Influence of mechanical integrity during pneumatic conveying on the bulk handling and rehydration properties of agglomerated dairy powders', Journal of Food Engineering, doi: 10.1016/ j.jfoodeng.2020.110103
Type of publication	Article (peer-reviewed)
Link to publisher's version	https://www.sciencedirect.com/science/article/pii/ S0260877420302016 - 10.1016/j.jfoodeng.2020.110103
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Download date	2025-07-02 01:40:52
Item downloaded from	https://hdl.handle.net/10468/9958



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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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PII: S0260-8774(20)30201-6

DOI: https://doi.org/10.1016/j.jfoodeng.2020.110103

Reference: JFOE 110103

To appear in: Journal of Food Engineering

Received Date: 27 January 2020

Revised Date: 22 April 2020

Accepted Date: 24 April 2020

Please cite this article as: Hazlett, R., Schmidmeier, C., O'Mahony, J.A., Influence of mechanical integrity during pneumatic conveying on the bulk handling and rehydration properties of agglomerated dairy powders, *Journal of Food Engineering* (2020), doi: https://doi.org/10.1016/j.jfoodeng.2020.110103.

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Credit Author Statement

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

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2	Influence of mechanical integrity during pneumatic conveying on
3	the bulk handling and rehydration properties of agglomerated dairy
4	powders
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25 Abstract

26 Agglomerated powders are susceptible to breakdown on handling, most notably, during 27 powder conveying. In this study, three agglomerated dairy powders (whey protein concentrate powder, WPC; fat-filled milk powder, FFMP and infant formula powder, IF) 28 were conveyed through a custom-fabricated dispersion rig to understand the effects of 29 agglomerate breakdown on dairy powder handling and application. All samples displayed 30 31 significant breakdown on dispersion, evidenced by reducing particle size and increasing bulk 32 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 33 34 stages of rehydration were impeded by agglomerate breakage (42.9-47.0% wettability 35 reduction and 7.22-16.4% dispersibility reduction), while powder solubility remained relatively unchanged. This study provides insights into the alterations of agglomerated dairy 36 powder properties on agglomerate breakdown, while identifying the effects these alterations 37 38 have on the functional properties of these powders.

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40 Key words: Agglomerate integrity, Powder conveying, Powder functionality, Flowability,41 Rehydration.

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47 1. Introduction

48 The production of dairy powders, via spray drying, concentrates the numerous 49 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 50 51 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 52 53 types display challenges with handling and rehydration, attributable mainly to their bulk 54 composition and/or physical properties (Fitzpatrick et al., 2016, 2007) and for such powders, agglomeration may be utilised to overcome these challenges. 55

56 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 57 particles are combined together into cluster-like structures where individual primary particles 58 may still be distinguished (Cuq et al., 2013; Iveson et al., 2001). Agglomeration has been 59 shown to alter the powder physical and bulk-handling properties, ultimately increasing their 60 flowability, by reducing the extent of interparticle interactions (e.g., van der Waals forces) 61 occurring between individual powder particles (Barkouti et al., 2013; Szulc and Lenart, 62 2013). The improvement of powder rehydration in the initial stages (i.e., wettability) on 63 agglomeration is another, well established, beneficial effect of agglomerating dairy powders 64 65 and is achieved mainly through increasing the volumes of interstitial air within the resulting powder (Gaiani et al., 2007; Ji et al., 2016). 66

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.

77 Numerous studies have been completed in the area of pharmaceutical sciences to study the breakage of granulated powder material, including understanding the causes, mechanisms 78 and means of measurement (Iveson et al., 2001; Reynolds et al., 2005; Subero and Ghadiri, 79 2001). Granulation, although another form of powder particle size enlargement, forms 80 81 particles that are very different to agglomerated dairy powder particles. These particles are 82 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 83 generally have low mechanical integrity. Therefore, it proves challenging to extend any 84 85 correlations between the breakdown of granulated pharmaceutical powders and agglomerated 86 dairy powders.

87 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 88 89 breakdown has on selected key quality attributes of the resultant powders. Three representative agglomerated dairy powders were chosen (i.e., whey protein concentrate 90 powder, fat filled milk powder and infant formula powder) which had diverse bulk 91 92 composition, physical properties and intended applications in order to generate inter-93 relationships between these properties applicable to many different forms of agglomerated dairy powders. The powders were dispersed through a custom-fabricated dispersion rig with 94 95 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 96

97 control of the functional and physical properties of agglomerated dairy powders during98 conveying.

99 **2.** Material and methods

100 2.1. Materials and composition

101 Three commercially significant agglomerated dairy powders were used in this study. 102 Agglomerated whey protein concentrate (WPC) and agglomerated fat-filled milk powder (FFMP) ingredients were kindly donated by Carbery Ingredients (Ballineen, Cork, Ireland) 103 104 and Lakelands Dairies (Bailieborough, Cavan, Ireland) respectively, while a first-age, wheydominant, infant formula (IF) powder was sourced from a local commercial outlet. Data for 105 composition was provided by the suppliers of the WPC (protein: 80.0% and fat: 8.50%) and 106 FFMP (protein: 26.4% and fat: 28.7%) ingredients and was calculated from the product 107 packaging for the IF powder (protein: 8.52% and fat: 25.6%). 108

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110 2.2. Powder dispersion process for agglomerate breakdown

A pressure dispersion rig was custom fabricated by Liam A. Barry Ltd. (Little Island, Cork, 111 112 Ireland) to achieve controlled breakage of agglomerated dairy powders in a simulated lean 113 phase pneumatic conveying configuration. The rig was composed of a compressed air source, an eductor (Figure 1), a powder hopper and stainless steel and flexible tubing. Compressed 114 115 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 116 117 slowly poured into the powder hopper to avoid bridging or arching at the exit of the hopper 118 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, 119 entered stainless steel tubing (20 mm in length) where particle-particle and particle-wall 120 121 collisions occurred, causing the agglomerated powder structures to break down further,

122 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 123 temperature (22°C) and protected from light until analysis. One kilogram of each powder was 124 125 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 3 126 127 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-128 129 contamination between samples.

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130

131 2.3. Powder physical properties

132 2.3.1. Agglomerate size and morphological analysis

The particle size distribution (PSD) and specific surface area (SSA) analysis of the powders 133 was measured using a Mastersizer 3000 equipped with an automated Aero S dry powder 134 disperser cell (Malvern Instruments, Worcestershire, UK). The lowest possible dispersion 135 pressure of 0.2 bar was used to minimise additional breakage on dispersion during all PSD 136 137 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 138 139 the agglomerated dairy powders was reported as the volume-weighted median particle 140 diameter (i.e., Dv50) and this data was used in later calculations such as agglomerate 141 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.

146

147 2.3.2. 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:

151

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

152

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.

158

159 2.3.3. Distribution of fat in powder particles

160 The surface free fat content of each powder sample was quantified post conveying using the 161 GEA Niro analytical methods No. 10a (GEA Niro, 2006a) with minor modifications 162 previously described by Schmidmeier *et al.* (2019). Confocal laser scanning microscopy 163 (CLSM) was used to visualise the distribution of fat throughout the powder particles and to 164 observe any changes occurring as a result of breakdown of agglomerated powders containing 165 high bulk fat levels following a method previously described by Drapala *et al.* (2017).

166

167 2.4. Powder physical and bulk-handling properties

168 2.4.1. Particle density, bulk density, occluded and interstitial air

169 The particle density of the samples was measured using the Micromeritics Accupyc II 1340

170 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).

176 2.4.2. 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).

180

181 2.5. Rehydration properties

182 2.5.1. Wettability

Initially, the IDF wettability standard method 87:2014 (IDF, 2014) was used to assess the 183 impact of agglomerate breakdown on the wetting properties of the powder samples. For 184 powders that took longer than 60 min to wet (i.e., WPC and FFMP), a modification to the 185 186 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 187 migrated below the water surface (i.e., have not been wetted) 60 min after powder addition. 188 189 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 190 191 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 192 193 weight. The mass of wetted particles is then used to determine wettability of each sample using the following calculation: 194

195
$$Wettability (\%) = \frac{mass of wetted particles (g)}{mass of initial powder (g)}. 100$$

196 2.5.2. Dispersibility

A modified version of the IDF dispersibility method 87:2014 (IDF, 2014) was utilised 197 whereby powder (10 g) was added to the surface of 250 ml of ultrapure water in an 80 mm 198 199 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 200 201 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 202 203 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 204 calculation as described in the IDF method to calculate the percent dispersibility of samples. 205

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207 2.5.3. 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.

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215 2.6. 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 another at a 95% confidence 221 level. Unless otherwise stated, results are expressed as mean \pm standard deviation from 222 triplicate analysis, with statistically significant differences identified using superscript letters. 223

224 3. Results and discussion

225 *3.1. Quantification and mechanisms of agglomerate breakage*

Initially, all three powders had significantly (P < 0.05) different particle sizes before 226 pressure dispersion, with the infant formula (IF) sample having the largest Dv50 value of 252 227 μm, followed by the whey protein concentrate (WPC) and the fat filled milk powder (FFMP) 228 229 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 230 extent of agglomerate breakage occurring in each sample. The results, presented in Table 1, 231 232 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 233 through the dispersion rig at both 1 and 3 bar dispersion pressures. The WPC and IF samples, 234 which had highest initial Dv50 values, displayed the greatest reductions in particle size on 235 236 dispersion at 1 and 3 bar. The reduction in Dv50 value for the FFMP sample, although 237 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 238 results indicate that agglomerated dairy powders, of larger initial particle size, are more 239 240 susceptible to reduction in particle size during powder conveying due to a greater extent of 241 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 242 the extent of agglomerate breakage from different infant formula powder conveying systems 243 244 (pneumatic and bucket elevator conveying).

245 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 246 agglomerate breakage. The percentage of agglomerate breakage was calculated for each 247 248 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 249 250 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 251 dispersion pressure, as only 5.29% agglomerate breakage occurred. At 3 bar dispersion, the 252 FFMP sample showed the lowest level of agglomerate breakage (21.7%), followed by the IF 253 254 (23.1%) and WPC (26.3%) samples. These results indicate that although larger agglomerates 255 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 256 257 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 258 259 physical properties all yielded similar levels of agglomerate breakage. This suggests that 260 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 261 262 pneumatic conveying of agglomerated dairy powders.

263

264 *3.2. Influence of agglomerate breakage on powder properties*

265 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 (Figure 2) resulted in a subsequent and significant (P < 0.05) increase in the SSA of each sample (WPC; 27.3 to 38.3 kg m⁻², FFMP; 53.9 to 69.2 kg m⁻² and IF; 23.5 to 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 275 materials in all samples, led to a further significant increase (P < 0.05) in the bulk density of 276 each sample, a key quality parameter for further application of powder, e.g., transport costs 277 and dosage control (Schulze, 2008). The WPC sample showed an 8% increase in bulk density 278 279 at 3 bar dispersion while the bulk density of the FFMP and IF samples increased by 13 and 280 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 281 packing together into closer arrangement due to their decreased size and increased 282 uniformity, therefore taking up a smaller volume in a given space. These results are in 283 agreement with work completed by Hanley et al. (2011a) who also showed an increase in 284 bulk density for a range of IF samples after lab-scale powder conveying. In the present study, 285 breakage-induced increases in the bulk density of these powders may significantly impact 286 287 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 288 289 composition of a feed serving (Renfrew et al., 2003).

The WPC sample had over twice the amount of occluded air $(11.0 \text{ ml } 100 \text{ g}^{-1})$ 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

295 of atomisation device (O'Sullivan et al., 2019; Pisecky, 2012). The higher bulk protein 296 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, 297 contributing to the more hollow primary powder particles, with large amounts of entrapped 298 air, formed during spray drying (Bouman et al., 2016) (Table 2). A similar finding was 299 reported by Crowley et al. (2014) in a study where the content of occluded air increased on 300 increasing protein content over a range of milk protein concentrate powders. The presence of 301 occluded air is clearly evident in the scanning electron micrographs where the WPC powder 302 particles seem to be agglomerates of hollow primary particle shells with large amounts of 303 304 entrapped occluded air, also present, but to a lesser extent, in the FFMP sample (Figure 3). It is hypothesised here that these hollow particle shells, of which the WPC agglomerates (and to 305 some extent the FFMP sample) were composed of, acted as fragile points in the agglomerate 306 307 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 < P308 0.05) in the presence of fine material present in the powder bulk of the WPC sample, which 309 was the highest of all three samples, which easily breaks off of the friable, hollow powder 310 311 particles present in the WPC powder bulk (Figure 4).

A significant reduction (P < 0.05) in the volume of interstitial air was experienced for 312 all samples, with the WPC sample, which had the largest initial volume, again, experiencing 313 the largest loss on agglomerate breakdown, reducing from 198 to 156 ml 100 g⁻¹ on pressure 314 dispersion at 3 bar. The IF sample had a decrease from 99.3 to 78.9 ml 100 g⁻¹ while the 315 volume of interstitial air in the FFMP was reduced from 103 to 84.0 ml 100 g⁻¹, at the same 316 dispersion pressure (Table 2). The presence of interstitial air located between agglomerates 317 318 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 319

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).

324

325 3.2.2. Fat distribution

Initially, all samples had relatively similar concentrations of surface free fat for the 326 agglomerated powder particles (WPC; 0.57 g 100 g⁻¹, FFMP; 0.53 g 100 g⁻¹ and IF; 0.66 g 327 100 g^{-1}) (Figure 5) even though large variations in the bulk fat concentrations were present 328 between the samples. Both the FFMP and IF samples contained high concentrations of bulk 329 fat (28.7 and 25.6% fat, respectively) while the WPC sample in comparison, contained a 330 331 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 332 powders, caused by the atomisation step of the spray drying process. Here, the spray dryer 333 334 feed is preferentially dispersed into discrete droplets at the presence of fat globules (lowest 335 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). 336

Although all samples had similar initial surface free fat contents, a significant increase 337 (P < 0.05) in the concentration of free fat on the powder surfaces was only distinguishable in 338 the FFMP (0.55 to 0.93 g 100 g⁻¹) and IF (0.66 to 0.82 g 100 g⁻¹) samples on increasing 339 dispersion pressure (Figure 5). As these powders contained much higher concentrations of 340 fat, a homogenisation step is utilised during their production in order to stabilise the fat by 341 342 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 343 powder particles, become exposed, leading to increases in the concentrations of free fat on 344 345 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 (Figure 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.

351 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 352 resulting micrographs (Figure 6) showed a distribution of small fat globules, dispersed 353 354 through the particle's bulk structure. However, also present in the CSLM micrographs were 355 large pools of coalesced fat, which was primarily located along the pockets of occluded air, 356 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 357 drying process (Fäldt and Bergenståhl, 1996; Kim and Pearce, 2009). As previously stated, 358 occluded air pockets provide weak points in powder particle structures, therefore, 359 360 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 361 influence (generally inversely) the resulting bulk handling and rehydration properties of a 362 363 powder. Increasing surface free fat leads to an increase in the surface hydrophobicity of the 364 bulk powder and contributes to increased interparticle interactions, critical to the wettability 365 and flowability properties, respectively (Fitzpatrick et al., 2017; Kim et al., 2005).

366

367 *3.3. Effect of agglomerate breakdown on powder flowability*

368 Agglomerate breakage led to a reduction in the flow index (*i*) value for all samples 369 (Table 2). This reduction was significant (P < 0.05) for the WPC and IF samples, as these 370 samples experienced the greatest reduction in particle size after dispersion, whereas for the

371 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 372 flowability, with the *i* value reducing from 9.32 to 5.07 (45.6% reduction) after dispersion at 373 374 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 1 375 and 2). In combination, this acts to bring the powder particles into closer arrangement, 376 resulting in a greater number and strength of interparticle interactions (e.g., van der Waals 377 and electrostatic interactions) occurring at bulk powder level, reducing powder flowability 378 through increased cohesive bulk strength (Zafar et al., 2017). A considerable reduction in the 379 380 flow index also occurred in the IF sample (16.1 to 10.2 i.e., 36.6% reduction) due to the same 381 resulting effects of a reduction in particle size as described above for the WPC sample. Additionally, the increase in surface free fat occurring in the FFMP and IF samples on 382 agglomerate breakdown may contribute to the reduction in powder flowability (Figures 5 and 383 6). Once exposed on a powder particle surface, fat has the propensity to form liquid bridges 384 385 between neighbouring particles, causing a further increase in the cohesive forces in the 386 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 387 388 the reduction in the flow index of the FFMP sample was not significant (P > 0.05) (5.58 to 4.93 i.e., 11.6% reduction), it must be noted that the flowability of the control FFMP (non-389 390 dispersed form) was considerably lower than that of the WPC and IF samples, due to the 391 smaller size of the initial agglomerated powder particles and therefore, even a relatively small 392 reduction in flowability could have considerable effects on storage, handling and further 393 applications (e.g., bridging, rat-holing and impaired silo discharge) (Barbosa-Cánovas, 2005). 394

395 *3.4. Effect of agglomerate breakdown on powder rehydration*

396 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).

401 Analysis of the wettability of both the WPC and FFMP samples required a modification 402 of the IDF method (Section 3.5.1), due to their non-instant nature (Schuck, 2012c). The 403 results showed that both samples also displayed a significant (P < 0.05) impairment in their 404 wettability on agglomerate breakdown. The WPC sample had a wettability of 68.7% but this 405 was reduced to 59.4 and 39.2% on pressure dispersion at 1 and 3 bar, respectively. Similarly, 406 the FFMP initially had a wettability of 65.9%, with reductions to 46.6 and 34.9% when 407 dispersed at 1 and 3 bar, respectively.

The impairment to powder wettability, resulting from agglomerate breakdown can be 408 409 attributed to two major causes, firstly; the reduction in the volume of interstitial air, and 410 secondly, the increase in concentrations of surface free fat. The presence of interstitial air, between agglomerates and between neighbouring powder particles within an agglomerate 411 412 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 413 414 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 415 by agglomerate breakdown, also acted to inhibit efficient wettability by causing an increase 416 in the hydrophobicity of the surfaces of the powder particles, which contributed to the 417 impairment of wettability of the FFMP and IF samples (Figures 5 and 6). For optimal 418 expression of functionality, these powders are mostly required to be efficiently and fully 419

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).

- 424
- 425 3.4.2. Dispersibility and solubility

A decrease in the dispersibility was also displayed by all samples after pneumatic 426 conveying. This reduction in dispersibility was significant (P < 0.05) for both the FFMP and 427 IF samples at each dispersion pressure (1 and 3 bar), with reductions in the levels of 428 429 dispersibility from 65.9 and 91.4% to 62.9 and 84.8%, respectively (Table 3). The reduction 430 of dispersibility for WPC was not significant (P > 0.05) between the control and 1 bar sample (89.4 to 87.9%), but at the 3 bar dispersion level, where further agglomerate breakdown 431 occurred, this reduction in powder dispersibility (82.8%) was significant (P < 0.05) (Table 3). 432 At the final stage of the rehydration process (i.e., powder solubility), enough time and energy 433 434 had been provided to overcome the negative effects of agglomerate breakdown as there was 435 no significant difference (P > 0.05) in the solubility at each dispersion pressure for all samples. 436

437

438 **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

445 pressure dispersion. The significant adverse effects agglomerate breakage can have on both the flowability and rehydration properties of the powder samples were demonstrated through 446 this study. Powder flowability was decreased for all samples, attributed to increased particle-447 448 particle interactions, as broken-down agglomerates could pack together into closer conformations. The initial stages of rehydration (i.e., wettability and dispersibility) were 449 450 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 451 of which limit the movement of water through the powder bulk on rehydration. Although 452 partial agglomerate breakdown is inevitable on powder conveying, the new insights provided 453 454 here may be utilised to improve exiting industrial powder handling processes and final 455 powder functionality.

456

457 5. Acknowledgments

The authors would like to acknowledge the Dairy Processing Technology Centre (DPTC), an Enterprise Ireland initiative, for financial support and permission to publish this work (Grant Number TC/2014/0016. The authors would also like to thank Mr. Jim McNamara (School of Food and Nutritional Sciences, University College Cork) for technical assistance provided during the set-up and running of the pressure dispersion unit and Dr. Laura Mascaraque from Teagasc Food Research Centre, Moorepark, (Fermoy, Ireland) for assistance with confocal laser microscopy analysis.

6. Tables and figures

- 467 Table 1: Powder physical properties, including particle size distribution parameters, specific surface area (SSA) and particle density (ρ_p) of whey
- 468 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	Dv50	Dv90	D[4,3]	D[3,2]	SSA	$ ho_p$
				μm			kg m ⁻²	g cm ⁻¹
WPC	С	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}	$192\pm0.58^{\text{b}}$	336 ± 2.52^{b}	$203\pm1.00^{\rm b}$	$152\pm0.58^{\text{b}}$	29.7 ± 0.08^{b}	1.24 ± 0.01^{b}
	3b	$67.1\pm0.15^{\rm c}$	$154 \pm 1.53^{\circ}$	$269 \pm 4.73^{\rm c}$	$162 \pm 1.53^{\circ}$	118 ± 0.58^{c}	$38.3\pm0.15^{\rm c}$	1.25 ± 0.01^{a}
FFMP	С	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}	$110 \pm 1.15^{\text{b}}$	213 ± 6.35^{b}	120 ± 2.31^{b}	76.3 ± 0.59^{b}	59.2 ± 0.44^{b}	$1.22\pm0.01^{\text{b}}$
	3b	$34.5\pm0.10^{\rm c}$	$94.5\pm0.45^{\rm c}$	$183 \pm 3.00^{\circ}$	$103\pm0.88^{\rm c}$	$65.2\pm0.26^{\rm c}$	$69.2\pm0.26^{\rm c}$	1.23 ± 0.01^{a}
	С	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}
IF	1b	99.2 ± 0.71^{b}	$239\pm3.79^{\text{b}}$	$455\pm9.17^{\text{b}}$	259 ± 0.10^{b}	178 ± 2.65^{b}	25.4 ± 0.35^{b}	1.26 ± 0.01^{a}
	<i>3b</i>	$76.1\pm0.15^{\rm c}$	$194 \pm 1.53^{\circ}$	$352\pm4.16^{\rm c}$	$205\pm2.08^{\rm c}$	$139\pm0.58^{\rm c}$	$32.5\pm0.16^{\rm c}$	1.26 ± 0.01^{b}

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

 ${}^{a}Dv10$ 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

^d D[4,3] volume-weighted mean particle diameter

474 ^e D[3,2] surface-weighted mean particle diameter

- 477 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
- 478 flow index (*i*) of whey protein concentrate (WPC), fat filled milk (FFMP) and infant formula (IF) powder before (C) and after dispersion at 1
- 479 (1b) and 3 (3b) bar.

		Agglomerate breakage	ρ_{bulk}	$ ho_{tapped}$	V _{ia}	V _{oa}	i
		%	g cm ⁻³		ml 1	-	
	С	n.a.	0.25 ± 0.01^{a}	0.36 ± 0.01^{a}	$198\pm2.17^{\rm s}$	11.0 ± 0.19^{a}	9.32 ± 0.64^{a}
WPC	1b	8.12 ± 0.79	$0.26\pm0.01^{\text{b}}$	0.38 ± 0.01^{b}	$184\pm4.17^{\rm b}$	11.6 ± 0.23^{b}	7.50 ± 0.29^{b}
	3b	26.3 ± 0.76	0.27 ± 0.01^{c}	$0.42\pm0.01^{\circ}$	$156\pm3.00^{\rm c}$	11.2 ± 0.23^{ab}	$5.07\pm0.67^{\rm c}$
FFMP	С	n.a.	0.39 ± 0.03^a	0.54 ± 0.01^{a}	$103\pm1.98^{\rm a}$	5.13 ± 0.04^{a}	$5.58\pm0.18^{\rm a}$
	1b	8.56 ± 0.92	0.40 ± 0.03^{b}	$0.58\pm0.01^{\rm b}$	91.0 ± 4.21^{b}	5.34 ± 0.06^{b}	5.42 ± 0.60^{a}
	<i>3b</i>	21.7 ± 0.44	0.44 ± 0.09^{c}	$0.60 \pm 0.01^{\circ}$	$84.0 \pm 1.09^{\circ}$	5.03 ± 0.09^{a}	4.93 ± 0.28^{a}
	С	n.a.	0.43 ± 0.03^{a}	0.56 ± 0.01^{a}	99.3 ± 1.13^{a}	$4.77\pm0.05^{\rm a}$	16.1 ± 0.68^{a}
IF	1b	5.29 ± 1.00	0.45 ± 0.03^{b}	0.58 ± 0.01^{b}	$92.6 \pm 1.98^{\text{b}}$	4.73 ± 0.04^{b}	$12.9 \pm 1.39^{\text{b}}$
	<i>3b</i>	23.1 ± 0.23	$0.49 \pm 0.09^{\circ}$	$0.63\pm0.01^{\circ}$	$78.9\pm2.00^{\rm c}$	4.52 ± 0.01^{ab}	$10.2\pm0.30^{\rm c}$

- 480 Values followed by different superscript letters in the same column, for each sample are significantly different (P < 0.05).
- 481 n.a. = not applicable.
- 482
- 483

485 Table 3: Rehydration properties of whey protein concentrate (WPC), fat filled milk (FFMP) and infant formula (IF) powder before (C) and after

486	dispersion at 1 (1b) and 3 (3b) bar.
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		Wet	Wettability		Solubility	
		S	%	%	%	
	С	> 1 h	68.7 ± 4.41^a	$89.4\pm0.95^{\rm a}$	95.8 ± 0.39^{a}	
WPC	1b	> 1 h	59.4 ± 2.71^{a}	$87.9\pm0.80^{\rm a}$	94.4 ± 0.48^{a}	
	<i>3b</i>	> 1 h	$39.2 \pm 3.88^{\text{b}}$	$82.8\pm1.28^{\text{b}}$	92.8 ± 0.34^{a}	
	С	> 1 h	65.9 ± 3.64^{a}	$75.2\pm1.90^{\rm a}$	73.8 ± 0.73^a	
FFMP	1b	> 1 h	$46.6\pm4.02^{\rm a}$	69.8 ± 3.38^{b}	$72.1\pm0.17^{\rm a}$	
	<i>3b</i>	> 1 h	34.9 ± 3.20^{b}	$62.9\pm3.14^{\rm c}$	71.0 ± 0.63^a	
	С	18.7 ± 0.61^{a}	100	91.4 ± 0.31^{a}	$99.5\pm0.27^{\rm a}$	
IF	<i>1b</i>	$21.3 \pm 1.24^{\text{b}}$	100	$89.4{\pm}~1.27^{b}$	$99.0\pm0.38^{\rm a}$	
	<i>3b</i>	$35.0 \pm 1.02^{\circ}$	100	$84.8\pm0.39^{\rm c}$	$98.6\pm0.34^{\rm a}$	

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





Figure 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.

FFMP

IF





WPC



Figure 3: Scanning electron micrographs of whey protein concentrate (WPC) – 370 X magnification, fat filled milk powder (FFMP) – 400 X 517 magnification and an infant formula (IF) powder sample - 150 X magnification; before (C) and after dispersion at 1 (1b) and 3 (3b) bar. 518



Figure 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; \Box) and after dispersion at 1 (1b; \Box) and 3 (3b; \Box) bar.

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



Figure 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.

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Highlights

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

- Large agglomerates experienced the greatest loss in particle size on conveying •
- Alterations to powder physical properties occurred, impairing powder functionality •
- Agglomerate breakage caused a significant decrease in powder flowability ٠
- Initial stages of powder rehydration were impeded by agglomerate breakdown •
- Final powder solubility was relatively unaffected by agglomerate breakage •

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Declaration of interests

 \boxtimes 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.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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