ISSN: 2347-467X, Vol. 11, No. (1) 2023, Pg. 77-87

Current Research in Nutrition and Food Science

www.foodandnutritionjournal.org

Valorization and Chemical Constituents Assessments of Khalas Dates Fruit, Syrup and Pits

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Abstract

This study was conducted to contribute to the national approach to achieving food security in Saudi Arabia, which is one of the world's largest producer of dates. Studying the chemical properties of the most commercial date's fruits cultivar in the eastern region will contribute to understanding to industrial potential Khalas dates. This is in addition to developing a method for extracting date syrup (DS) in a way that preserves as much nutritional properties as possible. Date fruits (DF) flesh and water mixture at a 1:3 ratio gave 18% total soluble solids (TSS) then concentrated to 70° Brix. The used date flesh to water ratio recaptured all sugars, acetic, butyric and propionic acids in DS. Sugars were the major components amounting 34.3 and 32.6% glucose and fructose, respectively. No crude or dietary fibers were present in DS due to their insolubility in water. In contrast, the major portion of minerals was recovered in DS. In the same way, 60% and 4.9% of the fat and protein respectively were recovered in the DS. It also notes that the main amino acid in DS is glutamic acid which represents 16.25 % of the total detected amino acids. In concentrated DS a total polyphenol (PP) tenor of 1448.65 mg/100 g gallic acid equivalent (GAE) was recovered. The presence of large amounts of PP in DS compared to whole dates can be explained by the use of high temperature DS extraction with vacuum evaporation of excess water. Crude and dietary fibers were the major components in date pits (DP) representing 24.1% and 56.1% respectively. Fat and sucrose were also present in a significant portion in DP showing 5.9% and 2.4%, respectively. Therefore, the results obtained will serve many food and nutrition industries as well as the pharmaceutical and therapeutic industries.

Article History
Received: 04 February 2023
Accepted: 15 March 2023

Keywords
Antioxidant Activity;
Date Syrup;
Date Syrup Extraction;
Date Pits;
Khalas Cultivar;
Total Polyphenol.

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Doi: http://dx.doi.org/10.12944/CRNFSJ.11.1.5

Introduction

World yield of date fruits (*Phoenix dactylifera* L.) was 9.45 million tons in 2020, of which Saudi Arabia produced 1.54 million tons.[1] There are more than 400 types of dates fruits in Saudi Arabia and each has a different taste and texture.[2] The Kingdom of Saudi Arabia takes advantage of being the second largest producer of date palm fruits in the world,[1] to contribute locally and globally for achieving food security, it is therefore necessary to evaluate chemical, nutritional and functional properties of their fruits. It must be mentioned that even before the outbreak of COVID-19, global food systems faced significant challenges in providing food security and adequate nutrition to a growing global population, as well as ensuring environmental sustainability. However, this pandemic has accelerated local, regional and international policy efforts to ensure food security and sustainable development. In fact, date palm products will contribute to providing food security and healthy nutrition in production countries.[3,4] Considering the biotechnological properties, date palm fruits consist of three essential parts: date flesh representing 85 - 90% 5, date pit (6 - 12%). The outer part of the fruit (the skin) is a thin, protective layer .[6,7] About 15 % of the dates produced in Saudi Arabia are considered as wastes. Presently, there are no suitable and optimal management systems for these wastes. Developing methods for the rational valorization of low quality DF can constitute a suitable solution for their disposal.[8] Such DF could be utilized in DS industry. In which, the fruits go through juice extraction, clarification, filtration, and concentration. Aleid[9] analyzed DF and found that fructose and glucose were their major chemical components representing 44% fructose, 39% glucose and 1% sucrose on a dry weight basis. In addition, DF and its various derivatives have been used as functional foods with therapeutic properties for some diseases and various health disorders, as they are an important source of biologically active compounds.[10,11,12] However, there are few studies dealing with chemical characterization of DS as a value added product. Knowing as much as possible about the physicochemical properties of DS is important from many perspectives.

Such data will be of great importance for knowing suitable functional components from DF derivatives for food, pharmaceutical and biotechnology industries without losing sight of the nutritional significance and potential health benefits.[13] Accordingly, the objective of the current study was to evaluate the chemical constituents (chemical composition, fatty acid profile and amino acid profile) of Khalas dates fruit, syrup and pits.

Material and Methods

Materials

Newly harvested DF (Khalas cultivar) were obtained from a local farm in in Alahsa City, Saudi Arabia at the beginning of the 2021 harvest season. All chemical analysis was run in triplicate and results are expressed as the mean ± SD (Standard Deviation) unless otherwise stated.

Chemicals and Reagents

All the chemicals used in the current study were of analytic grade and were obtained from Sigma Chemical Co., St. Louis, MO, USA unless mentioned elsewhere.

2,2-Diphenyl-1-picrylhydrazyl free radical (DPPH), Folin-Ciocalteu reagent, gallic acid, ascorbic acid, quercetin, and were obtained from Fluka Chemie GmbH, Buchs, Switzerland.

Extraction and Clarification of Date Fruit Syrup (DS)

DS was extracted according to Aleid[9] in a large industrial scale processing line in Al Ahsa National Food Processing Plant facilities (Al Ahsa, Saudi Arabia). In the extraction process, dates were placed in a vessel and water was added at water-to-date ratio of 3:1. The temperature of the mixture was adjusted to 65°C then mixed for 30 min. The mixing was held constant at 50 rpm and to reduce evaporation of water the vessel was covered.

Date Pits, as a byproduct were separated by sieving which is a main step in DS extraction industry. The extract was primarily filtered to separate DP and other solid materials before obtaining a clear solution with 18 Brix. Efficient clarification was obtained by applying ultrafiltration: [molecular weight membranes of 20,000 and 3.3 Bar]. The concentration of filtrate was realised under vacuum (0.8 Bar) at 70°C to 70° Brix. A flow diagram for the commercial DS process could be seen in (Figure 1).

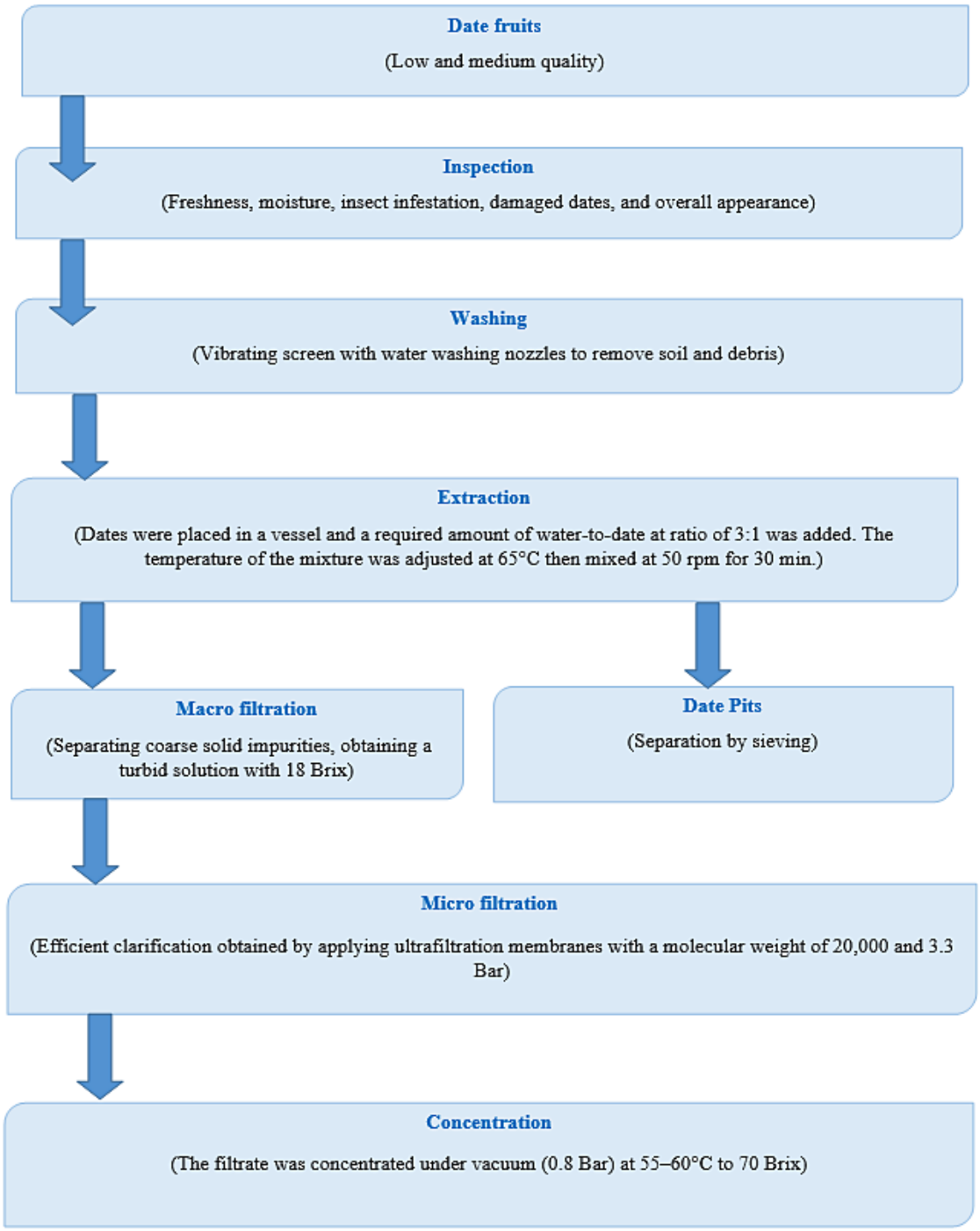

Fig. 1: Flow diagram for date fruit syrup extraction process.

Chemical Analysis

The A.O.A.C. (2005)[14] Official Method of Analysis have been used for the following analysis: moisture (A.O.A.C.-926.08), total fat (A.O.A.C.-933.05), crude protein (A.O.A.C.-930.33), crude fiber (A.O.A.C.-962.09), fructose, glucose, sucrose (A.O.A.C.-977.20), total dietary fiber (A.O.A.C.-993.21), potassium, calcium,

magnesium, sodium, copper, manganese, zinc, iron (A.O.A.C.-985.35), phosphorus (A.O.A.C.-991.25). Fatty and amino acids were measured (A.O.A.C.-996.01) and A.O.A.C.-933.40 methods respectively. Organic acids such as acetic, propionic and butyric were quantified in the hydrophilic phase, using an HPLC system as previously reported.[15,16]

Total Phenolic Content and Antioxidant Activity

The Folin-Ciocalteu method was used to measure the total phenolic contents of each extract.[17] The total phenolic content was expressed as mg Gallic Acid Equivalents (mgGAE)/100 g sample. The free radical scavenging assay of the DF, DS and DP was determined using 1,1-diphenyl-2-picrylhydrazyl (DPPH) according to the method previously described.[18]

Results and Discussion

Extraction and Clarification of DS

In an extraction experiment, about 600g syrup with a Brix of about 70 was obtained from one kilogram of dates. The solution from the water-to-date at ratio of (3:1) was heated, filtered and concentrated under vacuum to 70° Brix, which is suitable for a long term preservation method. To prevent the sugars burning, evaporation is usually done at 55-60°C. In the water-date fruits system, Emile[19] reported that in order to obtain a maximum extraction rate, the following optimal conditions were required: process temperature 65-70 ° C, using a higher temperature may burn the sugars; using a ratio of water to dates 3 (weight-to-weight), exceeded this ratio the energy needed for evaporation may compensate further sugars extraction; the duration of the procedure is 30 minutes, after which the increase in the amount of extract is insignificant. The sugars extraction efficiency from DF is substantial[20] and the water-to-date fruits ratio is an essential factor in solids extraction, from an economic and technical reasons.[21] Abbès *et al.*,[22] studied the manufacture of DS with high commercial value from second-grade firm texture dates. They noticed that a water-to-date paste ratio of 3:1 gave the most TSS recovery.

Macro Chemical Analysis

The average proximate composition of DS is summarized in Table 1. The most important carbohydrate components are glucose, fructose, small amounts of sucrose and other soluble materials. The concentration of sugars was 67.16 %, which is comparable to that previously obtained.[22] The DS has 34.3% glucose, 32.6% fructose and 0.2% sucrose. After analysis of DS, it has been proven that fructose and glucose constitute the main sugars.[9,23]

Moisture was the second predominant component in DS. The moisture content was 12 %, which is slightly lower than that in date fruits (14.7%). This is due to controlled DS concentration method. Aleid *et al.*,[24] investigated the chemical and physical parameters of DS and the impact of the extraction method on these parameters. They set up that the most effective way to extract TSS from dates is by mixing with a factor of 2.5 the mass of water and autoclaving at 15 psi for 10 minutes. However, a caramelized flavor and darkened color were obtained. Water content highly affects syrups storage stability, as it immediately affects the probability of unwanted fermentation.[24]

Protein in DS was 0.9 g/ 100g which is in agreement with the data previously obtained.[25,26] The DS from three date varieties (Deglet Nour, Allig and Kentichi) that were previously prepared showed protein content in the range from 1.0 to 1.5 g/100 g.[22] It can be noticed that the concentration of protein is about 2.5 times in date fruits than in DS.

In general, DF contain 8.1-12.7% total dietary fiber, with the insoluble fiber accounting for 84-94% and the soluble one accounting for 6-16%.[27] The results presented in Table 1 indicate that DPs are a rich source of carbohydrates and, in particular, significant amounts of fiber which may have potential health benefits. Moreover, DP had a substantial amount of fat Table 1. DPs contain about 15% fiber, characterized by a high rate of water-insoluble mannan fiber.[27] There was no crude fiber or dietary fiber presence in the DS as shown in (Table 1). The findings of this research are in agreement with the findings acquired by other researchers.[26]

Minerals Contents

Mineral content in DS indicates that it is a rich source of macronutrients like potassium, phosphorus, magnesium, calcium and sodium While its content of iron, zinc, manganese and copper is low Table 1. Potassium concentration was the highest (432 mg/100g), followed in descending order by magnesium (48.2 mg/100g), phosphorus (52.5

mg/100g), calcium (21.8 mg/100g), sodium (12.10 mg/100g) and the syrup contains traces of iron, zinc, manganese and copper and which is in agreement with the data on the mineral content of syrup previously tested.[9] Moreover, results of many researchers demonstrated the richness of DS in minerals.[22,25,28,29,30] Results of previous studies indicated that dates, in general, contain large quantities of minerals[31,32,33] and these quantities are estimated to be three to five times higher than the amounts detected in apples, oranges, grapes, and bananas.[34] In addition, dates are suitable for people who suffer from high blood pressure, as they contain a high percentage of potassium and low percentage of sodium.[35] Dates also contain elements associated to healthy bone growth and energy metabolism, such as calcium and magnesium, in addition to the benefits of dates in the production of red blood cells due to the presence of iron.[36,17] The results of chemical analysis show that DP is richer than DF and DS, not only in fat, protein, sucrose, crude fiber and dietary fiber, but also in mineral content of phosphorus, magnesium, calcium, sodium and iron Table 1. This makes it an important source of these elements, which can be used in the food industry, as well as for animal feed. Regarding the mineral content of DP, the following minerals were observed in descending order: potassium, phosphorus, magnesium, calcium, sodium, iron, zinc, manganese and copper Table 1.

Table 1: *Chemical analysis of date fruit, syrup and pits

Macro Component (g/100 g)	**Date fruit**	**Date Syrup**	**Date pits**
Moisture	14.7±0.01	12.0±0.11	4.28±0.21
Fat	0.10±0.01	0.06±0.01	5.95±0.11
Protein	2.20 ±0.05	0.90±0.05	4.40±0.22
Glucose	34.7±0.10	34.3±0.71	0.69±0.02
Fructose	31.9±0.12	32.6±0.15	0.95±0.03
Sucrose	0.20±0.05	0.20±0.01	2.36±0.02
Crude fiber	2.90±0.08	Nd	24.10±0.21
Dietary fiber	8.2±0.04	Nd	56.09±0.01
Acetic acid	0.30±0.01	0.30±0.02	0.01±0.01
Butyric acid	0.20±0.01	0.20±0.01	0.37±0.03
Propionic acid	0.20±0.02	0.20±0.01	Nd
Micro Component (mg/100 g)	Date fruit	Date Syrup	Date pits
Potassium	495.7±0.46	432.0±0.38	214.19±0.51
Phosphorus	50.30±0.11	48.2±0.08	103.23±0.21
Magnesium	59.80±0.15	52.5±0.41	70.68±0.85
Calcium	38.90±0.11	21.8±0.08	34.25±0.51
Sodium	18.8±0.11	12.10±0.03	9.32±0.07
Iron	1.40±0.01	0.40±0.03	7.14±0.03
Zinc	0.30±0.02	0.20±0.02	1.10±0.01
Manganese	0.52±0.05	0.18±0.01	0.91±0.01
Copper	0.20±0.01	0.08±0.01	0.28±0.02
Total polyphenol (mg GAE / 100g)	1833.52±8.5	1448.65±8.5	2954±9.22
Antioxidant activity (DPPH, IC_{50})	88.23±0.52	62.43±0.77	280±6.54

Nd= not detectable. *All results are means ± SD (n = 3).

Organic Acids Contents

The average organic acid contents (Acetic acid, Butyric acid) in date syrup were 0.50±0.02, 0.20±0.01 and g/100 g, respectively Table 1. Many parameters like climatic and geographic, season, amount of sunlight received, growing conditions, harvest

time, variety, maturity and storage conditions affect the presence and composition not only of organic acids[37] but also other date components.[26] The presence of organic acids as taste-active ingredients can ameliorate the organoleptic properties of DS as well as products to which DS is added. These acids give certain properties to the products in which they are present, such as: sour, sour, acidic, fruity taste. This is in addition to its role in the quality of product storage because of its impact on the growth of microorganisms.

Fatty Acids Profile

The fatty acids present in date's syrup constitute saturated fatty acids, palmitic, stearic and decanoic. It also contains monounsaturated fatty acid, oleic acid (omega-9) and polyunsaturated fatty acid, linoleic Table 2. The saturated fatty acids represent 55.31% of the total fatty acids whereas unsaturated fatty acids represent 44.69 % of which 21.44 % are monounsaturated fatty acids and 23.25 % polyunsaturated fatty acids. The results of chemical analysis show that DP are richer than date fruits and date syrup in alpha-linolenic acid, linoleic acid, and oleic acid. These acids are also known as Omega 3, 6, and 9, respectively. These are polyunsaturated fatty acids that are essential to human nutrition and health, in addition to important nutritional and health benefits to animals Table 2.

Table 2: Relative fatty acid profile (g/100 g total fats) of date fruit, syrup and pits

Fatty acid		Date fruit	Date Syrup	Date pits
Saturated fatty acids (g/100 g total fats)				
Palmitic	$C_{16}H_{32}O_2$	27.65±0.04	32.38±0.05	16.81±0.05
Stearic	$C_{18}H_{36}O_2$	3.94±0.04	22.00±0.08	6.07±0.02
Docosanoic (Behenic)	$C_{22}H_{44}O_2$	3.02±0.01	Nd	0.52±0.02
Pentadecanoic	$C_{15}H_{30}O_2$	1.13±0.03	Nd	Nd
Heptadecanoic (Margaric)	$C_{17}H_{34}O_2$	1.00±0.04	Nd	Nd
Myristic	$C_{14}H_{28}O_2$	0.90±0.02	Nd	10.14±0.06
Tetracosanoic (Lignoceric)	$C_{24}H_{48}O_2$	0.50±0.01	Nd	0.29±0.01
Undecanoic	$C_{11}H_{22}O_2$	0.93±0.01	Nd	Nd
Lauric	$C_{12}H_{24}O_2$	0.44±0.02	Nd	14.13±0.05
Eicosanoic (Arachidic)	$C_{20}H_{40}O_2$	0.42±0.02	Nd	0.43±0.02
Tricosanoic	$C_{23}H_{46}O_2$	0.21±0.03	Nd	Nd
Caprylic	$C_8H_{16}O_2$	Nd	Nd	0.15±0.03
Capric	$C_{10}H_{20}O_2$	0.16±0.01	Nd	0.24±0.01
Total*		**39.37±0.10**	**55.31±0.11**	**48.78±0.21**
Unsaturated fatty acids (g/100 g total fats)				
Oleic	$C_{18}H_{34}O_2$	28.16±0.12	21.44±0.07	41.48±0.10
Linoleic	$C_{18}H_{32}O_2$	27.08±0.20	23.25±0.21	8.08±0.09
Linolenic	$C_{18}H_{32}O_2$	4.97±0.05	Nd	0.52±0.02
Palmitoleic	$C_{16}H_{32}O_2$	0.42±0.01	Nd	0.19±0.01
Myristoleic	$C_{14}H_{26}O_2$	Nd	Nd	0.09±0.01
13-Docosenoic (Erucic)	$C_{22}H_{42}O_2$	Nd	Nd	0.35±0.02
6,9,12,15-Docosatetraenoic	$C_{22}H_{36}O_2$	Nd	Nd	0.51±0.03
Total*		**60.63±0.19**	**44.69±0.11**	**51.22±0.17**

*The total fatty acid content was calculated as the sum of all individual fatty acids, and the relative abundance of each fatty acid was calculated by dividing the concentration of each individual fatty acid by the total fatty acid content. All measurements were run in triplicate and results are indicated as means ± Standard Deviation (n = 3).

Amino Acids Composition

The analysis of amino acids content of DS indicated the presence of fifteen amino acids Table 3. The amino acids profile of DS showed a moderate amount of glutamic and aspartic acids and low concentrations of arginine, tyrosine, histidine, alanine, glycine, valine serine, isoleucine, leucine, threonine, methionine and lysine. Moreover, the analysis did not detect the presence of phenylalanine. It was found that the DS contains certain amount of essential amino acids (lysine: 0.08, isoleucine 0.06 and threonine 0.09 % w/w). It is noticeable that the concentration of amino acids in the DF is more than in the DS Table 3. Amino acid profiles revealed that DS includes most of the essential amino acids: leucine, isoleucine, lysine, methionine, threonine, valine and histidine. These results were in agreement with that previously reported.[38,39] The essential amino acid Threonine was detected and identified in the DP besides Alanine, Aspartic, Glutamic, Glycine and Serine. The glutamic acid was the prevailing amino acid Table 3. In previous study, 17 types of amino acids were revealed and specified in DP. The glutamic acid was the dominant amino acid. The essential amino acid threonine was present in the DP.[40]

Table 3: Amino acid composition (% w/w) of date fruit, syrup and pits

Amino acid (g/100 g protein)	Date fruit	Date Syrup	Date Pits
Alanine	0.09± 0.01	0.06± 0.01	0.01± 0.03
Aspartic	0.25± 0.01	0.10± 0.03	0.05± 0.04
Glutamic	0.53± 0.01	0.13± 0.03	0.07± 0.02
Glycine	0.10± 0.01	0.05± 0.04	0.02± 0.01
Serine	0.11± 0.03	0.05± 0.01	0.02± 0.01
Arginine	0.11± 0.08	0.09± 0.02	Nd
Histidine	0.07± 0.02	0.07± 0.03	Nd
Isoleucine	0.06± 0.01	0.04± 0.03	Nd
Leucine	0.14± 0.03	0.04± 0.02	Nd
Lysine	0.08± 0.01	0.02± 0.01	Nd
Methionine	0.04± 0.01	0.02± 0.01	Nd
Phenylalanine	0.11± 0.01	Nd	Nd
Threonine	0.09± 0.01	0.03± 0.01	0.01± 0.02
Tyrosine	0.10± 0.04	0.07± 0.03	Nd
Valine	0.16± 0.03	0.05± 0.05	Nd
Total amino acids	2.04± 0.05	0.80± 0.06	0.17± 0.08

* All measurements were run in triplicate and results are presented as means ± Standard Deviation (n = 3).

Total Phenolic Content and Antioxidant Activity

The total polyphenol compositions were 1833.52 and 1448.65.5mg GAE /100 g in DF and DS, respectively Table 1. Al-Farsi *et al.*,[26] showed that the total phenolic compositions varies from 172 to 246 mg GAE/100 g in three types of Omani sun-dried local dates. While, Wu *et al.*,.[41] indicated that Deglet Nour and Medjol varieties contained 661 and 572 mg GAE/100 g, respectively. However, Mansouri *et al.*,[42] indicated much lower contents (2.49-8.36 mg GAE/100 g) of phenolic profile of several Algerian date varieties.

The result of the analysis of total polyphenol and Antioxidant activity in DP were 2954±9.22 mg/100 g GAE and 280±6.54 DPPH, IC50, respectively Table 1. These results were lower than those obtained by previous studies, total phenolic in DS from three varieties (Mabseeli, Um-sellah, and Shahal) were 4430 ± 0.297, 4293 ± 0.180, and 3102 ± 0.58 mg GAE/100 g, respectively.[26]

Differences in the total phenolic compositions may be due to various parameters such as climatic and geographical factors, season, soil type, type of dates,

handling practices, growing conditions, maturity etc. From the study results, DS appears to have potential antioxidant activity Table 1. These findings are consistent with those previously reported.[17,42] Date pits can be considered as an inexpensive rich source for phenols with an antioxidant potency. as well as a source for essential minerals and fatty acids. Such biologically active components in DP can provide an added value to food industry.[43]

Conclusions

The current research confirmed that DS and DP from Khalas cultivar have a potential functional and nutritional property, in addition to being a good source of natural antioxidants and minerals. Due to the nutritional and healthy added value of dates and their various products, these products are of interest to consumers in almost all date-producing countries. Thus, the development of food products from dates is the focus of the food industry in those countries, which will help in achieving sustainable development and food security, especially with the great challenges facing the global food industry. According to the results obtained from this research, obviously, the method used to extract DS preserves to a large extent the chemical properties of DF, and this is very important in all nutritional, functional and health aspects of DS, which will make its uses in various food industries on a large scale imperative. Based on the observed chemical properties of DP, they can be considered as an important by-product from date industry and hence can be applied as functional food or functional food ingredient. Moreover, they can be used as a good nutritional and healthy choice in animal feed manufacturing. Since DP contain large amounts of sugars, dietary fiber, oils and protein; they also contain bioactive phenolic compounds, which gives them pharmacological properties.

Authors' contributions

Salah Mohammed Aleid- Conception and design of the study, Data curation, Investigation, Methodology, Writing – original draft, Visualization. Jamal Saleh Haddadin-Conception and design of the study, Data curation, Investigation, Methodology, Writing – original draft, Visualization.

Acknowledgments

The authors would like to thank Department of Food and Nutrition Sciences, King Faisal University, Alahsa, Saudi Arabia, and the University of Lorraine-LRGP, France, for the technical and scientific advices.

Funding

The authors would like to thank the Date Palm Research Center of Excellence, King Faisal University, Saudi Arabia, for the financial support to carry out this work.

Conflict of Interest

The authors confirm that there is no conflict of interest related to the manuscript.

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