



Contents lists available at ScienceDirect

## International Dairy Journal

journal homepage: [www.elsevier.com/locate/idairyj](http://www.elsevier.com/locate/idairyj)

Short communication

## Viscosity reduction in concentrated protein solutions by hydrodynamic cavitation

Sandra Beyer Gregersen<sup>a</sup>, Lars Wiking<sup>a</sup>, Karina Braad Bertelsen<sup>b</sup>,  
Janjira Tangsanthakun<sup>c</sup>, Bent Pedersen<sup>b</sup>, Kristian Raaby Poulsen<sup>d</sup>, Ulf Andersen<sup>e</sup>,  
Marianne Hammershøj<sup>a,\*</sup>

<sup>a</sup> Department of Food Science, Aarhus University, Blichers Allé 20, 8830, Tjele, Denmark

<sup>b</sup> SPX Flow Technology Danmark A/S, Pasteursvej 1, 8600, Silkeborg, Denmark

<sup>c</sup> Department of Food Technology, Silpakorn University, 73000, Nakhonpathom, Thailand

<sup>d</sup> Arla Foods Ingredients Group P/S, Sønderupvej 26, 6920, Videbæk, Denmark

<sup>e</sup> Arla Foods R&D, Agro Food Park 19, 8200, Aarhus N, Denmark

## ARTICLE INFO

## Article history:

Received 31 January 2019

Received in revised form

30 April 2019

Accepted 30 April 2019

Available online 28 May 2019

## ABSTRACT

Controlling viscosity of concentrated protein solutions is an integral part of dairy powder production. Hydrodynamic cavitation has been suggested as a new processing tool for reducing the viscosity of concentrates prior to spray drying. The aim of this study was to evaluate the viscosity reduction of whey protein concentrate as result of hydrodynamic cavitation. A whey protein concentrate (31% dry matter) was subjected to different hydrodynamic cavitation treatments and the viscosity was monitored during 14 days of storage, denaturation enthalpy was evaluated by differential scanning calorimetry and particle size by dynamic light scattering. Results showed that hydrodynamic cavitation treatment decreased viscosity by 7–8%, and this effect remained constant during the 14 days of storage. Based on the analysis of particle size distribution, the viscosity reduction was suggested to be linked to a reduction in the presence of large particles, possibly due to disruption of aggregates.

© 2019 Elsevier Ltd. All rights reserved.

### 1. Introduction

Controlling product viscosity and age thickening is an integral part of dairy processing, where powder production is becoming of great importance (Westergaard, 1994). Here, hydrodynamic cavitation has been suggested as a new processing tool for reducing the viscosity of protein concentrates.

Cavitation occurs due to the formation, growth and collapse of low-pressure bubbles in a liquid within a short time frame (Ashokkumar, 2011). Upon bubble collapse, energy is released resulting in high temperatures, pressure changes and shear forces created locally in the liquid, which may result in favourable or unfavourable changes; thus, cavitation technologies are regarded as material altering (Ashokkumar, 2011; Patist & Bates, 2008).

Exploitation of cavitation technologies in dairy processing is well studied in terms of ultrasound treatment in areas from milk fat globule homogenisation (Villamiel & de Jong, 2000), improved texture of yoghurt (Riener, Noci, Cronin, Morgan, & Lyng, 2009),

acceleration of milk fat crystallisation (Gregersen et al., 2019) as well as reduced viscosity of concentrated protein solutions (Zisu, Schleyer, & Chandrapala, 2013). However, difficulties with up-scaling has limited the implementation of ultrasound technologies.

Hydrodynamic cavitation may be an alternative to acoustic cavitation, due to a potential improvement in terms of energy efficiency and, above all, to the capacity of this technology to work at a large scale (Gogate, 2011). Hydrodynamic cavitation can be generated when a liquid is passed through a constriction, with the decrease and subsequent increase in local pressures creating bubble implosion (Carpenter et al., 2017). A recent study has illustrated a potential for the use of hydrodynamic cavitation to reduce the viscosity of concentrated milk protein solutions (Li, Woo, Patel, Metzger, & Selomulya, 2018). However, the time dependent behaviour of the viscosity reduction has, to the best of our knowledge, not been elucidated.

In this study, we evaluated the effect of hydrodynamic cavitation treatment on the viscosity of whey protein concentrate solution during a 14 days storage period. Furthermore, effects on protein denaturation enthalpy and particle size distribution were evaluated, thereby, this study contributes to elucidate the mechanism(s) responsible for the hydrodynamic cavitation assisted viscosity decrease in concentrated protein solutions.

\* Corresponding author. Tel.: +45 87157974.

E-mail address: [marianne.hammershoj@food.au.dk](mailto:marianne.hammershoj@food.au.dk) (M. Hammershøj).

## 2. Materials and methods

Whey protein concentrate at a dry matter content (DM) of 28.5% (80% protein) was used as starting material in this study, which was kindly provided by Arla Foods Ingredients (Viby, Denmark) and concentrated at the SPX Flow innovation centre (Silkeborg, Denmark) to a final DM of 31.2% using ultrafiltration membranes of polyethersulphone with a 10 kDa cut-off (Koch Membrane Filtration Systems, Inc., Wilmington, MA, USA) and allowing the temperature to increase to 23 °C. The protein and fat contents of the resulting whey protein concentrate was 25.6% and 1.5%, respectively.

A 12" APV Cavitator (SPX Flow technology, Silkeborg, Denmark) was used for hydrodynamic cavitation treatments. Here, cavitation is introduced by a spinning rotor with a speed of 3000 rpm, containing two rows each having 40 radial holes (cavities) with a volume of 8042.8 mm<sup>3</sup> (diameter: 16 mm, depth: 40 mm). Hydrodynamic cavitation treatments were conducted as outlined in Table 1 and treatments were conducted in duplicates within the same day.

After treatment, samples were cooled to 10 °C and stored at this temperature. Viscosity was determined at 10 °C with a shear rate profile of 1–200 s<sup>-1</sup> directly after treatment using a RheoCompass MCR72 rheometer (Anton Paar, Graz, Austria) and during the storage period with the same settings on a AR-G2 rheometer (TA Instruments, New Castle, DE, USA). Tests were performed with a concentric cylinder geometry and temperature control was ensured by a Peltier temperature unit. Viscosity was determined at different time points relative to sampling time of the first hydrodynamic cavitation treatment with measurements in randomised order at each time point: 4 h, 6 h, 12 h, 20 h, 28 h, 32 h, 48 h, 72 h, 120 h, 144 h, 192 h and 336 h.

Denaturation of whey proteins was analysed using differential scanning calorimetry (DSC) (Q1000 DSC, TA instruments). Samples of approximately 12 mg whey concentrate were loaded in aluminium pans, which were hermetically sealed and heated at 5 °C min<sup>-1</sup> from 20 to 85 °C. An empty pan was used as reference. Three samples were analysed by DSC for each replication. The denaturation enthalpy (J g<sup>-1</sup>) was determined as the integrated area of the endothermic peak (denaturation temperature of 73 °C).

Particle size analyses were performed using Dynamic Light Scattering (Nanoflex, Microtrac, Montgomeryville, PA, USA). Prior to the measurements, samples were diluted (1:10, w/w) in a phosphate buffer (100 mM, pH 7) and the analysis was performed in duplicates with three subsequent measurements on each

**Table 1**  
Overview of hydrodynamic cavitation treatments: flow rate, residence time, cooling, inlet temperature [T(in)] and outlet temperature [T(out)].<sup>a</sup>

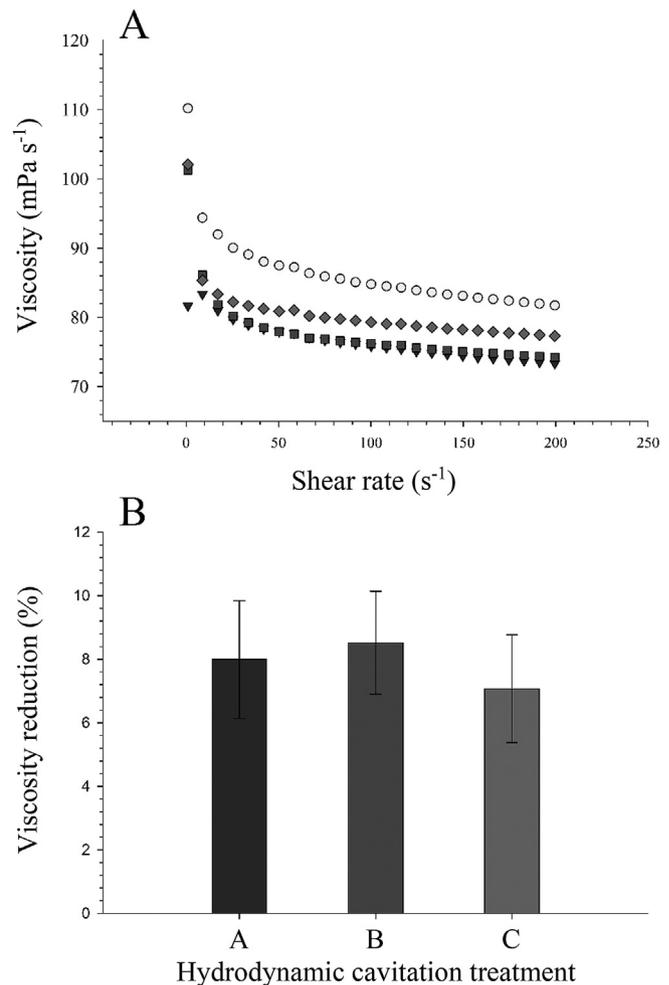
Treatment	Flow (L h <sup>-1</sup> )	Residence time (s)	Cooling	T(in) (°C)	T(out) (°C)
Hydrodynamic cavitation A (low flow, no cooling)	500	1.05	–	7.2 ± 0.2	39.85 ± 0.4
Hydrodynamic cavitation B (low flow, cooling)	500	1.05	+	6.9 ± 0.1	31.1 ± 0.1
Hydrodynamic cavitation C (high flow, no cooling)	830	0.62	–	7.2 ± 0.1	25.05 ± 4.3

<sup>a</sup> Residence time is time in cavitation zone; cooling is represented as +, with or –, without.

sample. The integrated software calculated the volume based mean and the 10th [D<sub>v</sub>(10)], 50th [D<sub>v</sub>(50)] and 90th [D<sub>v</sub>(90)] percentiles. Statistical analyses were carried out using RStudio version 0.96.330 (RStudio, Inc., Boston, MA, USA) by analysis of variance followed by Tukey's honest significance test for comparison (95% significance level).

## 3. Results and discussion

Hydrodynamic cavitation provided a reduction ( $P < 0.001$ ) in the viscosity of whey protein concentrated protein solutions (DM ~31%) of 7–8% independently of the treatment conditions (Fig. 1). This reduction is comparable with the reduction in viscosity reported by Zisu et al. (2013) for acoustic cavitation (ultrasound) treatment of concentrated skim milk with an initial viscosity in the same range as samples analysed in the current study. Decrease of milk protein concentrate viscosity (MPC80) of up to 56% by hydrodynamic cavitation has previously been shown by Li et al. (2018); however, the initial viscosity was higher (200 mPa s<sup>-1</sup>) than in the present study. Furthermore, milk protein concentrate may behave differently during hydrodynamic cavitation compared with a whey based concentrate,



**Fig. 1.** Example of flow curves (A) for an untreated sample (○) and for hydrodynamic cavitation treatments A (▼; low flow, no cooling), B (■; low flow, cooling) and C (◆; high flow, no cooling) and viscosity (100 s<sup>-1</sup>) reduction (B) for the different hydrodynamic cavitation treatments (A, B, C) in relation to an untreated sample, averaged over the total storage period of 14 days (error bars represent standard deviations; no significant differences were found between the hydrodynamic cavitation treatments).

due to the high concentration of caseins in the former, making the two systems very different.

In this study, we evaluated the time dependent behaviour of the hydrodynamic cavitation assisted viscosity reduction within a 14 days storage period, which to the best of our knowledge, has not been described before. Independently of treatment, a slight increase in viscosity occurred within the first 20 h of storage (data not shown), i.e., the decrease in viscosity of hydrodynamic cavitation treated concentrate relative to an unstated concentrate was unaffected by storage time. Thus, our results indicate that the hydrodynamic cavitation assisted decrease in viscosity is a result of irreversible changes.

For this particular application of hydrodynamic cavitation in the dairy industry, it is critical to prevent changes to the final functional properties of the milk powder based products. Thus, it is of great importance to understand the molecular basis for the viscosity decrease. The DSC analysis showed no changes in the denaturation enthalpy (Table 2), i.e., hydrodynamic cavitation results in no irreversible changes in the folding structure of the whey proteins.

Analysis of the particle size distribution showed the presence of relative large particles initially in the untreated concentrate (1.5%), which could be attributed to the presence of large protein aggregates as well as the presence of residual fat in terms of primarily polar lipids. Hydrodynamic cavitation treatments introduced a significant decrease in  $D_v(90)$ , and for treatment C a decrease in  $D_v(10)$ ,  $D_v(50)$  and the mean diameter was also observed (Table 3). Thus, results indicate that hydrodynamic cavitation reduces the amount of large particles in the systems, which could be explained by disruption of aggregates by forces introduced during cavitation events.

For concentrated protein systems, it is well known that the presence of aggregates can result in a pronounced increase in

viscosity (Lilyestrom, Yadav, Shire, & Scherer, 2013; Nicoud, Lattuada, Yates, & Morbidelli, 2015). Thus, based on the results it is suggested that the hydrodynamic cavitation assisted decrease in viscosity is due to disruption of aggregates. This is in line with findings by Pathania, Ho, Hogan, McCarthy, and Tobin (2018) in a study on hydrodynamic cavitation assisted milk powder hydration, relating reduced viscosity of rehydrated milk powder solution to a decrease in particle size. A reduction in particle size during acoustic cavitation of whey protein solutions has previously been reported, which was linked to changes in the molecular weight of protein fractions and changes in the intermolecular hydrophobic interactions (Jambrak, Mason, Lelas, Paniwnyk, & Herceg, 2014). Such changes may also occur during hydrodynamic cavitation and further studies are needed to fully elucidate the mechanism(s) responsible for the changes in particle size distribution by hydrodynamic cavitation.

#### 4. Conclusion

In conclusion, the results demonstrate that hydrodynamic cavitation can provide a viscosity reduction in liquid whey protein concentrate, which was still present after 14 days of storage. We suggest reduction in very large particles, possibly due to disruption of aggregates, as the underlying mechanism hereof. Thus, our findings support that hydrodynamic cavitation can provide a new processing tool to control the viscosity of high protein concentrates. In dairy powder production, this may allow for spray drying at higher solid contents and/or storage of liquid protein concentrates, without unacceptable viscosity increases. For such applications, it is critical to ensure that any treatment does not deteriorate the milk protein functionality. Here, we demonstrate that hydrodynamic cavitation does not introduce any significant changes in the denaturation enthalpy, indicating that no irreversible unfolding of the proteins occurs during treatment. However, further studies are needed to verify if hydrodynamic cavitation treatment affects the final functional properties of dairy powders.

#### Acknowledgements

The authors wish to thank the Danish Dairy Research Foundation, Arla Foods a.m.b.a, Arla Foods Ingredients, SPX flow technology and Future Food Innovation, Region Midjotland (Denmark) for financial support.

#### References

- Ashokkumar, M. (2011). The characterization of acoustic cavitation bubbles—an overview. *Ultrasonics Sonochemistry*, 18, 864–872.
- Carpenter, J., Badve, M., Rajoriya, S., George, S., Saharan, V. K., & Pandit, A. B. (2017). Hydrodynamic cavitation: An emerging technology for the intensification of various chemical and physical processes in a chemical process industry. *Reviews in Chemical Engineering*, 33, 433–468.
- Gogate, P. R. (2011). Hydrodynamic cavitation for food and water processing. *Food and Bioprocess Technology*, 4, 996–1011.
- Gregersen, S. B., Frydenberg, R. P., Hammershøj, M., Dalsgaard, T. K., Andersen, U., & Wiking, L. (2019). Application of high intensity ultrasound to accelerate crystallisation of anhydrous milk fat and rapeseed oil blends. *European Journal of Lipid Science and Technology*, 121, Article 1800200.
- Jambrak, A. R., Mason, T. J., Lelas, V., Paniwnyk, L., & Herceg, Z. (2014). Effect of ultrasound treatment on particle size and molecular weight of whey proteins. *Journal of Food Engineering*, 121, 15–23.
- Lilyestrom, W. G., Yadav, S., Shire, S. J., & Scherer, T. M. (2013). Monoclonal antibody self-association, cluster formation, and rheology at high concentrations. *Journal of Physical Chemistry B*, 117, 6373–6384.
- Li, K., Woo, M. W., Patel, H., Metzger, L., & Selomulya, C. (2018). Improvement of rheological and functional properties of milk protein concentrate by hydrodynamic cavitation. *Journal of Food Engineering*, 221, 106–113.
- Nicoud, L., Lattuada, M., Yates, A., & Morbidelli, M. (2015). Impact of aggregate formation on the viscosity of protein solutions. *Soft Matter*, 11, 5513–5522.

**Table 2**  
Effect of hydrodynamic cavitation on protein denaturation enthalpy determined by DSC analysis.<sup>a</sup>

Treatment	Enthalpy (J g <sup>-1</sup> )
Untreated	2.18 ± 0.07
Hydrodynamic cavitation A (low flow, no cooling)	2.20 ± 0.14
Hydrodynamic cavitation B (low flow, cooling)	2.20 ± 0.14
Hydrodynamic cavitation C (high flow, no cooling)	2.13 ± 0.22

<sup>a</sup> Values are expressed as mean ± standard deviation; there are no significant differences between the data.

**Table 3**  
Effect of hydrodynamic cavitation on the particle size distribution determined by dynamic light scattering (volume based distribution).<sup>a</sup>

Treatment	Mean (nm)	$D_v(10)$ (nm)	$D_v(50)$ (nm)	$D_v(90)$ (nm)
Untreated	373 ± 84 <sup>a</sup>	113.9 ± 37.5 <sup>a</sup>	292.2 ± 62.2 <sup>a</sup>	1162 ± 540 <sup>a</sup>
Hydrodynamic cavitation A (low flow, no cooling)	300 ± 63 <sup>a</sup>	85.0 ± 13.1 <sup>b</sup>	248.0 ± 30.7 <sup>a</sup>	533 ± 91 <sup>b</sup>
Hydrodynamic cavitation B (low flow, cooling)	300 ± 77 <sup>a</sup>	81.5 ± 24.4 <sup>b</sup>	214.7 ± 27.9 <sup>a</sup>	556 ± 177 <sup>b</sup>
Hydrodynamic cavitation C (high flow, no cooling)	264 ± 23 <sup>b</sup>	70.2 ± 24.4 <sup>b</sup>	202.2 ± 47.6 <sup>b</sup>	402 ± 114 <sup>b</sup>
Significance level	$P < 0.05$	$P < 0.05$	$P < 0.05$	$P < 0.001$

<sup>a</sup> Values are expressed as mean ± standard deviation; values in a column with different superscript letters differ significantly at the given  $P$ -value.

- Pathania, S., Ho, Q. T., Hogan, S. A., McCarthy, N., & Tobin, J. T. (2018). Applications of hydrodynamic cavitation for instant rehydration of high protein milk powders. *Journal of Food Engineering*, 225, 18–25.
- Patist, A., & Bates, D. (2008). Ultrasonic innovations in the food industry: From the laboratory to commercial production. *Innovative Food Science & Emerging Technologies*, 9, 147–154.
- Riener, J., Noci, F., Cronin, D. A., Morgan, D. J., & Lyng, J. G. (2009). The effect of thermosonication of milk on selected physicochemical and microstructural properties of yoghurt gels during fermentation. *Food Chemistry*, 114, 905–911.
- Villamiel, M., & de Jong, P. (2000). Influence of high-intensity ultrasound and heat treatment in continuous flow on fat, proteins, and native enzymes of milk. *Journal of Agricultural and Food Chemistry*, 48, 472–478.
- Westergaard, V. (1994). *Milk powder technology: Evaporation and spray drying*. Sørborg, Denmark: A/S NIRO.
- Zisu, B., Schleyer, M., & Chandrapala, J. (2013). Application of ultrasound to reduce viscosity and control the rate of age thickening of concentrated skim milk. *International Dairy Journal*, 31, 41–43.