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# Effects of composition and relative humidity on the functional and storage properties of spray dried model milk emulsions



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#### ABSTRACT

This study investigates the effect of relative humidity (RH) and milk composition on the changes in solubility, lactose crystallinity and protein structural form on a commercial infant formula, by identifying the interactions between water, lactose and protein at different storage conditions. Two spray dried model milk emulsions were studied for comparison, by storing these powders under varying relative humidity (RH) levels between 11 and 94% at room temperature. The properties of powders were evaluated from the degree of insolubility, the rate of browning, and the extent of protein denaturation. The model samples were found to be more stable than the commercial powder, even at high relative humidity (>50% RH). The difference was attributed to the presence of casein, which did not denature to the same extent as whey protein. The results also suggested that minerals present in the commercial powder could be responsible in enhancing protein denaturation, thus accelerating the rate of browning and decreasing solubility.

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#### 1. Introduction

Although human milk is very complex in nature, the dairy industry has attempted to replicate it using a complex combination of proteins, carbohydrates, fats, and vitamins to produce infant formula for bottle-feeding (Jacobsen, 2013). Previously, milk has been extensively dried using thin films on heated rollers, however this method was largely replaced by spray-drying in the 1960s (Bhandari et al., 2013), as a more efficient and economical method for dairy powder production. Commercialised dairy powders such as skimmed milk powder (SMP) or whole milk powder (WMP) are manufactured through processes such as evaporation, atomisation, spray-drying, and fluidised bed drying/cooling. However, the production of powdered infant formula differs from that of SMP or WMP due to the relatively high level of protein content in the products, preventing direct spray drying of the formulation. Generally, they are manufactured via two types of processes, i.e. dry mixing process or wet mixing – spray drying process. The former consists of mixing together dehydrated powdered ingredients to achieve a uniform blend of macro and micronutrients necessary for a complete infant formula product. Wet blending on the other hand

Much work has been done over the years to improve the properties of dairy powder. There are standard methods to quantify different properties such as foaming properties, flowability, instant properties, and heat stability. However, the amount of studies done on infant formula pales in comparison to those on skimmed milk or whole milk powder. Little is known on the parameters shortening the shelf-life of this complex dairy powder, besides the occurrence of lipid oxidation (Almansa et al., 2013; Angulo et al., 1998). The aim of this study is to quantify the influence of different relative humidity (11–91% RH) on the physical and functional properties of spray-dried milk model emulsions and commercial formula for comparison. These model emulsions have general compositions (fat, protein, lactose) similar to that of breast milk during the first six months of lactation.

#### 2. Materials and methods

#### 2.1. Preparation of model emulsions

Two model emulsions; model emulsion 1 and model emulsion 2 (ME1 and ME2) were prepared using  $\alpha$ -Lactose monohydrate (Sigma–Aldrich, Australia), whey protein isolate (WPI) (Mullins

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constitutes of blending different ingredients together with the addition of water, homogenizing, pasteurizing, and spray drying to produce the final powdered product.

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Whey, USA), sunflower oil (Crisco, Australia) and milk protein concentrate (MPC) (MG Nutritionals, Australia) based on the dry basis milk composition. Their basic compositions (fat, protein, lactose) were based on those of breast milk in the first fortnight and from three to six months of lactation, respectively. On dry basis, WPI (Mullins Whey, USA) contains 0.7 wt% lactose, 94.5 wt% protein, 1.8 wt% fat and 3.0 wt% contents while MPC (MG Nutritionals, Australia) contains 4.5 wt% lactose, 86.0 wt% protein, 1.6 wt% fat and 7.9 wt% ash. All ingredients used in this study were used as purchased.

To make up an emulsion of 500 g and of 25 wt.% concentration, specific amounts of lactose, sunflower oil, MPC and WPI were used with 350 g of deionised water added to the dry mixture. The suspension was placed in a water bath on a magnetic stirrer/hot plate to allow it to be constantly stirred for 1 h at 50 °C. The mixture was then passed through a high pressure homogenizer (Emulsiflex C5, Avestin, Canada) at two passes - 350 bars and 10 bars to yield a homogenous emulsion.

#### 2.2. Microfluidic jet spray drying

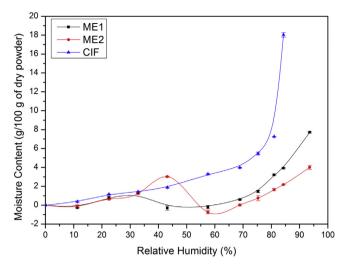
Monodisperse droplets were formed by a micro-fluidic aerosol nozzle system with an orifice diameter of 100 µm. The emulsion was fed into a standard steel reservoir with dehumidified instrument air to force the liquid to jet through the nozzle and the jet was broken up by disturbance from vibrating piezoceramics. The droplet formation was monitored using a digital SLR (Nikon D90) with a speed light (Nikon SB-400) and micro-lens (AF Micro-Nikkon 60 mm f/2.8D), while droplet spacing was optimized via adjustment of frequency and period of stimulation of piezoelectric nozzle under observation using high-speed photography, until monodisperse droplets were formed. The monodisperse droplets were well dispersed and dried in a micro-fluidic-jet spray dryer (MFJSD) at an inlet temperature of 180 °C and an outlet temperature of 80 °C. The average inlet air flow pressure was at 10 psi, while the inlet and outlet air relative humidity were on average between 52 and 58%.

### 2.3. Storage study of spray-dried milk powder

The equilibrium moisture content was determined using the static gravimetric method, employing the use of saturated salt solutions to maintain a fixed relative humidity at room temperature. Desiccators were used to ensure the required airtight environment. Excess salt were added to obtain saturated salt solutions, hence creating the required equilibrium relative humidity. Ten different environment with relative humidity values in the range of 11.3–93.6% were created using salts (sodium hydroxide, potassium acetate, magnesium chloride, potassium carbonate, sodium bromide, potassium iodide, sodium chloride, ammonium sulphate, potassium chloride and potassium nitrate with RH of 11.3, 22.5, 32.8, 43.2, 57.6, 68.9, 75.3, 81.0, 84.3 and 93.6% RH, respectively). The spray dried powders and the commercialised infant formula were placed in a vacuum oven at 30 °C for 3-4 days to remove as much moisture as possible. 2.0 g of the dried ME1, ME2 and commercialised powder (NAN H.A. 1 Gold Infant Formula, Nestle, Australia) were weighed in a plastic petri dish and then placed inside ten desiccators. The experiments were done in triplicate and were run until the constant weights (equilibrium) of the samples were reached (within 0.001 g) after 7 weeks of storage.

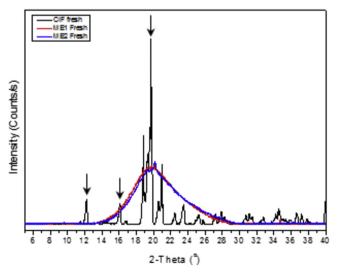
#### 2.4. Characterisation of samples

Scanning Electron Microscope (SEM) images of the spray-dried samples were examined using a FEI Nova NanoSEM 450 Field



**Fig. 1.** Moisture sorption isotherm of model emulsion 1 (ME1) and 2 (ME2) and commercial infant formula at room temperature.

Emission Electron SEM. The microscope was operated between a HV of 2-5 kV and with a spot size of 2.0. The X-ray diffraction patterns of the dairy powders were measured using a Rigaku MiniFlex 600 XRD, in the range of 5–40  $2\theta$  at a step size of  $2\theta = 0.02$ and reading speed of  $2\theta = 2/\min$ . Additionally, the insolubility index measurement was done according to GEA Niro method No.A3a (IDF, Standard 129), whereby  $3 \pm 0.01$  g of spray dried powder was reconstituted in 150 mL of deionised water at 50 °C. The solution was stirred for 30 min before being centrifuged at 4000 rpm for 10 min. Instead of only measuring the volume of supernatant left after centrifugation, the supernatant was removed and the insoluble matter was dried to constant weight at 50 °C for a period of 24 h and insolubility index was recorded in milligram (mg) Attenuated total reflectance (ATR) mid-infrared spectra were acquired using a Fourier transform infra-red spectroscopy (FTIR, PerkinElmer, Australia), over a wavenumber range of 600–4000 cm<sup>-1</sup>, with all measurement performed in duplicate. As for the measurement of denaturation of protein, a differential scanning calorimeter (DSC) was employed, whereby approximately 10 mg of the sample solution of concentration 10 g protein/100 ml



**Fig. 2.** XRD Pattern of fresh commercialised infant formula (with arrows indicating characteristic peaks of α-lactose monohydrate), ME1 and ME2.

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