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Lignin is the main determinant of total dietary fiber differences between date fruit (*Phoenix dactylifera* L.) varieties

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ABSTRACT

Date fruits (*Phoenix dactylifera*) of ten varieties, collected in the United Arab Emirates, were studied to determine their dietary fiber content and composition. Fourier transform infrared (FTIR) spectroscopy indicated that the dietary fiber components in all the date fruit varieties was similar. The major dietary fiber components, including cellulose, hemicellulosic components, lignin, and pectin, were analyzed by the Uppsala method. The total dietary fiber content in the date fruits analyzed (5.2%–8.3%) is comparable to commonly consumed dried fruits and is correlated with the content of lignin. The lignin was the main determinant of the total dietary fiber content in dates and its content was higher in semi-hard and hard fruit varieties.

1. Introduction

Meta-analyses of prospective epidemiological studies revealed that fruits and vegetables have moderate but significant associations with decreased risk of mortality and metabolic diseases including obesity, cardiovascular disease and some cancers [1,2]. The consumption of fruits and vegetables are found to improve the overall diet profile by increasing the intakes of carbohydrates, dietary fiber, and micronutrient vitamins and minerals [3]. The dietary fiber is effective in potentiating health, e.g. by increasing fecal bulking and viscosity and decreasing the contact time of inflammatory agents and carcinogens and mucosal cells, enhancing the production of short chain fatty acids and the binding between bile acids and harmful dietary components such as estrogen and carcinogenic compounds, and by supplying antioxidants to the body [4]. The Institute of Medicine (IOM) set a fiber allowance intake value of 14 g/1000 kcal, which is equivalent to 25 g/day for women and 38 g/day for men in the age range 19–50 years [5].

Dietary fiber is composed of various fractions including cellulose, non-cellulosic polysaccharides such as hemicelluloses, and non-carbohydrate fractions such as lignin. These are also are the main structural components of the cell wall in plants. Dietary fibers can typically be classified as soluble or insoluble according to their water solubility [6]. Major dietary fiber fractions such as cellulose, hemicelluloses, and lignin are insoluble; they have beneficial effects on the human intestine [7]. The soluble dietary fiber fractions include non-cellulosic polysaccharides, oligosaccharides, pectin, and gums; they have beneficial effects on blood glucose and serum cholesterol levels [8,9].

Date fruits represent a rich source of dietary fiber (6.5–11.5%), of which 84–94% is insoluble and 6–16% is soluble [10,11]. Cellulose, hemicelluloses, and lignin make up the insoluble dietary fiber fractions in dates, whereas pectin, fructooligosaccharides, inulin, galactomannan, and β-glucan (among others) contribute to the soluble dietary fiber fractions. Some of these dietary fiber fractions will now be described in more detail. Cellulose is an unbranched linear chain of several thousand glucose units with β-1,4-glucosidic linkages. It is not digested to any extent by the enzymes of the human gastrointestinal system [12]. Hemicelluloses also contain backbones of glucose units with β-1,4 glucosidic linkages, but they differ from cellulose as they are smaller in size, are usually branched, and contain a variety of sugars, which primarily include xylose with some galactose, mannose, arabinose, and other sugars [12,13]. Neither cellulose nor hemicelluloses, which together comprise the main constituents of insoluble dietary fiber in dates, are soluble in ethanol. Lignin is a complex polymer containing about 40 oxygenated phenyl propane units including coniferyl, sinapyl, and *p*-coumaryl alcohols that have undergone a complex dehydrogenative polymerization. Due to strong intramolecular bonding, which includes carbon to carbon linkages, lignin is relatively inert. It also demonstrates a greater resistance to digestion than any other naturally occurring polymer. Finally, pectic substances are a complex group of polysaccharides in which D-galacturonic acid is a chief constituent. They are also structural components of plant cell walls and act as intercellular cementing substances. Pectin is highly

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Fig. 1. The ten date varieties analyzed for dietary fiber composition in this study.

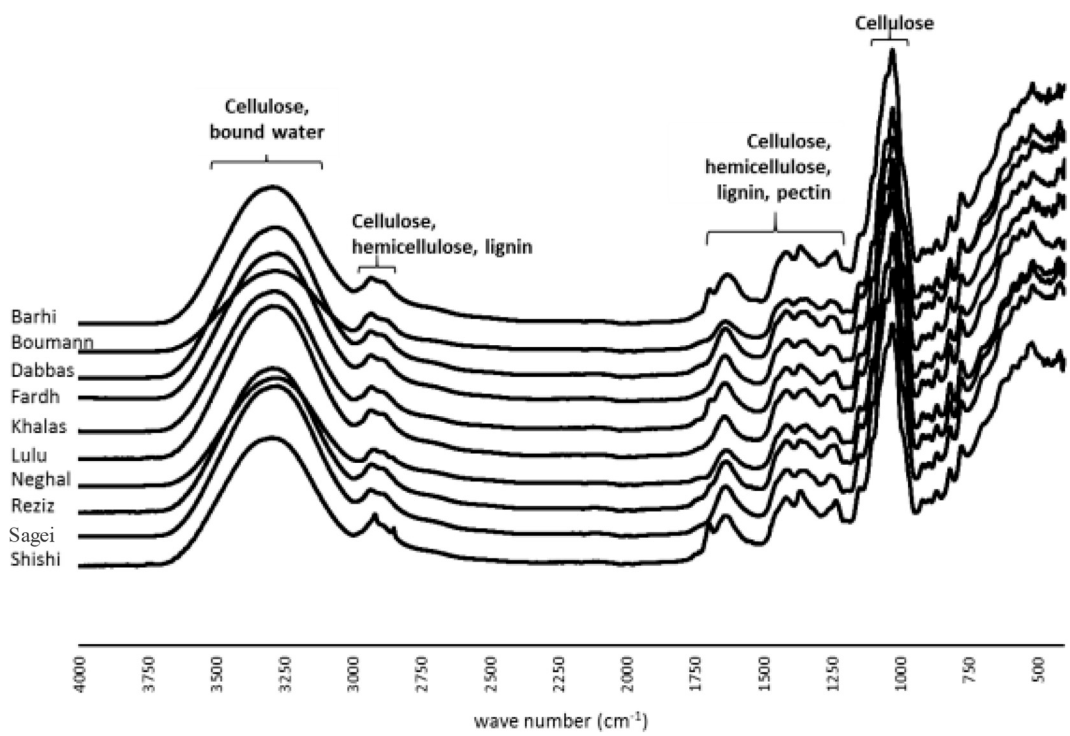

Fig. 2. Fourier transform infrared absorbance spectra of ten Emirati date fruit varieties.

Table 1
Peak positions and assignments of various chemical groups as evidenced from the Fourier transform infrared spectra of the UAE date fruit varieties.

Wave number for a region in the spectrum (cm^{-1})	Characteristic chemical groups and their behavior	Molecules responsible
3600–3200	O—H stretching vibrations of the hydroxyl groups, inter- and intramolecular hydrogen bond vibrations	Cellulose, water molecule due to the moisture content
2960–2830	Stretching vibrations of the C—H of alkyl groups	Cellulose, lignin, and hemicellulose
1700–1500	C═C aromatic skeletal vibrations, arising from acetyl, and ester groups	Lignin, hemicelluloses
1022	C—O stretching of the pyranose ring	Cellulose
1740–1600	From various carbonyl groups, bending vibrations	Pectins bound water
1645–1612	C—O stretching of conjugated or aromatic ketones and in flavones	Lignin
1200–1400	C—O stretching, C—H stretching, C—H vibrations, CH2 bending, and asymmetric stretching mode vibration of methyl ester	Hemicelluloses, cellulose, pectin
1050–1040	C—O, C—C, and C—OH stretching vibrations, aromatic C—H in plane deformation	Cellulose, hemicelluloses, lignin, arabinose
1010–1018	C—O, C═C, and C—C—O stretching backbone vibrations	Galactomannans, pectins
910–750	C—H bending of syringyl units, aromatic ring; antisymmetric out-of-phase ring stretching	Hemicellulosic compounds

Compiled from the references [18–21].

Table 2
Total dietary fiber (TDF) content and relative composition of fiber components (g/100 g dry weight) in UAE date fruit varieties*.

Variety	Sugar residues (% of dm)							Klason lignin	Uronic acids	Fructans	TDF including fructans
	Rhamnose	Fucose	Arabinose	Xylose	Mannose	Galactose	Glucose				
Barhi	0.08 ± 0.01	0.03 ± 0.01	0.28 ± 0.01	0.77 ± 0.04	0.22 ± 0.01	0.25 ± 0.02	1.24 ± 0.04	1.24 ± 0.09	0.78 ± 0.03	0.42 ± 0.03	5.30
Boumann	0.08 ± 0.01	0.03 ± 0.00	0.29 ± 0.01	0.80 ± 0.11	0.21 ± 0.00	0.52 ± 0.04	1.27 ± 0.07	1.93 ± 0.10	0.82 ± 0.08	0.13 ± 0.01	6.09
Dabbas	0.08 ± 0.01	0.03 ± 0.00	0.27 ± 0.02	0.88 ± 0.04	0.23 ± 0.01	0.58 ± 0.04	1.33 ± 0.05	2.94 ± 0.24	0.97 ± 0.01	0.24 ± 0.06	7.56
Fardh	0.08 ± 0.01	0.03 ± 0.00	0.29 ± 0.01	0.69 ± 0.09	0.23 ± 0.01	0.53 ± 0.07	1.31 ± 0.11	2.35 ± 0.14	0.92 ± 0.08	0.28 ± 0.04	6.71
Khalas	0.11 ± 0.02	0.03 ± 0.00	0.34 ± 0.04	0.77 ± 0.13	0.24 ± 0.02	0.32 ± 0.03	1.46 ± 0.23	1.64 ± 0.27	0.93 ± 0.16	0.36 ± 0.08	6.20
Lulu Red	0.08 ± 0.01	0.03 ± 0.00	0.27 ± 0.03	0.75 ± 0.06	0.24 ± 0.01	0.52 ± 0.04	1.31 ± 0.09	1.54 ± 0.17	0.83 ± 0.04	0.35 ± 0.08	5.92
Neghal	0.10 ± 0.00	0.03 ± 0.01	0.31 ± 0.01	1.11 ± 0.03	0.21 ± 0.00	0.53 ± 0.02	1.54 ± 0.02	3.18 ± 0.32	1.12 ± 0.01	0.28 ± 0.09	8.42
Reziz	0.10 ± 0.01	0.03 ± 0.01	0.34 ± 0.02	0.77 ± 0.04	0.22 ± 0.02	0.33 ± 0.01	1.42 ± 0.09	1.99 ± 0.32	1.02 ± 0.11	0.43 ± 0.19	6.65
Sagei	0.09 ± 0.00	0.03 ± 0.00	0.29 ± 0.02	0.77 ± 0.08	0.21 ± 0.01	0.68 ± 0.03	1.25 ± 0.07	3.00 ± 0.44	0.81 ± 0.22	0.30 ± 0.05	7.43
Shishi	0.09 ± 0.00	0.03 ± 0.00	0.32 ± 0.01	0.82 ± 0.01	0.24 ± 0.01	0.46 ± 0.00	1.30 ± 0.07	2.33 ± 0.23	0.91 ± 0.03	0.50 ± 0.06	7.01

* Values of the different dietary fiber component represent the mean ± SD for three samples per variety. TDF is obtained by adding all the presented dietary fiber components.

water-soluble and is almost completely metabolized by colonic bacteria [12].

Date fruits, with their inherent polyphenols, are considered to be a nutritious and healthy source of dietary fiber with the added benefit of antioxidant activity [14]. Given the potential health benefits of dates, it is important to understand how various varieties of the fruit can contribute to human nutrition and, ultimately, well-being. Therefore, the aim of this study was to analyze the content of total dietary fiber in dates from ten Emirati varieties of *Phoenix dactylifera* collected from different locations in the United Arab Emirates (UAE).

2. Materials and methods

2.1. Samples

Fully mature (tamar stage) fruits of ten *P. dactylifera* date varieties were collected in 2017, each from three locations of the following in UAE (Al Saad, Al Foah, Bak Riya, Gummed, Al Dahid, and Wadi Al Khar). The ten studied varieties are presented in Fig. 1.

2.2. Fourier transform infrared spectroscopy

The date fruit samples were placed directly into the sample holder of a Fourier transform infrared (FTIR) spectroscopy instrument (PerkinElmer Spectrum Two™ FT-IR spectrometer, USA). The absorbance was obtained after recording the background signal for the spectral range from 400 to 4000 cm^{-1} using Spectrum software.

2.3. Dietary fiber analysis

De-seeded fruit pieces (80 g) were mixed with cold, deionized water (160 mL) and grounded in a Sorwall Omni mixer until the sample is well homogenized. 0.5 g of the mixed sample was used for the analysis of dry matter (16 h at 60 °C in a vacuum oven). The dietary fiber content and composition were determined by the Uppsala method [15]. First, the simple sugars were washed and removed with 80% ethanol. Subsequently, the sample was treated with enzymes and the soluble dietary fibers were precipitated with 80% ethanol solution. Finally, the precipitated and insoluble polysaccharides were hydrolyzed with sulphuric acid. The released neutral sugars were quantitated as alditol acetates by gas-liquid chromatography (GC). Uronic acids in the acid hydrolysate were determined spectrophotometrically after hydrolysis. Klason lignin was determined gravimetrically as ash-free acid-insoluble residue.

A spectrophotometric method was followed using an enzymatic assay kit (k-FRUC, Megazyme, Bray, Ireland) to determine the fructan (fructo-oligosaccharides and fructan polysaccharide) content according to the methods of McCleary, Murphy, and Mugford [16] with some modifications. The extraction step was completed with a 0.5 g sample, which was accurately weighed into a glass tube with 10 mL of deionized water and kept at 80 °C for 20 min. The filtration step was replaced by centrifugation: 1 mL of the mixture was centrifuged for 15 min at 10,600 g and 200 μL of the supernatant was then used for analysis. The samples were treated with α-galactosidase for the removal of raffinose-type oligosaccharides. This was completed before the degradation of maltosaccharides and sucrose, as detailed in the kit manufacturer's instructions. The sum of the dietary fiber from the Uppsala method and fructan was defined as the total dietary fiber (TDF).

2.4. Statistical analysis

Statistical analysis was completed using Minitab, version 19 (https://www.minitab.com/en-us/). One-way ANOVA was used to test for differences in dietary fiber content and composition among date fruit varieties. $p < 0.05$ was used as a measure of statistical significance.

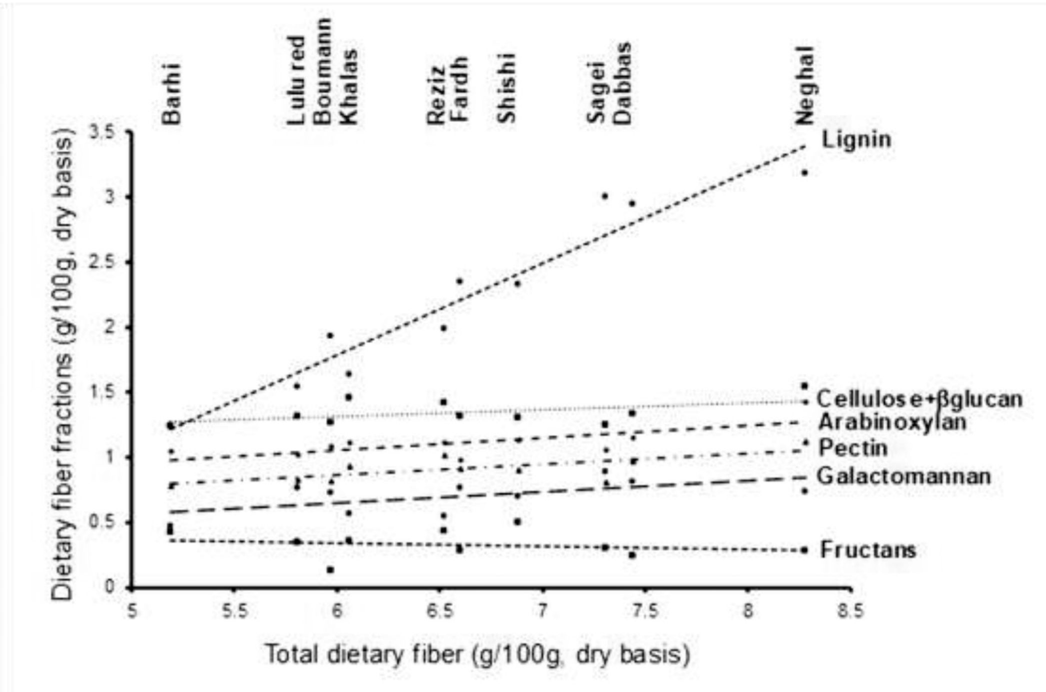

Fig. 3. Scatter plot showing the relationships among total dietary fiber contents and the content of the different fiber fractions (mean values for three samples).

Table 3
Dietary fiber in the skin and white parts of date fruits Fardh and Sagei (g/100 g dry matter)*.

Variety	TDF	Sugar residues							Klason lignin	Uronic acids
		Rhamnose	Fucose	Arabinose	Xylose	Mannose	Galactose	Glucose		
Skin (mainly collenchyma cells & sclereids)										
Fardh	21.8	0.17	0.06	1.10	6.40	0.57	0.87	4.04	6.42	2.22
Sagei	18.9	0.16	0.04	0.98	5.79	0.50	1.01	3.28	5.28	1.82
Inner white fiber bundles of the mesocarp (parenchyma cells)										
Fardh	6.13	0.12	0.04	0.46	0.63	0.42	0.47	2.32	nd	1.67
Sagei	5.34	0.11	0.04	0.40	0.61	0.38	0.50	1.94	nd	1.36

TDF, total dietary fiber. nd, not detected.
* Values are means of the analysis of two samples. Analysis of fructans was not performed for these samples.

3. Results and discussion

3.1. FTIR spectral fingerprints of date fruit fibers

The ten date fruit varieties studied here differed in their softness with Barhi and Lulu being soft varieties, Neghal and Dabbas being hard to semi-hard, and the rest of varieties lie in between. In general, the fruits are composed of skin, sugary flesh, a distinct white part composed of fibrous bundles that is edible and devoid of sugars, and a seed [17]. The FTIR analyses of the edible parts of the fruits, showing peaks for various absorbances from 400 to 4000 cm^{-1}, are shown in Fig. 2. All date fruits studied showed similar FTIR fingerprints, which suggests that they have comparable dietary fiber compositions. The spectra, interpreted according to Table 1, show the absorption bands associated with stretching and bending vibrations of the characteristic chemical groups of the various dietary fiber components including cellulose, hemicelluloses, lignin, and pectin [18–21]. The spectral fingerprints of the date fruit fibers reported here agree with those from previous studies of date fruit fibers [14].

3.2. Total dietary fiber and its constituents in date fruits

The sugar residues, Klason lignin, uronic acids, and fructan components of the analyzed date fruits are presented in Table 2. The observed sugar residues resulting from the hydrolysis of the fruit polysaccharides, analyzed by GC, show that these dates consisted mainly of glucose, xylose, galactose along with limited amounts of arabinose and mannose with minute quantities of rhamnose and fucose. The quantity of uronic acid was usually lower in soft dates such as Barhi (0.7–0.8%), whereas Neghal and Reziz had higher uronic acid values of around 1.1%. The average fructan content across varieties was 2–8% of the TDF with the soft variety Barhi having the highest fructan content (7.9% of TDF). The content of Klason lignin varied from around 25 to 40% of TDF in the samples. Lignin, the non-carbohydrate fraction of the dietary fiber, is the major dietary fiber component in the date fruits, which agrees with the findings of previous studies [11,14,22]. The varieties Barhi, Lulu, and Khalas had low lignin contents while Sagei and Negal had high lignin contents suggesting that the Klason lignin content increased with increasing fruit hardness.

The total dietary fiber (TDF) content found in the ten date varieties was 5.3–8.4% (Table 2); values that are in accordance with results from previous studies [10,13]. In general, the TDF content in dates is comparable to that of other dry fruits such as dried blueberries (7.5%), raisins (6.8%), apricots (7.3%), pears (6.4%), and cranberries (5.3%) [23] but is much higher than that observed in fresh fruits [24]. According to our results, the contribution of date fruits to the recommended daily intake of dietary fiber will vary according to variety. For example, three fruits of Barhi and Sagei, weighing approximately 13 and 35 g [25], will provide 2–3% and 7–10% of the recommended daily fiber intake, respectively.

The obtained neutral sugars are constituents of cellulose and hemicellulose. The pectin and lignin of the fruit cell walls contribute to the obtained values for uronic acids and Klason lignin. The glucose in date fruit polysaccharides comes mainly from cellulose and, to a lesser extent, from β-D-glucan [9]. According to literature, date fruit polysaccharides are mainly composed of cellulose, hemicelluloses (arabinoxylans and galactomannans), pectins (uronic acid derivatives), and lignin [11,14]. Ishrud et al. [26] isolated galactomannan from date seeds and hence we assume that the fruits must also have the same polysaccharide. The values of the released neutral sugars (galactose and mannose) in the dietary fiber analysis of our samples are thus added together and taken as galactomannan. Fig. 3 shows that presumed levels of fructans, galactomannan, pectin, arabinoxylan, and cellulose/β-glucan did not differ much between the ten date varieties but the level of lignin differed considerably in these varieties and had a high significant correlation with the TDF content ($r = 0.964$, $p < 0.01$).

3.3. The variability in the fiber composition within the fruit tissues

Table 3 presents the differences in TDF and its components between the skin and the inner white bundles of the mesocarp in two date varieties, Fardh and Sagei. The TDF and especially lignin, arabinose and xylose contents are much higher in the skin and that the uronic acid content in the skin and the inner white part of the date fruits was more than twice that in the whole fruit. Date fruit skin had more than twice as much lignin than the fruit whole fruit and that no lignin was detected in the inner white fiber bundles of the fruit. This variability in fiber components distribution is related to the types of cells constituting these tissues. The skin is rich in collenchyma cells in addition to sclereids, which are sclerenchyma cells highly thickened by lignin. In contrast, the mesocarp dominating the rest of the fruit is composed of parenchyma cells having thin primary cell walls mainly composed of cellulose [27].

4. Conclusions

Analysis of ten UAE date varieties showed that date fruit is a good source of dietary fiber and that these varieties did not differ significantly in their constituent sugar residues or fructans. This study showed that lignin is the main determinant of the total dietary fiber content in date fruits and that harder date varieties have higher levels of lignin and total dietary fiber than softer varieties. It is suggested that date fruit by-products that remain after the extraction of sugars or syrups could be a promising source of dietary fiber and can be used as functional ingredients in a variety of foods such as ice cream, sausages, bakery products, and beverages to modify textural and emulsification properties [25,28].

Credit author statement

Afaf Kamal-Eldin planned the study and obtained funding, Navomy George and Andersson A.A.M. performed the analysis, Roger Andersson supervised the study, and N. George and A. Kamal-Eldin wrote the manuscript. All authors reviewed and accepted the manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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