



Effects of hydrolysis on solid-state relaxation and stickiness behavior of sodium caseinate-lactose powders

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ABSTRACT

Hydrolyzed or nonhydrolyzed sodium caseinate-lactose dispersions were spray dried, at a protein:lactose ratio of 0.5, to examine the effects of protein hydrolysis on relaxation behavior and stickiness of model powders. Sodium caseinate (NC) used included a nonhydrolyzed control (DH 0) and 2 hydrolyzed variants (DH 8.3 and DH 15), where DH = degree of hydrolysis (%). Prior to spray drying, apparent viscosities of liquid feeds (at 70°C) at a shear rate of 20/s were 37.6, 3.14, and 3.19 mPa·s, respectively, for DH 0, DH 8, and DH 15 dispersions. Powders containing hydrolyzed casein were more susceptible to sticking than those containing intact NC. The former had also lower bulk densities and powder particle sizes. Scanning electron microscopy showed that hydrolyzed powders had thinner particle walls and were more friable than powders containing intact NC. Secondary structure of caseinates, determined by Fourier transform infrared spectroscopy, was affected by the relative humidity of storage and the presence of lactose as co-solvent rather than its physical state. Glass transition temperatures and lactose crystallization temperatures, determined by differential scanning calorimetry were not affected by caseinate hydrolysis, although the effects of protein hydrolysis on glass-rubber transitions (T_{gr}) could be determined by thermo-mechanical analysis. Powders containing hydrolyzed NC had lower T_{gr} values ($\sim 30^\circ\text{C}$) following storage at a higher subcrystallization relative humidity (33%) compared with powder with nonhydrolyzed NC (T_{gr} value of $\sim 40^\circ\text{C}$), an effect that reflects more extensive plasticization of powder matrices by moisture. Results support that sodium caseinate-lactose interactions were weak but that relaxation behavior, as determined by the susceptibility of powder to sticking, was affected by hydrolysis of sodium caseinate.

Key words: sodium caseinate, hydrolysis, glass transition, stickiness

INTRODUCTION

Sodium caseinate is extensively used in a wide range of food formulations because of its nutritional and functional attributes. Hydrolysis of proteins alters functional characteristics such as solubility, viscosity, emulsion and foam formation, stability, and organoleptic qualities. Enzymatic hydrolysis of sodium caseinate developed under mild conditions of pH (6–8) and temperature (40–60°C) may lead to the development of biologically active nutritional components for use as dairy ingredients.

The peptides produced by hydrolysis have lower molecular mass and less secondary structure than intact proteins (Flanagan and FitzGerald, 2002). Control of the molecular size of resultant peptides constitutes an essential step in the development of protein hydrolysates for dietary use. Extensively hydrolyzed proteins also show reduced immunological reactivities and can be used in formulas for hyperallergic infants and in the nutritional management of individuals who cannot digest whole or intact protein. The high biological value peptides generated by hydrolysis are readily absorbed, are attractive as a source of nitrogen in sports nutrition, and provide a general protein supplement in a wide variety of diets.

Such nutritional components can be incorporated as ingredients in food systems, which may be produced in powdered form for long shelf life. It is of interest to study the physical properties of such food systems to optimize their manufacture and storage stability.

It is well established that physical changes in food powders are often related to the glass transition (T_g) phenomenon (Chuy and Labuza, 1994; Roos, 1995). Below T_g , a powder exists in a pseudo-equilibrium condition and, although thermodynamically metastable, it can be stored for extended periods without significant physical change. Temperatures at and above T_g result in an exponential increase in molecular mobility, and subsequent decreases in surface particle viscosity of the particle surface result in a material softening and the onset of sticking (Roos, 1995; Foster et al., 2005). The presence of moisture accelerates physical destabilization of powders because of its profound effect in low-

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ering T_g . Low molecular weight sugars, most notably lactose, are the main sources of ingredient instability in conventional spray-dried milk powders.

Previous work has provided some clarification on the effects of milk protein on the physical behavior of lactose in spray-dried powders (Hogan and O'Callaghan, 2010). Powders with higher protein:lactose ratios were less susceptible to sticking. It was shown that stickiness was related both to the T_g temperature and to the temperature increment by which T_g must be exceeded before sticking occurred ($T - T_g$). Composition affects T_g and $T - T_g$ in different ways, suggesting that ingredient interactions in multi-component systems affect powder material behavior in a complex manner.

Netto et al. (1998) reported that the T_g of protein hydrolysates is dependent on the average molecular weight and suggested that such hydrolysates may be as important as sugars in lowering the overall T_g of food powders. Hydrolysate powders also tend to have higher hygroscopicity and thermoplasticity and poorer stability compared with powders containing intact proteins. Notwithstanding previous work, little information can be found on the influence of casein hydrolysates on the stickiness properties of milk-based powders.

The objectives of this study were to characterize the moisture relationships, physical characteristics, and stickiness behavior of powders prepared from intact or hydrolyzed sodium caseinate and lactose, to gain better insight into the relationships between ingredient types, and their effects on drying, state transitions, and relaxation behavior of powders. Such knowledge is necessary for the development of processing and spray-drying control systems that help minimize downtime due to powder blockages and make more efficient the production, storage, and transport of dairy powders.

MATERIALS AND METHODS

Materials

Sodium caseinate (87% protein, wt/wt) was obtained from Kerry Ingredients (Listowel, Co. Kerry, Ireland). Edible lactose (Lactopure, 94.2% lactose) was obtained from DOMO (Borculo, the Netherlands). Sodium caseinate hydrolysates (8.3 and 15% DH, where **DH** = degree of hydrolysis, the number of peptide bonds cleaved enzymatically and expressed as a percentage of the original number of peptide bonds) were made in-house (Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland) by an enzymatic process, using a proprietary method.

Preparation of Spray-Dried Powders

Three powders containing sodium caseinate (intact or hydrolyzed) and lactose, with a protein:lactose ratio of 0.5, were produced at Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland from 10-kg dispersions containing 11% (wt/wt) protein and 22% (wt/wt) lactose. Protein powders were reconstituted in reverse osmosis grade water at 60°C with shearing for 20 min at 6,000 rpm using a Silverson mixer (Silverson Machines Ltd., Chesham, UK). Lactose was added with constant mixing for 10 min and the temperature was brought to 70°C to maintain complete solubilization. Samples were subsequently spray dried in a pilot-scale drier (model Lab 3 Anhydro drier; APV Anhydro AS, Søborg, Copenhagen, Denmark) with pressure nozzle atomization, inlet and outlet temperatures of 185°C and 85°C, respectively, and final drying in an external fluidized bed. Powders were produced singly, packed in plastic-lined, paper bags, and stored at 15°C and ambient relative humidity [water activity (a_w) of approximately 0.8] before analyses. All analyses were carried out within 3 mo of manufacture. Sodium caseinate-lactose (**NC/L**) powders were designated **DH 0** (nonhydrolyzed), **DH 8**, and **DH 15** to reflect the degree of hydrolysis of the caseinate fractions.

Powder Characterization

Chemical Analyses. Free moisture content was determined by weight loss following overnight drying of 2 g of powder at 102°C. Protein ($N \times 6.38$) was determined by macro-Kjeldahl method (IDF, 2001). Ash content was determined after overnight incineration at 550°C. Lactose content was determined by difference and lactose was assumed to be in the amorphous state. Chemical analysis of powders was carried out in duplicate, immediately after manufacture, and the results are shown Table 1.

Molecular Mass Distribution of Protein Powders. Gel permeation HPLC was performed on a Spherogel-TSK G2000SW gel permeation column (600 × 7.5 mm i.d.; Tosoh Bioscience GmbH, Stuttgart, Germany) and preceding guard column, fitted to a Waters HPLC system (Waters Corp., Milford, MA). The mobile phase consisted of 30% (vol/vol) acetonitrile containing 0.1% (vol/vol) TFA. Samples were diluted in mobile phase to 0.25% (wt/vol) protein, filtered through 0.2- μ m syringe filters (Whatman, Maidstone, UK), and 20 μ L was applied to the column at a flow rate of 0.5 mL/min. Chromatography was carried out at room temperature. A calibration curve was prepared using the average retention volume of protein and peptide standards (Smyth and FitzGerald, 1998). The calibration standards used

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