Journal of Cereal Science 71 (2016) 246–249

Contents lists available at ScienceDirect

Journal of Cereal Science

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

Research note

Rapid prediction of moisture content of quinoa (*Chenopodium quinoa* Willd.) flour by Fourier transform infrared (FTIR) spectroscopy

Adrián González-Muñoz [a, b], Bernabé Montero [a], Javier Enrione [b], Silvia Matiacevich [a, *]

[a] Food Properties Research Group (INPROAL), Department of Food Science and Technology, Technological Faculty, Universidad de Santiago de Chile, Chile
[b] Biopolymer Research and Engineering Lab (BiopREL), Nutrition and Dietetics School, Faculty of Medicine, Universidad de los Andes, Chile

ARTICLE INFO

Article history:
Received 19 May 2016
Received in revised form
9 September 2016
Accepted 13 September 2016
Available online 14 September 2016

Keywords:
Quinoa ecotypes
Moisture measurement
FTIR
Prediction

ABSTRACT

Moisture content determination on quinoa flour is actually performed by gravimetric analysis, which is time, energy consuming and sample destructive. An emerging technique to measure moisture in an innovative way avoiding those problems is the Fourier transform infrared (FTIR) spectroscopy. The aim of this study is to obtain a trustable and validated model to predict moisture content of quinoa flour using FTIR. To perform the moisture measurements in five quinoa ecotypes, a gravimetric data and area under the curve obtained by FTIR, considering the −OH peak associated to water (3200 cm^{-1}), were compared at five different relative humidities (0, 33, 58, 75 and 100%). A good correlation between gravimetric measurements and FTIR area were observed ($R^2 = 0.8729$) and no differences were observed between quinoa ecotypes. A cross validation technique to predict moisture considering experimental and predicted data by area under de curve by FTIR was performed obtaining a general equation ($y = 35.73x + 46.04$) with a high repetitively and good prediction (100%) of the tested models.

1. Introduction

Quinoa (*Chenopodium quinoa* Willd.) is a dicotyledonous pseudocereal, native to the Andean regions of South America (Bhargava et al., 2006). Due to its high nutritional quality, quinoa is generally regarded as an extremely healthy food (Valencia-Chamorro, 2003; Bhargava et al., 2006) making it ideal as a supplement for utilization in the food industry. Quinoa flour has been mixed with other flour at different levels (13–60%) (Valencia-Chamorro, 2003) in order to supplement protein deficient flour (Bhargava et al., 2006).

An important parameter used for quality assessment of flours in industry is the moisture content, due to its relevance in microbiological stability, food safety but also in terms of commercial value (Rahman and Labuza, 2007).

Several analytical techniques commonly used to determine the moisture content in foods are based on gravimetric determinations (e.g. at 105 °C for 5–24 h, AOAC, 2000). However, this technique requires long periods of time and high energy consumption. An emerging alternative technique is related to the use of Fourier Transform Infrared Spectroscopy (FTIR), which is a rapid and nondestructive method that can detect functional groups of specific molecules. This method has been used to determine chemical constituents of beans (Holse et al., 2011), wheat bran (Hell et al., 2016) and quinoa grains (Ferreira et al., 2015). In addition, the moisture content of other food products has been analyzed, such as palm crude oil (Che Man and Mirghani, 2000), cocoa powder (Veselá et al., 2007) and high fat products (mayonnaise, peanut butter) (van de Voort et al., 1993). However, literature does not provide information about determining the moisture content in quinoa flour and no statistical model has been proposed for its prediction using this technique. Therefore, the objective of this work is to determine the moisture content of different quinoa ecotypes using FTIR and to compare this data with the AOAC standard methods, in order to build a trustable and validated model for its prediction without the need of gravimetric experimental protocols.

2. Material and methods

Ecotypes from Peru, Bolivia, and Chile (northern, central and southern location) were evaluated. In order to eliminate

Abbreviations: ATR, Attenuated Reflection Unit; d.w.b, dry weight basis; FTIR, Fourier Transform Infrared Spectroscopy; RH, relative humidity.

* Corresponding author. Departamento de Ciencia y Tecnología de los Alimentos, Universidad de Santiago de Chile, Obispo Manuel Umaña 050, Estación Central, 9170201, Santiago de Chile, Chile.
E-mail address: silvia.matiacevich@usach.cl (S. Matiacevich).

http://dx.doi.org/10.1016/j.jcs.2016.09.006
0733-5210/

Fig. 1. Diagram of cross-validation procedure. *N* is the total number of data, *F* is the number of groups and *K* is the quantity of data of each group.

saponins, quinoa grains were washed and dried in an oven at 30 °C for 24 h, resulting in moisture contents lower than 10% dry weight basis (d.w.b). Then, grains were milled (Fagor, ML2006×) to obtain quinoa flour with a particle size lower than 500 μm.

FTIR capsules (~4 mm diameter x 3 mm height) of quinoa flours (~100 mg) were shaped using a press (Perkin Elmer, USA). In order to achieve different moisture contents, the capsules (in triplicate) were put into hermetic desiccators at 20 °C (Incubator FOC225, Velp Sci, Italy) for 10 days with saturated salt solutions in the range of 0–100% (Greenspan, 1977); dried silica gel (RH 0%); MgCl (RH 33%), MgNi (RH 58%), NaCl (RH 75%) and distilled water (RH 100%). Thyme sachets were included for RH at 75% and 100% RH in order to avoid fungal growth. Equilibrium was achieved when variation in weight was not detected by measuring consecutively with an analytical balance (Shimadzu AUX120, USA).

FTIR spectra were recorded on a spectrometer equipped with a Universal Attenuated Reflection Unit (ATR) (Spectrum Two, Perkin Elmer, USA). The capsules were directly placed over ATR crystal at constant pressure. For each spectrum, in absorbance mode, 16 scans from 4000 cm^{-1} to 300 cm^{-1} at a resolution of 4 cm^{-1} were accumulated and averaged. Spectral analysis was performed using the Spectrum Software v.10.04.02. The spectral region between 3000 and 3600 cm^{-1}, corresponding to the –OH stretching vibration was selected for analysis (Barth, 2007). After baseline correction, the intensity of the peak was measured as the area under the curve obtained by integration. No significant variation of sample weights was observed before and after measurements, indicating no variation on moisture content by atmospheric conditions, considering the short time (30s) required for measurements.

For gravimetric measurements, moisture content was performed according to AOAC (2000) in a drying oven with forced air circulation (Wiseven Daihan WOF-105, Korea) at 105 °C until constant weight (30 h). Results were calculated as dry weight basis (d.w.b).

All experiments were done in triplicate.

Statistical differences of the data within groups and between groups were compared by ANOVA and by Student's *t*-test using GraphPad Prism v.4.0 program (GradPad Systems Inc.). Statistical significances were expressed at the $P < 0.05$.

A cross-validation technique was used to evaluate pattern predicted values by using training and testing matrices, in order to obtain a robust and accurate mathematic descriptor as described by Che Man and Mirghani (2000) and Matiacevich et al. (2012). To achieve this, some of the collected samples were removed to become the training set. The total data ($N = 65$) was divided into F (=5) folds randomly. Each group contains N/F (=13) samples. Then,

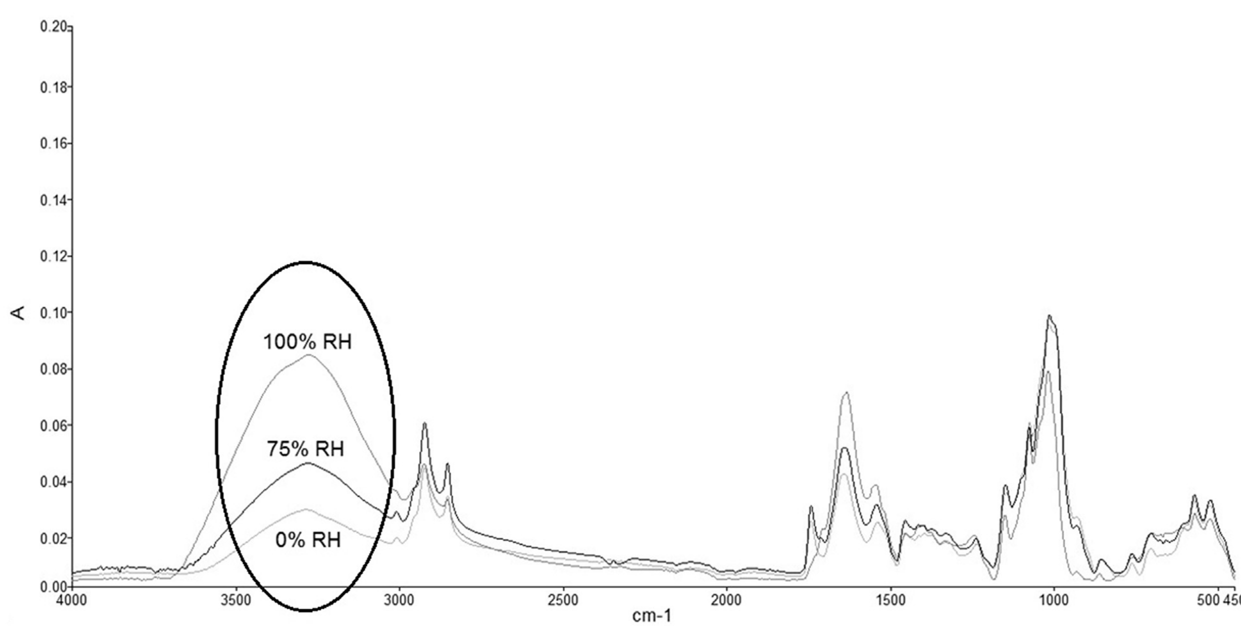

Fig. 2. Quinoa flour spectra by using ATR-FTIR Spectroscopy at different relative humidities, as example at 0, 75 and 100% of relative humidities (RH).