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Water sorption and diffusion properties of spray-dried dairy powders containing intact and hydrolysed whey protein

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ABSTRACT

The aim was to compare the effect of intact or hydrolysed whey protein in spray-dried lactose/protein powders on water diffusion properties and microstructure. Dispersions of protein/lactose (0.21:1) containing either intact or hydrolysed whey protein were spray-dried at pilot scale, and physical properties were determined. Lactose/hydrolysed whey protein powders had significantly increased ($P < 0.05$) particle density, resulting in lower bulk density and occluded air, and higher interstitial air. Moisture sorption analysis at 25 °C showed that dispersions containing intact whey protein exhibited lactose crystallisation at a lower relative humidity (RH) compared to the dispersions containing hydrolysed whey protein. Hydrolysed whey protein dispersions had a lower monolayer moisture value (m_m) than intact whey protein dispersions, as calculated using the Guggenheim–Anderson–de Boer (GAB) equation. Water diffusivity, determined at 25 °C from water sorption kinetics and the application of a mathematical model based on Fick's 2nd law, was significantly different ($P < 0.05$) with respect to the presence of intact or hydrolysed whey protein over the RH range examined (0–60% RH), except at 40% RH. The presence of hydrolysed whey protein resulted in a significantly higher ($P < 0.05$) water diffusivity in powders, with potential implications for hygroscopicity, caking, stickiness and flowability in humid environments.

1. Introduction

Carbohydrates and proteins are major non-fat solid components in food products, and their interaction with water and with each other influence their physical properties (Haque & Roos, 2005; Roos, 2002). These macronutrients are commonly derived from dairy sources. The proteins can be intact or hydrolysed, with the latter being composed of peptides produced from hydrolysis of intact proteins, giving lower average molecular mass and less secondary structure than intact proteins (Flanagan & FitzGerald, 2002). Hydrolysed proteins have reduced immunological reactivity and are commonly used in infant formulae for hypoallergenic infants and in the nutritional management of individuals who cannot digest whole or intact protein (Hochwallner et al., 2015). Peptides generated by hydrolysis are readily absorbed, and are therefore an attractive source of nitrogen in sports nutrition. Hydrolysis of proteins alters functional characteristics such as solubility, emulsification and foaming properties in food products.

Lactose, a low molecular weight carbohydrate in milk, commonly exists in the amorphous state in a number of low-moisture foods, i.e., dairy powders. The state of lactose in dairy powders may change during storage to a glassy or crystalline state, depending on composition and storage conditions, e.g., relative humidity, temperature and duration of storage (Jouppila, Kansikas & Roos, 1997; Kelly et al., 2015; McCarthy et al., 2012).

Water transfer in porous food products is complex, with different mechanisms occurring; vapour diffusion in air-filled pores as a result of vapour pressure gradients and movement of liquid due to capillary action (Gekas, 1992). The relative impact of these mechanisms to overall water diffusivity is difficult to determine, and therefore apparent water diffusivity is obtained using Fick's second law. Water diffusion of flour has been studied using sorption isotherms (Lomauro, Bakshi & Labuza, 1985), with limited studies on water diffusion within dairy powders. Murrieta-Pazos et al. (2011) examined water transfer in skimmed milk powder (SMP) and whole milk powder (WMP), and showed that WMP had a lower diffusion coefficient than SMP, suggesting that the presence of fat

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retards the diffusion of moisture. Diffusion of water is affected by the physical structure of the food material (Murrieta-Pazos, Galet, Gaiani & Scher, 2014); an increase in moisture gives rise to a change in porosity, which can lead to collapse of the food structure.

Powders with higher protein:lactose ratios are less susceptible to sticking (Kelly et al., 2015). Hydrolysed protein powders also tend to have higher hygroscopicity and thermoplasticity compared with powders containing intact proteins.

The aim of this work was to compare the effect of inclusion of intact versus hydrolysed whey protein in spray-dried lactose/protein dairy dispersions on water diffusion properties and the effects of storage under different RH levels on microstructure.

2. Materials and methods

2.1. Materials

Intact whey protein concentrate powder (80 g protein/100 g powder) and hydrolysed whey protein concentrate powder (80 g protein/100 g powder) with a degree of hydrolysis (DH) value of 12 were obtained from Carbery Ingredients Ltd. (Ballineen, Co. Cork, Ireland). According to the supplier, the hydrolysed whey protein had an average molecular weight of 5.84 kDa, with more than 70% > 5 kDa. Edible-grade α-lactose monohydrate was obtained from Glanbia Ingredients (Ballyraggett, Co. Kilkenny, Ireland), and SMP was purchased from Dairygold Food Ingredients (Mitchelstown, Co. Cork, Ireland).

2.2. Preparation of spray-dried powders and experimental design

The experiment was carried out in triplicate, where the trials for each replicate were carried out in random order using Design Expert Version 7.1.6 (Stat-Ease, USA). The dispersion (20% total solids) consisted of 3.34 g protein/100 g (whey protein: casein, 60:40; either intact or hydrolysed whey protein), and 16.04 g lactose/100 g. Batches (15 kg) were prepared as follows; lactose powder was dissolved in water at ~70 °C, using a Silverson L4RT (Silverson Machines Ltd., Waterside, Chesham, Bucks, England) mixer to aid reconstitution. SMP was then added slowly, followed by the whey protein. The batches were tempered to 60 °C and adjusted to pH 6.9 by adding 4 mol/L KOH and maintained under high shear for 30 min to ensure complete hydration of the whey protein. The blend was agitated prior to heat treatment at 100 °C for 30 s using a pilot scale tubular heat exchanger (Microthermics Model 25HV; North Carolina, USA). The product was then spray-dried in a pilot-scale Anhydro Spray dryer (Model Plant No. 3 type I KA, Copenhagen, Denmark), equipped with a two-fluid nozzle atomisation system (Type 1/8 JAC 316ss) and counter-current drying. Dryer inlet temperature was maintained constant at 185 °C and outlet temperature was maintained at 80 °C. Powders were labelled IF and HF, for powders containing intact and hydrolysed whey protein, respectively. Samples of powder were stored at 10 °C in sealed foil bags until analysis was completed.

2.3. Viscosity

Viscosity for each formulation post heat-treatment was measured at 55 °C and reconstituted product at 12.5 g/100 g was measured at 20 °C using the method described by Kelly, O'Mahony, Kelly and O'Callaghan (2014). The apparent viscosity measured at 500 s^{-1} was used for comparison of formulations.

2.4. Powder characterisation

Water content of powders was determined using a halogen rapid moisture analyser (HR-83 Halogen, Mettler Toledo, Switzerland). The samples were dried at a temperature of 105 °C until a constant weight was attained (<1 mg change over 140 s, equivalent to ±0.025%). Water activity (a_w) was measured with a Novasina LabMaster aw water activity meter (Novatron Scientific Ltd., West Sussex, UK). Protein (N x 6.38) was determined by macro-Kjeldahl (IDF 2001). Ash content was determined after overnight incineration at 550 °C. Lactose content was estimated by difference in weight. Bulk density and particle density (ρ_p) were measured as described by GEA Niro methods (GEA Niro, 2006, b), respectively. The volume of interstitial air (V_{ia}) and occluded air (V_{oa}) were determined as per GEA Niro (2006).

2.5. Powder particle size distribution

Powder particle size was determined by laser light scattering using a Malvern Mastersizer 3000 with the Aero S unit (Malvern Instruments Ltd., Malvern, Worcestershire, UK). Powder sample was added to the standard venturi disperser with a hopper gap of 4 mm and then fed into the dispersion system. Compressed air at 50 000 Pa was used to transport and suspend the powder particles through the optical cell, with a measurement duration of 10 s. Background measurements were made using air for 20 s.

2.6. Water sorption isotherms

Sorption isotherms were determined gravimetrically as described by Kelly et al. (2015) using a dynamic vapour sorption (DVS) technique (DVS Advantage 1, Surface Measurement Systems Ltd., London, UK). Dried samples (~30 mg) were loaded into the sample pan and humidified from 0 to 90% RH in 10% RH increments. Equilibrium was considered to be reached when change in mass with time (dm/dt) was <0.001 mg/min for at least 10 min for each step. DVS Data Analysis Suite, which runs with a Microsoft Excel add-on (Microsoft Office Excel, 2003), was used to graph and analyse data.

The Guggenheim–Anderson de Boer (GAB) equation (Van den Berg, 1984) was used to model water sorption isotherms and to determine the critical water content and water activity (a_w).

$$\frac{m}{m_m} = \frac{CKa_w}{(1 - Ka_w)(1 + Ka_w(C - 1))} \quad (1)$$

where m is the moisture content (g/100 g dry solids), m_m is the monolayer value (g/100 g), a_w is water activity, and C and K are constants, K having units inverse to a_w and C being dimensionless.

Bizot (1983) showed that this equation could be converted to a second-order polynomial giving a quadratic equation (Eq. (2)):

$$\frac{a_w}{m} = \alpha a_w^2 + \beta a_w + \gamma \quad (2)$$

Values for the parameters, α, β, γ were determined by quadratic regression analysis of a_w/m as a function of a_w using DVS data, e.g., over a range of a_w, from 0 to 0.4. The solution to equations (1) and (2) give the values for m_m, K and C as;

$$m_m = \frac{1}{\sqrt{\beta^2 - 4\alpha\gamma}} \quad (3)$$

$$K = \frac{\beta - \left(\frac{1}{m_m}\right)}{-2\gamma} \quad (4)$$