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# The compositional effects of high solids model emulsions on drying behaviour and particle formation processes



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### ABSTRACT

This paper investigates the drying behaviour and particle formation of emulsions modelled after a range of compositions observed in human milk at different lactation periods. The knowledge on drying kinetics would be relevant to understand important drying conditions in processing dairy products with similar compositional makeup while shrinkage kinetics and surface characterization provide a greater comprehension of the influence surface-active milk components have on product functionality. Droplet drying histories of model emulsions are presented through the glass-filament single droplet drying (SDD) method combined with the Reaction Engineering Approach (REA) modelling. For droplets with both initial solids of 20 wt.% and 40 wt.%, model emulsion (ME) with the highest lactose content exhibited the most shrinkage (normalized particle size being 0.68 and 0.81 respectively), followed by those with highest protein content (0.70 and 0.81), whereas those with the highest fat content showed the least shrinkage (0.73 and 0.84). In situ rehydration study found that ME with the highest fat content exhibited the earliest skin formation but delayed crust formation. The X-ray photoelectron spectroscopy (XPS) analysis which observed an overrepresentation of surface fat across all three MEs regardless of the drying technique indicated the occurrence of surface fat enrichment during ME droplet generation and drying processes.

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## 1. Introduction

Spray drying is utilized in a wide range of industries to transform feed from fluid to dry solid state, in order to extend the shelf-life of commercial materials. Greater control over the final product quality could be achieved through accurate modelling of the spray drying operations using computational fluid dynamics (CFD) which requires a sufficiently realistic drying model (Woo et al., 2008b). Drying kinetics data for CFD simulation could be obtained by interpreting droplet temperature and moisture content histories during convective dehydration using the Reaction Engineering Approach (REA) drying model. An overview of the REA has been reported in previous studies (Chew et al., 2013; Fu et al., 2012b) and has demonstrated its robustness in drying performance prediction (Putranto et al., 2010; Woo et al., 2008a).

From the perspective of the REA model, droplet evaporation is driven by vapour density difference between the drying medium and droplet surface (Chew et al., 2014). The foundation of the model being a material-specific normalized activation energy curve that accurately describes the thermal-moisture history of droplet dried under varied conditions (Chew et al., 2014). Glass-filament single droplet drying (SDD) conveniently generate the necessary experimental data by allowing one to study the drying behaviour of a single droplet in a controlled drying environment. This approach has been used extensively to study a range of materials (Fu et al., 2011; Lin and Chen, 2007).

Infant formula is an alternative source of liquid nutrition for newborns where breastfeeding is not possible. Extensive study on infant formula has been conducted through spray drying where the functional, nutritional, and physicochemical properties of powdered product were determined (Kane et al., 2010; Singh and Mathur, 1992; Zhang, 2009). However, the drying behaviour and



*Abbreviations:* C, carbon; CFD, computational fluid dynamics; Dev., standard deviation; ME, model emulsion; MPC, milk protein concentrate; N, nitrogen; O, oxygen; REA, Reaction Engineering Approach; SDD, single droplet drying; SMP, skim milk; SSD, single stream drying; WPI, whey protein isolate; XPS, X-ray photoelectron spectroscopy.

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Nomenclature				
wt.% D <sub>o</sub> D/D <sub>o</sub> X X <sub>b</sub>	weight solute/weight solution after mixing initial droplet diameter (mm) normalized droplet diameter droplet moisture content (kg/kg) bulk moisture content (kg/kg)	$\begin{array}{lll} X-X_b & \mbox{reduced moisture content (kg/kg)} \\ \Delta E_{v,max} & \mbox{equilibrium activation energy (J/kmol)} \\ \Delta E_v/\Delta E_{v,max} & \mbox{normalized activation energy} \\ \gamma & \mbox{relative fraction of component at the particle surface} \\ I & \mbox{molar fraction of chemical element present} \end{array}$		

particle formation of infant formula as well as compositional influence have not been previously observed.

Droplets containing dissolved solids would often undergo a constant-rate drying period in the presence of an outer free moisture evaporative front. Insufficient outward moisture diffusion leads to the appearance of surface skin. The dissolved solids would accumulate on the droplet outer layer during rapid drying and form shrinkage-inhibiting crust. Particle functionality will be dependent on surface properties that dictate particle-liquid interactions during reconstitution. It is noteworthy that surface composition may differ from bulk as the distinct diffusion coefficient of each component led to material segregation during particle/powder production which involved atomization (droplet generation) and drying (Kim, 2008).

In this work, drying of high solids model emulsion (ME) systems of different compositions was carried out to investigate compositional effects on their drying behaviour and particle formation process. The emulsions were very simplified version of human milk at different lactation period. This work was not intended to mimic human milk but motivated by interests to determine the degree of influence exerted by the predominant presence of different milk components on droplet drying rate and product quality. In situ rehydration study and X-ray photoelectron spectroscopy (XPS) analysis were also conducted to enable qualitative determination of compositional influence on droplet drying behaviour and particle formation process.

#### 2. Materials & method

#### 2.1. Material preparation

Three model emulsions (ME) were prepared using lactose monohydrate (Sigma–Aldrich, Australia), whey protein isolate (WPI) (Mullins Whey, USA), sunflower oil (Crisco, Australia) and milk protein concentrate (MPC) (MG Nutritionals, Australia) based on the bulk composition on dry basis displayed in Table 1. On dry basis, WPI (Mullins Whey, USA) contains 0.7 wt.% lactose, 94.5 wt.% protein, 1.8 wt.% fat and 3.0 wt.% ash content while MPC (MG Nutritionals, Australia) contains 4.5 wt.% lactose, 86.0 wt.% protein, 1.6 wt.% fat and 7.9 wt.% ash. 240 g of deionised water (Millipore, Australia) was weighted and heated in a water bath (Büchi, Switzerland) to 50 °C. 160 g of combined solids based

#### Table 1

Bulk composition of three model emulsions on dry basis (Dewey et	al., 1984; Dewey
and Lönnerdal, 1983; Hibberd et al., 1982).	

Composition	Model emulsion (ME) Mean ± Dev. (% dry basis)			
	1	2	3	
Lactose	59.07 ± 0.20	45.26 ± 0.10	42.55 ± 0.12	
Fat	29.27 ± 0.02	23.97 ± 0.03	42.43 ± 0.12	
Protein	11.66 ± 0.22	30.77 ± 0.10	15.03 ± 0.24	
	Lactation period 1 7–11 months	representation 1–2 days	1 month	

on their respective amount (Table 2) were mixed briefly with the heated deionised water to obtain a 40 wt.% emulsion solution. As for 20 wt.% emulsion solution, 80 g of combined solids were added to 320 g of heated deionised water. WPI and MPC were added at a ratio of 1:0.81 to achieve the whey protein-casein micelle ratio in human milk (65:35). Emulsion mixture was then sent through a high pressure homogenizer (Emulsiflex C5, Avestin, Canada) with three passes at the pressure range of 150–172 bar and two passes at 30-34.5 bar to yield a homogenous emulsion. Emulsion stability was determined using an optical analyser (Turbiscan MA 2000, Formulaction, USA) and the size of oil droplets  $(10 \,\mu m)$  was observed and measured using an optical microscope. A sample from each emulsion was oven dried at 102 °C ± 2 °C for 2 h and cooled to room temperature before measuring the dry weight. The exact concentrations of ME1, ME2 and ME3 were 37.0 wt.%, 38.6 wt.% and 39.6 wt.% as the powdered form of individual components (Table 2) contained pre-existing moisture content and possible solids loss during the homogenization process.

#### 2.2. Experimental method

## 2.2.1. Glass-filament single droplet drying

 $2\pm0.05~\mu$ L single droplets with initial mean diameter of 1.64 mm were generated using a positive displacement pipette (Microman M10, Gilson, France) and transferred onto a fine glass filament inside a purpose-built drying chamber. Compressed air was filtered and dehumidified (KF-DDF-125, Knight Pneumatics, Australia) prior to entering the heater and maintained at a flow rate of 0.75 m/s, temperature of 70 °C and humidity of 0.0001 kg/kg.

Using the single droplet drying rig, change in droplet temperature, diameter and mass were obtained independently in separate experiments with four repetitions. Droplet diameter change was determined from images recorded via a camcorder (Sony DCR-HC36 Camcorder, Sony Corporation, Japan) and extracted using Adobe After Effects 7.0 (Adobe Systems, USA). Droplet temperature was monitored by hanging the droplet across a thermocouple (Type K, Part# CHAL-001, Omega Engineering, USA), linked to the computer via a picometer (TC-08, Pico Technology, UK). Droplet mass change was derived based on the displacement of a mass-measuring glass filament which deflects when a single droplet is present at its tip. A standard curve that correlates displacement distance to droplet mass change was generated through

Table 2	
Mass of components in three model emulsions to make 4	0 wt.%.

Composition	Mass (g)			
	ME1	ME2	ME3	
Lactose	94.01	71.09	67.42	
Sunflower oil	46.48	37.42	67.43	
WPI	10.78	28.46	13.90	
MPC	8.73	23.03	11.25	
Water	240.00	240.00	240.00	
Total	400.00	400.00	400.00	

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