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Moisture sorption isotherm and caking properties of infant formulas

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ABSTRACT

The moisture sorption isotherms of three different infant formulas (denoted as P1, P2 and P3) that contained almost the same amount, but different types, of protein were analysed and their physicochemical changes characterised. Although P3 showed a different trend as compared to both P1 and P2, all the powders had the highest moisture content at 0.428 a_w, signifying the occurrence of lactose crystallisation as confirmed by both XRD analysis and glass transition temperature (T_g) analysis. The particle size (D_{90}) decreased for all the powders due to distortion of particle structure. Caking was observed in all samples conditioned between 0.330 and 0.753 a_w. The caking strength of P2 was significantly higher as compared to the other powders at 0.529 a_w, which suggests that magnesium content played a significant role in the hygroscopicity of the powders. These results demonstrate that small compositional differences have a profound effect on the moisture sorption behaviour and caking strength of the infant formulas.

1. Introduction

Infant formula comprises of essential nutrients and bioactive components that are important for both the nutrition and development of an infant. Supplementation of infant formulas with nutritionally functional components has been an increasingly popular trend in order to achieve better composition and functionalities similar to that of human breast milk (O'Callaghan et al., 2011). Due to further supplementation, the infant formula matrix becomes increasingly complex, raising concerns with the stability and its impact on the functionality of the finished product. In general, the physicochemical stability of infant formula is influenced by its composition, storage condition and handling.

One of the main components used in formulating infant formulas is lactose. During the spray drying process of infant formula, lactose may be dried under its saturation point and form amorphous lactose (Haque and Roos, 2006). Amorphous lactose is extremely hygroscopic and susceptible to changes in the environmental relative humidity and temperature, and through prolonged storage, can undergo physicochemical changes (Chuy and Labuza, 1994; Jouppila et al., 1997; Vuataz, 2002).

In addition, infant formulas that are formulated for infants (between 0 and 6 months of age) have a casein:whey ratio of 40:60. Caseins, such as α, β, κ, are flexible proteins with disordered structure while whey proteins such as β-lactalglobulin A, β-lactalglobulin B, α-lactalbumin, immunoglobulin G and bovine serum albumin, have disulphide bridges, tertiary structure and can maintain their globular structure even after interfacial adsorption (Dickinson, 2001). The changes in their molecular structures have a profound effect on the physicochemical and interfacial properties of infant formulas (Gaiani et al., 2011). Due to the ability of α-lactalbumin to bind with strong calcium sites via cation binding, it is able to form intramolecular ionic bonds in the presence of calcium. This enhances the molecule's resistance to thermal unfolding and therefore its heat stability (Kilara, 2008).

Deteriorative changes occur when moisture is absorbed from the environment typically on the powder surface. The temperature whereby amorphous lactose changes from the “meta-stable” glass state to the rubbery state is known as the glass transition temperature (T_g) (Chuy and Labuza, 1994; Jouppila and Roos, 1994; Roos, 2003). Even with a minimal increase in moisture content, water is known to have a strong plasticising effect that reduces T_g significantly and induces degradative changes in infant formula (Nasirpour et al., 2007). Lactose crystallisation in infant formula can lead to increased stickiness, increased water activity of the overall product, increased free fat, development of off-flavours, increased non-enzymatic browning reactions (Kim et al., 1981) and caking.

Caking is a phenomenon that causes an initially free-flowing

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powder containing low moisture content to gradually form lumps and subsequently progress to create a hardened agglomerated solid structure (Hartmann and Palzer, 2011). Caking of fine, hygroscopic crystalline powders is possible due to the partial dissolution and recrystallisation of their crystalline components (Wahl et al., 2008; Mauer and Taylor, 2010). In addition, fat-containing powders suffer from caking problems due to partial melting and recrystallisation of triglycerides that are present.

The majority of nutrition for infants who are formula fed is obtained from the digestion of the reconstituted powder formula. Physicochemical changes that occur during shelf-life of the powder formula such as caking might influence the rheological properties and particle size of the infant formulas and their subsequent nutrient absorption (Chuy and Labuza, 1994; Prakash et al., 2014).

In light of the increasing global demand of increased fortification and highly value added infant formula, it is important to understand the relationship between the moisture present within these nutritional powders and the consequent physicochemical changes occurring when exposed to a range of relative humidity. As such, the main objectives of this study were to characterise three infant formulas of different compositions that were conditioned under various relative humidities, to establish the changes in their physicochemical properties and to determine their moisture sorption isotherms and changes in their caking strength.

2. Materials and methods

2.1. Materials

Three industrially spray-dried infant formulas denoted by P1, P2 and P3 were obtained from Wyeth Nutritionals Pte Ltd. (Singapore). Their macro compositions are summarised in Table 1 and Fig. 1 shows the levels of calcium, phosphorous, chlorine and magnesium present in the powders, which were estimated from the amounts of all mineral-containing components in the ingredients. The lactose content was determined using an Automatic Polarimeter (MCP 300, Anton Paar, Austria). The protein content was measured using Kjeldahl method with a nitrogen-protein conversion factor of 6.38. The fat content of each infant formula was determined using a Soxhlet extractor. The amount of infant formulas used for the extraction was 1.74 (±0.03) g and the solvent used for the extraction was hexane and it was heated to reflux at 80 °C for 40 h. The standard gravimetric method of drying was employed for moisture content analysis. Samples of 2.5–3.0 g were weighed into aluminium dishes and allowed to dry at 102 °C ± 2 °C for 2 h under atmospheric pressure. The mass was subsequently determined using additional drying steps of 1 h until the weight difference was no more than 0.5 mg. The samples were then incinerated at 550 °C in a furnace to determine the ash content.

The powders were commercially available products that were freshly produced and packed into cans for consumer use. The major protein components present in the infant formulas were as follows: P1 consisted of skim milk powder (SMP), P2 consisted of a combination of SMP and demineralised whey proteins (DWP) while P3 consisted of a combination of SMP and whey protein concentrate α-lactalbumin. The amount of α-lactalbumin present in P1, P2 and P3 were approximately 0.389, 0.189 and 1.976 g/100 g powder, respectively. The vegetable oil blends for the three infant formulas consisted of palm oil, coconut oil, soybean oil, sunflower oil and lecithin at approximately 8.8, 5.6, 7.2, 5.6 and 0.24 g/100 g powder, respectively. For this study, two separate batches of infant formula (i.e. P1, P2 and P3) were used. Triplicates were conducted for each batch and a total of six readings were obtained for each analysis.

Table 1
Composition of the 3 infant formulas (n = 6).

Product	Composition (% w/w)				
	Lactose	Protein	Fat	Moisture	Ash
P1	56.0 ± 0.2	12.0 ± 0.2	29.0 ± 0.4	1.99 ± 0.05	3.00 ± 0.19
P2	56.6 ± 0.2	11.8 ± 0.1	28.3 ± 0.2	2.02 ± 0.03	2.00 ± 0.09
P3	58.0 ± 1.3	11.0 ± 0.1	29.0 ± 1.0	1.97 ± 0.03	2.50 ± 0.09

2.2. Moisture adsorption analysis

Seven different salts in their saturated solutions providing gradient relative humidity (RH) values were employed to create desired RH in separate dessicators at 25 °C. They were LiCl (11.1% RH), CH_3COOK (22.5% RH), $MgCl_2$ (33.0% RH), K_2CO_3 (42.8% RH), $MgNO_3$ (52.9% RH), KI (68.9% RH) and NaCl (75.3% RH) (Greenspan, 1954). One gram of sample from each powder product was placed in a plastic bottle (without capping) and was stored in these desiccators until equilibrium (i.e. 21 days) (Shrestha et al., 2007). The equilibrium moisture content of the powders was measured gravimetrically by drying in a vacuum oven at 70 °C for 6 h. The moisture content was measured and expressed as percentage dry basis.

2.3. Glass transition temperature

Glass transition temperature (T_g) was measured by a Differential Scanning Calorimeter (Mettler-Toledo DSC822e, Switzerland) equipped with liquid nitrogen cooling accessories. Dry nitrogen gas was used to purge the surroundings of the furnace and furnace chamber at 200 ml/min and 80 ml/min, respectively, to avoid the condensation of water. A RH equilibrated powder sample (5–10 mg) was weighed into a 40 μl aluminium standard crucible (ME-51119870, Mettler-Toledo, Switzerland) and hermetically sealed with an aluminium standard lid (ME-51119871, Mettler-Toledo, Switzerland). All the samples were cooled at a rate of 20 °C/min to reach a temperature that was about 60 °C below their T_g midpoints (±5 °C). Then the samples were reheated at 10 °C/min to a temperature about 40 °C above the T_g midpoints (±5 °C) to analyse the glass transition temperature. T_g was analysed using the STARe software (Version 8.01, Mettler-Toledo, Switzerland). The midpoint of the glass transition was considered as the characteristic temperature of the transition and was determined as shown in Fig. 2.

2.4. Particle size analysis

The particle size distribution of powders was measured in isopropyl alcohol (IPA) by using a LA-950 V2 Laser Scattering Particle Size Analyser (Horiba, Japan) at room temperature. The refractive index of isopropyl alcohol was set to be 1.378 and a stationary iteration number of 15 was used. The particle size distribution was characterised by the D_{90} value (i.e. the diameter at which 90% of the powder had smaller particle sizes while 10% were bigger).

2.5. Caking analysis

Caking tests were performed based on the method described in Wang and Zhou (2012) with slight modifications. Four grams of powder sample were placed in a cylindrical plastic bottle lined with paper (3.5 cm diameter and 6 cm height), tapped gently to form a uniform flat layer, and compacted under a Texture Analyser-XT2i (Stable Micro System, UK) with a load force of 2 kg for 1 min. The bottle containing the compacted powder was stored in desiccators containing saturated salt solutions that created the relative humidity condition of 0.330, 0.428, 0.529, 0.689 and 0.753 a_w