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The effect of high velocity steam injection on the colloidal stability of concentrated emulsions for the manufacture of infant formulations

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Abstract

A major challenge for the infant formula industry is to develop more energy efficient processes while maintaining product quality and robust manufacturing practices. An effective way of improving energy utilisation is to reduce the number of processing steps during manufacture. This study examines a novel high solids process with reduced processing steps paying particular attention to emulsion stability. Model infant formulations (whey to casein ratio, 60:40) were homogenised at a solids content of 60% w/w using an in-line colloid-mill type mixer, yielding a stable emulsion with a fat globule size distribution (D(v,0.9)) of 2.99 µm. These formulations were heat-treated using a high velocity direct steam injection device, whereby steam is accelerated using a De Laval nozzle before injection. The steam condenses on contact with the formulation, giving up latent heat, thus heating the mix. The process was found to increase the colloidal stability of the formulations, as measured in an analytical centrifuge. The fat globule size distribution was significantly ($p < 0.05$) decreased to 2.69 µm after processing by the injector with a concomitant significant ($p < 0.05$) increase in emulsion viscosity. In conclusion, in-line homogenisation followed by high velocity steam injection, using a De Laval geometry, was successfully used for heat treatment of a high solids infant formulation.

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Keywords: high solids processing; steam injection; colloid stability

1. Introduction

Currently infant formula is produced utilising multiple processing steps e.g. mixing of ingredients, heat treatment, homogenisation, evaporation and drying. The intensive energy usage and thermal load on the product means that a shorter process is desirable both in terms of economic/environmental sustainability

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and nutritional quality of the product. The concentration of infant formulations during processing is limited by fouling during heat treatment at higher solids contents. The fouling deposits, consisting of milk protein and minerals, are formed around the heat transfer surface reducing the efficiency of heat transfer and possibly compromising quality. The severity of this fouling increases as the concentration and viscosity of the formulation increases, necessitating more frequent cleaning of processing lines and thus increased process down-time [1]. Therefore, there is a maximum concentration that can be efficiently processed. Milk-based emulsions, such as infant formlations, undergo a variety of changes when heat treated. Whey proteins are particularly heat-sensitive and unfold, losing their secondary and tertiary structure when exposed to temperatures greater than 60 °C. This unfolding exposes hydrophobic groups previously located within the interior of the protein leading to interactions with similarly exposed groups of other proteins i.e. aggregation. In emulsions it has been proposed that these interactions lead to the flocculation of fat globules. Whey proteins in the continuous phase unfold on heating and can interact with protein adsorbed on fat globules causing flocculation [2,3]. As emulsion concentration increases, emulsion particles come into closer proximity increasing the probability of interactions. Caseins are not susceptible to thermal unfolding and the presence of sodium caseinate has been shown to protect protein-stabilised emulsions against flocculation [4]. However, complexes of κ-casein and β-lactoglobulin are known to occur during heating [5] and it is possible that, in high solids content emulsions, this could contribute to fat globule flocculation. Homogenisation downstream of heat treatment ensures the dissociation of any flocculated material formed during heat treatment. The effect of upstream homogenisation of milk on its microstructure and separation stability has been studied [6]. It was found that both the microstructure and extent of creaming were similar for UHT milk processed using upstream and downstream homogenisation. However, the same may not be true for concentrated dairy emulsions as constituents are in much closer proximity.

Infant formulas are susceptible to changes in nutrient composition if subjected to high heat treatment during manufacturing [7]. Heat induced changes include non-enzymatic glycosylation or glycation of protein which occurs in the early stages of Maillard reaction [8], where lactose becomes covalently attached to the lysine residues of proteins during heating. The formation of this reaction product, called lactulosyllysine, renders lysine biologically unavailable. It has been reported that the heat load and nutritional changes incurred during direct heat treatment are less than that of indirect heat treatment for the same bactericidal effect [9].

The aim of this study is to determine the effect of upstream colloid mill homogenisation followed by heat treatment using a high velocity steam injector on the colloidal stability of concentrated (60% total solids) model infant formulations (60:40 whey:casein). The high velocity steam injection used in this study is a direct heat transfer process whereby steam is accelerated using a De Laval nozzle before injection into the product stream, forming a two-phase mixture of product and steam travelling at supersonic velocity. The internal geometry of the unit forces the steam to condense, heating the product as it gives up its latent heat.

2. Materials & Methods

2.1 Composition

Model first age infant formulae (3 x 200 kg batches) were formulated at a target solids content of 60% w/w, using medium heat skim milk powder (SMP, protein 34%, = 19.2 kg), electro-dialyzed whey powder (EDW, protein 12.5%, = 54.8 kg), lactose (12 kg), sunflower oil (34 kg) and RO water (80 kg). The protein:fat:lactose ratio was 1:2.6:5.1. SMP and EDW were supplied by Dairygold Food Ingredients (Ireland). Lactose was supplied by Friesland Foods Domo, (The Netherlands), under the product name Lactopure. Sunflower oil (SO) was supplied by Trilby Trading (Ireland). Potassium Hydroxide was supplied by Sigma Aldrich (Ireland).

2.2 Processing

Reconstitution and mixing of the dried ingredients was achieved using a YTRON ZC powder induction unit/colloid mill (YTRON Process Technology GmbH, Germany) equipped with a high shear dispersion head (3 mm tooling). The negative pressure created as the liquid stream was pumped through the head was used to induct the powders. After powder induction the oil was added and formulations were homogenised by recirculating through the colloid mill dispersion head of the YTRON ZC for 10 minutes. pH was adjusted to 6.9 with 4 N potassium hydroxide prior to heat treatment (at 120 °C for 3 s) using a Maklad high velocity steam injector (Maklad Innovative Fluid & Systemtechnik Gmbh, Austria). Formulations were then cooled using a plate heat exchanger.

2.3 Analysis

Fat globule size distribution (FGSD) of formulations was determined, in duplicate, using a Mastersizer 2000S (Malvern Instruments Ltd., UK). The optical parameters selected were a sample refractive index of 1.46, a particle absorbance of 0.1 and a dispersant refractive index of 1.33 (water). The parameter D(v,0.9) was chosen to represent fat globule size and corresponds to a theoretical particle size beneath which 90% of the particles by volume are found. Solids content (% w/w) was determined, in duplicate, by Smart System 5, Smart Trac System (CEM Corporation, USA). Viscosity was determined using an AR2000ex Rheometer (TA Instruments, UK). Measurements were carried out at 20 °C on samples standardised to a solids content of 12.5% w/w, using a 60 mm diameter parallel plate. Samples were pre-sheared at 300 1/s for 1 minute, equilibrated for 1 minute and then ramped from 1 to 300 1/s while operating under conditions of steady state flow. Stability of formulations was determined by a Lumifuge analytical centrifuge (L.U.M Gmbh, Germany) which operates on the basis of continuous measurement of light transmitted through a specimen over a defined length in a measurement cell. Samples were centrifuged at 1500 rpm (221 – 287 *g*, dependent on the distance from the rotor within the length of the measurement cell) for 7.5 hours, simulating approximately 3 months ageing under conditions of normal gravity. Separation behaviour was analysed using Sepview 4.1 (L.U.M Gmbh, Germany) software which calculated the time course of the separation in percentage transmission/hour. Levels of native whey pre- and post-heat treatment were measured, in duplicate, by reverse-phase high performance liquid chromatography (RP-HPLC) using a 1200 Series system (Agilent Technologies, USA). The pH of the samples were adjusted to 4.6 to remove the casein, non native whey and fat from the samples prior to RP-HPLC analysis. The column used was an XBridge BEH300 C4 3.5 μm (Waters, USA). The solvents used were - Aqueous Phase: 0.1% trifluoroacetic acid (TFA) v/v, and Organic Phase: 90% acetonitrile (ACN), 0.1% TFA. The gradient used was as follows: 30% Organic Phase for 2.5 minutes, increasing to 50% Organic Phase over the next 12.5 minutes. Absorbance was measured at 214 nm and column temperature was 28 °C. Statistical significance of variables pre- and post-heat treatment was determined by means of a paired t-test.

3. Results & Discussion

Table 1 shows the averaged FGSD and solids content of samples pre- and post-heat treatment for of all three trials. Reconstitution and mixing using the YTRON ZC device produced stable emulsions of approximately 60% w/w solids content with, on average, a FGSD (D(v,0.9)) of 2.99 μm. To the best knowledge of the authors, there is no work published on the homogenisation of high solids (approx. 60% w/w) infant formulations; therefore no comparison can be drawn between the fat globule size distribution obtained from the trials and published work. However, no destabilisation of the emulsion was apparent during the trials which can be attributed to the viscosity of the emulsion. Heat treatment by high velocity steam injection resulted in a significant ($p < 0.05$) decrease in FGSD to a D(v,0.9) of 2.69 μm, most

likely due to the cavitation induced shear created as the steam condenses. The condensing steam also had the effect of diluting the final product to approximately 55% w/w, suitable for spray drying.

Table 1. Fat Globule Size Distribution and Solids Content (average of 3 trials) pre- and post-heat treatment.

Stage	D(v,0.9) μm	% w/w
Pre-heat treatment	2.99	59.09
Post-heat treatment	2.69	54.76

To avoid the effect of age thickening and lactose crystallisation at high solids content, all emulsions were standardised to a concentration of 12.5% w/w before rheological analysis. This also compensated for variation in the solids content between samples taken pre- and post-heat treatment (Table 1). Figure 1 shows the averaged viscosity profile pre- and post- heat treatment.

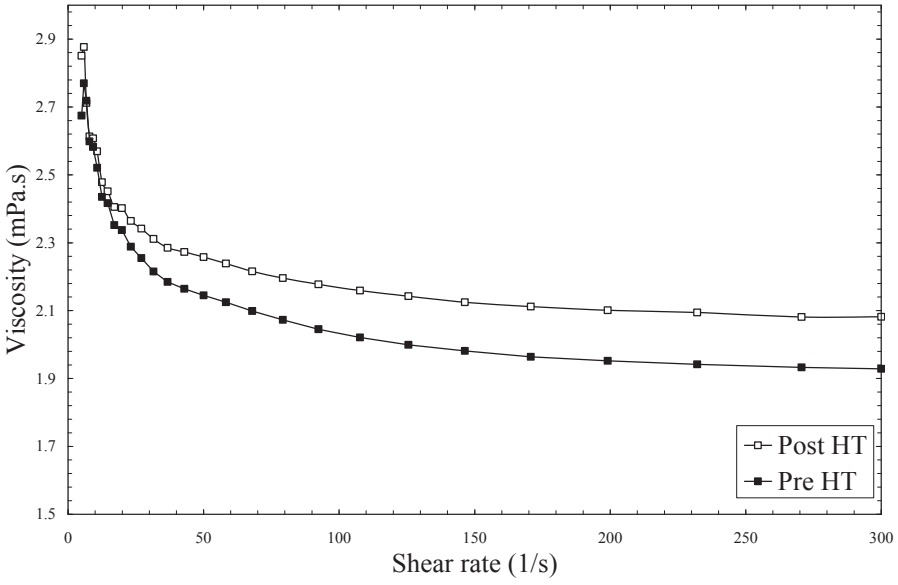

Fig. 1. Viscosity of standardised (12.5% w/w) formulations pre- and post-heat treatment (HT) by high velocity steam injection. Each profile is an average of three replicate trials

The apparent viscosity (at 300 1/s) of the heat treated samples was found to be significantly ($p < 0.05$) greater than the non-heated, i.e. 2.08 compared to 1.93 mPa.s. Emulsification of oil-in-water occurs in two steps, 1) disruption of the oil phase into smaller droplets of increased surface area followed by 2) stabilisation by adsorption of an emulsifier to the droplet surface [10] and an increase in viscosity [11]. The increase in viscosity along with the concomitant reduction in FGSD (approx. 10%) suggests that there was a homogenisation effect within the injector.

Separation of native whey protein was achieved by RP-HPLC. The samples were diluted to 0.1% w/v total whey protein (native and non-native inclusive) with the level of denaturation given by the difference between the actual RP-HPLC response and the response of 0.1% w/v native whey. The level of denatured protein for Trial 3 is represented in Figure 2, along with standards of 0.01% w/v of α-lactalbumin (α-lac) and β-lactoglobulin (β-lg). It can be seen that when compared with the 0.01% w/v standards, the native

whey protein remaining in the pre-heat treatment sample is low, in the range of 10%. The peak areas shown in Table 2 were found to be quite variable as illustrated by the high standard deviation of β-lg. In all cases the amount of native whey in the emulsions was reduced post heat treatment as a result of thermally induced denaturation. The broadening of the α-lac and β-lg peaks, in comparison to the standards, could be attributed to glycated isoforms eluting, as shoulders, before the main peaks of α-lac and β-lg [8]; the hydrophilic lactose moieties attached to the protein reduce its overall hydrophobicity [12]. It is possible that the broadening of the peaks seen could be due to glycation of the whey protein or partial unfolding of the whey protein, however it is likely to be the former as in skim milk powder it has been reported that up to 50% of lysine residues are converted to lactuloslysine [7]. Therefore, there was likely partial glycation in the powders used to formulate the emulsion which may have increased during batch make-up at high temperature (60 – 70 °C) resulting in the broad peaks.

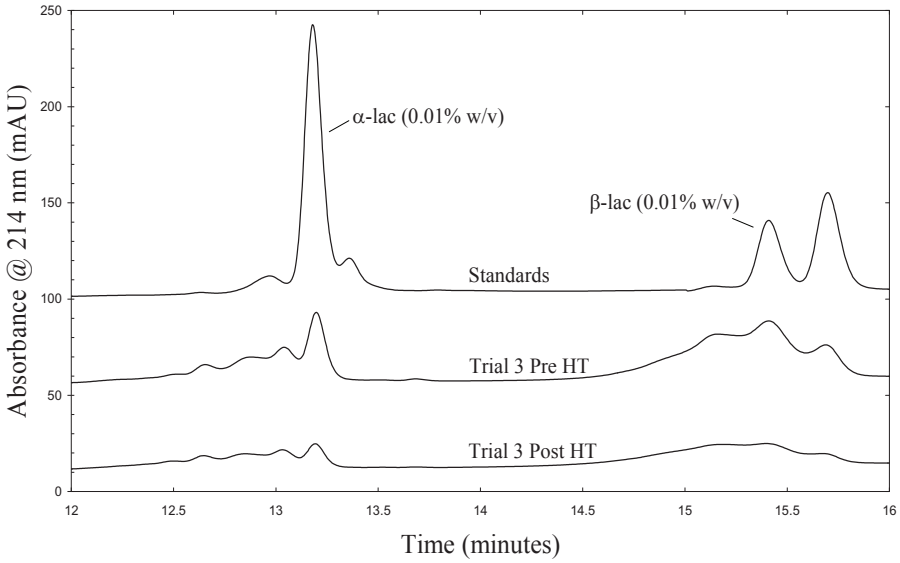

Fig. 2. RP-HPLC Profiles for Trial 3 vs. 0.01% protein standards

Table 2. Integrated Chromatograph Areas

Trial	α-lac (mAU.min)	β-lg (mAU.min)
Pre Heat Treatment	724 ±69	1759±615
Post Heat Treatment	385±15	805±245

The stability of the formulations to separation (measured as change in % transmission of light through the sample per hour, %/h) is shown in Figure 3. Stability increased after heat treatment, with the initial rate of change of transmission of light i.e. separation, decreasing from 10.8 to 4.9%/h. This is in accordance with Stokes' Terminal Velocity equation, which gives separation rate as proportional to the square of the droplet diameter and inversely proportional to viscosity [13]. Therefore the reduced FGSD and increased viscosity post-heat treatment resulted in lower separation rates.

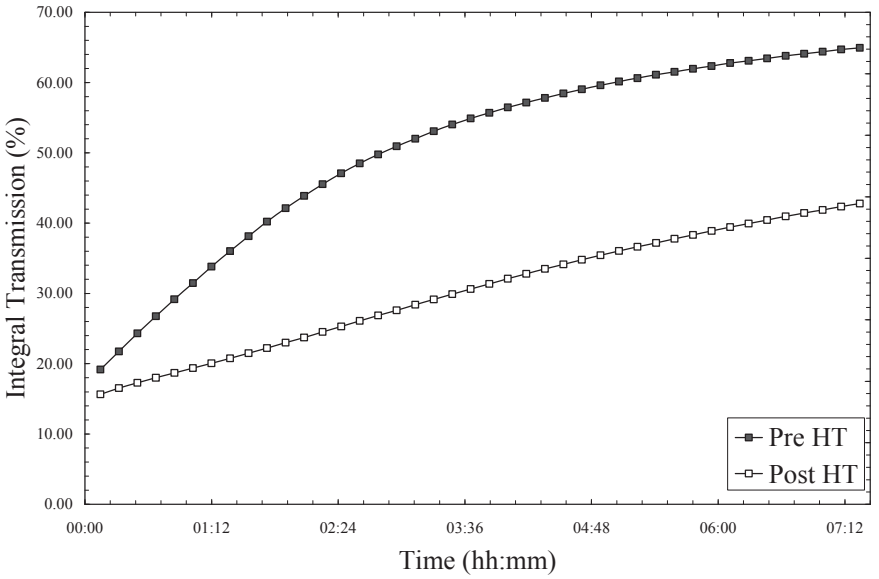

Fig. 3. Averaged separation profiles of standardised (12.5% w/w) formulations

4. Conclusion

This study presents a feasible alternate process for the production of infant formula utilising less processing steps. Upstream homogenisation of model infant formulations was achieved using a high shear dispersion head/colloid mill, which also served to hydrate the dried ingredients. The benefit of this type of homogenisation is a more efficient process. Heat treatment of formulations using high velocity steam injection increased the colloidal stability of the formulations. Overall the new process is capable of producing stable high solids emulsion suitable for spray drying, without the use of evaporation.

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