LWT - Food Science and Technology 73 (2016) 675–682

Contents lists available at ScienceDirect

LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt

Storage stability of freeze-dried, spray-dried and drum-dried skim milk powders evaluated by available lysine

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ARTICLE INFO

Article history:
Received 3 February 2016
Received in revised form
17 May 2016
Accepted 4 July 2016
Available online 5 July 2016

Keywords:
Skim milk powder
Available lysine
Storage stability
Drying technology
Dye-binding methods

ABSTRACT

Formation of Maillard products and the influencing factors, which are of crucial importance for both manufacturers and consumers, are still not fully understood. Thus in this study available lysine was used as a marker to monitor the extent of Maillard reactions in freeze-dried, spray-dried and drum-dried skim milk powders during 200 days of storage at highly controlled atmospheres. Storage variables included two temperatures (20 °C, 30 °C) and two relative humidities (33%, 52%). The available lysine in five replicates was quantified at pre-determined intervals by a dye-binding method using Acid-orange 12, validated in our previous work. Findings of this study show that temperature and relative humidity during storage have a profound influence on the rate of available lysine loss. Choice of the drying technology as the other investigated variable also had a significant impact. The drying process least affected the available lysine content in freeze-dried powders, followed by spray-dried and drum-dried powders. Storage at 52% relative humidity and 30 °C for 200 days led to a 39.2–45.9% decrease in the available lysine content, regardless of the drying of skim milk powder, while the powders stored at 33% relative humidity and 20 °C did not show a significant loss during the same period of time.

1. Introduction

During recent years the application of skim milk powder (SMP) in the food industry has increased due to its long shelf-life and various functional properties. Based on a report by the Food and Agriculture Organization of the United Nations, the annual production of skim milk powder reached 1.9 million tons in 2013, which was a 3.3% increase compared to the previous year (Griffin, 2013). Considering its composition, which is mainly proteins and carbohydrates and bearing in mind that it is usually stored for a significant period of time before being used, the possibility of skim milk powder undergoing Maillard reactions during storage cannot be ruled out. These controversial and complex chemical reactions may lead to the formation of pro-inflammatory molecules, specifically advanced glycation end products (AGEs), under certain circumstances (Bengmark, 2007; Goldberg et al., 2004; Henle, 2005; Nguyen, van der Fels-Klerx, & van Boekel, 2013; Siciliano, Mazzeo, Arena, Renzone, & Scaloni, 2015). Therefore, understanding and monitoring this reaction is of vital importance. This can be achieved by means of optimizing the parameters of processing as well as controlling the conditions during transport and storage.

Available lysine is one of the markers that can be employed to understand the early stages of the Maillard reaction and its content is decreased with respect to the increased reaction rate. This reduction in the amount of available lysine is considered to be the first step of the Maillard reaction and is due to binding with lactose, the main carbohydrate in milk (Contreras-Calderón, Guerra-Hernández, & García-Villanova, 2009; El & Kavas, 1997; Hurrell, Finot, & Ford, 1983; Malec, Pereyra Gonzales, Naranjo, & Vigo, 2002; Mehta & Deeth, 2016; Ramírez-Jiménez, García-Villanova, & Guerra-Hernández, 2004; Pereyra Gonzales, Naranjo, Leiva, & Malec, 2010; Rutherfurd & Moughan, 2008; Schmitz, Gianfrancesco, Kulozik, & Foerst, 2011;). In order to quantify the available lysine, different approaches can be adopted depending on the type of sample material. Regarding skim milk powder, a convenient and reliable method is the dye-binding method using Acid-orange 12 which was validated in our previous work for casein (the main protein in milk), bovine serum albumin and a wide range of skim milk powders (Aalaei, Rayner, Tareke, & Sjöholm, 2016).

There have been only a few studies done on the storage of skim milk powder at mild temperatures (37 °C, 50 °C, 60 °C), (Pereyra Gonzales et al., 2010) and in those studies the sample was always

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http://dx.doi.org/10.1016/j.lwt.2016.07.011
0023-6438/

commercial. In this study, the skim milk was obtained from a known source (local dairy) and extra effort had been put into manufacturing the different powders in the pilot plant where all the processing parameters were controlled. In order to obtain a clear picture of the impact of processing on the progress of the Maillard reaction, three types of skim milk powders produced by a pilot-scale freeze-dryer, spray-dryer and drum-dryer were investigated, and their available lysine contents were quantified. Subsequently, the storage effect was studied by storing the above-mentioned skim milk powders at various temperatures and relative humidities in highly controlled atmospheres for 200 days. The storage variables included two temperatures (20 °C, 30 °C) and two relative humidities (33%, 52%) and available lysine was measured at intervals during 200 days.

Therefore, the aim of this project was to understand the effects of different drying technologies and various storage conditions on the level of remaining available lysine in order to determine the extent of early Maillard reactions. The hypothesis was that the impacts of the processing and further handling, including the storage and transportation, are more profound than previously thought and mostly underestimated. In other words, this study aims to increase the awareness that the Maillard reaction does not necessarily need extreme conditions to occur and it can take place at temperatures and humidities that are common in many places. The storage conditions investigated in this study can easily occur when consumers store an opened package at room temperature and consume the product intermittently over a long time. This was the criterion behind the selection of storage variables in the way that they closely resembled typical climate conditions during transport and storage, particularly at home.

2. Materials and methods

2.1. Chemicals and instruments

In order to analyze available lysine, the following chemicals were used: sodium acetate anhydrous reagent grade (CAS: 127-09-3) was purchased from Scharlau (Sentmenat, Spain); Acid Orange 12 (Crocein Orange G) (CAS: 1934-20-9 and MW = 350.32) was supplied by Tokyo Chemical Industry (Tokyo, Japan); potassium dihydrogen phosphate for analysis (CAS: 7778-77-0) and magnesium nitrate hexahydrate for analysis (CAS: 13446-18-9) were obtained from Merck (Darmstadt, Germany); propionic anhydride 99% (CAS: 123-62-6) was acquired from ACROS Organics (Geel, Belgium); magnesium chloride hexahydrate (CAS: 7791-18-6) was purchased from VWR International (Leuven, Belgium); oxalic acid dehydrate >99.5% (CAS: 6153-56-6) and casein sodium salt from bovine milk (CAS: 9005-46-3) were supplied by Sigma-Aldrich (Steinheim, Germany), while albumin, bovine 96–99% (CAS: 9048-46-8) was obtained from Sigma-Aldrich (St. Louis, USA).

The instruments used included a 3005 orbital lab shaker type from Gesellschaft für Labortechnik, an Aqualab Series 3 water activity meter from Decagon Devices, a wireless Hygroclip with temperature and air humidity sensors, a Mätman 3 from Eltex AB of Sweden, an Optima LE-80 k ultracentrifuge from Beckman Coulter and a Varian Cary 50 UV–Vis spectrophotometer from Agilent Technologies.

2.2. Drying experiments

2.2.1. Freeze-drying

Freeze-drying was carried out using a pilot-scale freeze-dryer (Labconco, Missouri, USA). Skim milk (0.1% fat) was placed into aluminum trays (1 cm thickness). The samples were then put into the freezer at −20 °C for 24 h before freeze-drying. The freeze-drying temperature was −20 °C in the beginning and reached 20 °C with a 1 °C/h increase. The condenser had a temperature of −50 °C and the vacuum pressure was 0.02 mbar. The freeze-drying duration was seven days and the samples were immediately ground, vacuum-packed and placed into the freezer at −20 °C until further analysis.

2.2.2. Drum-drying

The process was carried out using a Goudsche Machinefabriek drum-dryer (Waddinxveen, Netherlands). The surface temperature of the drum was 115 °C on average and the drying was completed in 40 s. The obtained flakes were then ground, vacuum packed and placed into the freezer at −20 °C until further analysis.

2.2.3. Spray-drying

A lab-scale Büchi mini spray-dryer B-290 (Flawil, Switzerland) was utilized for spray-drying. The inlet temperature of 150 °C and outlet temperature of 85 °C was applied. The flow rate of the feed was 0.6 L/h and the pre-heated air had a flow rate of 540 L/h. The powder was immediately collected, vacuum-packed and put into the freezer at −20 °C until further analysis.

2.3. Storage of the samples

Storage of the samples was carried out in desiccators at two temperatures (20 °C and 30 °C) and two relative humidities (33% and 52%). In order to achieve 33% relative humidity inside the desiccator, 200 g magnesium chloride was mixed with 25 ml distilled water and stirred until a homogeneous solution was obtained, while 52% relative humidity was obtained by dissolving 200 g magnesium nitrate in 30 ml distilled water (Motarjemi, 1988). The solutions were placed in the desiccators and allowed to equilibrate for one week before adding the samples. This is an important step often neglected in other studies. The temperature and relative humidity in the desiccators were monitored once prior to putting the samples in the desiccators, and regularly during storage, with a wireless Hygroclip.

Several glass Petri dishes each containing 7 g milk powder were placed in the desiccator, which was then placed in an incubator to reach the desired storage temperature. The temperature of the incubators was controlled regularly with the thermometer. At each pre-determined sampling point one plate was taken out and analyzed. Water activity and water content of the samples were determined before analysis of available lysine.

2.4. Quantification of available lysine

The dye-binding method with Acid-orange 12 used for the determinations of available lysine in the samples is fully described earlier (Aalaei et al., 2016) and is based on work presented by Hurrell, Lerman & Carpenter, with slight modifications (Hurrell, Lerman, & Carpenter, 1979). As a brief explanation, 200 mg of the sample was mixed with 2 ml of sodium acetate solution for 20 min. Then, 0.2 ml propionic anhydride was added to half of the flasks and continued mixing for another 20 min. Subsequently, 40 ml of the dye solution (1.36 mg/ml) was added and mixed for 1 h. The analysis continued with the centrifugation step (5000 rpm, 10 min) and measurement of the absorbance of the supernatant with the spectrophotometer at 475 nm and concentration of the available lysine was calculated using the equation from the calibration curve $y = 47.37\ x + 0.01$ ($R^2 = 0.9999$). The analysis was always carried out with five replicates. A significance test was performed on the results by the student t-test for a significance level of 95%.