

**UCC Library and UCC researchers have made this item openly available.
Please [let us know](#) how this has helped you. Thanks!**

Title	Monitoring of pilot-scale induction processes for dairy powders using inline and offline approaches
Author(s)	O'Sullivan, Jonathan J.; Schmidmeier, Christiane; Drapala, Kamil P.; O'Mahony, James A.; Kelly, Alan L.
Publication date	2016-10-27
Original citation	O'Sullivan, J. J., Schmidmeier, C., Drapala, K. P., O'Mahony, J. A. and Kelly, A. L. (2016) 'Monitoring of pilot-scale induction processes for dairy powders using inline and offline approaches', Journal of Food Engineering, 197, pp. 9-16. doi: 10.1016/j.jfoodeng.2016.10.023
Type of publication	Article (peer-reviewed)
Link to publisher's version	http://dx.doi.org/10.1016/j.jfoodeng.2016.10.023 Access to the full text of the published version may require a subscription.
Rights	© 2016, Elsevier Ltd. All rights reserved. This manuscript version is made available under the CC BY-NC-ND 4.0 license. https://creativecommons.org/licenses/by-nc-nd/4.0/
Item downloaded from	http://hdl.handle.net/10468/11777

Downloaded on 2021-11-27T15:50:48Z

Accepted Manuscript

Monitoring of pilot-scale induction processes for dairy powders using inline and offline approaches

Jonathan J. O'Sullivan, Christiane Schmidmeier, Kamil P. Drapala, James A. O'Mahony, Alan L. Kelly



PII: S0260-8774(16)30384-3

DOI: [10.1016/j.jfoodeng.2016.10.023](https://doi.org/10.1016/j.jfoodeng.2016.10.023)

Reference: JFOE 8700

To appear in: *Journal of Food Engineering*

Received Date: 9 September 2016

Revised Date: 21 October 2016

Accepted Date: 23 October 2016

Please cite this article as: O'Sullivan, J.J., Schmidmeier, C., Drapala, K.P., O'Mahony, J.A., Kelly, A.L., Monitoring of pilot-scale induction processes for dairy powders using inline and offline approaches, *Journal of Food Engineering* (2016), doi: 10.1016/j.jfoodeng.2016.10.023.

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

1 **Monitoring of pilot-scale induction processes for dairy powders using inline and offline**
2 **approaches**

3

4 Jonathan J. O'Sullivan^{a,b}, Christiane Schmidmeier^{a,b}, Kamil P. Drapala^{a,b}, James A. O'Mahony^{a,b}, Alan L.
5 Kelly^{a,b*}

6

7 ^a*School of Food and Nutritional Sciences, University College Cork, Cork, Ireland*

8 ^b*Dairy Processing Technology Centre, University College Cork, Cork, Ireland*

9

10 * Corresponding author: *Email address:* a.kelly@ucc.ie

11

12

13 **ABSTRACT**

14

15 The induction of two dairy powders, skim milk powder (SMP; low-protein content), and milk
16 protein isolate (MPI, high-protein content), was studied. The powder induction approaches
17 investigated were (1) eductor alone, (2) eductor with a static mixer, and (3) eductor with high
18 shear inline mixing. Measurement of pressure drop, from which viscosity was determined
19 inline using the Hagen-Poiseuille equation, offline viscometry and particle size analyses were
20 performed. High shear inline mixing provided the most efficient induction of powders. In
21 addition, more rapid powder induction, as observed from particle size analysis, was achieved
22 for SMP in comparison to MPI, owing to its better rehydration properties. Inline pressure
23 drop data demonstrated that dissolution of MPI had two distinct phases: (i) powder
24 introduction, and (ii) powder breakdown, irrespective of configuration and concentration
25 employed.

26

27 **Keywords:** Powder induction, Eductor, static mixer, High shear inline mixer, Milk protein
28 isolate, Skim milk powder

29 1. Introduction

30 In the food industry, supply chains from primary production to finished product often
31 require several transformations of physical state. In the case of dairy ingredients, the raw
32 material is milk, with the derived ingredients often dried to a powder state to increase shelf-
33 life, reduce bulk and facilitate use as food ingredients (O'Connell & Flynn, 2007; O'Sullivan
34 & O'Mahony, 2016). For utilisation of these ingredients in food formulations, it is normally a
35 prerequisite that the powder is completely rehydrated. Dairy ingredients that possess a high
36 protein content and have a casein-dominant protein profile are challenging to reconstitute
37 quickly and completely, and thus processors of these ingredients and end-users often employ
38 a range of approaches to achieve homogeneous solutions, such as in-tank agitation, high
39 shear mixing, ultrasonic processing, or hydrodynamic cavitation (Crowley *et al.*, 2015;
40 McCarthy *et al.*, 2014; Schuck *et al.*, 2007; Vos *et al.*, 2016).

41 Powder induction is typically achieved through a two-step approach, although, for
42 powders demonstrating good dissolution behaviour, the first step is adequate: (1) initial
43 mixing of the powder with the solvent, using a powder inductor (also known as eductors),
44 and (2) a means for achieving a uniform dispersion, through shear-induced disruption of
45 powder agglomerates (Bete Fog Nozzle Inc., 1999; Forny *et al.*, 2011; Venegas *et al.*, 2014).
46 Eductor technologies are widely used in industrial applications, such as lean phase pneumatic
47 conveying, powder induction and liquid blending. Eductors usually consist of two inlets and
48 a single outlet (Fig. 1d). One of the inlets narrows to a constricted point, referred to as a
49 nozzle, while the second inlet is typically perpendicular to the exit of the first inlet, where at
50 this point both streams intersect at a locus point, converge, and exit through a single outlet.
51 At the locus point, the contents of the perpendicular inlet are drawn into contact with the
52 tangentially flowing fluid from the nozzle by means of the venturi effect (Douglas *et al.*,
53 2005; Gogate & Kabadi, 2009; Venegas *et al.*, 2014). Powder induction can be achieved in

54 either a batch (*e.g.*, batch stirred tank), continuous (*e.g.*, powder eductor) or semi-continuous
55 configuration (*e.g.*, eductor with a recirculation loop).

56 Static mixers are devices that are readily used in continuous processing for mixing
57 operations. Static mixers are motionless inserts, also known as elements, within a pipeline,
58 which redirect fluid flow in directions transverse to the main direction of flow (Thakur *et al.*,
59 2003). SMX static mixers (Sulzer Chemtech, Winterthur, Switzerland; Fig. 1e) disrupt bulk
60 fluid flow through the development of striations due to their structure, and further disrupt
61 flow by each consecutive element being oriented by 90° to the preceding one (Ghanem *et al.*,
62 2014; Mihailova *et al.*, 2015; 2016).

63 High shear mixing technologies are widely used for the disruption of powder
64 aggregates to form homogeneous solutions and in emulsification applications (Hall *et al.*,
65 2013). The configuration of these mixers is that of a rotor-stator, and they can be used as
66 inline devices for either continuous processing (*i.e.*, single pass mode) or batch processing
67 (*i.e.*, multiple pass mode) (Hall *et al.*, 2011). The shear rate range for high shear mixers is
68 typically within the range $20,000 - 100,000 \text{ s}^{-1}$ (Pacek *et al.*, 2007).

69 In this study, three powder induction approaches were investigated: (1) eductor alone,
70 (2) eductor integrated with an SMX static mixer, and (3) eductor integrated with a high shear
71 inline mixer. The powders examined were low (skim milk powder; SMP) and high (milk
72 protein isolate; MPI) protein content dairy ingredients, in order to comparatively assess the
73 processing performance and industrial relevance of these approaches for rehydration of dairy
74 powders across a wide range of protein content. The objectives of this research were to
75 discern differences in rehydration properties of the selected dairy powders, SMP and MPI, in
76 terms of wettability, dispersibility and changes in particle size, and relate these differences to
77 variations in the rate of powder induction, as monitored inline using a pressure drop approach

78 to calculate viscosity, by applying the Hagen-Poiseuille equation. This approach could allow
79 for the real-time monitoring of industrial dissolution processes for dairy ingredients, and
80 allow manufacturers to optimise such processes for shear energy and time, with major
81 energy-saving potential.

82

83 **2. Materials and methods**

84 *2.1. Materials*

85 Milk protein isolate (MPI) was kindly provided by Kerry Ingredients and Flavours
86 (Listowel, Ireland). The skim milk powder (SMP) used in this study was sourced from a local
87 commercial outlet. The composition of the SMP and MPI is presented in Table 1. The water
88 used throughout this study was deionised water, unless stated otherwise.

89

90 *2.2. Powder induction configuration*

91 Powder induction was conducted at two protein concentrations, 3.6 and 7.2% (w/w),
92 for both SMP and MPI. Three configurations were used to induct the dairy ingredients into
93 solution: (a) eductor alone, (b) eductor and SMX static mixer, and (c) eductor and inline high
94 shear mixer (Fig. 1). The induction process was started by filling the closed-loop liquid
95 system with the required amount of deionised water to achieve the desired protein
96 concentration for the different ingredients, and initialising the progressive cavity pump
97 (Torqueflow, Sydex, UK) to a volumetric flow rate of 675 L h⁻¹. The required mass of
98 powder was loaded carefully into the powder hopper, and introduced to the liquid system by
99 means of a ball valve (25.4 mm internal diameter) and an in-house-designed and custom-
100 fabricated (Liam A. Barry Ltd., Cork, Ireland; Fig. 1d) eductor, whereby the powder is drawn

101 into the liquid stream by means of the venturi effect (Douglas *et al.*, 2005; Gogate & Kabadi,
102 2009). The total mass within the system after powder induction was 2 kg for all experimental
103 instances, and samples for offline analysis were collected from a sampling port located before
104 the inlet to the pump. The temperature at the start of the induction process was 20°C, and
105 increased by *ca.* 8°C during the induction process due to the action of the pump.

106 The SMX static mixer employed in this study was an 8-element 19.05 mm mixer (*i.e.*,
107 D20) and 3D printed (Shapeways, USA) in stainless steel from a CAD file. SMX static mixer
108 elements have a characteristic pattern with six planes of blades, with each opposing plane at
109 90° to the preceding one (Fig. 1e). SMX static mixers are designed for flow within the
110 laminar flow regime and rely upon disrupting and recombining the bulk of the inlet into
111 smaller streams, using a series of channels (Mihailova *et al.*, 2015; 2016). The maximum
112 observed Reynolds number (Re) within the SMX mixer was *ca.* 10, as determined from Eq. 1:

$$113 \quad Re = \frac{\rho v d}{\eta} \quad (1)$$

114 where ρ is the density (kg/m^3), v is the average velocity (m s^{-1}), d is the internal diameter
115 (19.05 mm) and η is the viscosity (Pa.s). The approximate shear rate observed within the
116 SMX static mixer was calculated using the Streiff-Jaffer correlation as follows (Mihailova *et*
117 *al.*, 2016; Streiff *et al.*, 1999):

$$118 \quad \dot{\gamma} = \frac{64v}{d} \quad (2)$$

119 where $\dot{\gamma}$ is the shear rate (s^{-1}). The maximum observed shear rate within the SMX static mixer
120 was calculated as *ca.* 2,200 s^{-1} .

121 The inline high shear mixer used in this study was a YTRON-Z (1.50FC, YTRON
122 Process Technology GmbH, Germany), operating at 100%, yielding *ca.* 6,000 rpm. The

123 typical shear rate range of high shear mixers is between *ca.* 20,000 and 100,000 s⁻¹ (Pacek *et*
124 *al.*, 2007).

125

126 2.3. Wettability and dispersibility

127 Wettability was determined as described by Schuck *et al.* (2012) and powders
128 possessing wettability times of 30, 60 or > 120 s are categorised as very wettable, weattable,
129 and non-wetting, respectively (Schuck *et al.*, 2012). Dispersibility measurements were
130 conducted as described by Schuck *et al.* (2012), and dispersibility index was calculated as
131 follows:

$$132 \text{ Dispersibility Index} = \frac{(100+w) \cdot X_{DM}}{(100 - X_{RW}/100) \cdot w} \quad (3)$$

133 where *w* is the mass of powder used (10 g), X_{DM} is the dry matter content of the filtrate after
134 sieving (% w/w), and X_{RW} is the moisture content of the powder (% w/w).

135

136 2.4. Contact angle characterisation

137 The contact angle (θ) of SMP and MPI powders was assessed on powder samples that
138 had been compressed in order to produce cylindrical tablets, to minimise surface variations
139 between the investigated powders. SMP and MPI were compressed to form cylindrical tablets
140 through application of ~78.5 kN for 10 s using a stamp die with a diameter of 1.3 cm (15 Ton
141 Manual Hydraulic Press, Specac, UK). The contact angle between cylindrical tablets of SMP
142 or MPI and ultrapure water was measured using optical tensiometry (Attension Theta, Biolin
143 Scientific Holding AB, Sweden). A drop (10 μ L) of water was deposited centrally on the
144 surface of the tablets of either SMP or MPI as a sessile drop and contact angle was measured
145 over 5 min.

146 2.5. Particle size and microstructure of powders

147 The particle size distribution (PSD) for SMP and MPI powders was measured by
148 static light-scattering using a Mastersizer 3000 (Aero S, Malvern Instruments, UK). Powder
149 particle size was reported as $d_{4,3}$ (*i.e.*, volume-weighted mean particle size) and PSD data
150 (volume *vs.* size class). Scanning electron microscopy (SEM; JSM-5510, Jeol Ltd., Japan)
151 was used to visualize the microstructure of powder samples and determine if there were any
152 morphological differences between SMP and MPI. The investigated powder samples were
153 placed upon double-sided adhesive conductive carbon tape, attached to SEM stubs, sputter-
154 coated with gold/palladium (80:20) and scanned at 5 kV.

155

156 2.6. Viscosity determination: calculated versus experimental approaches

157 Viscosity was calculated from experimentally-measured pressure drop readings, and
158 compared to experimentally-measured viscosity, in order to validate the calculated viscosity
159 results. Pressure drop was recorded for SMP and MPI solutions, at both protein
160 concentrations, using the three experimental setups (Fig. 1), and was recorded using two
161 pressure transducers (PR-33X, Keller, UK), positioned 1.08 m apart. Pressure differential
162 data was collected, before powder induction, during the powder induction process, and for 15
163 min after completion of powder addition. Calculated viscosity values were determined from
164 *Eq. 4*, the Hagen-Poiseuille equation, using experimentally-measured pressure drop values as
165 follows (Douglas *et al.*, 2005):

$$166 \eta_{\text{calculated}} = \frac{\pi \Delta P d^4}{128 L Q} \quad (4)$$

167 where $\eta_{calculated}$ is the calculated viscosity (Pa.s), ΔP is the pressure differential across a given
 168 straight section of pipeline (Pa), d is the internal diameter (19.05 mm), L is the length over
 169 which the pressure drop was recorded (1.08 m), and Q is the volumetric flow rate (m^3s^{-1}).

170 The experimental viscosity was measured for SMP and MPI dispersions/solutions, 15
 171 min after complete powder addition from each of the three investigated configurations at a
 172 protein concentration of 7.2% (w/w), and control solutions, which were prepared at a protein
 173 concentration of 7.2% (w/w) using overhead stirring (250 rpm with a 4-bladed, 99 mm
 174 diameter impeller, at 22°C) for 2 h. The beaker in which the control solutions were prepared
 175 had an internal diameter of 178 mm and a liquid height of 81 mm, with the impeller being
 176 positioned centrally. The control solutions were prepared and analysed as a comparison to
 177 solutions produced using the powder induction setups (Fig. 1). The experimental viscosity
 178 (η_{exp}) was measured using a rotational viscometer (Haake RotoVisco 1 Rotational
 179 Viscometer, Thermo Fisher Scientific, USA) equipped with a cylindrical double-gap cup and
 180 rotor (DG43, Thermo Fisher scientific, USA) as described by Mulcahy *et al.* (2016).
 181 Apparent viscosity was measured at a temperature of 24°C, the mean temperature at which
 182 the powder induction was conducted (Section 2.2). A shear rate of 275 s^{-1} was used for
 183 viscosity determination, as this was the calculated shear rate within the 1.08 m section from
 184 which the pressure drop was recorded, using Eq. 5 (Douglas *et al.*, 2005):

$$185 \quad \dot{\gamma} = \frac{8v}{d}, \text{ where } v = \frac{Q}{A} \quad (5)$$

186 where $\dot{\gamma}$ is the shear rate (s^{-1}), d is the internal diameter (19.05 mm), v is the average velocity
 187 (m s^{-1}), Q is the volumetric flowrate (m^3s^{-1}), and A is the cross sectional area (m^2).

188

189 *2.7. Particle size of protein dispersions*

190 The changes in particle size for inducted SMP and MPI solutions (1, 5 and 15 min),
191 and control solutions (1, 15 and 120 min; Section 2.6), as a function of time, were measured
192 by static light-scattering using a Mastersizer 3000 (Hydro EV, Malvern Instruments, UK).
193 *Eq. 6* was used in order to determine the number of times which the protein dispersions had
194 been recirculated through the system at the investigated time points (1, 5 and 15 min) for all
195 configurations (O'Sullivan *et al.*, 2015):

$$196 \text{ Pass number} = \frac{Q \times t}{V} \quad (6)$$

197 where Q is the volumetric flowrate (m^3s^{-1}), t is the residence time (s), and V is the volume
198 within the system (m^3). The mean number of passes for which the protein dispersions would
199 have been subjected to time intervals of 1, 5 and 15 min was 5, 28 and 84 passes,
200 respectively.

201

202 2.8. Statistical analysis

203 Data presented are the average and standard deviation of at least three repeat
204 measurements, from one lot of each powder. Student's t-test with a 95% confidence interval
205 was used to assess the significance of the results obtained; t-test data with $P < 0.05$ were
206 considered statistically significant.

207

208 3. Results and discussion

209 3.1. Comparison of the physical and rehydration properties of SMP and MPI

210 The size distribution of particles in skim milk powder (SMP) and milk protein isolate
211 (MPI) was initially investigated (Fig. 2). SMP powder had a significantly ($P < 0.05$) larger

212 particle size than that of MPI powder, and in addition demonstrated a mono-modal size
213 distribution, whereas MPI exhibited a broader distribution, with shoulders either side of the
214 main peak. The observed size of particles in MPI is in agreement with results presented by
215 Crowley *et al.* (2015), for MPC90 (Milk Protein Concentrate); however, SMP, which had a
216 composition analogous to that of MPC35, exhibited a significantly ($P < 0.05$) larger size than
217 MPC35. This was attributed to the nature of commercial retail SMP, which is typically
218 agglomerated in order to enhance its instant properties (Turchiuli *et al.*, 2013), in comparison
219 to the powders used within the study of Crowley *et al.* (2015), which had predominantly
220 discrete powder particles rather than agglomerated structures, as observed by SEM analysis
221 (Vos *et al.*, 2016).

222 In order to investigate these observations further, SMP and MPI powders were
223 examined by SEM (Fig. 3). Particles in SMP (Fig. 3a) appeared to be agglomerated
224 structures, where the agglomerates consisted of many individual powder particles. In the case
225 of MPI (Fig. 3b), discrete powder particles can be seen, possessing a wide range of sizes from
226 larger particles ($\sim 40 \mu\text{m}$) to smaller particles ($\sim 10 \mu\text{m}$). These results are in agreement with
227 the previously discussed particle size measurements (Fig. 2), and highlight the morphological
228 differences between the two ingredients investigated.

229 The time taken to wet SMP powder was significantly ($P < 0.05$) lower than that of
230 MPI, where SMP was classified as wettable ($> 30 \text{ s}$, and $\leq 60 \text{ s}$), while MPI was categorised
231 as a non-wetting powder ($> 120 \text{ s}$). In addition, the dispersibility index of SMP was
232 significantly ($P < 0.05$) greater than that of MPI, whereby SMP possessed a lower standard
233 deviation (± 1.41), in comparison to MPI (± 34.51). The high degree of variability associated
234 with the dispersibility index of MPI is ascribed to a combination of its poor wetting
235 behaviour, and the nature of the dispersibility test, where non-wetting powders may get
236 mixed to varying degrees over the prescribed 15 s of mixing (Section 2.3.). These observed

237 differences in wetting and dispersibility behaviour are attributed to compositional differences
238 between SMP and MPI (Table 1), as the high content of lactose within SMP allows for more
239 rapid ingress of water into powder particles during rehydration. The obtained values for
240 wettability and dispersibility (Table 1) are in agreement with those of Schuck *et al.* (2012),
241 for similar types of powders.

242 The contact angle (θ) between SMP and MPI and ultrapure water was investigated in
243 order to further evaluate the wetting behaviour of these powders (Fig. 4). SMP had a
244 significantly ($P < 0.05$) lower θ value than that of MPI. The higher content of lactose within
245 SMP makes it more hygroscopic than MPI, allowing for greater rates of moisture imbibition.
246 Crowley *et al.* (2015) determined θ values for MPC35 and MPC90, equivalent to SMP and
247 MPI used in this study, respectively. Contact angle results for SMP used in this study and
248 MPC35 used in the study of Crowley *et al.* (2015) were comparable, with MPC35 having a
249 marginally lower θ than that of SMP. However, the MPI used in this study yielded a
250 significantly ($P < 0.05$) higher θ in comparison to the MPC90 used in the study of Crowley *et*
251 *al.* (2015), even though they had comparable composition profiles. These differences are
252 ascribed to differences in terms of methodology (*i.e.*, different drop volumes and equipment
253 employed), timescale of measurement, which was 300 s rather than 5 s in the study of
254 Crowley *et al.* (2015), and potential differences in heat treatment applied to the skim milk or
255 liquid concentrates in the manufacture of the ingredients. Regardless, the same trend in terms
256 of contact angle value was observed.

257

258 3.2. Comparative assessment of powder induction approaches

259 The calculated viscosity ($\eta_{calculated}$) as a function of time (up to 15 min after complete
260 powder addition) is shown in Fig. 5 for MPI at protein concentrations of 3.6 and 7.2% (w/w),

261 for the three configurations investigated. Data for SMP was also recorded, however, the
262 obtained pressure drop results exhibited high variability owing to the low viscosity of SMP
263 solutions (data not shown). Unexpectedly, no significant differences ($P > 0.05$) were
264 observed when comparing the development of calculated viscosity over induction time
265 between the three different induction approaches, at either concentration for MPI.
266 Nevertheless, significant ($P < 0.05$) differences were observed in the calculated viscosity
267 upon powder addition to the system between 3.6 and 7.2% (w/w), where the sample with
268 higher concentration demonstrated higher initial viscosity values. This greater value was
269 attributed to ~twice the mass of powder being present within the system.

270 MPI exhibited two distinct phases in the development of calculated viscosity as a
271 function of time. In all cases, there was an initial increase in viscosity, followed by a gradual
272 decrease. These distinct phases correspond to: (1) contact of powder with water and swelling,
273 and (2) breakdown of swollen powder agglomerates. A similar trend was observed for the
274 dissolution of native phosphocaseinate in the study of Gaiani *et al.* (2006), who used a
275 rheological approach to monitor rehydration. Two peaks in viscosity were observed, the first
276 peak corresponding to powder wetting, and the second peak corresponding to powder
277 swelling (Gaiani *et al.*, 2006; Schuck *et al.*, 2007). The initial peak and the decrease in
278 viscosity following this peak as presented in the study of Gaiani *et al.* (2006) are comparable
279 to the initial increase in calculated viscosity in the current study, and the trough between
280 peaks to the gradual decrease in calculated viscosity; however, it should be noted that native
281 phosphocaseinate was used in the study of Gaiani *et al.* (2006), rather than MPI, as used in
282 this study (Fig. 5) – the former would have had a much higher casein:whey protein than the
283 latter. Gaiani *et al.* (2006) also used longer times than those in this study (up to 3 h) to
284 achieve complete rehydration; nonetheless, the obtained calculated viscosity results (Fig. 5)

285 are in agreement with those reported by Gaiani *et al.* (2006), as they focus upon the initial
286 stages of rehydration over shorter timescales.

287 The validity of calculated viscosity results was assessed through direct comparisons to
288 experimentally obtained viscosity values at the same shear rate value at which the pressure
289 drop was measured (275 s^{-1}) and the average temperature recorded during the powder
290 induction process (24°C). The values of calculated viscosity ($\eta_{\text{calculated}}$) for MPI and
291 experimental viscosity ($\eta_{\text{experimental}}$) for SMP and MPI solutions (7.2% w/w), compared to
292 control solutions, prepared using overhead stirring (2 h at 250 rpm), are provided in Table 2.
293 Similar trends in comparisons of calculated and experimental viscosities were observed for
294 both SMP and MPI at a concentration of 3.6% (w/w) (data not shown).

295 The trends in $\eta_{\text{experimental}}$ values for SMP and MPI processed using the three
296 investigated induction approaches highlights that, with increasing degree of shear in the
297 process, there was an increase in the viscosity, owing to enhanced protein hydration (García
298 De La Torre *et al.*, 2000; O'Connell & Flynn, 2007). This behaviour was attributed to
299 differences in the level of applied shear between the three approaches, where high-shear
300 inline mixing with an eductor provides shear rates $> 20,000 \text{ s}^{-1}$ (Pacek *et al.*, 2007), SMX
301 static mixing with an eductor provides *ca.* $2,200 \text{ s}^{-1}$ at a volumetric flowrate of 675 L/h (Eq.
302 2; Mihailova *et al.*, 2016), and the eductor alone yields *ca.* 275 s^{-1} (Eq. 6; Douglas *et al.*,
303 2005). In the case of control solutions, higher viscosity values were observed in comparison
304 to solutions prepared using the induction configurations (Table 2), owing to the prolonged
305 preparation time (2 h), allowing for enhanced protein hydration (García De La Torre *et al.*,
306 2000).

307 A comparison of the $\eta_{\text{calculated}}$ and $\eta_{\text{experimental}}$ values for MPI at a concentration of
308 7.2% (w/w) highlight that there is a discrepancy in the values, by a factor of *ca.* 2, whereby

309 the calculated value is overestimated in all instances. This observed difference between
310 experimental and calculated values were ascribed to the nature of the Hagen-Poiseuille
311 equation, which assumes that the fluid demonstrates Newtonian behaviour, whereas it has
312 been established that protein solutions typically exhibit shear-thinning behaviour (Morris *et*
313 *al.*, 1981; O’Sullivan *et al.*, 2014). Nevertheless, the pressure drop approach highlighted that
314 it was suitable as an industrial approach for inline monitoring of dissolution of high-protein-
315 content dairy ingredients, demonstrating variations in viscosity as a function of dissolution
316 time.

317 The changes in particle size as a function of induction time for each of the three
318 dissolution approaches for both of the studied powders was also investigated. Size
319 distribution data for powder particles, and induced dispersions/solutions at time points of 1,
320 5 and 15 min after powder addition, for both SMP and MPI (7.2% w/w), are shown in Fig. 6,
321 along with control samples prepared using overhead stirring as described in Section 2.6, and
322 measured at time intervals of 1, 15 and 120 min. Similar trends in terms of change of particle
323 size distribution as a function of processing time were observed for both SMP and MPI at a
324 concentration of 3.6% (w/w) (data not shown).

325 There were significant differences ($P < 0.05$) in the rate of reduction in size between
326 SMP and MPI, for all dissolution approaches studied, while SMP generally achieved a
327 submicron peak (mean particle size of ~250 nm) more rapidly than MPI. SMP and MPI both
328 have casein-dominant protein profiles, where the diameter of casein micelles is within the
329 range 100–250 nm (O’Connell and Flynn, 2007). Thus, the development of the submicron
330 peak for both powders on reconstitution is associated with the release of casein micelles,
331 where differences in dissolution rate are ascribed to compositional differences between SMP
332 and MPI (Table 1), particularly in terms of SMP having higher lactose content than MPI.
333 This behaviour was previously observed through non-invasive acoustic spectroscopic

334 approaches (*i.e.*, broadband acoustic resonance dissolution spectroscopy; BARDS) and cryo-
335 SEM visualisation as a function of dissolution time by Vos *et al.* (2016), and direct particle
336 size measurements using static light scattering by Crowley *et al.* (2015), whereby a slower
337 release of casein micelles was observed for MPC90 (similar to MPI) in comparison to
338 MPC35 (similar to SMP).

339 The rate of powder dissolution, in terms of development of the nano-sized peak (*i.e.*,
340 casein), was also affected significantly ($P < 0.05$) by the induction technology employed, as
341 the highest shear process (*ca.* 20,000–100,000 s^{-1}), inline high shear mixing with the eductor
342 demonstrated the highest rates of powder rehydration (Pacek *et al.*, 2007), followed by the
343 SMX static mixer in conjunction with the eductor (*ca.* 2,200 s^{-1} ; Mihailova *et al.*, 2016), and
344 lastly by eductor alone (*ca.* 275 s^{-1} ; Douglas *et al.*, 2005). This trend was observed for both of
345 the powders studied. However, in the case of SMP induction using inline high shear mixing
346 (7.2% w/w), an increase in the size of the micron-sized peak was observed at the 15 min
347 processing time. This behaviour is attributed to formation of stable air bubbles, with the air
348 most likely originating from both occluded and interstitial air contained within the SMP
349 powder agglomerates (Fig. 3a).

350 In comparison to the conventional overhead stirring (250 rpm for 120 min), all of the
351 investigated powder induction approaches demonstrated significantly ($P < 0.05$) greater rates
352 of powder dissolution, as observed by the greater rate of development of the submicron peak
353 over a significantly shorter timescale. Furthermore, induction achieved a greater degree of
354 submicron particles in comparison to overhead stirring, for both SMP and MPI, and over a
355 shorter timescale, *i.e.*, 15 min rather than 120 min. The differences between conventional
356 overhead stirring and the investigated induction approaches was due to the extent of
357 processing (*i.e.*, shear rate), whereby, for the solutions prepared using the studied powder
358 induction configurations, all of the material is processed, as there were no conceivable dead-

359 zones in the setup, with the exception of the wall boundary layer (Douglas *et al.*, 2005).
360 However, for overhead stirring of a 2 L batch, dead-zones were inevitable, which would
361 greatly reduce mixing efficiency (Hall *et al.*, 2005).

362

363 **4. Conclusions**

364 This study showed that inline measurement of pressure drop is an effective approach
365 for monitoring in real-time the dissolution kinetics of high-protein dairy ingredients. Pressure
366 drop results were used to determine real-time viscosity data, by means of the Hagen-
367 Poiseuille equation. Inline high shear mixing yielded the most efficient generation of protein
368 solutions, for SMP and MPI, as shown by off-line particle size and viscosity measurements,
369 compared to either an eductor alone or eductor integrated with an SMX static mixer. MPI
370 demonstrated two distinct stages during dissolution as observed by pressure drop results: (1)
371 initial mixing of powder with water and swelling (an increase in viscosity), and (2) disruption
372 of powder agglomerates (a decrease in viscosity). From a technological perspective, this
373 study highlighted the importance of selection of the appropriate induction technology for
374 efficient formation of solutions, whereby processes giving high shear rates are desirable for
375 the induction of high-protein ingredients (MPI), whereas low shear rate technologies may be
376 adequate for low-protein ingredients (SMP). Moreover, this study showed that pressure drop
377 is a suitable inline approach to monitor powder dissolution processes.

378

379 **Acknowledgements**

380 The authors would like to acknowledge the Dairy Processing Technology Centre
381 (DPTC), an Enterprise Ireland initiative, for financial support and permission to publish this
382 work. This work was supported by the Irish State through funding from the Technology

383 Centres programme (Grant Number TC/2014/0016). The authors would like to thank Dr Olga
384 Mihailova of Unilever Research (Port Sunlight, UK) for useful discussions and assistance
385 with respect to the SMX static mixer and data processing. The authors would also like to
386 thank Mike Barry and Kevin McEvoy of Liam A Barry Ltd. for the custom fabrication of
387 many of the components of the experimental setup, and the Department of Anatomy &
388 Neuroscience Imaging Centre, BioSciences Institute, UCC, for assistance with sample
389 preparation and scanning electron microscopy imaging.

390

391 **References**

- 392 Bete Fog Nozzle Inc., 1999. *New Equipment, Processes and Materials - Eductor nozzles.*
393 *Met. Finish.* 97, 73.
- 394 Crowley, S. V., Desautel, B., Gazi, I., Kelly, A.L., Huppertz, T., O'Mahony, J.A., 2015.
395 *Rehydration characteristics of milk protein concentrate powders. J. Food Eng.* 149, 105–
396 113.
- 397 Douglas, J., Gasoriek, J., Swaffield, J., Jack, L., 2005. *Fluid Mechanics*, 5th ed. Prentice Hall.
- 398 Forny, L., Marabi, A., Palzer, S., 2011. Wetting, disintegration and dissolution of
399 agglomerated water soluble powders. *Powder Technol.* 206, 72–78.
- 400 Gaiani, C., Scher, J., Schuck, P., Hardy, J., Desobry, S., Banon, S., 2006. The dissolution
401 behaviour of native phosphocaseinate as a function of concentration and temperature
402 using a rheological approach. *Int. Dairy J.* 16, 1427–1434.
- 403 García De La Torre, J., Huertas, M.L., Carrasco, B., 2000. Calculation of hydrodynamic
404 properties of globular proteins from their atomic-level structure. *Biophys. J.* 78, 719–30.
- 405 Ghanem, A., Lemenand, T., Della Valle, D., Peerhossaini, H., 2014. Static mixers:

- 406 Mechanisms, applications, and characterization methods – A review. *Chem. Eng. Res.*
407 *Des.* 92, 205–228.
- 408 Gogate, P.R., Kabadi, A.M., 2009. A review of applications of cavitation in biochemical
409 engineering/biotechnology. *Biochem. Eng. J.* 44, 60–72.
- 410 Hall, J.F., Barigou, M., Simmons, M.J.H., Stitt, E.H., 2005. Just Because It's Small Doesn't
411 Mean It's Well Mixed: Ensuring Good Mixing in Mesoscale Reactors. *Ind. Eng. Chem.*
412 *Res.* 44, 9695–9704.
- 413 Hall, S., Cooke, M., El-Hamouz, A., Kowalski, A.J., 2011. Droplet break-up by in-line
414 Silverson rotor–stator mixer. *Chem. Eng. Sci.* 66, 2068–2079.
- 415 Hall, S., Pacek, A.W., Kowalski, A.J., Cooke, M., Rothman, D., 2013. The effect of scale and
416 interfacial tension on liquid–liquid dispersion in in-line Silverson rotor–stator mixers.
417 *Chem. Eng. Res. Des.* 91, 2156–2168.
- 418 McCarthy, N.A., Kelly, P.M., Maher, P.G., Fenelon, M.A., 2014. Dissolution of milk protein
419 concentrate (MPC) powders by ultrasonication. *J. Food Eng.* 126, 142–148.
- 420 Mihailova, O., Lim, V., McCarthy, M.J., McCarthy, K.L., Bakalis, S., 2015. Laminar mixing
421 in a SMX static mixer evaluated by positron emission particle tracking (PEPT) and
422 magnetic resonance imaging (MRI). *Chem. Eng. Sci.* 137, 1014–1023.
- 423 Mihailova, O., O'Sullivan, D., Ingram, A., Bakalis, S., 2016. Velocity Field Characterisation
424 of Newtonian and Non-Newtonian Fluids in SMX Mixers Using PEPT. *Chem. Eng.*
425 *Res. Des.*
- 426 Morris, E.R., Cutler, A.N., Ross-Murphy, S.B., Rees, D.A., Price, J., 1981. Concentration
427 and shear rate dependence of viscosity in random coil polysaccharide solutions.
428 *Carbohydr. Polym.* 1, 5–21.

- 429 Mulcahy, E.M., Mulvihill, D.M., O'Mahony, J.A., 2016. Physicochemical properties of whey
430 protein conjugated with starch hydrolysis products of different dextrose equivalent
431 values. *Int. Dairy J.* 53, 20–28.
- 432 O'Connell, J.E., Flynn, C., 2007. The manufacture and application of casein-derived
433 ingredients, in: Hui, Y.H. (Ed.), *Handbook of Food Products Manufacturing*. John Wiley
434 & Sons, New Jersey, pp. 557–593.
- 435 O'Sullivan, J., Arellano, M., Pichot, R., Norton, I., 2014. The Effect of Ultrasound Treatment
436 on the Structural, Physical and Emulsifying Properties of Dairy Proteins. *Food*
437 *Hydrocoll.* 42, 386–396.
- 438 O'Sullivan, J., Murray, B., Flynn, C., Norton, I., 2015. Comparison of batch and continuous
439 ultrasonic emulsification processes. *J. Food Eng.* 167(B), 141–121.
- 440 O'Sullivan, J.J., O'Mahony, J.A., 2016. Food Ingredients, in: *Reference Module in Food*
441 *Science*. pp. 1–3.
- 442 Pacek, A., Baker, M., Utomo, A., 2007. Characterisation of Flow Pattern in a Rotor Stator
443 High Shear Mixer, in: *Proceedings of European Congress of Chemical Engineering*
444 (ECCE-6).
- 445 Schuck, P., Jeantet, R., Dolivet, A., 2012. Determination of Rehydration Ability, in:
446 *Analytical Methods for Food and Dairy Powders*. Wiley-Blackwell, pp. 203–216.
- 447 Schuck, P., Méjean, S., Dolivet, A., Gaiani, C., Banon, S., Scher, J., Jeantet, R., 2007. Water
448 transfer during rehydration of micellar casein powders. *Lait* 87, 425–432.
- 449 Streiff, F.A., Jaffer, S., Schneider, G., 1999. Design and application of motionless mixer
450 technology, in: *ISMIP3*. Osaka, pp. 107–114.
- 451 Thakur, R.K., Vial, C., Nigam, K.D.P., Nauman, E.B., Djelveh, G., 2003. Static Mixers in the

- 452 Process Industries—A Review. *Chem. Eng. Res. Des.* 81, 787–826.
- 453 Turchiuli, C., Smail, R., Dumoulin, E., 2013. Fluidized bed agglomeration of skim milk
454 powder: Analysis of sampling for the follow-up of agglomerate growth. *Powder*
455 *Technol.* 238, 161–168.
- 456 Venegas, P.A., Narváez, A.L., Arriagada, A.E., Llancaleo, K.A., 2014. Hydrodynamic effects
457 of use of eductors (Jet-Mixing Eductor) for water inlet on circular tank fish culture.
458 *Aquac. Eng.* 59, 13–22.
- 459 Vos, B., Crowley, S. V., O’Sullivan, J., Evans-Hurson, R., McSweeney, S., Krüse, J.,
460 Ahmed, M.R., Fitzpatrick, D., O’Mahony, J.A., 2016. New insights into the mechanism
461 of rehydration of milk protein concentrate powders determined by Broadband Acoustic
462 Resonance Dissolution Spectroscopy (BARDS). *Food Hydrocoll.* 61, 933–945.

463 **Figure Legends**

464 **Fig. 1.** Schematic representation of the experimental configurations employed: (a) educator
465 alone, (b) educator and SMX static mixer, and (c) educator and high shear inline mixer. All
466 configurations show a pump and pressure transducers. Panel (d) shows a schematic of the
467 educator configuration and (e) is a CAD diagram of a five element section of a standard SMX
468 static mixer, for which rights of use were acquired from O. Mihailova (Mihailova *et al.*,
469 2015).

470 **Fig. 2.** Particle size distributions for skim milk powder (SMP; solid line; $d_{4,3} = 128.7 \mu\text{m}$) and
471 milk protein isolate (MPI; dashed line; $d_{4,3} = 36.8 \mu\text{m}$).

472 **Fig. 3.** Scanning electron micrographs of (a) skim milk powder (SMP) and (b) milk protein
473 isolate (MPI). Scale bar is 100 μm in both micrographs.

474 **Fig. 4.** Contact angle between skim milk powder (SMP; ●) or milk protein isolate (MPI; ○),
475 and distilled water, measured over 300 s.

476 **Fig. 5.** Development of calculated viscosity upon addition of powder to the system as a
477 function of time for educator alone (solid line), educator and SMX static mixer (long-dashed
478 line), and educator and high shear inline mixer (short-dashed line): (a) 3.6% (w/w) milk
479 protein isolate (MPI), and (b) 7.2% (w/w) MPI.

480 **Fig. 6.** Changes in particle size distribution as a function of processing time, showing powder
481 initially (solid line), and 1 (long-dashed line), 5 (medium-dashed line), and 15 (short-dashed
482 line) min after induction for: (a) skim milk powder (SMP) – educator, (b) milk protein isolate
483 (MPI) – educator, (c) SMP – educator + SMX, (d) MPI – educator + SMX, (e) SMP – educator +
484 YTRON, (f) MPI – educator + YTRON, (g) SMP – control, and (h) MPI – control. The time
485 increments for control samples were 1 (long-dashed line), 15 (medium-dashed line), and 120
486 (short-dashed line) min after powder addition. The concentration in all cases was 7.2% (w/w).

Table 1

Composition of skim milk powder (SMP) and milk protein isolate (MPI), acquired from supplier specification sheets, and measured values for wettability and dispersibility for SMP and MPI.

		SMP	MPI
Composition	Protein (%)	35.9	86
	Moisture (%)	6.5	4
	Fat (%)	0.6	1.5
	Carbohydrate (%)	50.5	1
	Ash (%)	7.9	6
Rehydration Properties	Wettability (s)	59 ± 10	> 120
	Dispersibility (%)	99.9 ± 1.4	27.1 ± 34.5

Table 2

Comparison of calculated viscosity (15 min after powder induction) and experimentally measured viscosity (at a shear rate of 275 s^{-1}) for skim milk powder (SMP) and milk protein isolate (MPI) at protein concentrations of 7.2% (w/w) for the three investigated powder induction approaches.

		$\eta_{\text{calculated}}$ (mPa.s)	$\eta_{\text{experimental}}$ (mPa.s)
	Control solution	-	4.03 ± 0.04
SMP (7.2% w/w)	Eductor	-	2.89 ± 0.07
	Eductor + Static Mixer	-	3.43 ± 0.05
	Eductor + High Shear Mixer	-	4.44 ± 0.12
	Control solution	-	25.2 ± 0.5
MPI (7.2% w/w)	Eductor	7.7 ± 0.7	2.83 ± 0.13
	Eductor + Static Mixer	8.2 ± 0.9	4.21 ± 0.05
	Eductor + High Shear Mixer	9.6 ± 0.6	5.83 ± 0.11

Figures

Fig. 1.

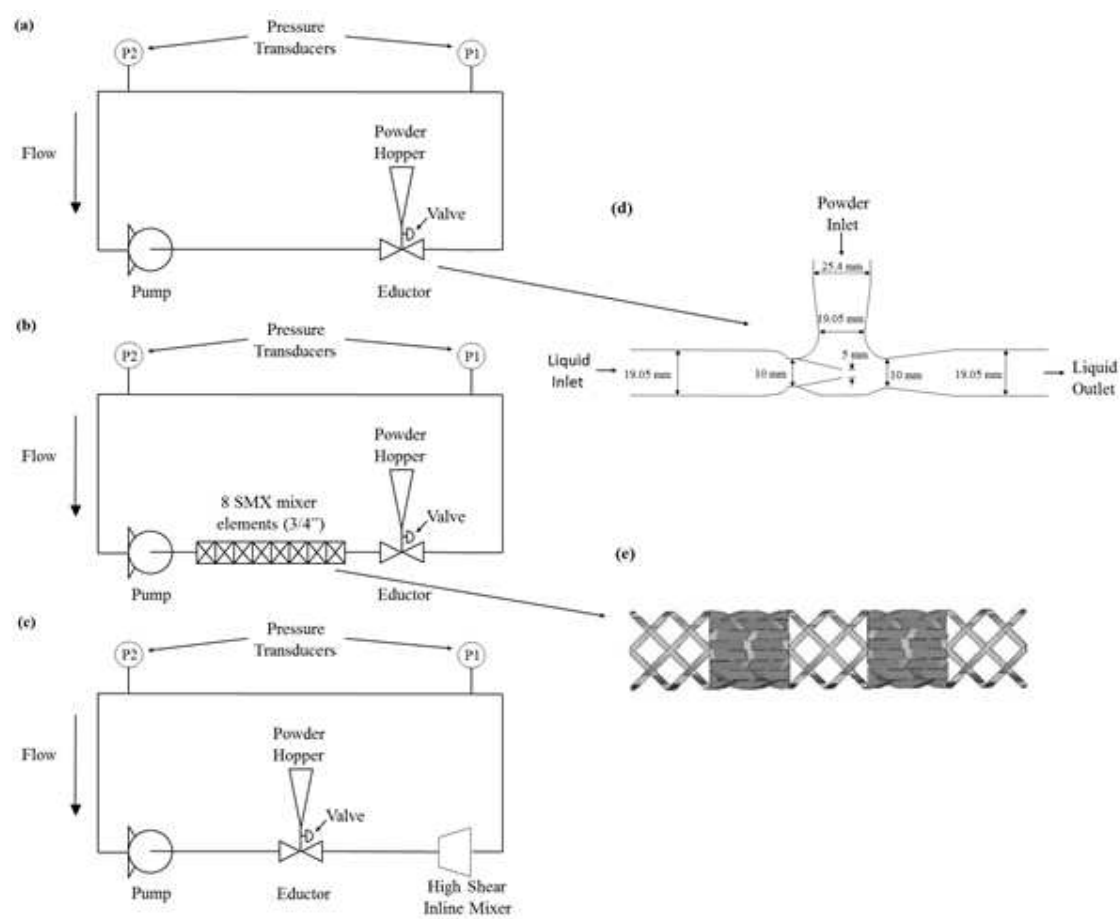


Fig. 2.

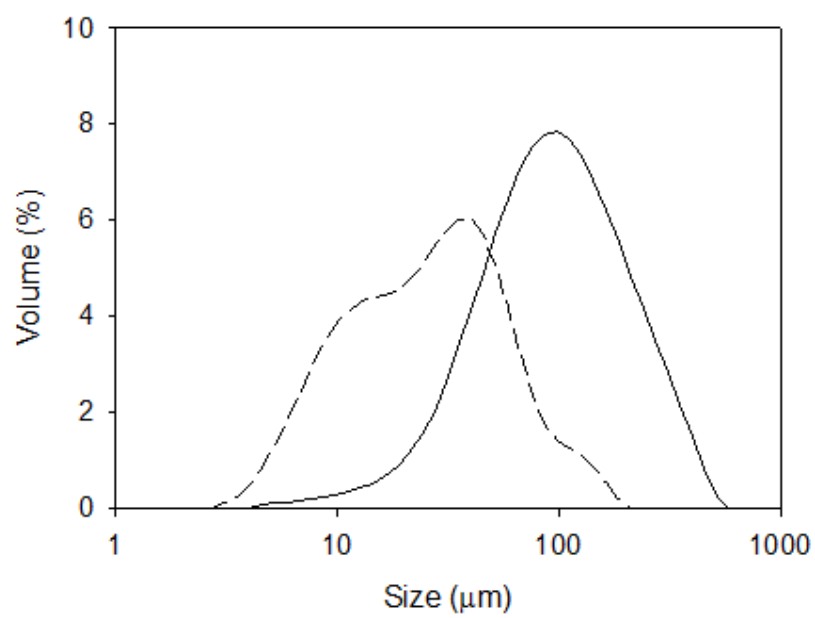


Fig. 3.

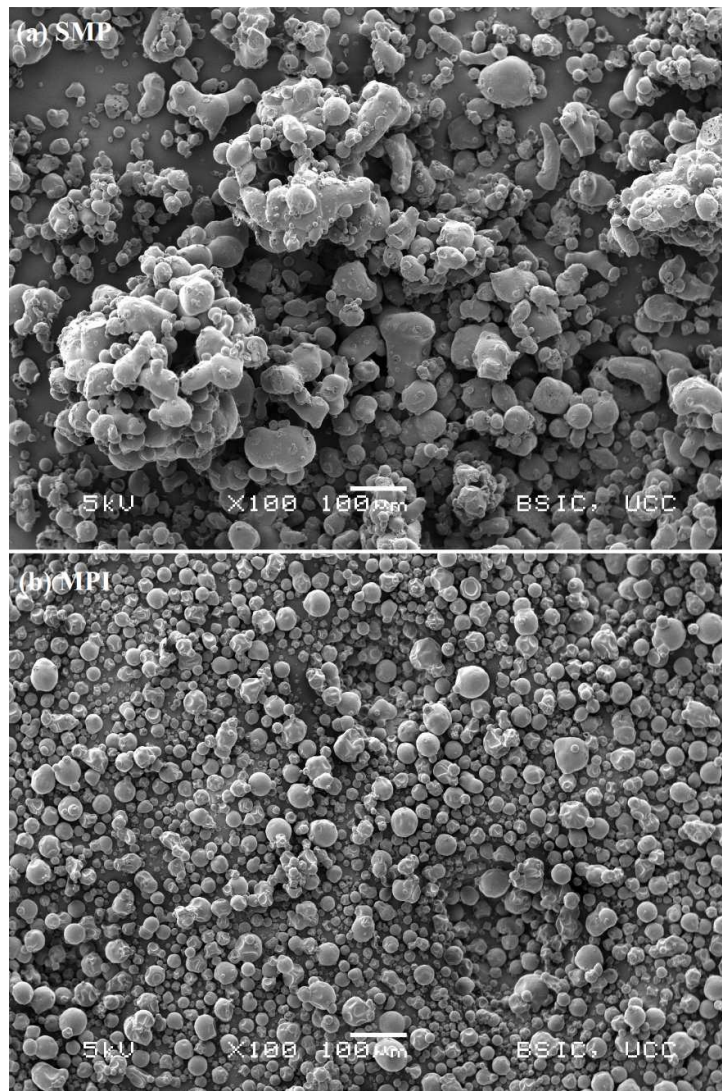


Fig. 4.

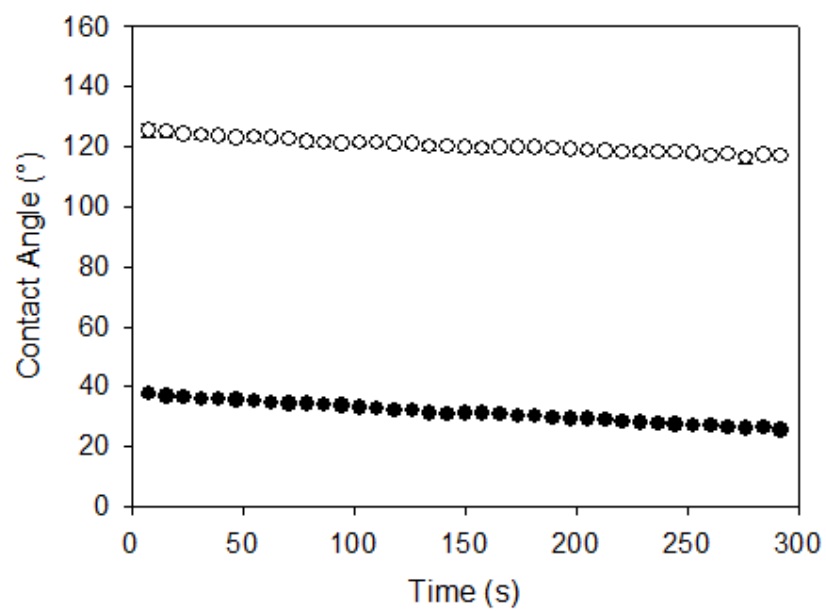


Fig. 5.

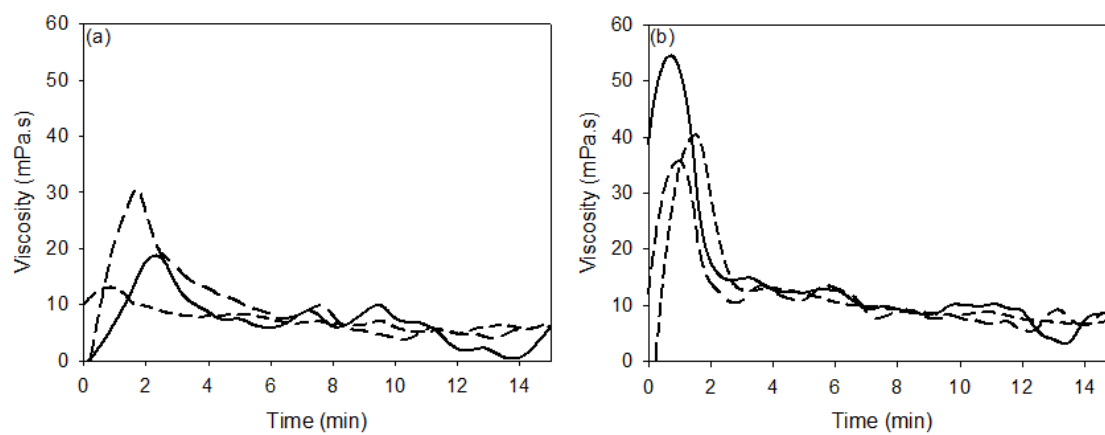
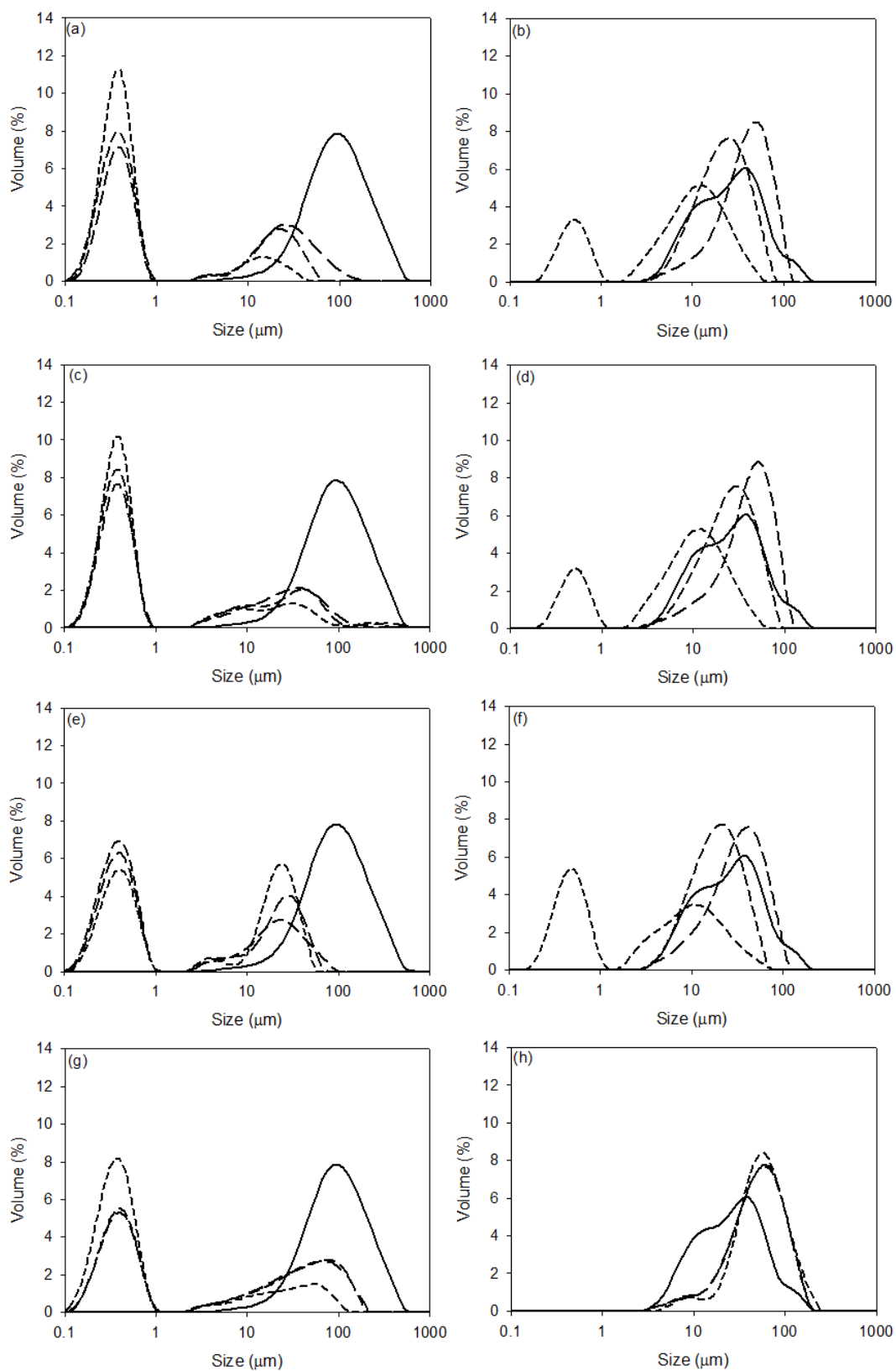


Fig. 6.



Highlights

- Induction of dairy powders, SMP and MPI, was investigated.
- The induction process was monitored inline using pressure drop analysis.
- Pressure drop data allowed for estimation of viscosity during powder dissolution.
- SMP was inducted more rapidly than MPI, due to compositional differences.
- Inline high shear mixing was most effective compared to the other technologies.