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

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

3

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12

**13 ABSTRACT**

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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

## 1. Introduction

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

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

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

## 2. Materials and methods

### 2.1. Materials

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

### 2.2. Powder induction configuration

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

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

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

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

where  $\rho$  is the density (kg/m<sup>3</sup>),  $v$  is the average velocity (m s<sup>-1</sup>),  $d$  is the internal diameter (19.05 mm) and  $\eta$  is the viscosity (Pa.s). The approximate shear rate observed within the SMX static mixer was calculated using the Streiff-Jaffer correlation as follows (Mihailova *et al.*, 2016; Streiff *et al.*, 1999):

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

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

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



typical shear rate range of high shear mixers is between *ca.* 20,000 and 100,000 s<sup>-1</sup> (Pacek *et al.*, 2007).

### 2.3. Wettability and dispersibility

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

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

where *w* is the mass of powder used (10 g), *X<sub>DM</sub>* is the dry matter content of the filtrate after sieving (% w/w), and *X<sub>RW</sub>* is the moisture content of the powder (% w/w).

### 2.4. Contact angle characterisation

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

## 2.5. Particle size and microstructure of powders

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

## 2.6. Viscosity determination: calculated versus experimental approaches

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

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

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

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

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

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

## 2.7. Particle size of protein dispersions

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

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

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

## 2.8. Statistical analysis

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

## 3. Results and discussion

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

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

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

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

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

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

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

### 3.2. Comparative assessment of powder induction approaches

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

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

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

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

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

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

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



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

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

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

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

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

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

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

#### 4. Conclusions

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

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## References

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

## Figure Legends

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

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

**Fig. 3.** Scanning electron micrographs of (a) skim milk powder (SMP) and (b) milk protein isolate (MPI). Scale bar is  $100 \mu\text{m}$  in both micrographs.

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

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

**Fig. 6.** Changes in particle size distribution as a function of processing time, showing powder initially (solid line), and 1 (long-dashed line), 5 (medium-dashed line), and 15 (short-dashed line) min after induction for: (a) skim milk powder (SMP) – educator, (b) milk protein isolate (MPI) – educator, (c) SMP – educator + SMX, (d) MPI – educator + SMX, (e) SMP – educator + YTRON, (f) MPI – educator + YTRON, (g) SMP – control, and (h) MPI – control. The time increments for control samples were 1 (long-dashed line), 15 (medium-dashed line), and 120 (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)	$59 \pm 10$	> 120
	Dispersibility (%)	$99.9 \pm 1.4$	$27.1 \pm 34.5$

**Table 2**

Comparison of calculated viscosity (15 min after powder induction) and experimentally measured viscosity (at a shear rate of  $275 \text{ s}^{-1}$ ) 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)
SMP (7.2% w/w)	Control solution	-	$4.03 \pm 0.04$
	Eductor	-	$2.89 \pm 0.07$
	Eductor + Static Mixer	-	$3.43 \pm 0.05$
	Eductor + High Shear Mixer	-	$4.44 \pm 0.12$
MPI (7.2% w/w)	Control solution	-	$25.2 \pm 0.5$
	Eductor	$7.7 \pm 0.7$	$2.83 \pm 0.13$
	Eductor + Static Mixer	$8.2 \pm 0.9$	$4.21 \pm 0.05$
	Eductor + High Shear Mixer	$9.6 \pm 0.6$	$5.83 \pm 0.11$

## Figures

Fig. 1.

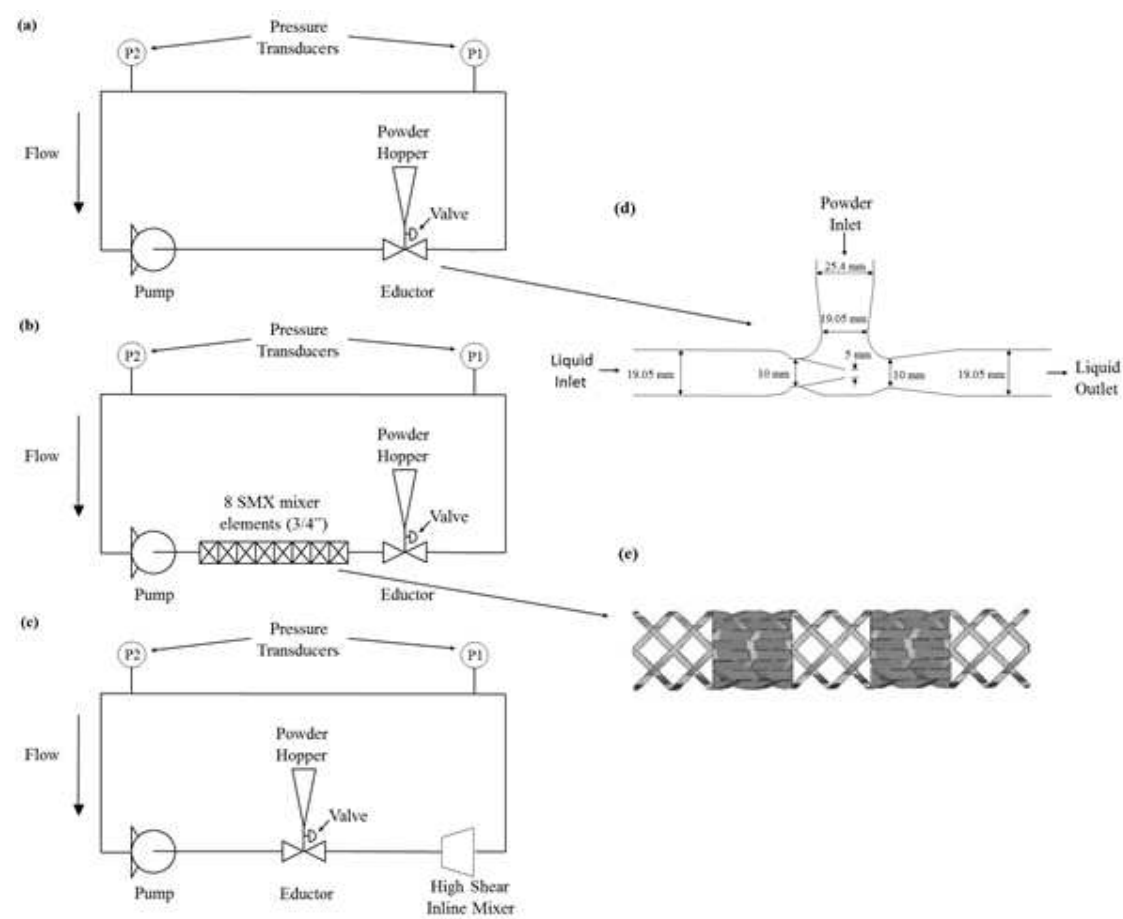


Fig. 2.

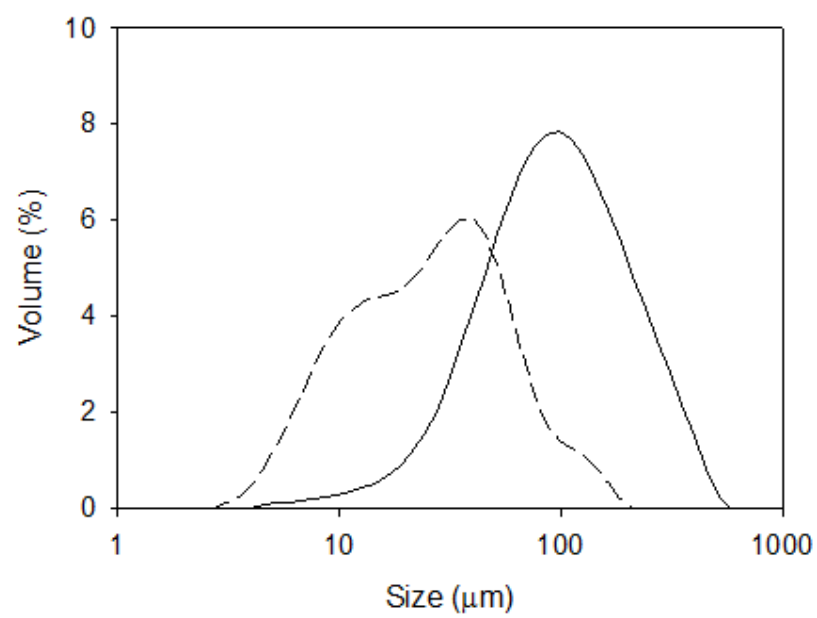


Fig. 3.

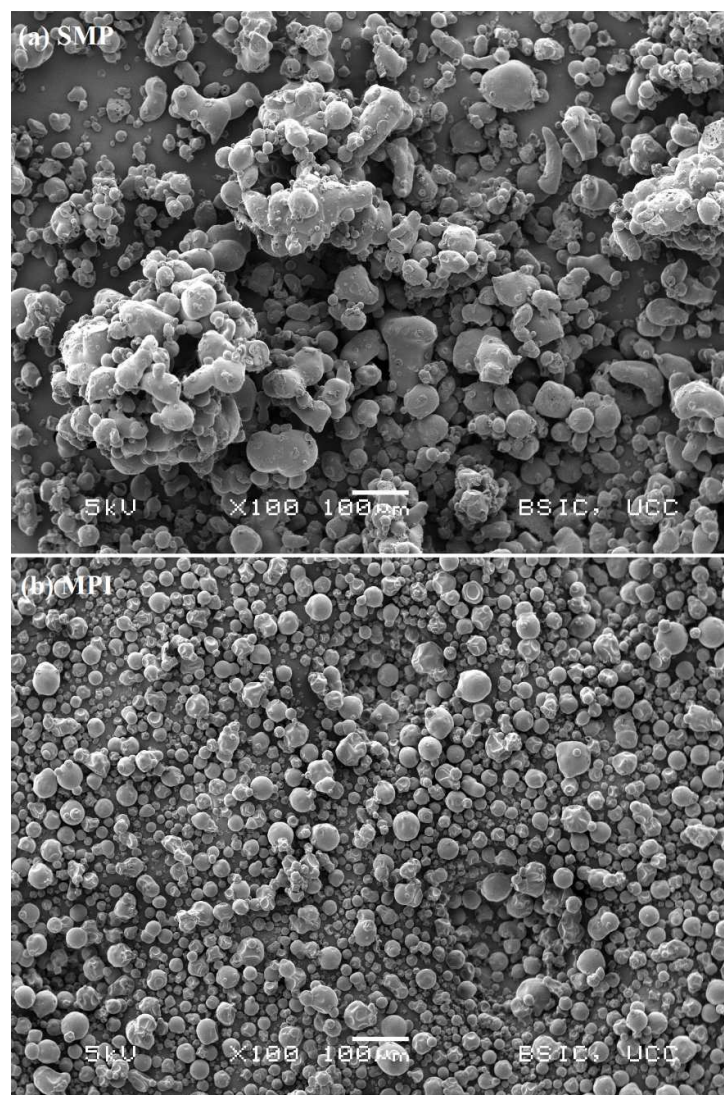


Fig. 4.

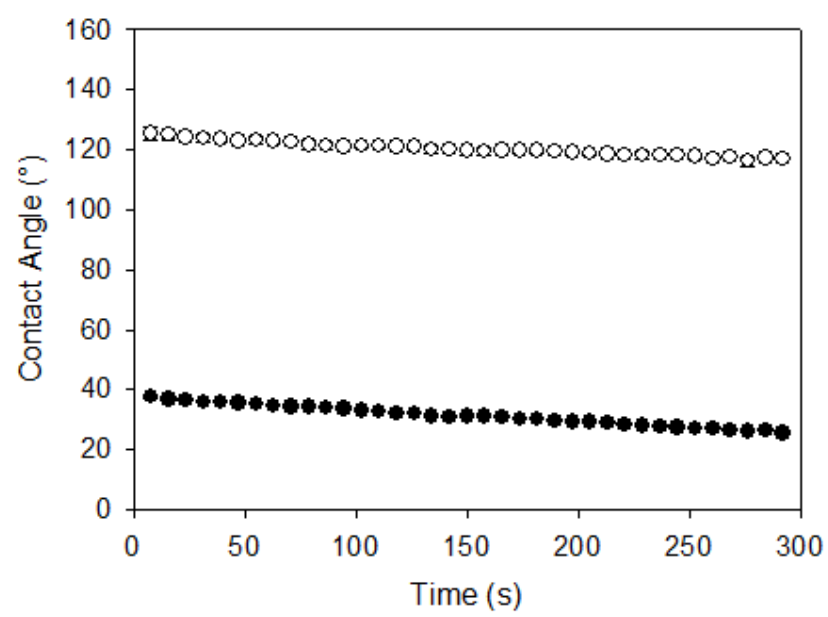


Fig. 5.

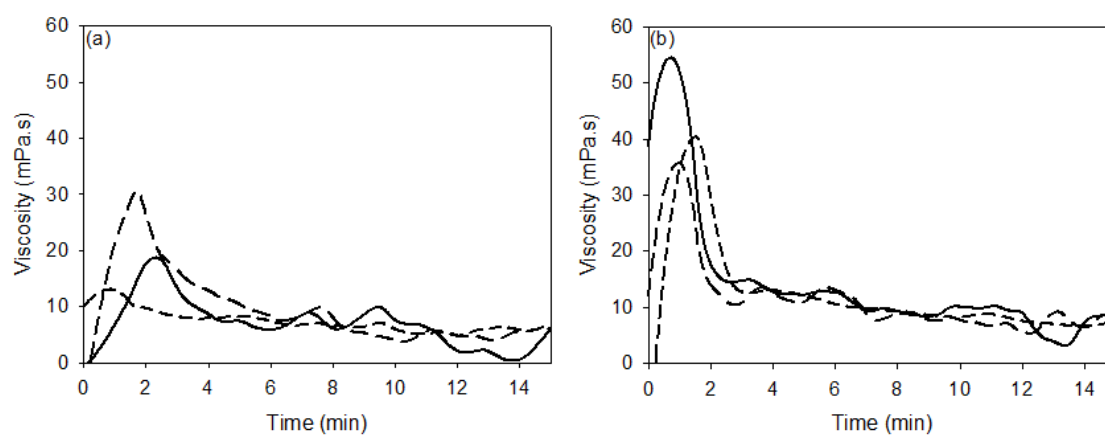
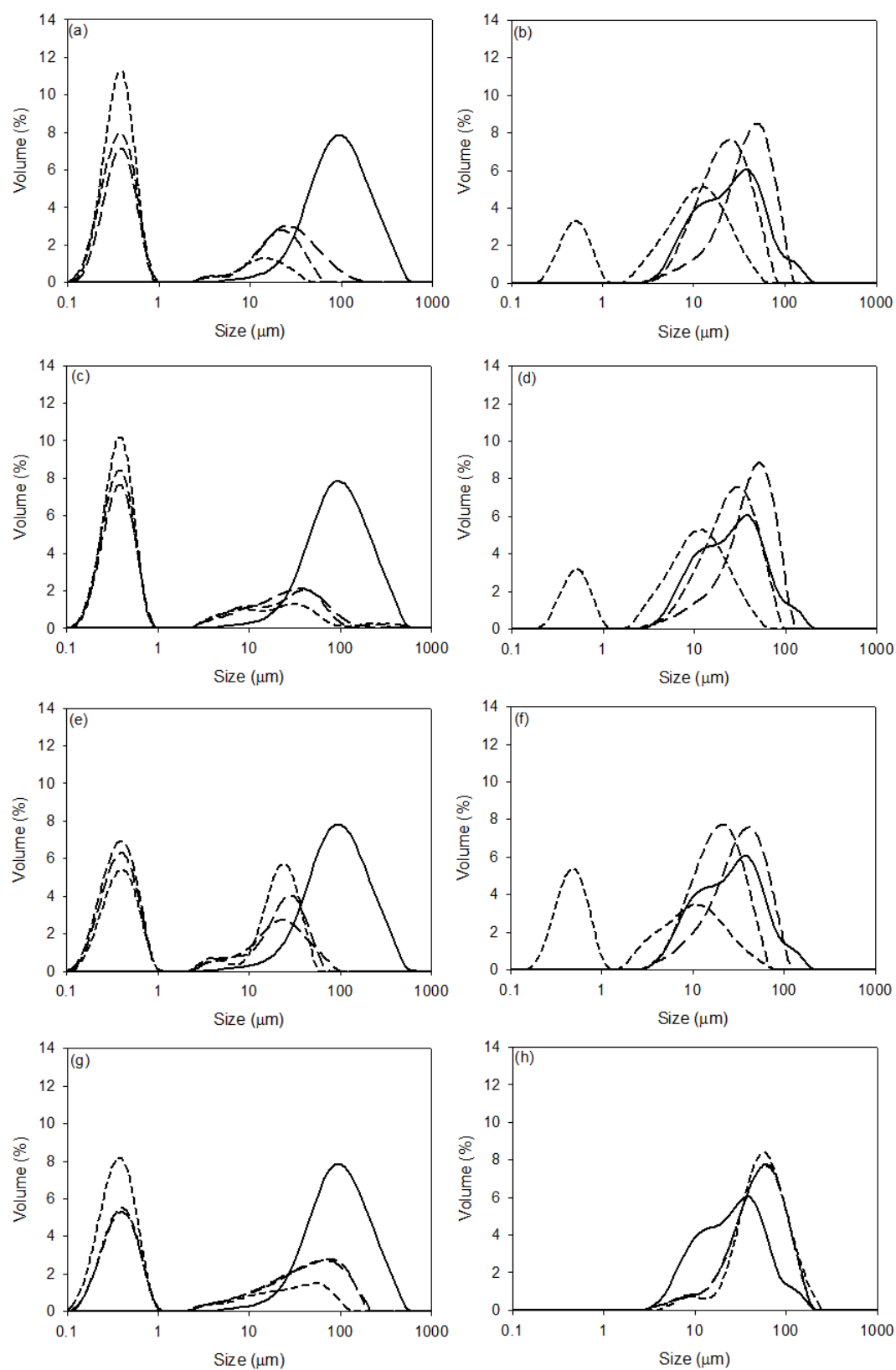


Fig. 6.





**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.