



Extraction of Insoluble Dietary Fibre from Date (*Phoenix dactylifera*) and its Characterization

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In the present study our aim was to extract water insoluble dietary fibre from the date fruit using microwave assisted heating. Physicochemical properties, rheological behavior, elemental analysis, FTIR and, SEM of fibre were determined. Particle size was in the range of 800–1067 nm. Elemental analysis of water insoluble fibre confirms the presence of protein and absence of sugar. Extracted dietary fibre has excellent oil and water holding capacity. FTIR confirms the presence of polysaccharides, cellulose, hemicelluloses and amides which indicate the presence of protein in fibre sample. Atomic absorption spectroscopic study confirmed the existence of higher amount of calcium, magnesium, manganese, iron, zinc, phosphorous, but absence of sodium.

Keyword: Date, Dietary Fibre, Extraction, Particle Size, SEM

Introduction

Date (*Phoenix dactylifera*) is one of the delicious, sweet, single seeded fruit of *Arecaceae* family of genus *phoenix* and its scientific name is originated from land of bank of Nile and Euphrate of ancient Egypt. Nowadays dates are grown worldwide brimming with much needed energy due to the presence of dextrose, fructose, vitamin, mineral, dietary fibre, flavonoids, polyphenolics, antioxidants having anti-inflammatory, anti-hemorrhagic, anti-infective properties to facilitate healthy and fit lifestyle.^{1,2} Diabetic patients can consume date without any fatal change of glycaemic index. The fruit is rich in dietary fibre and has good oil absorption properties that prevent harmful LDL cholesterol absorption. Dietary fibre has good water binding properties; it prevents constipation, colon, pancreatic, prostate cancer etc.³ A significant amount of under sized or inferior quality dates are wasted in date producing country might be due to the fluctuation of agro-climatic condition, lack of storage facility etc. To prevent wastage, date dietary fibre can be extracted from insoluble part as it is an excellent source of fibre, polyphenols, phytonutrient and antioxidants to utilize that as food additives. The objective of the study was to extract insoluble fibre from date palm and its characterization.

Material and Methods

Materials

The Indian variety of date fruit (Tamar soft dates) was purchased from the local market, Chandigarh and stored at ($4 \pm 2^\circ\text{C}$) before extraction. All the chemicals were of analytical grade and purchased from Hi-Media Chemicals, Mumbai, India.

Fibre isolation from dates and Composition analysis

A flow diagram for the fibre extraction process is shown in Fig. 1. Moisture, protein, ash, fat, total protein (Kjeldahl method) and dietary fibre (DF) were determined following AOAC, 2007 method.⁴ Mineral contents were determined using Atomic Absorption Spectrometer (Thermo Fisher Scientific Inc., MA, USA). Sugar content was determined by Miller's method using 50–100 $\mu\text{g/mL}$ standard D-glucose measuring absorbance at 600 nm using UV-visible spectrophotometer (UV 2401 PC, Shimadzu, Kyoto, Japan).⁵ Particle size of dry fibre powder was determined using Zetasizer (Nano ZS-90 model, Malvern, UK). Water activity was measured using water activity meter (Aqualab, Decagon, USA). Bulk density of the dried fibre was determined by pouring freely the dried fibre powder sample in a 50 mL graduated measuring cylinder taking 25 g fibre sample powder. Water holding capacity (WHC) and oil holding capacity (OHC) of fibre samples were measured according to Elleuch *et al.*, (2008) method and expressed as g of water or oil absorbed/g of fibre

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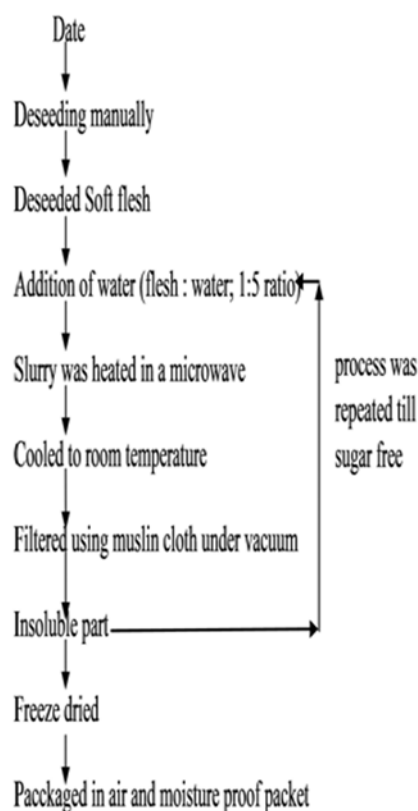


Fig 1 — Date Fibre Extraction method

sample.⁴ Rheological properties at selected concentrations (3%, 4%, 5%) of dried fibre powder were measured using cup and bob (CC27) assembly of dynamic rheometer (MCR 102 Anton Paar, Austria). The shear stress (Pa) was measured in duplicate with linearly increasing shear rate till 1000 s^{-1} at 25°C . Carbon, hydrogen, nitrogen and sulphur (CHNS) analyses were performed on a Flash 2000 Organic Elemental Analyzer (Thermo Fisher Scientific Inc., MA, USA) following standard instrumental protocols. Visual color of the extracted dietary fibre was measured using a Hunter colorimeter Color Flex model (Hunter Associates Laboratory, Reston, VA) in terms of L^* (lightness), a^* (redness and greenness) and b^* (yellowness and blueness).⁶

Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM)

The dry powdered date fibre sample was characterized by FTIR spectroscopy (Perkin-Elmer IR spectroscope, Waltham, Massachusetts, USA) and the spectra were recorded in the range of $4000\text{--}400 \text{ cm}^{-1}$ on Perkin Elmer Spectrum software (Version 10.03.08). Scanning Electron Microscopy (Ultra-high Resolution Scanning Electron Microscope JSM-6100,

Digital SEM) was recorded to study the morphology and appearance of the surface of the date sample and the extracted dietary fibre following the instrumental protocol.⁷

Glucose Diffusion Rate (GDR) and Glucose Diffusion Reduction Index (GDRI)

Dietary fibres control the glucose diffusion rate in human body. Quantity of glucose released during metabolism and absorbed in human body is defined as Glucose Diffusion Rate (GDR). The Glucose Diffusion Reduction Index (GDRI) was determined according to Saikia and Mahanta, 2016.⁸

Statistical analysis

The outcomes were expressed as mean \pm SD. Statistical analysis was done using Microsoft Excel. $p > 0.05$ stands for not significant.

Results and discussion

Yield and Chemical composition

Yield of total fibre from the date fruit was 28.7 g per 100 g sample, which was quite good in amount but higher than Crude fibre in date pits was 21 g (on wet basis) per 100 g of sample. Date flesh reducing sugar was 5.32 g and non-reducing sugar was 2.02 g, and no residual sugar was found after 5th extraction. Remarkably lower water activity 0.18 indicates longer shelf life of the product with proper storage and packaging. The bulk density of the fibre powder was $928 \pm 0.15 \text{ Kg/m}^3$ (Table 1). The moisture and ash content were 1.53% and $4.45 \pm 0.2\%$ respectively which were lower than other fruit peel (except dried palm 1.6%) but similar to cereal fibre.⁸ The fat content of the fibre sample was 0.16% whereas in date sample it was 5.96%, it was significantly lower as compared to other fruit fibre samples.¹ Significantly higher amount (18.56%) of crude fibre was present in date. Date pit also contains 21.35% fibre.

Elemental analysis results revealed that carbon content in fibre sample was more than nitrogen and hydrogen. The carbon, hydrogen, nitrogen and oxygen content were 33.828%, 6.068%, 0.839% and 59.256 % per 4g of sample respectively. Protein content in fruit and extracted fibre samples were found 10.98% and 13.32% respectively by Kjeldhal method and 10.76% and 14.14% respectively by CHNS method. According to Elluech 2008 though Kjeldahl method was a more reliable method for true protein, combustion method was used to determine

Table 1 — Characterization of date sample and fibre sample

Parameter	Date fruit	Dietary Fibre	Parameter	Date fruit	Dietary Fibre
Crude fibre (g/100g)	18.56±0.12	28.7± 1.05	P	—	1.25 ppm
Total Sugar	7.34	0.38± 0.12	Ca	—	8.5 ppm
Water activity (a_w)	0.34± 0.05	0.18 ± 0.08	Cu	—	0.085 ppm
Bulk density (kg/m ³)	—	928 ± 0.15	Mn	—	0.125 ppm
WHC (g water per g of fibre)	—	18.30	Fe	—	1.27 ppm
OHC (g of oil per g of fibre)	—	9.98	Zn	—	0.84 ppm
Color	—	—	Moisture %	14.6±0.1	4.45 ± 0.15
Particle diameter (nm)	—	800–1067	Ash %	2.4± 0.01	1.53 ± 0.15
Reducing Sugar	5.32	0.15 ± 0.86	Fat %	5.96 ± 0.11	0.16 ± 0.14
Non reducing Sugar	2.02	0.23± 0.32	(L*)	26.71	45.25
Protein (%)	10.76±0.23	13.32 ± 0.75	(a*)	11.68	5.65
Protein (%)	10.98±0.12	14.14 ± 0.03	(b*)	18.34	8.28

crude protein content as it was faster, very sensitive to low concentrations of nitrogen, requires small amount of sample.¹ As good amount of mineral like P (1.25 ppm) Ca (8.5 ppm), Cu (0.085 ppm), Mn (0.125 ppm), Fe (1.27 ppm), Zn (1.84 ppm) were present in date fibre (Table 1), therefore extracted fibre was a superior source of dietary fibre and can be incorporated in processed foods.

The water holding capacity (WHC) of the date fibres was 18.30 g per 100 g fibre (on dry basis) which was significantly higher as compared to other fruits and vegetables fibres. The high WHC attributed to the presence of large amounts of insoluble dietary fibres.⁸ OHC of date fibre sample was found 9.98 g per 100 g fibre. The higher the OHC of date fibre it could be used as an ingredient to stabilize foods with a high percentage of fat.¹ The rheological behaviour of 3%, 4%, and 5% date fibre solution are shown in Fig. 2. At lower shear rate small increase in shear stress, but for the higher value of shear rate, shear stress increased continuously and finally reached constant (Fig. 2a). The fluid showed non-Newtonian, pseudo-plastic behaviour at all the concentrations. Viscosity-shear rate relationship of date fibres at 25°C is shown in Fig. 2b. It showed that for all concentrations, the apparent viscosity of all samples decreased with increasing shear rate, indicating shear-thinning characteristic of fibre solution. With increasing shear rate, the hydrodynamic forces would dominate and molecules become aligned resulting in reduction of viscosity. The colour values (L*, a*, b*) for date and extracted fibre are shown in Table 1. The brightness of date fibre was higher as compared to fruit flesh, repeated washing during extraction cause fading of dark color. Significantly lower a* value

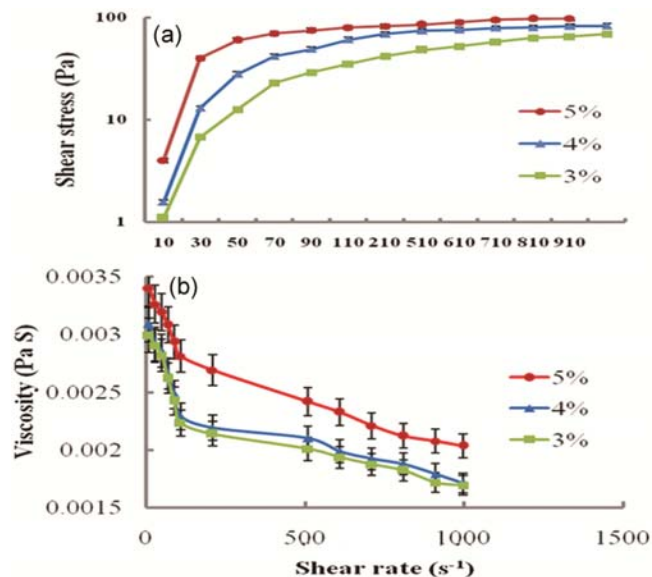


Fig. 2 — (a) Rheological behaviour of fibre shear stress vs. shear rate; (b) shear stress vs. viscosity;

indicated lower redness and b* value was higher which indicated yellowness.

The FTIR of the dietary fibres revealed the presence of C=O stretch in frequency range of 1760–1665 cm⁻¹. This stretch might be due to the presence of carboxylic acid. In the frequency range 1650–1580 cm⁻¹, the N-H bends due to the 1^o amines. Carbon stretching is showing the nitrogen and hydrogen bonding in the range of 1250–1020 cm⁻¹ which indicated the presence of protein in the fibre sample.

SEM picture of extracted fibre from date fruit was shown in Fig. 3. The picture showed large number of regularly homogenized well scattered and exfoliated fibres on the particle's surface with average range of fibre thread length 123.20–126.35 μm. From the

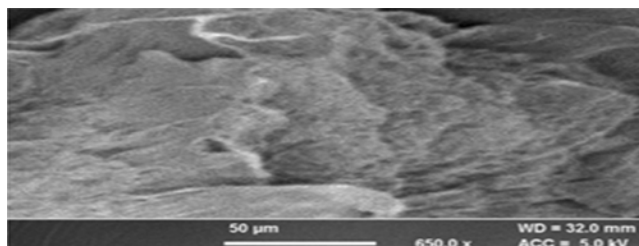


Fig 3 — SEM of the extracted fibres from date fruit

given figure the shape of the fibres was found thread like, bundle forming, regularly repeated transverse pattern with analogous orientation was very clear. The particle size ranged between 800–1067 nm. GDR and GDRI of extracted fibre after 30, 60, 90 and 120 min was determined and it was found that amount of glucose in the dialysate increased with time and the values of GDRI was in the range of 25–18 respectively. Therefore it can be used as nutraceuticals in low calorie food products. The particles did not aggregated till 30 minutes in water which revealed that date fibres have potential to use as nano-particles. Spongy structure of the date dietary fibres was found as an essential element for preparation of new products in frameworks for micro propagation, as wound healings after surgery similar to polyurethane.

Conclusion

In the present study dietary fibres were extracted from date using microwave assisted water extraction method with 28.7% yield. From the characterization study of physicochemical properties it is confirmed that the extracted fibre has beneficial effect and it is

an excellent source of nutrient and minerals, therefore it can be used as an additive in low calorie glucose lowering functional food products.

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