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Isolation and characterization of insoluble date (*Phoenix dactylifera* L.) fibers

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ABSTRACT

A comparative study of water insoluble fiber extraction and characterization was carried out in three potential cultivars of palm dates (*Phoenix dactylifera* L.): *Barhee*, *Sultana* and *Owadi*. Insoluble date fiber was extracted by multistage water extraction of date flesh using microwave (MW) heating followed by freeze-drying, and grinding. High-performance liquid chromatography (HPLC) analysis confirmed the absence of sugars in the fiber after the 6th extraction. The focus of this study was to characterize water insoluble date fiber through various techniques so that it can be used as an important ingredient in food formulation. The particle size of the fiber was measured by dynamic light scattering, and the particle diameters ranged between 700 and 1000 nm. The elemental determinations were performed by inductively coupled plasma optical emission spectrometry (ICP OES) which confirmed that date fibers are rich in potassium, calcium and magnesium (1.5–2.4 g/kg) and low in sodium content. Date fibers showed a high water and oil holding capacity. There were significant differences in tristimulus color values, mineral contents, water and oil holding capacities among date cultivars. These date fibers are good dietary sources with rich in mineral contents and have potential for development of health related foods.

1. Introduction

Palm date (*Phoenix dactylifera* L. Arecaceae) is an important fruit in Middle Eastern countries and it is one of the oldest fruit trees in the world (Marzouk & Kassem, 2011). The fruit is composed of a seed and fleshy pericarp which constitutes between 85 g/100 g and 90 g/100 g of date fruit weight (Hussein, Alhadrami, & Khalil, 1998). Dates are rich in sugar, protein, dietary fiber, minerals, and some vitamins and provide a good source of rapid energy (Al-Hooti, Sidhu, Al-Saqer, & Al-Othman, 2002; Al-Shahib & Marshall, 2003). Recent studies indicated that date fruit contains significant amount of flavonoid glycosides including luteolin, quercetin, apigenin, p-coumaric, ferulic acid and sinapic acids (Abdelhak, Guendez, Eugene, & Panagiotis, 2005; Bilgari, Alkarkhi, & Easa, 2008; Hong, Tomas-Barberan, Kader, & Mitchel, 2006).

The date is mostly consumed as the whole of the sound fruit flesh. Sound date is graded and packaged commercially which provides the highest reward to the producer. The annual world date production in 2010 was about 7.91 million tons-increase of 6.6% from 2009 (FAO, 2011). However, a significant portion of dates are wasted in date producing countries (e.g. 30% loss of total produce in Tunisia) due to its inferior quality, damage, and undersized fruit of unattractive appearance (Besbes, Drira, Blecker, Deroanne, & Attia, 2009). It is also reported that dates are also wasted during the sorting, the storage and the conditioning (Cheikh-Rouhou et al., 2006). The non-use of lesser-quality dates for human food constitutes a real economic loss since it is rich in bioactive compounds which can be extracted and used as value added materials (Elleuch et al., 2008). Attempts should be made to utilize these unused portions into value added products to increase economic feasibility of date industries and processors. Converting unutilized or lower grade date into date fiber and biomass production (e.g. yeast) could be a solution for the problem. Detailed studies on the subject should be carried out in date producing countries to get a boost in economy by producing value added products.

Increasingly, consumers are looking for foods with health benefits which eventually change the diet patterns. High-fiber diets are in great demand in the market which is associated with the prevention and treatment of some diseases such as coronary heart-related diseases, diabetes, constipation, diverticular disease, colonic cancer etc (Brighenti, 1999; Cassidy, Bingham, & Cummings, 1994; Mendeloff, 1987). Furthermore, the most common form of fiber is insoluble fiber (cellulose, lignin and some hemicelluloses), which reduces constipation and is being studied for its potential to reduce the risk of colon/rectal cancer (Peressini & Sensidoni, 2009). Cereals, fruits and vegetables are major source of dietary fibers. Fibers from other sources are welcome by food professionals to meet the continuous demands of the industries. In this regard, date

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fiber-a by-product of the date processing industry has potential to serve as a functional ingredient to formulate health related food products. Limited studies have been reported in the literature on date-based dietary fibers (Borchani et al., 2011; Elleuch et al., 2008). Nevertheless, the mineral profiles, elemental analysis, particle size had not been characterized before. Therefore, further studies on the subject will provide more detailed information on functionality and commercial viability of the date fiber.

The objective of this work was to extract insoluble date fiber from three date cultivars using multistage water extraction through microwave, and characterize them based on their physiochemical properties. This information will be advantageous for food product development and formulate functional foods.

2. Materials and methods

2.1. Materials

Date palm fruits were obtained from the tissue culture centre of Kuwait Institute for Scientific Research, Kuwait. Three cultivars of date samples (*Barhee, Sultana and Owadi*) at the "Tamr stage" (full ripeness) were used for dietary fiber isolation. Date samples were stored at cold storage (5 ± 0.5 °C) before the fiber extraction.

2.2. Fiber isolation

Date samples were depitted manually; cut into small pieces; weighed and put in a glass beaker. Distilled water was added to flesh sample (500 ml/100 g date flesh) and stirred well. The mass was heated in a microwave oven (1000 W; Samsung, South Korea) for 7 min (optimized the time from few preliminary studies) at full power level (10), cool to room temperature (25 °C), and the mixture was filtrated through a fine cloth (pore size of 300 μm). The step was repeated for 6 times to make it sugar free. A flow diagram for the process is shown in Fig. 1. The filtrate was sampled after each extraction for sugar analysis.

After extraction, the wet sample was dried using different types of driers: hot air-flow, vacuum and freeze-dryer. However, only the freeze dryer (VirTis, SP Scientific, NY, USA) at a vacuum pressure of 1×10^{-2} Pa at −55 °C and 25 °C (sample temperature not controlled) for 72 h produced the best result, and therefore, all reported results are based on freeze-dried samples. The dried date fibers were grounded and passed through #200 mesh screen aperture (74 μm sieve opening as per ASTME II).

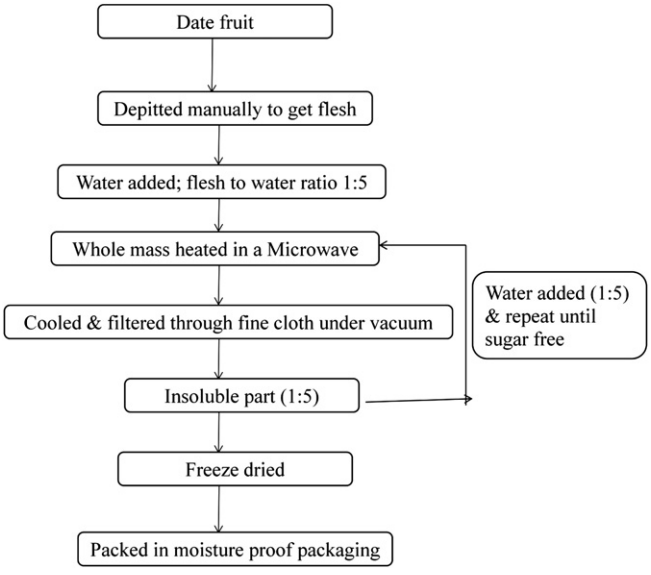

Fig. 1. Flow diagram for freeze-dried water insoluble date fiber.

2.3. Composition analysis

Moisture, ash, lipid and crude fiber contents of freeze dried date fiber samples were determined according to the AOAC methods (1990).

2.4. Protein content

Protein content was estimated from nitrogen ($N = 6.25$) using Gerhardt semi-micro Kjeldahl method (AOAC, 1990) and the CHNS analysis based on combustion method (AOAC, 1990).

2.5. Particle size

Dynamic light scattering (DLS) was performed on a Malvern Instruments Zetasizer Nano-ZS instrument (Malvern Instruments Ltd, UK) for characterization of size of the fiber particle in solution. The device uses a 4 mW He–Ne 633 nm laser to analyze the samples. One gram date fiber sample was dissolved in 100 mL deionized water and sonicated for 45 min before measurement. One and half milliliter was transferred to a square cuvette for DLS measurements. The Dispersion Technology Software (DTS) (V4.20) was used for data collection and analysis. The mean particle diameter is calculated by the software from the particle distributions measured.

2.6. Water activity

Water activity of the samples was measured by a water activity meter (AquaLab, Decagon, USA) as per method described by Ahmed and Ramaswamy (2006).

2.7. Bulk density

Bulk density is measured by the weight of the dried fiber sample which freely poured in a 100 mL graduated cylinder and expressed as weight per unit volume (kg/m^3).

2.8. Elemental analysis

Carbon, hydrogen, nitrogen and sulfur (CHNS) analyses were performed on a Flash 2000 Organic Elemental Analyzer (Thermo Fisher Scientific Inc., MA, USA). Lyophilized samples (about 4 mg) were weighed in small tin capsules and then dropped into an oxidation/reduction reactor for combustion at 950 °C. Pure oxygen and helium were used as the combustion and the carrier gas, respectively. Carbon, hydrogen, nitrogen and sulfur present in samples were converted into CO_2, H_2O, N_2, and CO_2 respectively. These gases are separated in a chromatographic column and finally detected by a highly sensitive thermal conductivity detector (TCD). Cystine (C 29.99.09%; N 11.66%; H 5.03% and S 26.69%) was used for calibrating the instrument. Final concentrations of C, H, N, and S were directly obtained from the manufacturer supplied Eager Xperience software (Thermo Fisher Scientific Inc., MA, USA).

2.9. Mineral analysis

2.9.1. Preparation of samples

Approximately 2 g of date fiber was added into the Teflon beaker, and 30 mL of concentrated nitric acid was added and kept