



## Technological aspects of lactose-hydrolyzed milk powder



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### ARTICLE INFO

#### Keywords:

Spray drying  
 Hydrolyzed lactose milk powder  
 Microstructure  
 Glass transition

### ABSTRACT

Few reports describe the effect of lactose hydrolysis on the properties of milk powder during production and storage. Hence, the aim of this study was to evaluate the effects of five different levels of enzymatic lactose hydrolysis during the production and storage of milk powder. As the lactose hydrolysis rate increased, adhesion to the drying chamber also increased, due to higher levels of particle agglomeration. Additionally, more brown powder was obtained when the lactose hydrolysis rate was increased, which in turn negatively affected rehydration ability. Using Raman spectroscopy, crystallization of the lactose residues in various samples was assessed over 6 weeks of accelerated aging at a room temperature environment with 75.5% of air moisture. Products with 25% or greater lactose hydrolysis showed no signs of crystallization, in contrast to the non-hydrolyzed sample.

### 1. Introduction

Lactose maldigestion in human adults can result in gastrointestinal discomfort after consuming lactose-containing products (a condition known as lactose intolerance) which is why many people avoid dairy products. The important nutritional value of dairy products has therefore led to the production of low-lactose or lactose-free products. (Bailey et al., 2013; Rong et al., 2011; Troise et al., 2016). In Brazil, the lactase persistence allele, LCT-13910T, was found in about 43% of both white and “pardo” (mixed ethnicity) Brazilians and 20% of black Brazilians, but was absent among all Brazilians of Japanese descent studied (Mattar et al., 2009). Lactose-free and low-lactose products such as yoghurt, UHT milk and beverages, as well as cheese, are well-established on the market in many countries (Moreira et al., 2017; Milkovska-Stamenova & Hoffmann, 2017; Ruiz-Matute et al., 2012; Adhikari, Dooley, Chambers, & Bhumiratana, 2010), but the production of lactose-free milk powder remains an under-studied area.

Spray drying is widely used in the food industry and is applied mainly for food preservation, constituent and emulsion stabilizing, and microencapsulation of microorganisms, enzymes and molecules (Janiszewska-Turak, 2017; Sánchez, Cuvelier, & Turchiuli, 2016; Noello, Carvalho, Silva, & Hubinger, 2016; Zheng, Fu, Huang,

Jeantet, & Chen, 2016; Pinto et al., 2015, chap. 5; Estevinho, Damas, Martins, & Rocha, 2014).

The glassy state formation and the degree of crystallinity in dried dairy powders are essential steps for controlling properties such as stickiness, caking, porosity, solubility and dissolution rates, flowability, and bioavailability (Carpin et al., 2016; Islam & Langrish, 2010; Langrish, 2008; Roos, 2010; Schmitz-Schug, Gianfrancesco, Kulozik, & Foerst, 2013). The relationships among product composition, glass transition temperature (related to the glassy state formation and the degree of crystallinity), spray-drying settings, and powder properties play a key role in the industrial production of foods and have been widely researched. (Carpin et al., 2016; Norwood et al., 2017; Sadek et al., 2016; Schuck et al., 2009; Schuck et al., 2016; Schuck, le Floch-Fouere, & Jeantet, 2013; Zhu, Méjean, Blanchard, Jeantet, & Schuck, 2011). In the case of hydrolyzed milk powders, spray drying is very difficult because most of the powder sticks to the insides of the dryer even at very low inlet/outlet air temperatures (Shrestha, Howes, Adhikari, & Bhandari, 2007). During the production and storage of low-lactose powdered milk (LLPM), technological problems may occur, including unwanted adhesion of agglomerated particles to the equipment, caking, and darkening. These issues can lead to low production yield, operational problems and difficulty in powder handling (Fernández,

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Schebor, & Chirife, 2003). According to Schuck et al. (2015), lactose-hydrolyzed milk has a greater number of molecules (glucose and galactose, as opposed to lactose) in its amorphous state during drying and the product becomes highly hygroscopic, which makes processing productivity challenging due to chamber clogging and powder conservation.

Therefore, this study's objective was to elucidate the technological and storage characteristics of various LLPMS produced by spray-drying after concentration, using enzymatic lactose hydrolysis. This work contributes to the development of technology for hydrolyzed milk powder manufacturing.

## 2. Materials and methods

### 2.1. Obtaining powder products

Concentrated whole milk was obtained from the reconstitution of milk powder in water at 25 °C and contained approximately 40% total solids. The trials were performed using whole milk powder from the Brazilian dairy producer Itambé. This whole milk powder was produced without agglomeration and without lecithin addition. The composition of the powder was solely milk. In order to hydrolyze the milk, 0.2% w/w of Lactomax Super enzyme by Prozyn® (São Paulo, Brazil) was added, and samples were incubated at 34 °C ± 1 °C. The analytical report of the enzyme applied in the experiment indicated enzymatic activity of 60,000 ONPGU·g<sup>-1</sup> (*o*-nitrophenyl- $\beta$ -D-galactopyranoside Units·g<sup>-1</sup>) and density between 1.1 and 1.3 g·mL<sup>-1</sup>.

To quantify the degree of lactose hydrolysis, samples were collected before the addition of lactase, then collected every 10 min afterwards until the desired hydrolysis level was achieved. Prior to the cryoscopic lactose hydrolysis degree evaluation, samples of concentrated milk were diluted to 10% w/v total solids using distilled water to achieve the characteristics of fluid milk. The degree of hydrolysis was monitored by measuring the freezing point depression in each diluted sample using an RTI MK540 Flex II Cryoscope (São Paulo, Brazil). The same approach that was applied by Rodrigues Júnior et al. (2016). The enzyme was inactivated by heating the milk to between 90 and 95 °C for 60 s. The percentage of lactose hydrolysis was determined after the heat-induced inactivation of the enzyme.

Five treatments of lactose hydrolysis were obtained: non-hydrolyzed milk concentrate (0H) and 25% (25H), 50% (50H), 75% (75H) and > 99% (99H) hydrolyzed milk concentrates. Four replicates (n = 4) of each condition were performed, totaling 20 experiments.

All products were dried in a Spray Dryer MSD 1.0 (using a flow rate of 1.0 ± 0.1 kg·h<sup>-1</sup>, with compressed air flow rate of 30 L·min<sup>-1</sup> and blower air flow rate of 2.0 m<sup>3</sup>·min<sup>-1</sup> through a pressure nozzle (1 mm diameter) atomization system LabMaq (Ribeirão Preto, Brazil). The drying parameters were set to an inlet air temperature of 170 °C ± 5 °C and an outlet air temperature of 85 °C ± 5 °C. At the end of the drying process, the powder from each sample was collected from the equipment, vacuum-packed, protected from light, and stored in a temperature-controlled location (25 °C).

Photo documentation of the drying equipment was gathered with a Motorola Moto G 13 Megapixel model camera at the end of each sample preparation.

### 2.2. Physical-chemical analyses

The milk powder samples obtained were analyzed for moisture, protein, lipid, ash, and water activity according to Zenebon, Pascuet, and Tiglea (2008). Moisture was determined using a gravimetric oven technique at 105 °C. Total protein was determined using the micro-Kjeldahl method; ash content was determined using a gravimetric method. Weight loss of the material subjected to incineration in a muffle furnace at 550 °C was recorded, and the lipid content was determined using the Gerber method. Powders were analyzed for water

activity ( $a_w$ ) using a Decagon3TE Aqualab instrument (Pullman, USA). The lactose amounts in the powders were determined using the enzymatic method (McCleary & Charnock, 2004) using the kit K-Lacgar (Megaenzymes, USA).

### 2.3. Particle size distribution of the rehydrated powders by laser diffraction

The size distribution of the powder particles during rehydration was obtained using a Beckman Coulter LS 13 320 laser-diffraction analyzer (Beckman Coulter, Miami, FL, USA) coupled to an aqueous liquid module (Beckman Coulter, Miami, FL, USA).

A sufficient amount of sample to generate turbidity readings was added to the liquid analysis module tank, which contained water at room temperature. Samples were added slowly to prevent the formation of agglomerates. The rehydration process was monitored every 3 min for 15 min. During this time, the samples remained under recirculation into the equipment. At the end of the data collection, stable particle size distributions were obtained. Data were collected in the particle size region of 0.04 to 2000  $\mu$ m, with an acquisition time of 100 s. The results were obtained using 1.332 as the refractive index for the dispersing medium (water) and 1.57 for particles. This method, as described by Mimouni, Deeth, Whittaker, Gidley, and Bhandari (2009), has been used to measure the size of dairy components in suspension in individually fat globules, lactose crystals, casein micelles, or composite media such as skim milk or whole milk. Following the administration of this method, the results were represented as the volume (%) occupied by the particles in relation to their size.

### 2.4. Scanning electron microscopy

The morphology and agglomeration characteristics of sample particles were evaluated without prior preparation using scanning electron microscopy (Hitachi TM 3000, Hitachi Ltd., Tokyo, Japan). A magnification of 400 × was used to characterize the samples.

### 2.5. Obtaining Raman spectra during accelerated aging

Approximately 5 g of each of the five LLPMS treatments were stored in a vacuum desiccator in a saturated NaCl solution to obtain a relative humidity of 75.5% at approximately 23 °C. Raman spectra of these samples were obtained weekly for 6 consecutive weeks. The Raman spectra of all LLPMS samples represented in this study were obtained using a Bruker FT-Raman RFS 100 spectrometer equipped with a liquid nitrogen-cooled Ge detector and a Nd:YAG laser. Spectra were collected using a 100 mW laser beam with near-infrared excitation at 1064 nm, and the scattered radiation was collected at 180°. For all spectra, good signal/noise ratios were obtained by performing an average of 512 scans, which were collected with a spectral resolution of 4 cm<sup>-1</sup> in the region from 3500 cm<sup>-1</sup> to 50 cm<sup>-1</sup>. OPUS platform 6.0 was used for the acquisition of Raman spectra. All spectra were obtained in duplicate to ensure that the intensity and spectral regions of the respective vibrational modes were reproducible.

### 2.6. Chemometrics

To perform the exploratory analysis, the Raman spectra were evaluated using Matlab software version 7.10.0 (R2010a). A potential complication in the interpretation of Raman spectra is the contribution and effect of factors such as particle size and morphological differences. These effects are dependent on experimental conditions and must be removed before the use of chemometric tools. To address these effects, Raman spectra were preprocessed using a weighted least-squares baseline, Raman intensities were normalized to a unit vector length and mean centering, and principal components analysis was employed. The number of main components was chosen according to the explained variance.

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