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Analytical Methods

Development and in house validation of a new thermogravimetric method for water content analysis in soft brown sugar

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ABSTRACT

Recently the use of brown sugar has increased due to its nutritional characteristics, thus requiring a more rigid quality control. The development of a method for water content analysis in soft brown sugar is carried out for the first time by TG/DTA with application of different statistical tests. The results of the optimization study suggest that heating rates of 5 °C min^{-1} and an alumina sample holder improve the efficiency of the drying process. The validation study showed that thermo gravimetry presents good accuracy and precision for water content analysis in soft brown sugar samples. This technique offers advantages over other analytical methods as it does not use toxic and costly reagents or solvents, it does not need any sample preparation, and it allows the identification of the temperature at which water is completely eliminated in relation to other volatile degradation products. This is an important advantage over the official method (loss on drying).

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1. Introduction

Soft brown sugar is a product derived from sugar cane, usually produced in an artisanal way by small agribusinesses through the cooling of highly concentrated cane syrup. Due to the type of processing applied, in soft brown sugar most of the components of sugar cane are preserved. It is composed of sugars of high biological value, like sucrose, glucose and fructose and other components such as water, proteins, insoluble solids, and a group of important minerals (K, Ca, P, Mg, Na, Fe, Mn, Zn and Cu). So it is considered a food of high nutritional value (Guerra & Mujica, 2010; Jesus, 2010; Lopes & Borges, 1998; Tomasseto, Lima, & Shikida, 2009). Its nutritional properties and type of processing has raised the interest of (i) consumers concerned in adopting a healthy way of eating by consuming products that have not undergone a rigorous processing or have not received the addition of chemicals and (ii) by food and beverage industries aiming to meet the needs of these consumers (Verruma-Bernardi, Borges, Lopes, Della-Modesta, & Ceccato-Antonini, 2007). Thus, recently an increase in the consumption of brown sugar has been observed, especially in Brazil, as a substitute for white sugar and as an ingredient in the formulation of industrialized foods and beverages (Costa & Jongen, 2006).

Being a natural product, brown sugar presents a quite variable physicochemical composition and does not support long storage, mainly due to the presence of impurities and moisture originating from the raw material (sugar cane) and practices undertaken during the production (Jesus, 2010; Lopes & Borges, 1998; Verruma-Bernardi et al., 2007). This causes its marketing to more distant markets to be difficult. To ensure its food security and meet the quality requirements imposed by the food industry, it should therefore be subjected to a rigorous quality control (Guerra & Mujica, 2010; Jesus, 2010).

Among the physicochemical parameters adopted for the quality control of this food, moisture is one of the most critical, since the water content directly influences the growth of microorganisms and the texture, thus affecting stability, shelf life and commercial value (Isengard, 2001; Jesus, 2010). The Codex Alimentarius (1999) sets a maximum limit for moisture of 4.5% for soft brown sugar. Although the Brazilian legislation has not set a maximum value for this parameter, several authors have recommended that the moisture content should not exceed 2.4% (Lopes & Borges, 1998; Tomasseto et al., 2009).

The official method for determination of moisture in brown sugar is loss on drying performed by oven drying with a previously established temperature and time (105 °C and 3 h) (IAL, 2008; ICUMSA, 2007; Nielsen, 1994). Although this methodology is simple and cheap, it does not allow distinguishing between the end of the elimination of water and the beginning of the degradation

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reactions of foods rich in sugars such as brown sugar, thus causing systematic errors in moisture analysis (Nielsen, 1994; Wrolstad, 2012). In the literature a tendency can be observed however to propose alternative analytical methods that are more accurate and reliable than oven drying for water content analysis in foods with high carbohydrates contents, such as Karl Fischer titration and infrared radiation drying (Acquistucci, Bucci, Magri, & Magri, 1991; Felsner, 2001; Heinze & Isengard, 2001; Isengard & Heinze, 2003; Isengard & Präger, 2003; Schöffski, 2001; Tomassetti, Campanella, & Aureli, 1989).

Simultaneous 'thermogravimetry – differential thermal analysis' (TG/DTA) is a technique that allows determination of the loss of mass and thermal response (temperature difference or heat flow range) of any homogeneous material subjected to a heating rate under controlled conditions, as well as the identification of events related to dehydration and degradation reactions (Haines, 2002). The TG/DTA was described as an accurate method to determine the water content in various food products, such as starch, flour, roasted coffee, milk powder (Tomassetti et al., 1989), wheat flour (Acquistucci et al., 1991) and seafood (Silva, Silva, de Andrade, Veloso, & Santos, 2008), but has been applied less frequently to foods containing high sugar contents (Felsner, 2001).

These facts have stimulated the development of a new analytical method for the drying of soft brown sugar using thermogravimetry due to its simplicity and ease of implementation. The aim of this study was thus to develop, in an unprecedented way, a thermogravimetric methodology for water content analysis in this food by performing optimization and validation studies applying different statistical techniques. To our knowledge no previous effort has been made to optimize and validate this technique for water content analysis in soft brown sugar.

2. Materials and methods

2.1. Samples

In this study twelve soft brown sugar samples, (also known as muscovado brown sugar) obtained by artisanal processing from five different manufacturers were used. The samples 01–11 had a moist and sticky texture, fine grains, strong molasses flavor and dark brown color while the sample 12 presented light brown color and slightly coarse grains. They were stored in polyethylene containers and kept in a cool and dry environment until the realization of the analyses.

2.2. Experimental methods

2.2.1. Simultaneous thermogravimetry and differential thermal analysis (TG/DTA)

A simultaneous TG/DTA instrument (6000 series Exstar model of Seiko) was used to obtain TG/DTA curves of soft brown sugar samples under a constant dynamic atmosphere of synthetic air (50.0 mLmin^{-1}) from room temperature until 250 °C. Alumina or platinum sample holders of 50 μL for both the sample and the reference were used with heating rates of 5 °C min^{-1} or 10 °C min^{-1}. Each sample had a mass of approximately 5.0 mg. The samples were weighed as quickly as possible to minimize the moisture uptake or release from the sample. Therefore, the scans were initiated without awaiting thermal equilibration. Approximately 5.0 mg of α-alumina (α-Al_2O_3) powder (Sigma–Aldrich, Germany) was used as an inert reference.

2.2.2. Karl Fischer titration

The water content was measured by a volumetric Karl Fischer titration with titrator Q349-1 from Quimis using Karl Fischer reagent without pyridine (Biotec, Brazil) and anhydrous methanol (J.T. Baker, USA) according to the method 014/IV of the Physicochemical Methods for the Food Analyses (IAL, 2008). All measurements were carried out in duplicate.

2.2.3. Sugar moisture by loss on drying

The moisture content was determined by a loss on drying technique according to the GS 2/1/3/9-15 Method, from ICUMSA (2007). All measurements were carried out in duplicate.

2.2.4. Other physicochemical analyses

To better understand thermal degradation of soft brown sugar samples other physicochemical analyses such as reducing sugars and conduct metric ashes were carried out according to methods from ICUMSA (2007), and acidity according to method 174/IV of the Physicochemical Methods for the Food Analyses (IAL, 2008). All measurements were carried out in duplicate.

2.3. Optimization study

To optimize the thermogravimetric method a 2^2 factorial design was applied investigating the influence of heating rate and sample holder material on the soft brown sugar samples' water contents. Duplicate TG/DTA curves were obtained in random order for all combinations of the factors in each level.

The factor effects were calculated by:

$$E_f = (\bar{R}_+) - (\bar{R}_-)$$

where (R^+) and (R^-) are the means at the (+) and (−) levels of the factors.

The factor effects on water content values were tested for statistical significance by calculating standard errors and 95% confidence intervals. All the statistical data analyses were carried out using the software Minitab for Windows version 16.2.2 (MINITAB, 2010, *Minitab Incorporation, USA*, 2010).

2.4. Validation studies

For the validation study, the two parameters precision and accuracy were evaluated according to AOAC guidelines (Wernimont, 1985). Both parameters were determined by comparing the results of the thermogravimetric method developed with the values of Karl Fischer titration (reference method). To evaluate the accuracy of thermogravimetry, a paired *t*-test for the mean differences of water contents obtained for the analytical methods was applied (Wernimont, 1985). To evaluate the developed method's precision, the weighted variances (Sp^2) of each of the analytical methods were compared by applying an appropriate *F*-test and by calculating confidence intervals for the ratio between the variances and for standard deviations weighted (Sp). The precision and accuracy of the official method (loss on drying) was also evaluated by applying the same statistical techniques. All statistical analyses were performed at 95% confidence level by using the software Minitab for Windows version 16.2.2 (MINITAB, 2010, *Minitab Incorporation, USA*, 2010).

3. Results and discussion

3.1. Thermogravimetric drying profile

To evaluate thermogravimetric drying profile of a brown sugar sample, TG/DTA curves in the optimized conditions described in item 3.2 were obtained. The process of drying is characteristic of foods with high sugar contents as illustrated in Fig. 1a. In the TG curve, between 25 °C and 250 °C three very subtle inflection points were observed that indicate changes in velocity of mass loss during

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