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Formation and structure of insoluble particles in reconstituted model infant formula powders

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ABSTRACT

Phase separation of insoluble particles, so called “white flecks”, is a physical defect in reconstituted milk obtained from spray-dried, fat-containing dairy powders. Compared with bulk powder particles, white flecks have quite different chemical and morphological characteristics. They consist mainly of proteins and fat, forming a dense network. Here, two concurrent formation mechanisms are suggested based on the structures revealed by confocal microscopy imaging. Fused spherical particles with free fat on the surface were the most abundant and likely formed during the manufacturing process. Sharp-edged particles containing shapes typical of lactose crystals formed during storage. Fat globule stability was thus determined to be critical in preventing white fleck formation. Stability was increased with (a) the presence of lecithin in the oil phase; (b) systems with high protein-to-fat ratio and (c) storage in dry conditions since humidity leads to lactose crystallisation and disruption of the fat globules in powders.

1. Introduction

Milk powder production is based on pre-concentrating of milk by thermal evaporation followed by drying with a spray dryer or a rotary wheel atomiser. During spray drying, the concentrate is sprayed through an orifice with high pressure and exposed to a flow of hot air. Water is rapidly removed and solid particles are formed (Kelly & Fox, 2016). Reconstituted milk powder should ideally reflect the organoleptic, nutritional and colloidal properties of fresh milk. However, an issue that is often encountered upon dispersion of the powder in water medium is the presence of insoluble particles (Schuck et al., 2016; Singh & Ye, 2010).

The definition of the insoluble materials is not straightforward as they may have several different origins. Schuck et al. (2016) used the term “flecking” in infant milk formula (IMF) powder covering all insoluble material originating from protein interactions, fat coalescence or flocculation, or insoluble powder particles. Písecký (1997) classified the insoluble material in three categories of defects: sediment, slowly dispersible particles (SDP) and white flecks (WF). Sediment can be collected by centrifugation, but the differentiation of SDP and WF is not straightforward. According to McKenna, Lloyd, Munro, and Singh (1999), the structure of SDP is analogous to powder particles. The reason for their slow dispersion is therefore likely due to either agglomerate structure or surface composition. WF are completely insoluble and therefore possibly comprise a composition and structure different from that of bulk powder particles.

In this work, WF is defined as the insoluble material that suspends atop the aqueous dispersion forming a thin layer (Písecký, 1997). Therefore, the WF is assigned to fat. They typically have diameters of a few hundred micrometres and are prone to adhesion onto the contacting surfaces of the container. Their presence is easily determined by pipetting reconstituted milk onto a black plate. Besides the unappealing appearance of WF in reconstituted milk, they also promote clogging of the orifices in bottle nipples, reducing usability of the IMF. WF are only observed in reconstituted fat-containing milk powders, while they are absent in the case of skim milk powder. Therefore, their formation in milk powders is ascribed to the presence of milk fat. In IMF, the role of other components, such as vegetable fat, protein or lactose in the formation of WF is still unclear, as well as the morphology and composition of the particles. Thorough characterisation of the particles is needed to understand the mechanisms of formation and the conditions to minimise their occurrence in the reconstituted IMF.

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Insoluble particles with various sizes may form with several mechanisms at different stages of the manufacturing process. Insolubility originates from heat instability of the system before or during drying and depends on the composition, pretreatment (homogenisation, pasteurisation, lecithination) characteristics of the concentrate (pH, solids content, viscosity), dryer parameters (nozzle design, inlet and outlet temperatures) and finally, the transport and storage conditions (Sadek et al., 2014; Schuck et al., 2016; Sharma, Jana, & Chavan, 2012). Lactose, native whey proteins and some inorganic salts are the only completely water soluble components in milk powder. Therefore, fat globules, casein micelles, denatured whey protein and their combinations have the ability to form insoluble material (McKenna, 2000).

Protein insolubility is initiated by the unfolding of whey proteins followed by aggregation with casein (Baldwin, 2010; Sharma et al., 2012; Straatsma, Van Houwelingen, Steenbergen, & De Jong, 1999) and in fat-containing systems this results in formation of protein-bridged fat globule clusters (Singh & Ye, 2010). Based on the previous research (De Ruyck, 1991; McKenna et al., 1999; Mol, 1975; Ohba, Takahashi, & Igarashi, 1989) the formation of insoluble material is hypothesised to originate from heat and shear applied during spray drying resulting in changes in fat-protein interactions. Also, emulsion quality before drying (Schmidmeier, O'Gorman, Drapala, & O'Mahony, 2017) and fat crystallisation (Regost, 2016) as well as whey protein denaturation (Joyce, Brodkorb, Kelly, & O'Mahony, 2017; McKenna et al., 1999) at different stages of drying may play a role. Lactose crystallisation during humid storage has been reported to result in disruption of fat globules, higher amount of free fat and rougher particles in model infant formula powders made with different protein-to-fat ratios (McCarthy et al., 2013). This could contribute to insoluble material formation during storage.

This study presents the isolation and characterisation of white flecks from a model IMF powder and a suggestion of their origin. WF morphology and composition was determined with confocal imaging, chemical and thermal analyses. Fat globule distribution, fat globule clustering and size in the native powders were characterised to elucidate what type of powder structure is related to the WF formation. The experimental results suggest at least two different formation mechanisms, the control of which may assist in minimising the formation of such insoluble particles.

2. Materials and methods

2.1. Infant formula powders

IMF powders were composed from fresh fat standardised milk, demineralised whey powder (Valio Demi™90, Lapinlahti, Finland), infant formula fat OP3 (Cargill, Izegem, Belgium) containing sunflower, rapeseed and coconut oil and lecithin, and minerals to meet the standard IMF for stages 1–3 according to Technical Regulation of the Customs Union "On Safety of Milk and Dairy Products" (TR TS 033/2013). Target values are reported in Table 1. IMF powders were prepared with an industrial scale drying process in controlled conditions in a series of 33 preparations at Valio Ltd, Lapinlahti, Finland. Vegetable oil was homogenised at 140/40 bar (Tetra Alex 350, Tetra Pak, Lund, Sweden) and the mixture was evaporated to average dry matter of 50% using mechanical vapour recompression (MVR) type evaporator (GEA, Skanderborg, Denmark) and spray dried (Tetra Magna, Tetra Pak, Lund, Sweden).

In this study, detailed microstructural analysis of two stage 1 IMF powders (Powder A and Powder B) from the test series is presented and correlations between composition, structure and white flecks formation are evaluated from the 33 test powders. Apart from the lecithin addition in Powder B, the composition of the powders was similar. Moisture content of powder A was lower (2.3% in powder A and 1.4% in powder B) but this was not significant for white fleck formation as described later in section 4, Results and discussion. Six of the test runs (including Powder A) had a second homogenisation with a high pressure pump in the feed line (pressures 80/40 bar). Three test runs (including Powder A) had a higher second stage homogenisation pressure (140/80 bar). The composition of the powders (averages ± standard deviations) is shown in Table 1. Eight powders were stage 1–2 powders without lecithin, 15 were stage 1–2 powders with lecithin, seven were stage 3 powders without lecithin and three were stage 3 powders with lecithin. Target levels of lecithin in the powder were 0.2–0.3%. Free calcium was determined from reconstituted solution (12%) using an ion selective electrode (ionised calcium micro volume electrode, Konelab™/T-series) and an Arena 30 analyser (Thermo Scientific, Vantaa, Finland). The effect of the compositional variables on white fleck formation was evaluated by calculating the correlation coefficient between variables in Table 1. A correlation coefficient >0.5 indicates positive correlation between variables whereas values < 0.5 indicate negative correlation.

The powders were stored in closed containers in ambient conditions. To demonstrate the effect of increased humidity, the powders were kept for 3 weeks in evacuated desiccators in the presence of saturated NaCl and K_2CO_3 salt solutions (Merck KGaA, Darmstadt, Germany) for water activity (a_w) of 0.08 and a_w 0.43, respectively.

2.2. Extraction and quantification of white flecks

WF were extracted while trying to minimise the interference of any other insoluble material or material originating from the soluble fractions and dried during sample preparation. In particular, the extraction of SDP and WF has not been well established. SDP are extracted and quantified based on the fact that they remain on the walls of the test tube after pouring off the reconstituted milk or by filtration after 2 min of standing time (Niro, 2005; Písecký, 1997; GEA). The WF number (WFN) test (ISO/IDF, 2009), unlike SDP, is based on the clogging of a 63-μm sieve that results in slower filtration. However, these tests do not distinguish between WF and SDP. For quantification of WF, the IDF method cannot be used to appropriately distinguish between samples with low number of WF.

Here, WF were determined by dissolving 30 g of powder in 250 mL water at 50 °C followed by stirring for 1 min. The solution was allowed to stand for 10 min and 2 mL was pipetted from the top onto a black plate. The visible WF were counted and a lower number of particles was taken as an indication of a higher powder quality. Powders with a WF count >45 were assigned unacceptable for consumption. The particles were counted only when the total count was <45, otherwise the powder was assigned with the score 45.

2.3. Compositional analysis of white flecks

For compositional analysis, reconstituted solution was filtered through a 63 μm sieve, the insoluble material was washed with water at 50 °C two times and collected from the sieve. Protein, fat and dry matter contents were analysed in duplicate from the WF material extracted by sieving. Protein was determined with the Kjeldahl method (ISO/IDF, 2014) and fat with the Schmid-Bodzynski-Ratzlaff method (ISO/IDF, 2004).

2.3.1. SDS-PAGE

Protein composition was determined with SDS-PAGE ran in duplicate from reconstituted stage 1–3 bulk powders and