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A comparison of pilot-scale supersonic direct steam injection to conventional steam infusion and tubular heating systems for the heat treatment of protein-enriched skim milk-based beverages

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Keywords: supersonic steam injection; steam infusion; tubular; heat treatment; skim milk

Abstract

Direct supersonic steam injection, direct steam infusion, and indirect tubular heating were each applied to protein-enriched skim milk-based beverages with 4, 6 and 8% (w/w) total protein, and the effect of final heat temperature on the physical properties of these beverages was investigated. Supersonic steam injection resulted in significantly lower levels of denaturation of β-lactoglobulin (34.5%), compared to both infusion (76.3%) and tubular (97.1%) heating technologies. Viscosity, particle size and accelerated physical stability of formulations did not differ significantly between the heating technologies, while noticeable colour differences due to heat treatment (mainly attributed to increasing b* value) were observed, particularly for tubular heating. Overall, the extent of protein denaturation in high-protein dairy products was significantly influenced by the particular heating technology applied. The application of supersonic steam injection technology, with rapid heating and high shear characteristics, may enable differenciated product characteristics for ready-to-drink ambient-delivery high-protein dairy beverages.

Industrial Relevance

The design and application of novel direct supersonic steam injection technology was comprehensively studied and found to provide significant benefits over direct steam infusion and indirect tubular heating technologies for skim milk-based protein beverages. This type of injection heating system resulted in heat-treated formulations with lower levels of denatured whey proteins, compared to tubular and infusion heating, offering an alternative opportunity to the industry in terms of producing shelf-stable dairy protein beverages.

1. Introduction

Extended shelf life (ESL) and ultra-high temperature (UHT) treated milk have increased in popularity worldwide, providing long shelf-life and eliminating cold chain requirements, thereby reducing economic costs to producers, distributors and consumers (Bertolini *et al.*, 2016, Malmgren *et al.*, 2017). High-heat dairy treatments, like ESL heating (120-135 °C for 2-4 s) and UHT (135–145 °C for 2-4 s), can negatively impact the nutritional quality and sensory properties of the final product due to the severity of the heat treatment applied. In addition, the choice of thermal processing technology used to achieve ESL or UHT treatment can have a significant impact on physical properties and consumer acceptability of the final product (Deeth and Lewis, 2016; Roux *et al.*, 2016).

Thermal processing technologies may be classified as direct or indirect, which have different heat transfer mechanisms. For indirectly heated systems, heat is transferred from the heating medium to the product through a thermally conducting but otherwise impermeable barrier, while direct heating achieves almost instantaneous heating through the direct addition of the heating medium, steam, to the product (Hsu, 1970; Burton, 1994; Schroyer, 1997; Lewis *et al.*, 2000). Direct heating imparts a lower thermal load on the product due to significantly faster heating and cooling rates, thereby reducing thermally-induced changes in the final product (Kelleher et al., 2018b). However, there are challenges associated with direct systems such as the requirement for culinary-grade steam, lower heat regeneration capacity, and concerns with product dilution, resulting in indirect technologies being more commonly used industrially (Datta *et al.*, 2002; Britz and Robinson, 2008; Dickow *et al.*, 2012b; Karayannakidis *et al.*, 2014; Lee *et al.*, 2017).

Traditional direct technologies, such as steam injection and infusion systems, have been available for decades and their use with various dairy products has been investigated throughout the years (Ford et al., 1969; Patrick and Swaisgood, 1976; Lyster et al., 1971,

Datta et al., 2002, Dumpler and Kulozik, 2016). Over the years there have been engineering improvements and reconfigurations made to the original formats, such as the lenient steam injection patented process (Dickow et al., 2012a, Dickow et al., 2012b) or supersonic steam injection (Murphy, 2011, Murphy et al., 2013). There has been little investigation into the application and potential benefits of supersonic steam injection to dairy products. This study used a patented supersonic steam injector (Maklad Fluid GmbH), which makes use of a De Laval nozzle to achieve better mixing and potentially attaining supersonic flow within the injection unit. These nozzles are commonly referred to as converging-diverging nozzles, where an inlet section converges into a narrow throat and subsequently expands into a divergent outlet (Canosa et al., 2016). The converging inlet accelerates the fluid, in this case a mixture of steam and liquid product, until the flow becomes choked or sonic at the throat, where the cross-sectional area is smallest, and a Mach number (Ma) of 1 is achieved. Due to conservation of volumetric flow, temperature and pressure reduce with increasing area in the diverging outlet, thereby increasing kinetic energy and resulting in supersonic flow (Ma > 1). The application of a supersonic steam injector in dairy processing can theoretically produce better product mixing due to high shear from (i) the high throat velocities and (ii) the shockwave produced, with reduced residence time in the injection chamber, compared to traditional direct systems in dairy processing (Murphy *et al.*, 2011; Murphy *et al.*, 2013).

Increased consumer awareness has led to market demands for healthy, protein-enriched foods for general consumption, in addition to clinical uses such as for the treatment of malnutrition, sarcopenia in the elderly, high-performance sports nutrition, and body-building (Hayes *et al.*, 2008; Jelen, 2009; Shiby, 2013; Withers *et al.*, 2014; Chen and O'Mahony, 2016). Milk proteins have many health-promoting and nutritionally beneficial properties for the consumer, such as supplying essential amino acids for tissue growth and repair, metabolic

regulation for weight control, and anti-oxidant functions for immune-enhancing properties (Beucler, 2005; Smithers; 2008; Wijayanti *et al.*, 2014; Gupta and Prakash, 2015). However, protein-enriched beverages can pose thermal processing challenges, particularly in relation to the denaturation, aggregation and fouling of heat-labile dairy proteins, with the selection of thermal processing technology having a significant impact on the occurrence of these phenomena (Joyce *et al.*, 2017; Kelleher *et al.*, 2018). The nutritional value of proteins can be impaired by severe heat treatment, resulting in decreased protein digestibility and the availability of substrate to enzymatic digestion (Resmini *et al.*, 2003). 'Cooked' off-flavours commonly associated with ESL and UHT milks are connected to the level of whey protein denaturation, particularly β-lactoglobulin, as free sulfhydryl groups are exposed leading to the development of sulphur compounds in the milk (Al-Attabi *et al.*, 2009; Zabbia *et al.*, 2012; Lee *et al.*, 2017). Incorporation of ingredients can also pose challenges in high protein beverage systems, with commonly used powder ingredients such as milk protein concentrates (MPC) exhibiting poor solubility. The application of high temperatures, shear and increased hydration time can improve MPC solubility and incorporation into beverage formulations (Pathania *et al.*, 2018). Novel thermal processing technologies may prove to be important tools for the food industry in the development of protein-enriched beverages with differentiated physical properties which can satisfy changing market demands.

The aim of this study was to investigate the impact of direct supersonic steam injection heating on the physical characteristics of ready-to-drink protein-enriched dairy-based beverages with ambient distribution, compared to standard direct infusion and indirect tubular heating technologies. The three heating technologies were applied to beverages having three different protein levels, operated at three final heat treatment temperatures, and compared in terms of final product quality and stability. As little has been published in relation to the use of supersonic injectors in dairy processing, the focus of this study was to determine the

implications of high shear and rapid heat transfer during processing using the supersonic injector and analysing for protein denaturation, and beverage viscosity and physical stability.

2. Materials and methods

2.1. Materials and formulation

Medium-heat skim milk powder, SMP (33.93 % protein, 0.78 % fat, 6.04 % moisture, 48.88 % lactose and 8.13 % ash), and milk protein concentrate, MPC80 (83.03% protein, 0.96% fat, 4.03 % moisture, 3.99 % lactose and 6.96 % ash) were supplied by Glanbia Ingredients Ireland Ltd. (Kilkenny, Ireland).

Model protein-enriched beverages were formulated at 4, 6 and 8 % w/w protein concentrations using a skim milk base, reconstituted to 10 % total solids (w/w) in reverse osmosis water at 45 °C using a YTRON ZC powder induction unit (YTRON Process Technology GmbH, Bad Endorf, Germany). MPC80 was added to each formulation to yield desired protein concentration (0.73, 3.14, 5.55 % MPC80 (w/w) for 4, 6 and 8 %, respectively) and inducted with a high shear mixer (Silverson EX, Silverson Machines Ltd, UK). The formulations were held overnight in stirred tanks at 4 °C to allow for powder hydration. The pH was measured before and after overnight storage and was adjusted to pH 6.7 using 0.1 M HCl or KOH, if required.

2.2. Heat Treatment

Three types of heat treatment technology were applied to the formulations: direct steam infusion, direct supersonic steam injection, and indirect tubular heating (Fig. 1). All heat treatment conditions consisted of a preheat treatment (70 °C for 30 s) and final heat treatment (121, 135 or 142 °C, for 3 s) with a flowrate of 100 L/h. Infusion heating employed a UHT pilot-scale plate exchanger Model 422463 (APV, Denmark), and as in the injection system,

initial cooling to 70 °C was achieved using vacuum flash cooling. Preheat and final cooling operations were carried out using plate heat exchangers. Indirect tubular heating was applied using a MicroThermics tubular UHT 25HV pilot plant (MicroThermics, NC, USA), consisting of four tubular heat exchangers; preheat, final heat and two cooling exchangers. The direct steam injection was achieved by integrating a purpose-built process line with a Maklad supersonic injector Model 700-143-60 (Maklad Innovative Fluid- and Systemtechnik GmbH, Austria) for final heat treatment into the MicroThermics plant (Fig. 1B). The process line consisted of the Maklad injector, flash cooler, condenser, vacuum pump, product pump, culinary steam and product filters and an independent cleaning-in place (CIP) system. Vacuum flash cooling to 70 °C was applied after final heat treatment as part of the injection process line, while tubular heat exchangers from the Microthermics systems were used for preheating and final cooling operations. The injector had a de Laval converging-diverging nozzle with a flow rate range of 50 – 150 L/h for the steam-product mix and had a Teflon coating in the steam-product mixing zone to reduce burn-on. All heat treatment trials were carried out in triplicate.

2.3. Protein analysis

Total protein content was determined using the Kjeldhal method with a nitrogen-to-protein conversion factor of 6.38 (IDF, 2001). Native protein levels were determined using reverse-phase high-performance liquid chromatography (RP-HPLC) equipped with a Waters 2695 separation module, Waters 2487 dual wavelength absorbance detector at 214 nm and Empower® software (Milford, MA, USA). The HPLC was equipped with a PolymerX 5µm RP-1, 150 x 4.6 mm column (Phenomenex, Cheshire, UK). α-Lactalbumin (α-la), β-lactoglobulin A and B (β-lg A and B) standards (Sigma Aldrich, Ireland) were used to calibrate the method. Sample preparation required pH adjustment to 4.6 with 0.1 M acetate buffer to 2.5 g/L protein, centrifugation at 20,000 g for 20 min at 4 °C, and filtration of the

supernatant using 0.2 µm PES filters (Agilent Technologies, Santa Clara, CA, United States) (Kehoe *et al.*, 2011; Kelleher *et al.*, 2018). Total solids content was measured using a Smart System 5, Smart Trac (CEM Corporation, Matthews, NC, USA).

2.4. Viscosity

Viscosity was determined using a shear rate sweep at 25 °C, using an AR-G2 controlled stress rheometer (TA Instruments, Crawley, UK) with a concentric cylinder geometry (Murphy *et al.*, 2013). The apparent viscosity values presented are the average viscosity on holding at 500 1/s for 1 min.

2.5. Particle size

A Malvern Zetasizer Nano ZS combined dynamic light scattering analyser (Malvern Instruments Ltd., UK) was used to determine particle size at 25 °C. Samples were dispersed using ultra-pure water in polystyrene disposable cuvettes with refractive index for protein and the water dispersant of 1.45 and 1.33, respectively. Particle size is reported in terms of intensity mean (d.nm).

2.6. Colour analysis

Colour of samples in disposable cuvettes was measured using a Minolta Chroma meter CR-400 colorimeter (Minolta Ltd., Milton Keynes, UK) and expressed in L*, a* and b* values. Colour difference from unheated formulations, ΔE, was determined using the CIE76 Euclidean distance formula, given as described by (Morales and Jiménez-Pérez, 2001):

$$\Delta E = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2} \qquad 2.1$$

2.7. Accelerated physical stability

Accelerated physical stability of formulations was investigated using a LUMiSizer analytical centrifuge (Lum GMBH, Berlin, Germany), equipped with SepView 4.1 software. Samples

(0.4 mL) were filled into PC100-131XX polycarbonate cells to a 20 mm depth and centrifuged at 2300 g for 3 h at 25 °C (Chen and O'Mahony, 2016). The software integrates with respect to particle position on each transmission profile to characterise instability over time as a second order polynomial. To calculate the change in transmission over time, integration limits were set along the length of the filled tube, at 110 to 130 mm. The average slope of this polynomial, calculated from the polynomial coefficients, was used as an instability index to compare the stability of different samples under accelerated conditions.

2.8. Statistical analysis

Heat treatment trials were carried out in triplicate. The Minitab® 17 (Minitab Ltd., Coventry, UK) statistical analysis package was used to carry out one-way ANOVA with Tukey *post hoc* and three-way ANOVA analysis using protein content, heating technology and heat treatment temperatures as factors.

3. Results and discussion

3.1. Protein and total solids analysis

Total solids and total protein were analysed before and after heat treatment and steam injection and tubular heat treatment did not significantly differ in their effects on the level of total solids and total protein for 4, 6 and 8 % (w/w) protein formulations (Table 1). However, steam infusion technology resulted in a significant reduction in total solids and protein levels at each protein concentration and treatment temperature applied ($p < 0.001$). It is likely that the reductions in solids content following steam infusion are related to product dilution by the condensed steam heating medium, due to incomplete removal of water by flash cooling, which is commonly reported for pilot-scale operation of direct heating systems, (Dickow *et al.*, 2012a; Dickow *et al.*, 2012b; Murphy *et al.*, 2013; Dumpler *et al.*, 2017). The application of injection and tubular heating resulted in no significant change in total protein content being

observed and while infusion treatment affected the level of total protein in the final formulation (possibly due to fouling), injection heating did not.

The level of native protein was significantly affected by the type of heating technology, protein concentration and temperature applied ($p < 0.001$, $p < 0.001$ and $p < 0.05$, respectively). The concentrations of native α-la were most greatly reduced by tubular heating, resulting in a final native α-la content that was 11.7 - 55.9 % of the initial content in unheated formulations. Both direct injection and infusion heating resulted in significantly less denaturation of α-la, compared to tubular heating. For infusion technology, treatment at 121 °C did not affect the level of native α-la at any concentration ($p > 0.05$), while higher temperatures (135 and 142 °C) resulted in a reduction in levels of native α-la (on average 65.8 % native α-la from the initial content; $p < 0.05$). Injection heating resulted in the lowest level of α-la denaturation of all technologies investigated, with no significant change in native α-la level (on average 82 % native α-la from the initial content; $p > 0.05$) despite the application of high temperatures, with the exception of the treatment at 135 °C for the 4 % w/w protein formulation (with 71 % native α-la from the initial content; $p < 0.05$).

Despite extensive denaturation of β-lactoglobulin (β-lg) for all heat treatments, significant differences between heating technologies were evident (Fig. 2). Injection heating resulted in heat-treated formulations with significantly greater levels of native β-Lg A and B, compared to tubular and infusion heating ($p < 0.001$). The average levels of native β-lg A and B were greater after injection (64.7 and 66.4 %, respectively) compared to tubular heating (3.16 and 2.72 %, respectively), indicating substantial denaturation with the use of indirect tubular heating. For 4 % (w/w) protein formulations, the differences between infusion and tubular heating were statistically significant ($p < 0.05$); however, at higher protein concentrations, these differences were not significant.

While both infusion and injection systems resulted in higher levels of native protein than indirect tubular heating, supersonic injection technology resulted in the lowest whey protein denaturation levels at high processing temperatures. This may be due to the accelerated product flow within the injector chamber for supersonic injection, allowing the required heat to be imparted with reduced residence time, reduced thermal load and more uniform temperature (Murphy *et al.*, 2011; Murphy *et al.*, 2013). The application of high levels of shear, due to shockwaves produced in the supersonic injection system, may also contribute to the lower levels of protein denaturation observed. It has been shown that high shear can reduce whey protein aggregate formation leading to greater retention of native protein post heat treatment (Dissanayake and Vasiljevic, 2009; Çakır-Fuller, 2015; Wolz *et al.*, 2016). Reduced degree of β-lg denaturation have been shown to reduce the levels of 'cooked' off flavours and sulphur volatiles in milk (Lee et al., 2017; Kelleher et al., 2018b). The substantially lower levels of denatured whey protein in injection-heated formulations is a significant differentiating attribute (e.g. with respect to sensory, colloidal stability and protein quality) for the final product, compared to infusion- and tubular-heated formulations.

3.2. Viscosity

While beverage viscosity increased with increasing protein and total solids content ($p < 0.001$), heat-treated formulations were not significantly affected by heating technology or temperature *per se* ($p > 0.05$; Table 1). For 4 and 6 % (w/w) protein formulations, viscosity was not significantly affected by heat treatment using infusion, injection or tubular heating. Heat treatment significantly reduced the viscosity of 8 % (w/w) protein formulations in all cases, by an average of 28.4 % ($p < 0.05$) relative to the unheated formulation, with no significant effect of increasing heat treatment temperature or technology. This reduction in viscosity may be due to increased solubilisation of the added MPC powder at the high heat

treatment temperatures for the more concentrated 8 % (w/w) protein formulation as reported by Pathania *et al.* (2018).

3.3. Particle size

The average particle size for 4 and 6 % (w/w) protein formulations was not significantly affected by heat treatment ($p > 0.05$; Table 1). For 8 % (w/w) formulations, the unheated and injection 142 °C heated formulations resulted in the greatest particle size. This increase in the unheated formulation is likely due to the dissolution of MPC, an ingredient which is notoriously difficult to fully solubilise under standard processing conditions (McCarthy et al., 2014). With the application of heat treatment and, in the case of steam injection, shear effects, solubilisation of the MPC is improved and the average particle size is reduced for 8 % (w/w) formulations.

While, overall, the average particle size did not differ significantly between heating technologies for most protein formulations, differences in particle size distribution were observed (Fig. 3). Injection heating resulted in a broader size distribution than infusion and tubular heating, for each treatment temperature and protein concentration. The high levels of shear produced by the supersonic injector may be the cause of the broadening distribution, as the degree of protein aggregation is reduced, and a higher quantity of smaller soluble aggregates are present in the system (Wolz *et al.*, 2016).

3.4. Accelerated storage stability

The level of protein had a significant impact on stability of formulations, with increasing protein concentration resulting in improved accelerated storage stability (Fig. 4). This is likely due to the increase in viscosity with increasing protein concentration (Table 1) (Karlsson et al., 2005; Lim and Roos, 2015). As with viscosity and particle size, there was no significant difference in accelerated storage stability of unheated, infusion-heated, tubular-

heated or injection-heated formulations at any protein concentration ($p > 0.05$), while there were consistent trends for those parameters across protein levels

3.5. Colour analysis

The protein content of formulations was found to have a significant effect on the lightness, L*, which increased in unheated formulations as the total solids and protein content increased ($p < 0.001$; Table 2). The L* value is largely attributed to particle size and total solids; therefore, as the level of casein micelles increases with protein concentration the L* value increases for the unheated formulations (Chung *et al.*, 2014). Heating technology had a significant effect on L* values of formulations ($p < 0.001$), with tubular heating resulting in a higher L* value for all 6 and 8 % protein formulations compared to other heating technologies. Similar L* values were obtained for the 4, 6 and 8 % protein formulations using infusion and injection direct heating systems (Table 2).

The a* value (red-greenness) of beverages generally increased upon heat treatment, an effect that become more significant with increasing protein concentration ($p < 0.001$). At 4 % protein, significant increases in a* value were identified for infusion treatments at 121 °C and 135 °C and tubular treatment at 142 °C only (Table 2). Heating technology and temperature were shown to affect a* value ($p < 0.01$ and 0.05, respectively), with tubular heating causing the greatest increase. Changes in a* value increased with increasing heating temperature. There was no significant difference between a* values following injection and infusion for the protein concentrations investigated.

The b* value increased with increasing protein concentration (Table 2). Heat treatment had a significant impact on the b* value, with infusion resulting in the lowest b* value and tubular treatment resulting in the highest b* value overall ($p < 0.001$). Tubular and injection heating significantly increased the b* value with increasing temperature, while infusion did not. For

tubular-heated milks the b* value was shown to increase with increasing final heat temperature, while injection-treated milks did not. Increases in b* values can result from the occurrence of Maillard browning in a system (Morales and van Boekel, 1998). The increased b* values in tubular- and injection- treated formulations indicates a greater level of Maillard browning compared to that in infusion- treated formulations.

Euclidean distance, ΔE, provides information on the overall colour change from the unheated formulation for each of the heat-treated protein-enriched beverages (Table 2). Protein concentration, heating technology and final heat temperature all significantly affected the ΔE ($p < 0.001$, for each factor in terms of three-way ANOVA). Tubular heating resulted in the greatest overall colour change, particularly at 6 and 8% protein, and all heating temperatures resulted in a visibly observable colour difference ($\Delta E > 2.3$). It should be noted that these colour changes are not thought to be of an order of magnitude that would be undesirable from a consumer perspective.

4. Conclusion

Supersonic steam injection heating provides substantial retention of native whey protein, particularly heat labile β-lg, across three ESL and UHT temperatures, compared to traditional tubular and direct steam infusion heating. Physical characteristics such as viscosity, particle size and accelerated storage stability did not significantly differ between the differently heat-treated formulations. It is well established that direct heating imparts less thermal damage on a product than indirect heating; however, the more novel supersonic direct steam injection technology provides an opportunity to further reduce thermal damage of dairy beverages, particularly in terms of protein denaturation. The application of this technology could enable opportunities differentiated product characteristics in long-life ready-to-drink high-protein beverages with high levels of native whey protein.

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Appendix I

The ratio of steam to product, on a water basis, required to achieve the desired increase in product temperature, ΔT, can be calculated as:

$$z = \frac{c_p \Delta T}{h_1 - h_f} \quad \text{A.1}$$

where h_1 is the specific enthalpy of the steam at the nozzle inlet, h_f is the specific enthalpy of the product at the final temperature and c_p is the specific heat of the product; the value of c_p is made up of contributions from water (the major component) and also of fat and non-fat solids. If the incoming product is preheated, to T_i = 70 °C, with a typical final heat temperature, T_f = 121 °C, after mixing with steam at 3 bar(a), the ratio of condensed steam to incoming product to achieve the desired product temperature (121 °C) is 9. 5% by weight.

The thermodynamic conditions in the supersonic steam injector unit were analysed to understand the thermodynamic conditions in the deLaval nozzle. While the mass flowrate is constant at successive sections of the nozzle, the volumetric flowrate changes as it goes through, mainly for two reasons (i) the cross-sectional area is changing, and (ii) steam is being condensed as it moves through the nozzle. The percentage of the total steam load that is condensed before the throat is not known and hence in the simulation this percentage is treated as an arbitrary constant which can be set between 0 and 100%. The velocity profile for steam at successive sections can be described by the steady flow enthalpy equation, based on the law of conservation of energy as applied to compressible fluids:

$$h_1 + v_1^2/2 = h_2 + v_2^2/2 \quad \text{A.2}$$

where h is the specific enthalpy of steam and v is the velocity of fluid flow. For cases in which v_1 is very small in comparison to v_2, v_1 may be neglected (Gupta, 2013), resulting in the equation:

$$v_2 = \sqrt{2(h_1 - h_2)} \quad \text{A.3}$$

For a system using saturated steam supply (6 bar (a)) at nozzle inlet (1) and throat (2) pressure of 3 bar (a), we obtain using saturated steam tables, h_1 = 2756 kJ/kg, and near supersonic conditions, using wet steam tables, h_2= 2639 kJ/kg, giving

$$v_2 = 485\ m/s$$

The Mach number can be written as:

$$Ma = \frac{v}{c} \quad \text{A.4}$$

where c is the local speed of sound, 440 m/s. Thus, Ma at the throat is calculated as 1.1; however, as the flow would become choked at the throat, to a maximum of Ma = 1, the maximum throat velocity would equal c.

If the fluid flow reaches sonic velocity at the throat, the velocity increases in the diverging section of the de Laval nozzle, as the area increases, as described in equation A.4 below (derived from the Bernoulli continuity equation), i.e. giving supersonic flow, since the combination of $Ma \geq 1$ and $dA > 0$ gives $dv > 0$, hence increasing v:

$$\frac{dA}{A} = -\frac{dv}{v}(1 - Ma^2) \quad \text{A.5}$$

However, after supersonic flow has been achieved within the diffuser, boundary conditions will ensure that the velocity will eventually decrease at a point which depends on the ratio of inlet pressure to exit or back pressure of the injector and drop below *Ma* = 1 to subsonic flow. This irreversible adjustment of velocity results in a shock wave. It should be noted that achieving this condition depends on matching the nozzle dimensions to the flow of product and steam, using

$$Q = vA \qquad \text{A.6}$$

where *Q* is the volumetric flowrate of steam at the throat and *A* is the available cross sectional area, with allowance for a slight restriction due to product flow determined by the densities of product and steam.

The mass flowrate of water removed from the steam-product mix, m_l, by flash cooling can be determined by calculating the weight percent vapourised, X:

$$X = \frac{100(h_u^L - h_d^L)}{h_d^v - h_d^L} \qquad \text{A.7}$$

where h^L is the liquid enthalpy upstream (u) and downstream (d) from the flash cooler, and h^v is the vapour enthalpy at the flash cooler.

The mass flowrate of vapourised liquid in the flash cooler, m_l, can be determined using:

$$m_l = X\left({}^{m_i}/_{100}\right) \qquad \text{A.8}$$

Best operating practices of direct heating systems recommend that the flash cooling system is operated so that the flash cooling temperature is equal to the preheat temperature used (Burton, 1968). For a product at 121 °C where the flash cooler is operated at 0.3 bar(a)

resulting in a flash cooling temperature of 70 °C, the mass flowrate of liquid being removed from the flash cooler can be calculated as:

$$X = 11.40$$

$$m_l = 12.5\ kg/hr$$

Note that there is still a differential between the calculated values for m_s and m_l, which may result in a 2.98 % concentration of the product despite ideal operating conditions. As described by Burton and Lewis (2009), the total solids of the product should be monitored for dilution or concentration and the temperature of the flash cooler adjusted if required, as dilution of product is commonly reported in pilot-scale operation of direct heating systems (Dickow *et al.*, 2012b; Murphy *et al.*, 2013; Dumpler *et al.*, 2017). For the SSIH system, increasing the flash cooling pressure to 0.6 bar(a), related to a temperature of 86 °C, could limit the change in product solids to a dilution of 0.16 %. However, for flash cooler operation at 0.3 bar(a), a simple mass balance can determine the quantity of product leaving the flash cooler to be 97 kg/h.

Tables

Table 1. Physico-chemical characteristics of protein-enriched skim milk beverage formulations at 4, 6, and 8% protein before and after heat treatment with direct infusion, direct injection or indirect tubular heat treatment at a preheat temperature of 70°C for 30 s and final heat temperatures of 121, 135 and 142°C for 3s.[1]

	Treatment		pH	Total Solids	Total Protein	Viscosity	Particle Size diameter[2]
	Technology	Temp	-	% (w/w)	% (w/w)	m.Pas	(nm)
4% Protein	Unheated		6.74 ± 0.05^{a}	10.3 ± 0.09^{a}	4.02 ± 0.16^{ab}	3.86 ± 0.12^{a}	222 ± 8^{a}
	Infusion	121	6.75 ± 0.05^{a}	9.15 ± 0.09^{b}	3.82 ± 0.05^{bc}	3.83 ± 0.06^{a}	220 ± 11^{a}
		135	6.75 ± 0.07^{a}	9.09 ± 0.00^{b}	3.74 ± 0.03^{c}	3.86 ± 0.14^{a}	212 ± 1^{a}
		142	6.76 ± 0.06^{a}	8.94 ± 0.14^{b}	3.73 ± 0.07^{c}	3.86 ± 0.04^{a}	211 ± 2^{a}
	Injection	121	6.68 ± 0.02^{a}	10.1 ± 0.01^{a}	3.95 ± 0.06^{abc}	3.64 ± 0.09^{a}	238 ± 4^{a}
		135	6.68 ± 0.02^{a}	10.0 ± 0.0^{a}	3.92 ± 0.03^{abc}	3.72 ± 0.05^{a}	222 ± 9^{a}
		142	6.68 ± 0.01^{a}	10.1 ± 0.02^{a}	3.93 ± 0.01^{abc}	3.67 ± 0.03^{a}	280 ± 9^{a}
	Tubular	121	6.76 ± 0.06^{a}	10.1 ± 0.08^{a}	3.99 ± 0.19^{abc}	3.82 ± 0.10^{a}	205 ± 1^{a}
		135	6.74 ± 0.06^{a}	10.1 ± 0.08^{a}	4.13 ± 0.05^{a}	3.73 ± 0.17^{a}	214 ± 8^{a}
		142	6.74 ± 0.05^{a}	10.1 ± 0.03^{a}	4.09 ± 0.05^{ab}	3.82 ± 0.07^{a}	230 ± 10^{a}
6% Protein	Unheated		6.73 ± 0.06^{a}	12.3 ± 0.13^{a}	5.81 ± 0.63^{abc}	4.33 ± 0.16^{a}	291 ± 75^{a}
	Infusion	121	6.73 ± 0.09^{a}	11.5 ± 0.12^{b}	5.51 ± 0.08^{abc}	4.21 ± 0.21^{a}	238 ± 6^{a}
		135	6.73 ± 0.08^{a}	11.1 ± 0.05^{bc}	5.41 ± 0.16^{c}	4.10 ± 0.02^{a}	230 ± 8^{a}
		142	6.73 ± 0.06^{a}	11.0 ± 0.13^{c}	5.41 ± 0.09^{bc}	4.36 ± 0.12^{a}	231 ± 10^{a}
	Injection	121	6.64 ± 0.02^{a}	12.1 ± 0.03^{a}	5.95 ± 0.01^{abc}	4.25 ± 0.06^{a}	259 ± 4^{a}
		135	6.64 ± 0.02^{a}	12.1 ± 0.03^{a}	5.95 ± 0.01^{abc}	4.19 ± 0.04^{a}	250 ± 1^{a}
		142	6.64 ± 0.01^{a}	12.1 ± 0.06^{a}	5.95 ± 0.04^{abc}	4.27 ± 0.06^{a}	282 ± 8^{a}
	Tubular	121	6.72 ± 0.08^{a}	12.2 ± 0.09^{a}	5.93 ± 0.29^{a}	4.11 ± 0.14^{a}	227 ± 26^{a}

		135	6.71 ± 0.08[a]	12.1 ± 0.25[a]	5.88 ± 0.16[abc]	4.18 ± 0.06[a]	269 ± 48[a]
		142	6.67 ± 0.07[a]	12.1 ± 0.12[a]	5.91 ± 0.20[abc]	4.32 ± 0.05[a]	272 ± 12[a]
8% Protein	Unheated		6.69 ± 0.06[a]	14.5 ± 0.12[a]	7.75 ± 0.38[a]	6.66 ± 1.33[a]	366 ± 14[a]
	Infusion	121	6.70 ± 0.08[a]	12.8 ± 0.28[b]	7.29 ± 0.30[a]	4.69 ± 0.52[b]	286 ± 6[bc]
		135	6.72 ± 0.06[a]	12.8 ± 0.29[b]	7.25 ± 0.23[a]	4.57 ± 0.45[b]	284 ± 26[bc]
		142	6.72 ± 0.06[a]	12.7 ± 0.27[b]	7.34 ± 0.20[a]	4.66 ± 0.06[b]	286 ± 10[bc]
	Injection	121	6.62 ± 0.01[a]	14.5 ± 0.06[a]	7.88 ± 0.08[a]	4.59 ± 0.13[b]	282 ± 17[bc]
		135	6.62 ± 0.00[a]	14.4 ± 0.07[a]	7.85 ± 0.03[a]	4.94 ± 0.48[b]	250 ± 6[c]
		142	6.63 ± 0.00[a]	14.2 ± 0.06[a]	7.82 ± 0.07[a]	4.66 ± 0.13[b]	316 ± 20[ab]
	Tubular	121	6.72 ± 0.06[a]	14.4 ± 0.20[a]	7.68 ± 0.47[a]	5.15 ± 0.19[b]	274 ± 20[bc]
		135	6.69 ± 0.06[a]	14.4 ± 0.19[a]	7.73 ± 0.44[a]	4.88 ± 0.43[b]	289 ± 13[bc]
		142	6.65 ± 0.05[a]	14.2 ± 0.08[a]	7.70 ± 0.51[a]	4.79 ± 0.26[b]	284 ± 13[bc]

[1] For each formulation (protein concentration), mean values with a common superscript letter in the same column are not significantly different ($p > 0.05$).

[2] Average particle size is presented in terms of intensity mean.

Table 2. Colour analysis of protein–enriched skim milks at 4, 6 and 8 % protein (w/w) before and after heat treatment with direct infusion, direct injection or indirect tubular heat treatment at a preheat temperature of 70°C for 30 s and final heat temperatures of 121, 135 and 142°C for 3s.

	Tech.	Temp. (°C)	L*	a*	b*	ΔE
4% Protein	Unheated		75.44 ± 1.12^{a}	-5.32 ± 0.16^{c}	-0.23 ± 0.20^{d}	
	Infusion	121	75.54 ± 1.51^{a}	-4.75 ± 0.05^{ab}	-0.17 ± 0.17^{d}	0.91 ± 0.18^{c}
		135	76.15 ± 1.39^{a}	-4.86 ± 0.03^{ab}	-0.18 ± 0.39^{d}	0.92 ± 0.28^{c}
		142	76.95 ± 0.89^{a}	-5.06 ± 0.18^{abc}	0.18 ± 0.10^{d}	1.64 ± 0.29^{c}
	Injection	121	75.81 ± 0.13^{a}	-5.08 ± 0.02^{abc}	2.62 ± 0.04^{ab}	2.89 ± 0.09^{b}
		135	76.09 ± 0.39^{a}	-5.05 ± 0.07^{abc}	2.71 ± 0.02^{ab}	3.11 ± 0.24^{b}
		142	76.52 ± 0.05^{a}	-5.05 ± 0.03^{abc}	2.87 ± 0.02^{ab}	3.35 ± 0.09^{b}
	Tubular	121	77.07 ± 1.04^{a}	-5.18 ± 0.07^{bc}	0.43 ± 0.27^{cd}	1.77 ± 0.07^{c}
		135	77.75 ± 1.03^{a}	-5.17 ± 0.10^{bc}	1.68 ± 0.46^{bc}	3.01 ± 0.45^{b}
		142	78.80 ± 1.42^{a}	-4.70 ± 0.25^{a}	3.32 ± 0.73^{a}	4.95 ± 0.62^{a}
6% Protein	Unheated		78.08 ± 1.16^{b}	-5.05 ± 0.56^{b}	1.56 ± 0.43^{c}	
	Infusion	121	78.64 ± 0.58^{b}	-4.72 ± 0.19^{ab}	3.00 ± 1.11^{bc}	2.26 ± 1.56^{b}
		135	78.70 ± 0.48^{b}	-4.63 ± 0.21^{ab}	2.75 ± 0.97^{bc}	2.15 ± 1.40^{b}
		142	80.20 ± 1.37^{ab}	-4.66 ± 0.08^{ab}	2.65 ± 0.44^{bc}	2.34 ± 0.67^{b}
	Injection	121	78.06 ± 0.13^{b}	-4.82 ± 0.01^{ab}	4.08 ± 0.00^{abc}	2.16 ± 0.18^{b}
		135	78.93 ± 0.04^{ab}	-4.74 ± 0.03^{ab}	4.48 ± 0.13^{abc}	3.13 ± 0.14^{ab}
		142	79.12 ± 0.13^{ab}	-4.62 ± 0.00^{ab}	4.71 ± 0.14^{ab}	3.41 ± 0.26^{ab}
	Tubular	121	80.58 ± 1.26^{ab}	-4.24 ± 0.92^{ab}	3.49 ± 1.00^{bc}	3.26 ± 1.41^{b}
		135	81.40 ± 1.33^{ab}	-3.86 ± 1.00^{ab}	5.12 ± 1.30^{ab}	5.08 ± 1.77^{ab}
		142	82.85 ± 1.07^{a}	-2.57 ± 1.07^{a}	7.25 ± 1.07^{a}	7.95 ± 2.49^{a}
8 %	Unheated		80.20 ± 0.44^{c}	-5.36 ± 0.18^{c}	2.82 ± 0.40^{e}	

Infusion	121	81.81 ± 1.19^{abc}	-4.54 ± 0.08^{b}	3.71 ± 0.41^{d}	2.75 ± 0.62^{d}
	135	82.08 ± 1.20^{abc}	-4.52 ± 0.09^{b}	3.84 ± 0.29^{d}	3.31 ± 0.78^{cd}
	142	82.28 ± 1.20^{abc}	-4.39 ± 0.04^{b}	4.28 ± 0.28^{cd}	3.77 ± 0.74^{bc}
Injection	121	80.14 ± 0.27^{c}	-4.51 ± 0.02^{b}	5.22 ± 0.16^{bc}	1.36 ± 0.42^{e}
	135	80.72 ± 0.08^{bc}	-4.38 ± 0.04^{b}	5.58 ± 0.10^{b}	2.03 ± 0.03^{de}
	142	80.92 ± 0.22^{bc}	-4.26 ± 0.02^{b}	5.85 ± 0.19^{b}	2.38 ± 0.43^{cde}
Tubular	121	81.90 ± 0.80^{abc}	-4.79 ± 0.16^{b}	4.13 ± 0.34^{cd}	2.90 ± 0.37^{cd}
	135	82.57 ± 0.78^{ab}	-4.44 ± 0.38^{b}	5.53 ± 0.80^{b}	3.95 ± 0.09^{b}
	142	84.04 ± 1.40^{a}	-3.72 ± 0.57^{a}	7.11 ± 1.22^{a}	5.87 ± 0.55^{a}

Figures

Fig. 1. Process flow diagram of (A) direct steam infusion, (B) direct steam injection and (C) indirect tubular heat exchange pilot plants across preheating, final heating, initial and final cooling operations. Common preheat and final cooling operations are used for the direct injection and indirect tubular plants (----).

Fig. 2. Levels of native (a) α-la, (b) β-lg A and (c) β-lg B protein in 4, 6, and 8% protein (w/w) formulations heat-treated using direct steam infusion (■), direct steam injection (■) and indirect tubular heating (■) at final heat temperatures of 121, 135 and 142 °C, expressed as a percentage of the respective native protein content of the unheated formulation. The error bars represent the standard error determined from three trial replicates.

Fig. 3. Particle size distribution, on an intensity basis, of unheated (– – –) beverages, and infusion (– · –), injection (—), and tubular (·····) heated 4% (w/w) protein formulation at final heat temperatures of (A) 121, (B) 135 and (C) 142 °C.

Fig. 4. Instability index of formulations, following accelerated storage stability using an analytical centrifuge at 2300 x g, for 3h at 25°C, with 4 (■), 6 (■), and 8% (■) protein (w/w) before and after heat treatment with direct infusion, direct injection or indirect tubular heat treatment at final heat temperatures of 121, 135 and 142 °C for 3 s.

Highlights

Supersonic steam injection provides rapid heating and high shear

Substantial native whey protein retention compared to tubular and infusion heating

No difference in accelerated physical stability for heating technologies

May enable new product development opportunities

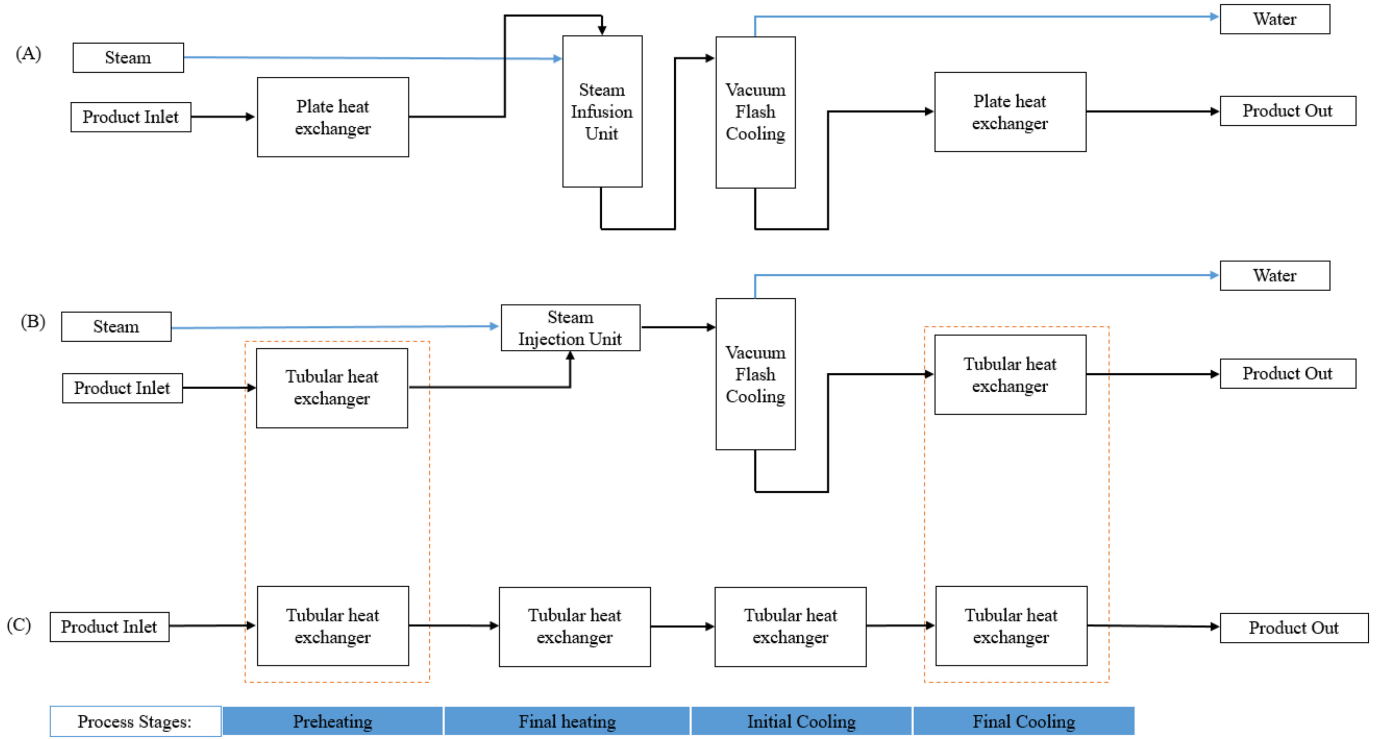

(A)
Steam
Product Inlet
Plate heat exchanger
Steam Infusion Unit
Vacuum Flash Cooling
Water
Plate heat exchanger
Product Out
(B)
Steam
Product Inlet
Tubular heat exchanger
Steam Injection Unit
Vacuum Flash Cooling
Water
Tubular heat exchanger
Product Out
(C)
Product Inlet
Tubular heat exchanger
Tubular heat exchanger
Tubular heat exchanger
Tubular heat exchanger
Product Out
Process Stages:
Preheating
Final heating
Initial Cooling
Final Cooling

Figure 1

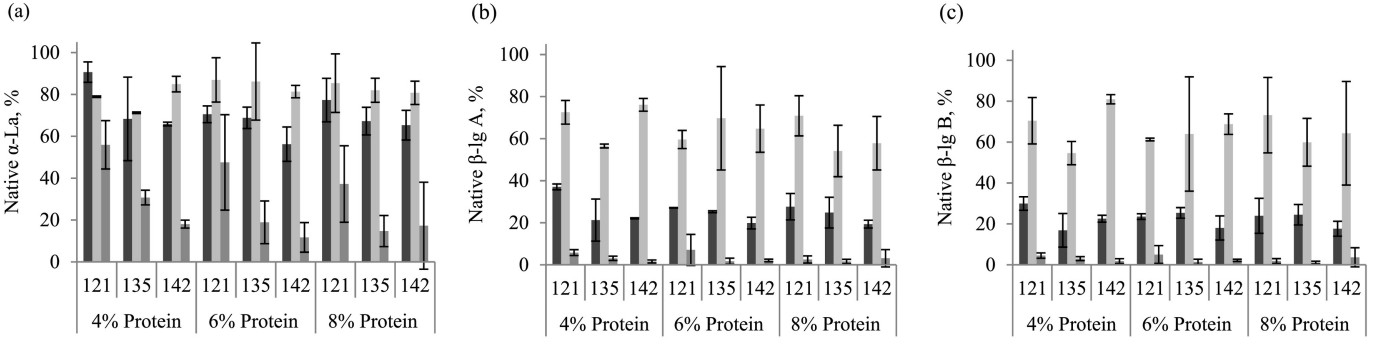
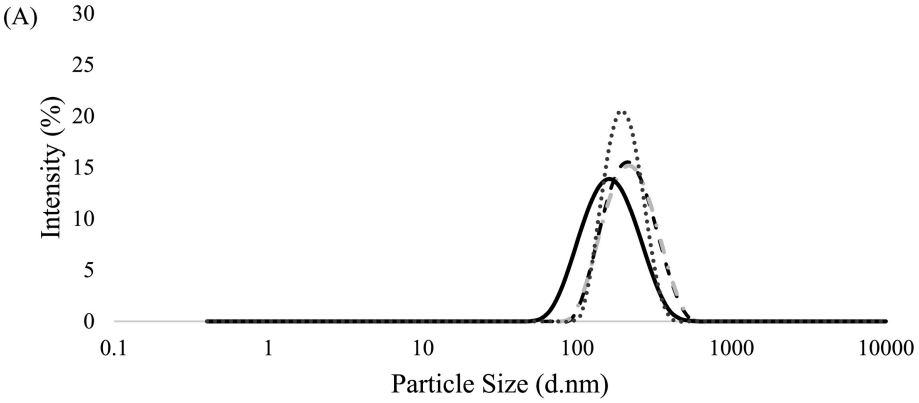
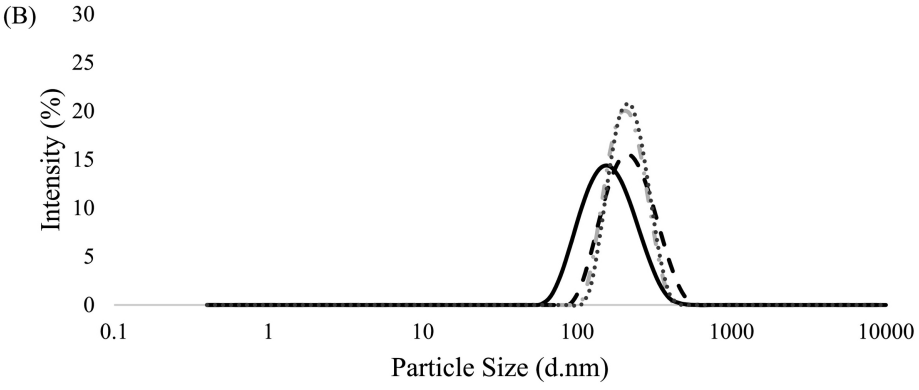

(a)
Native α-La, %
100
80
60
40
20
0
121
135
142
4% Protein
6% Protein
8% Protein
(b)
Native β-lg A, %
(c)
Native β-lg B, %

Figure 2


(A)
30
25
20
15
10
5
0
Intensity (%)
0.1
1
10
100
1000
10000
Particle Size (d.nm)


(B)
30
25
20
15
10
5
0
Intensity (%)
0.1
1
10
100
1000
10000
Particle Size (d.nm)

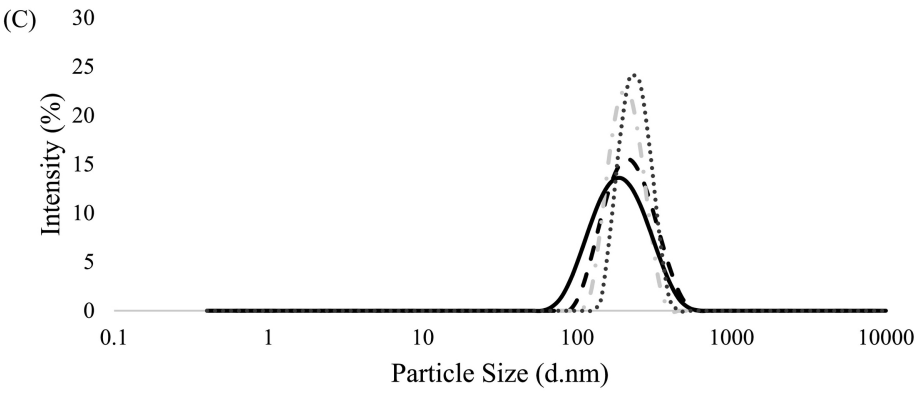

(C)
30
25
20
15
10
5
0
Intensity (%)
0.1
1
10
100
1000
10000
Particle Size (d.nm)

Figure 3

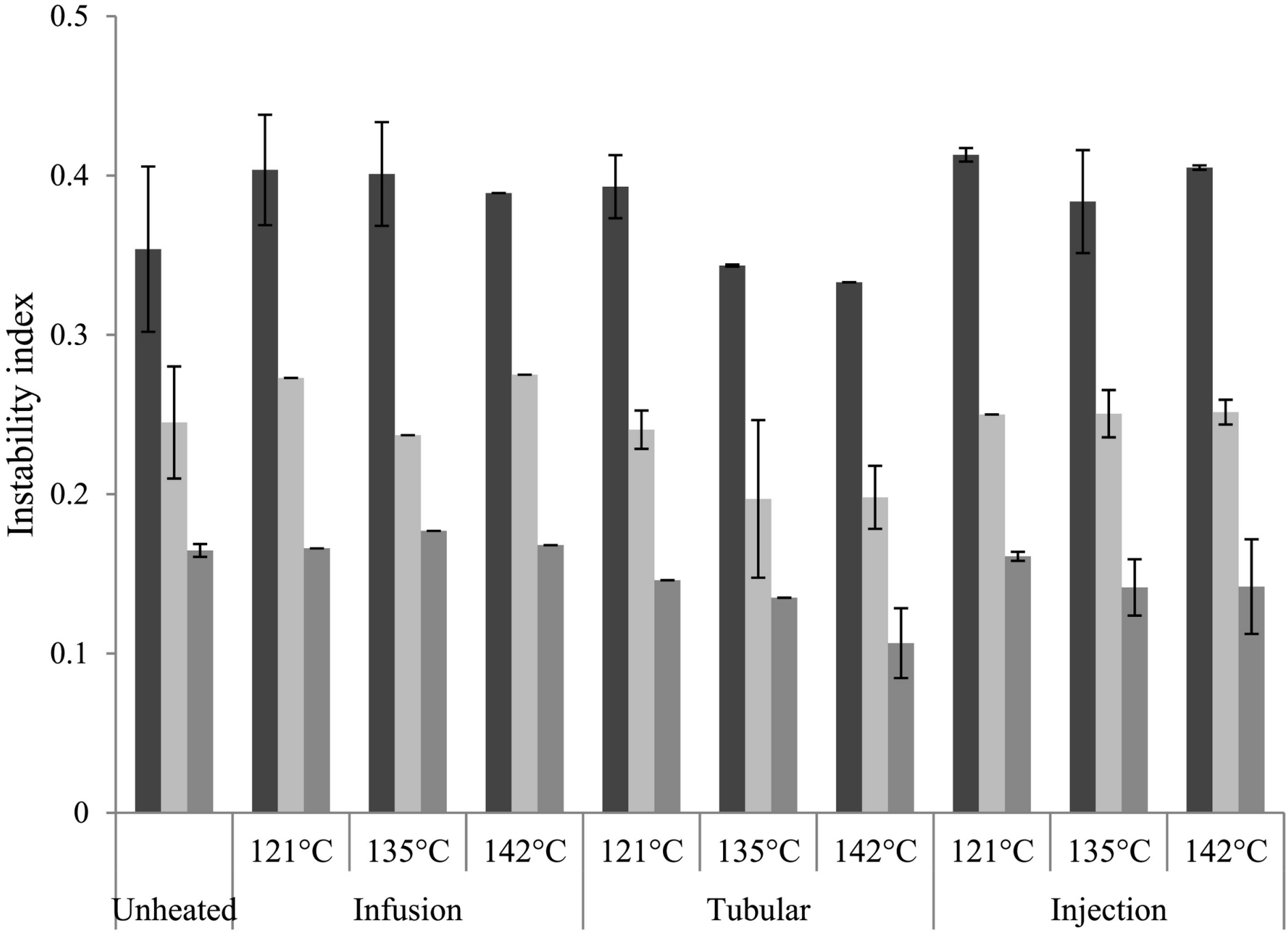

0.5
0.4
0.3
0.2
0.1
0
Instability index
121°C
135°C
142°C
121°C
135°C
142°C
121°C
135°C
142°C
Unheated
Infusion
Tubular
Injection

Figure 4