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10 Abstract

The BARDS technique was applied in this study to acoustically assess the 11 rehydration behaviour of milk protein isolate (MPI) agglomerates and to compare 12 with regular MPI powder. The results showed that BARDS has potential to monitor 13 the rehydration behaviour of agglomerates. The greater porosity (> 70%) of 14 15 agglomerated powders introduced more compressible gas into the water. The BARDS profile showed that there was faster initial gas release from the 16 agglomerates, indicating better wetting and dispersion ability of the agglomerates 17 with shorter t_{M} (time of maximum gas volume in solution). At 0.10% powder addition, 18 agglomerated MPI reached t_M within 109 s, which was significantly less than the 19 control MPI at 140 s. MPI with lactose binder (MPI-L) had a t_M of 80 s at 0.10% 20 powder addition and, larger size MPI-L had a t_M of 60 s. At 0.20% and 0.30% powder 21 addition, more time was required to wet and disperse the powders. 22

23 Key words: BARDS, milk protein isolate powder, rehydration, agglomeration

24 1. Introduction

Fluid-bed agglomeration is a technique that binds primary particles together to 25 form aggregates with larger particle size. It can be applied to improve the physical 26 properties of powders such as flowability and rehydration ability (Chever et al., 2017), 27 and is used widely in the food industry. MPI (milk protein isolate) powder is an 28 important product produced by the dairy industry, and is used as a milk protein 29 ingredient in many food products. A major problem with MPI powder is its 30 rehydration ability (Fitzpatrick et al., 2016). It is both a poor wetting and slowly 31 dissolving powder (Crowley et al., 2015; Ji et al., 2015; Wu et al., 2019). The high 32 content of casein present in MPI powder is considered as the major reason for its 33 slow dissolution (Mimouni et al., 2010). Crowley et al. (2015) characterised the 34 rehydration ability of milk protein concentrate (MPC) powders with milk protein 35 concentrations ranging from 35 to 90%. They showed that increasing protein content 36 reduced powder wettability. It also reduced powder dissolution ability especially at 37 protein contents of 70% and higher. Ji et al (2015) showed that fluid-bed 38 agglomeration could greatly improve the wettability of MPI powder, as the larger 39 agglomerate particle size made it easier for water to penetrate the interstitial spaces 40 between the particles. However, it did not improve the speed of dissolution of the 41 particles once they were wetted and dispersed. In fact, agglomeration caused the 42 dissolution to be slightly slower because a short period of time was required to 43 break-up the agglomerate structure into the primary particles, which then dissolved 44 slowly like in the regular MPI powder. 45

The dissolution process of powder in principle includes the gas exchange between 46 powder and solution, that is, the gas inside the powder particles is released into the 47 water as water penetrates into the particles and dissolves the powder material. 48 However, the literature mainly focuses on the dissolution of material in powder form 49 (Felix da Silva et al., 2018). Furthermore, when powder is re-structured by 50 agglomeration, the air distribution in the powder is inevitably altered. The air release 51 behaviour of agglomerated powder during rehydration is rarely discussed even 52 though this can potentially provide important insights into the rehydration process. 53

Broadband acoustic resonance dissolution spectroscopy (BARDS) is a technique 54 that can monitor gas release from powder in solution. The gas release has response 55 in the acoustic resonance, which is influenced by the changes in the medium due to 56 the release of air bubbles into the solvent during powder dissolution. The frequency 57 change of BARDS results from the alteration of the speed of sound in the solvent 58 containing air bubbles. Hence, the real-time information in the solvent can be 59 monitored acoustically by changes in frequency (Fitzpatrick et al., 2014). So 60 theoretically, the rehydration process of a dissolving powder can be described 61 acoustically by BARDS. The first application of BARDS to the rehydration of dairy 62 powder was reported for MPC powders with protein contents ranging from 35 to 90% 63 (Vos et al., 2016). Rehydration of the different MPCs was monitored using BARDS, 64 from which their rehydration characteristics were acoustically distinguished. The 65 higher protein content powders showed slower gas release, which is indicative of 66 slower rehydration behaviour. (Peddapatla et al., 2017) applied BARDS to 67 investigate the wettability of pharmaceutical blends. Very poor wetting blends did not 68 disperse into water and thus there was no gas release, while good wetting blends 69

readily dispersed and dissolved, releasing air bubbles that altered the frequencyresponse measured by BARDS.

The objective of this study is to apply the BARDS technique for investigating the gas release of fluid-bed agglomerated MPI powders during rehydration and to compare this behaviour with that of regular unagglomerated MPI powder. Agglomeration of MPI was achieved using both water and lactose solution as binders to investigate the influence of binder type. The agglomerated MPI was sieved into two size fractions to investigate the influence of agglomerate particle size.

78

79 2. Materials and methods

80 2.1. Materials

Milk protein isolate (MPI) powder was purchased from Kerry Ingredients (Country Kerry, Ireland). The solids composition of the MPI powder is 86% protein, 1.5 % fat, ash and <1 % carbohydrate. Crystalline lactose powder was supplied by Arla Food Ingredients (Viby J, Aarhus, Denmark).

85 2.2. Powder preparation

The agglomerated MPI was prepared in a top-spray fluidised bed granulator (VFC-Lab Micro flo-coater, Vector Corporation, Lowa, USA). The quantity of MPI produced in each trial was 100 g. The volume of binder solution was 50 mL, and this was transferred by a peristaltic pump at a flowrate of 1.2 mL· min⁻¹. The powder was fluidised in the bottom of the chamber by air at 50°C, while the binder was sprayed through a nozzle with a 1 bar pressure drop. After agglomeration, the powder was dried for 15 minutes by the air at 50°C. MPI agglomerates were produced using two

binders, one being water (MPI-W) and the other being a 15%, w/v lactose solution
(MPI-L). Both MPI agglomerates were sieved to produce two size fractions with
particle sizes between 106 and 180 µm and 180 and 300 µm.

96 2.3. Physical properties of powder

97 2.3.1. Particle size

Malvern Morphologi G3 (Malvern Instruments Ltd, Worcestershire, UK) was used to characterize the particle size distribution. A 15 mm³ volume of powder was dispersed by air as a single layer of powder on a glass plate in the dispersion unit. The Morphologi G3 measured the number size distribution from which the volume size distribution was evaluated, along with corresponding D₁₀, D₅₀, and D₉₀ values. The experiment was performed in duplicate for each size distribution.

104 2.3.2. Density and gas volume

The loose bulk density (ρ_B) and tapped bulk density (ρ_T) of the MPI powder and its 105 agglomerates were measured using a measuring cylinder and a tapping machine (Ji 106 et al., 2015). The powder was tapped 500 times by the tapping machine (Funke 107 Gerber, Berlin, Germany). The apparent density (ρ_A) was measured by a helium gas 108 pycnometer (AccuPyc II 1340, Micromeritics Instrument Corporation, Georgia, USA). 109 The measurements were done in triplicate. The solid density ($\rho_{\rm S}$) of MPI and MPI-W 110 was 1.53 g/mL, while the MPI-L was 1.54 g/mL. These were evaluated using the 111 composition of MPI and its agglomerates (Niro, 2006). 112

In the MPI powder and its agglomerates, there are a number of void spaces where air resides, as illustrated in Fig. 1. In the MPI powder, there are vacuoles within the powder particles and interstitial air between powder particles in the bulk. A fluid-bed

(1)

agglomerate particle consists of primary particles bound together, thus there is
 additional vacuole space between the primary MPI particles within the agglomerate
 particle.

119 The porosity of the MPI powder and its agglomerates is defined in equation (1).

120
$$\mathcal{E}=(\rho_A - \rho_T)/\rho_A \times 100$$

For the MPI powder, this represents the void fraction associated with interstitial voids in the tapped powder and possibly some vacuole voids within the primary particles, but this does depend on whether or not the pycnometer gas penetrates inside the primary particles. For the MPI agglomerates, the porosity includes additional voidage associated with the voids between primary particles within the agglomerate particles, as these are easily penetrated by the pycnometer gas.

The following specific air volumes are defined using the density data. These are the bulk interstitial air (BIA), tapped occluded air (TOA) and vacuole occluded air (VOA) specific volumes. These are defined in equations (2) to (4), and they are expressed in units of mL / 100 g of powder.

131
$$BIA = \left(\frac{1}{\rho_B} - \frac{1}{\rho_T}\right)$$
 (2)

132
$$TOA = \left(\frac{1}{\rho_T} - \frac{1}{\rho_A}\right)$$
 (3)

133
$$VOA = \left(\frac{1}{\rho_A} - \frac{1}{\rho_S}\right)$$
 (4)

Chever et al. (2017) and GEA Niro (Niro, 2006) use similar definitions to characterise
specific air volumes in powder and agglomerates.

136 **2.4. The measurement of BARDS**

The BARDS apparatus consists of a rehydration vessel, which is a glass vessel 137 with a magnetic stirring bar, and a microphone placed 5 cm above the top rim of the 138 glass vessel. A known mass of powder is dropped onto the surface of the water in 139 the glass vessel. The broadband acoustic excitation is obtained from the tapping 140 behaviour of a magnetic bar on the inner glass wall. The microphone captures the 141 acoustic resonance from the sound travelling through the liquid, typically in the 142 frequency range of 0-20 kHz. The BARDS instrument was stabilized for 30 s to allow 143 a steady-state frequency to be achieved before powder is added (Ahmed et al., 144 2018). Gas is released from the powder which alters the measured frequency as the 145 powder wets and dissolves. The frequency decreases over time until it attains a 146 minimum value (f_{min}) , after which the frequency gradually returns to its original 147 steady state value. 148

The volume of distilled water in the measuring vessel of BARDS was 25 mL. 149 Three masses of each powder were used, that is 0.025 g, 0.0375 g and 0.05 g, 150 giving powder concentrations in water of 0.1%, 0.15% and 0.2% w/v, respectively. 151 The duration of the BARDS measurement was recorded over an 800 s duration and 152 each test was carried out in duplicate. The stirring rate was 500 rpm in ambient 153 temperature. The small masses of powder readily wetted and dispersed. A typical 154 frequency profile over time is presented in Fig. 2. As stated above, the essence of 155 the BARDS measurement technique is that as a powder dissolves in a liquid, it 156 releases its entrained air to the (already saturated) liquid in the form of micro-157 bubbles and this correspondingly affects the frequency of sound in the liquid. The 158 relationship between the frequency response and the fractional volume occupied by 159

the air bubbles is presented in equation (5), and details of this equation are provided
by Fitzpatrick et al. (2012) and Crawford (1982).

162
$$freq = \frac{freq_w}{\sqrt{1+1.49 \times 10^4 \cdot f_a}}$$
(5)

where f_a is the fractional volume occupied by air bubbles. The $freq_w$ and freq are the frequency response of pure water in steady-state and bubble-filled water after adding sample, respectively. The equation and its parameter values are valid for the conditions prevailing for our work. The volume of gas (V_g) in the water in the BARDS glass vessel can be calculated from equation (6), where V_w is the volume of water for rehydrating powders in BARDS.

$$169 V_g = f_a \cdot V_w (6)$$

170 **2.5 Statistical analysis**

One-way analysis of variance (ANOVA) was performed by SPSS software (IBM SPSS Statistics version 24). Duncan test was run for comparing the significance of multiple groups. The significance was set as P<0.05.

174

175 3. Results and discussion

176 **3.1. Powder particle size, densities and specific gas volumes**

Particle size values for the MPI powder and its agglomerates are presented in Table 1. As expected, the particle size of agglomerated powders is much larger than that of the non-agglomerated powder. The D[v, 0.5] value of the MPI powder is 49.8 µm. The D[v,0.5] values of the smaller size agglomerate fractions are 119 µm for

MPI-W1 and 117 μ m μ m for MPI-L1. The D[v,0.5] values of the larger size agglomerate fractions are 207 μ m for MPI-W2 and 168 μ m for MPI-L2. Powder agglomerated by water is larger than the powder agglomerated by lactose, especially for the larger size fractions. Jinapong et al. (2008) also observed that smaller sized soymilk agglomerates were obtained by increasing the concentration of maltodextrin binder from 0 to 10 %. Similarly, Szulc and Lenart (2013) showed that larger sized agglomerates were achieved using distilled water as the binder.

The powder densities of the MPI powder and its agglomerates are presented in 188 Table 2. The apparent densities of the MPI powder and its applomerates are all fairly 189 similar with the agglomerates having slightly greater apparent densities ranging from 190 1082 to 1119 g/L, and the MPI having a value of 1073 g/L, which is in line with a 191 study by (Szulc and Lenart, 2013). The similar apparent densities are to be expected 192 as the fluid-bed agglomerates are composed of primary MPI particles, and the 193 pycnometer gas will penetrate them to the same extent whether they are in the MPI 194 powder or the MPI agglomerates. The solid bridges in the agglomerate are possibly 195 the reason why the applomerates have slightly greater apparent densities. Table 2 196 shows that both the loose and tapped bulk densities of the agglomerates are smaller 197 than the MPI powder. This is to be expected because of the additional voids that 198 exist between the primary particles within the agglomerate particles themselves. In 199 relation to the agglomerates, the different particle size fractions showed different 200 tapped bulk densities, with the smaller size fractions having larger tapped bulk 201 densities. 202

The porosity of the powders was calculated in equation (1), using the apparent density and tapped bulk density. The porosity of the MPI powder and its agglomerates are presented in Table 2. The porosity of the agglomerates is greater

than that of the MPI powder, due to their lower tapped bulk densities as highlighted
above. Likewise, the porosity of the larger sized agglomerates was significantly
greater than the smaller sized agglomerates, while the type of binder had little effect
on porosity.

The BIA, TOA and VOA specific volumes (equations 2-4) are presented in Table 3 210 for the MPI powder and its agglomerates. The VOA had similar specific volumes in 211 accordance with the apparent densities. This suggests that the pycnometer gas 212 easily penetrates the porous structure between the primary particles in the 213 agglomerates. The open pores in agglomeration possibly favour the gas penetration 214 as well (AI hassn et al., 2018). Consequently, the VOA represents some or all of the 215 specific air volume within the primary MPI particles, depending on the ability of 216 pycnometer gas to penetrate into these particles. The VOA of primary MPI particle is 217 probably dependent on the powder shrinkage during spray-drying (Foerster et al., 218 2016a; Foerster et al., 2016b; Fu et al., 2013). The TOA specific volumes have the 219 same trend as porosity, with the agglomerates having larger TOA values than the 220 MPI powder and the larger agglomerates have larger values than the smaller 221 agglomerates. The TOA values represent the specific air volume between particles 222 in the tapped bulk plus the specific air volume within the structure of the 223 agglomerates and possibly some air volume within the primary MPI particles 224 (depending on the ability of pycnometer gas to penetrate into these particles). The 225 BIA values represent the change in specific volume between the loose and tapped 226 bulks. 227

228 **3.2 BARDS profiles for MPI and agglomerated MPI**

This section compares the behaviour of agglomerated and non-agglomerated MPI, 229 by initially comparing the BARDS profile of the MPI powder with one of the 230 agglomerates, i.e. MPI-W2, at 0.1% w/v concentration of powder in water. The effect 231 of agglomerate particle size, binder type and powder concentration in water are 232 considered in subsequent sections. The BARDS acoustic profiles for the MPI powder 233 and MPI-W2 are presented in Fig. 3a. For both powders, the frequency initially 234 decreases to a minimum value and then increases gradually back to the original 235 steady-state frequency. The change in frequency during powder rehydration is due 236 to the change of the volume of gas in the water. The gas volume in water was 237 estimated from equations (5) and (6) using the BARDS frequency data. The gas 238 volume profiles for MPI and its agglomerate are presented in Fig. 3b. This shows a 239 240 greater volume of gas release for the MPI agglomerate, which is to be expected considering its porosity is greater than the MPI powder. During the test, gas is being 241 generated as gas is transferred from the particles into the water, and gas is being 242 eliminated as gas leaves the surface of the water at the top of the vessel. The up-243 slope (Fig. 3b) indicates that gas generation rate is greater than gas elimination rate, 244 and vice-versa during the down-slope. The frequency minimum or gas volume 245 maximum indicates where the two rates are equal. 246

The time (t_M) at which the maximum gas volume (or minimum frequency) occurs is indicative of the rehydration ability of the powder, with shorter times being indicative of faster or better rehydration ability (Peddapatla et al., 2017). Fig. 3 shows that the agglomerated MPI powder had a shorter t_M than the MPI powder. Table 4 shows that t_M for the agglomerated powder and MPI powder were 85 s and 140 s, respectively. After t_M , Fig. 3 shows that return trajectories towards steady-state became similar with similar return times of about 450 s.

Previous work in the research group, conducted by Ji et al. (2015), experimentally 254 compared the rehydration behaviour of MPI and agglomerated MPI. The wetting and 255 dissolution behaviour of MPI and MPI agglomerates were measured. The MPI 256 agglomerates in this BARDS study were prepared using the same techniques as 257 presented by Ji et al. (2015) and had similar sizes and used the same binders. Ji et 258 al. (2015) showed that agglomeration improved the wetting ability of the powder 259 because it was easier for water to penetrate into the spaces between the larger sized 260 agglomerate powder. Consequently, the agglomerated powder particles wetted more 261 quickly and the agglomerate structure broke down readily liberating the primary MPI 262 particles. This concurs with the BARDS profiles whereby the guicker wetting leads to 263 faster release of interstitial gas and the break-up of the agglomerate structure 264 leading to the release of vacuole air between the primary particles within the 265 agglomerate structure. This results in greater gas release and a shorter t_M , as 266 illustrated in Fig. 3b. This figure also shows that the MPI and the MPI agglomerate 267 have similar return trajectories to steady-state, with both attaining steady-state at 268 about 450 s. This can be explained by the gradual dissolution of the primary MPI 269 particles, which was experimentally shown by Ji et al. (2015). Once the 270 agglomerates wet and disintegrate to release the primary MPI particles, which is 271 rapid, the rehydration is rate-limited by the dissolution of the MPI primary particles. 272

273 3.3 Effect of particle size and binder

Table 4 shows that all the agglomerates have shorter t_M times than the MPI powder and this is consistent for all three concentrations, highlighting the better wetting behaviour of the agglomerates. This is in line with the effect of granulation on powder wetting (Schuck, 2009). Furthermore, the return to steady-state trajectories

of all the agglomerates and the MPI powders are similar, as illustrated in Figs. 4 and
5, which is indicative of the gradual dissolution of the MPI primary particles.

For the MPI agglomerates themselves, Table 4 shows that the larger sized 280 agglomerates had shorter t_M times. This is due to faster wetting of larger sized 281 particles, as water will penetrate more rapidly into the interstitial void spaces 282 between the agglomerates. The faster wetting is also potentially due to the higher 283 porosities (Table 2) and higher tapped occluded specific air volumes (Table 3) of the 284 larger agglomerates. This may give rise to a more open porous structure within the 285 larger agglomerates allowing water to penetrate more easily into the void spaces 286 within the agglomerates and allowing gas to escape more rapidly. This is consistent 287 with another study showing that large particles forming large pores and high porosity 288 favour fast wetting (Gaiani et al., 2005). Furthermore, the effect of agglomerate size 289 is consistent for all three concentrations. 290

291 The effect of the binder (water vs lactose solution) is also presented in Table 4. Shorter t_M times were obtained for lactose agglomerates containing the lactose 292 binder. This occurs consistently at all three concentrations. The agglomerates with 293 lactose and water binders have very similar porosities (Table 2), thus it is not a 294 porosity effect. It is most likely because the lactose containing bridges are more 295 hydrophilic (Li et al., 2016), which more rapidly dissolve resulting in easier break-up 296 of agglomerates and release of air into the water. The presence of lactose can inhibit 297 protein interaction and pave the way for water transfer within micelles during the 298 rehydration process (Baldwin, 2010). 299

The trends presented by BARDS above are in line with work presented by Ji. et al. (2016), which also showed that larger agglomerate particle size and lactose binder

resulted in faster wetting. This shows that the BARDS technique is effective at
 assessing these effects on the rehydration behaviour of the MPI agglomerates.

304 **3.4 Effect of concentration**

As expected, Fig. 5 shows that there is more gas volume released by powder at 305 higher concentrations of powder in water, simply because there is more powder 306 present. Table 4 shows that there is a general trend that increasing the concentration 307 of powder in water from 0.1% to 0.15% to 0.2% results in an increase in the t_M times 308 for the MPI and all the MPI agglomerates. For example increasing the concentration 309 from 0.1% to 0.2% resulted in t_M increasing from 140 s to 244 s for the MPI powder, 310 and from 60 s to 80 s for the MPI-L2 powder. This is indicative of slower wetting at 311 higher concentrations for all the powders, and shows that BARDS is effectively 312 monitoring the influence of concentration. Furthermore, the return to steady-state 313 took longer with increased concentrations, as illustrated in Figs. 4 and 5, with return 314 times being about 450 s for the MPI powders and greater than 800 s for the higher 315 concentrations. This is consistent with work conducted by Vos et al. (2016). 316

There are a number of reasons that potentially explain the effect of concentration 317 on slowing the rehydration behaviour. The more powder that is added the longer it 318 will take for all of the powder to wet and disperse, which is in agreement with a 319 previous study (Fitzpatrick et al., 2014). A notable shift of time taken for the acoustic 320 profiles returning to steady state from t_M was observed with more powder added. It 321 will also result in more gas in the water which will take longer to be eliminated from 322 the water. The more powder present will lead to more powder in solution which 323 lowers the mass transfer driver and thus slows the rehydration process. The more 324 powder present will also tend to increase viscosity which will slow the mass transfer 325

of water and gas between the particles and water, and slows the elimination of gas from the water.

328 **4. Conclusion**

BARDS is a promising technique for studying the rehydration behaviour of powders 329 by measuring the gas release behaviour over time. In this work, it was shown that 330 the BARDS technique could be applied for assessing the rehydration behaviour of 331 MPI agglomerates. Their faster gas generation showed that the agglomerates wetted 332 and dispersed more rapidly than the regular MPI powder. The similar return 333 trajectories to steady-state showed and the dissolution behaviours of the 334 agglomerates were similar to that of the MPI and were rate-limited by the slow 335 dissolution of the MPI primary particles. The technique was able to distinguish the 336 different wetting and rehydration behaviours of different size agglomerates and 337 agglomerates formed with different binders, because these factors influence the rate 338 at which the agglomerates are wetted and rate at which the agglomerate structure 339 disintegrates and releases gas. Overall, the study has shown that BARDS is a 340 convenient, easy to use technique that can be applied to study the rehydration 341 behaviour of agglomerates and to compare with the non-agglomerated powder. 342

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Sample	MPI	MPI-W1	MPI-W2	MPI-L1	MPI-L2
D[V, 0.1] / µm	22.6 ± 1.4^{a}	47.7 ± 2.0^{b}	$69.3 \pm 9.3^{\circ}$	53.0 ± 0.7^{b}	$71.2\pm2.2^{\circ}$
$D[V,0.5]/\mu m$	49.8 ± 2.5^{a}	118.8 ± 1.7^{b}	207.3 ± 15.5^{d}	117.3 ± 3.5^{b}	$168.8 \pm 9.3^{\circ}$
$D[V,0.9]/\mu m$	95.6 ± 2.1^{a}	193.0±2.3 ^b	334.7 ± 20.0^{d}	189.8±9.3 ^b	$290.2 \pm 14.6^{\circ}$

Table 1		
Particle size	of MPI and MPI	agglomerates

Table 2

Densities and porosity of MPI and MPI agglomerates.

	MPI	MPI-W1	MPI-W2	MPI-L1	MPI-L2
Loose bulk density (g/L)	261±2	226 <u>+</u> 6	223±18	218±4	200±6
Tapped bulk density (g/L)	431±5	330 <u>+</u> 6	277 <u>+</u> 4	311 <u>±</u> 0	265 <u>+</u> 1
Apparent density (g/L)	1073 <u>+</u> 83	1119 <u>+</u> 22	1108 <u>+</u> 39	1103±5	1082 <u>+</u> 31
Porosity %	59.5 <u>+</u>	70.5 <u>+</u>	$75.0\pm$	71.8 <u>+</u>	75.5±
	0.42	0.49	0.37	0.04	0.06

 Table 3

 Specific air volumes of MPI and MPI agglomerates: bulk interstitial air, tapped occluded air, vacuole occluded air.

	Unit	MPI	MPI-W1	MPI-W2	MPI-L1	MPI-L2
Bulk interstitial air	mL/	151 ± 0^{b}	140 <u>+</u> 16 ^b	89 ± 22^{a}	139 <u>+</u> 9 ^b	122 <u>+</u> 16 ^{ab}
	100g					
Tapped occluded air	mL/	139 <u>+</u> 2 ^a	214 <u>+</u> 5 ^b	271 <u>+</u> 5 ^d	230 ± 0^{c}	284 ± 1^{e}
Ć	100g					
Vacuole occluded air	mL/	$27.8 \pm 1.0^{\circ}$	24.0 ± 0.3^{a}	24.8 ± 0.4^{ab}	25.7 ± 0^{b}	$27.5 \pm 0.4^{\circ}$
	100g					

Table 4

Time (t_M) at frequency minimum (or gas volume maximum) during BARDS test for MPI and MPI agglomerates.

Concentration (w/v)	Components	t _M (s)	
	MPI	140	
	MPI-W1	109	
0.10 %	MPI-W2	85	
	MPI-L1	80	
0.10 % 0.15 % 0.20 %	MPI-L2	60	
	MPI	140	
	MPI-W1	118	
0.15 %	MPI-W2	109	
	MPI-L1	93	
	MPI-L2	80	
	MPI	244	
	MPI-W1	163	
0.20 %	MPI-W2	118	
	MPI-L1	101	
	MPI-L2	80	
	R		

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Fig.3. (A) BARDS frequency spectrum and (B) gas volume profile of MPI and MPI-W2 agglomerate at 0.1 % concentration.

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Fig.1



Fig.1. Illustration of air in agglomerated MPI powder: vacuole occluded air within primary MPI particles, occluded air between primary particles within agglomerates, and tapped interstitial air between agglomerates.

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Fig.2



Fig.2. A representative spectrum of 0.025 g agglomerated MPI in BARDS, showing different phases of the spectrum.

Fig.3



Fig.3. (A) BARDS frequency spectrum and (B) gas volume profile of MPI and MPI-W2 agglomerate at 0.1 % concentration.

Fig.4



Fig.4. The BARDS frequency spectrum for MPI, MPI-W, MPI-L at concentrations of A: 0.1 %, B: 0.15 %, C: 0.2 %.



Fig.5

Fig.5. The gas volume profile for MPI, MPI-W, MPI-L at concentrations of A: 0.1 %, B: 0.15 %, C: 0.2 %.

Highlights:

- 1. BARDS successfully monitored rehydration of MPI agglomerates.
- 2. BARDS showed that MPI agglomerates wetted more quickly than MPI powder.
- 3. BARDS showed that agglomerate size and binder type influenced rehydration.
- 4. Dissolution of MPI agglomerates was rate-limited by dissolution of primary MPI particles.