



Characterization analysis of date fruit pomace: An underutilized waste bioresource rich in dietary fiber and phenolic antioxidants

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ARTICLE INFO

Keywords:

Date fruit pomace
Waste management
Characterization
Total dietary fiber
Phenolic compounds
Antioxidants

ABSTRACT

Date fruit pomace (DFP), an abundant byproduct of the date syrup industry, is currently underutilized. It is either fed to animals or landfilled where it ferments, causing serious environmental issues. Proper waste management through valorization strategies is required to reduce the environmental impact of date waste and foster the transition of the date processing industry towards sustainability. The implementation of valorization strategies requires characterizing the DFP composition. Herein, four DFP samples of Emirati varieties were characterized in terms of compositional and functional properties. The fresh DFPs contained moisture (~60.0 g/100 g). On a dry matter basis, DFPs contained total dietary fiber (~45.5 g/100 g), residual sugars (~35.3 g/100 g), protein (~10.6 g/100 g), ash (~4.1 g/100 g), and fat (~1.3 g/100 g). They were also rich in nutritionally important elements such as potassium (~145.7 ppm), calcium (~46 ppm), phosphorus (~28.7 ppm), and magnesium (~26.7 ppm). The total phenolic content varied from 170 to 260 mg gallic acid equivalents GAE/100 g, making DFP a good source of antioxidants. Scanning electron microscopy measurements revealed the presence of fibrous bundle-like structures, whose thermal stability was confirmed by thermogravimetric analysis, showing the dominance of insoluble over soluble fiber. Higher water- and oil-holding capacities were found for dried desugared pomace. The high fiber, phenolic, and elemental content highlight the high nutritional value of DFP, which can be used as dietary supplement, while its sugar content can be used to produce value-added biochemicals via fermentation. Thus, DFP may represent a valuable bioresource for food and non-food applications.

1. Introduction

Industries that process food such as fruit and vegetables generate large amounts of waste in form of peels, seeds, and pomace, which when not managed properly can cause environmental problems (Joshi, 2020). As these waste sources are rich in critical bioactive molecules such as proteins, minerals and phenolic compounds (Arun et al., 2022), a sustainable green approach for their utilization via conversion to value-added products would not only improve the economic efficiency of the industry, but also help to reduce the pollution caused by the disposal of such waste. Environmental law has greatly supported the adoption of sustainable waste management strategies. Among the latter, the top priority is represented by waste minimization, which includes waste prevention (i.e., waste reduction through the adoption of more efficient production technologies), internal recycling of production waste,

source-oriented waste quality improvement (e.g., hazardous substance replacement), and re-use of products or parts of products for the same or other purposes (Joshi, 2020). In United Arab Emirates (UAE), these challenges are being addressed by recycling and transforming waste into energy and value-added resources, as well as by adopting new technologies or enhanced waste separation and collection methods (UAE, 2021). The wine industry is among the food-related activities that produce a significant amount of waste worldwide (Jin et al., 2018). However, in the Middle East region, including UAE, date syrup industries are the major food processing industries. Date palm (*Phoenix dactylifera* L.) is one of the main crops that play important roles not only as a source of nutrients, but also for cultural identity and economic growth. The date fruit is very valuable from a nutritional point of view, being rich in dietary fiber (5.3–8.2 g/100 g) and in a wide variety of phenolic antioxidants (George et al., 2020; Alam et al., 2021). On an industrial scale,

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<https://doi.org/10.1016/j.wasman.2023.03.027>

Received 11 January 2022; Received in revised form 27 February 2023; Accepted 21 March 2023

Available online 29 March 2023

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the main date fruit-based products are date syrup and paste; however, these products are not sufficient to absorb the large production of date fruit in the Middle East region, which leads to economic losses. Thus, there is an increasing demand for adding value to date fruits and increasing their industrial use, for example, by valorizing certain varieties for fresh consumption, increasing the utilization of lower-quality fruits, and/or making better use of the secondary products of date syrup and paste processing (Kamal-Eldin et al., 2012; Ghnimi et al., 2017).

Date honey or syrup (also called dibs in the Middle East) is extracted from the fruits using hot water, leaving behind two types of industrial waste: the seeds and the fibrous material of the fruit pulp, named date fruit pomace (DFP) or date press cake (Heidarinejad et al., 2018). According to Al-Farsi et al. (2007), date syrup production yields approximately 52–64 g/100 g syrup, 17–24 g/100 g DFP, and 8–15 g/100 g seeds, on a dry matter basis. Currently, the large amount of DFP waste is either fed to animals or dumped into landfills, where its fermentation causes serious waste management issues (Oladzad et al., 2021). DFP, which consists of the exhausted date flesh with some residual sugars, is currently considered as a waste, rather than a resource (Barreveld, 2013). In UAE, the Al Baraka Dates Company (Dubai) produces approximately 1560 tons of DFP annually (Al-Farsi et al., 2019); with seven such factories in the country, DFP could be potentially used as a large-scale feedstock for making value-added products.

Currently, these byproducts are only used in a few applications, such as animal feed (Chandrasekaran and Bahkali, 2013). The water footprint associated with such waste is also enormous. Bazrafshan et al. (2020) have reported that in Iran the date palm has a water footprint volume of approximately 2437 million m³/year, of which soft and dry date palms account for 1949 and 59.7 million m³/year, respectively. Hence, if the large volume of residues produced by the date processing industry is not efficiently valorized, an indirect wastage of water and arable land can result. Furthermore, the uncontrolled decomposition of date waste can cause serious health and environmental issues through the generation of leachate and odor emissions and increases the risk of disease transmission by facilitating the growth of bacteria and the occurrence of pests and mice (Cheok et al., 2018). Proper waste management and disposal strategies are required to reduce the environmental impact of date waste and promote the transition of the date processing industry towards sustainability.

A recent review (Oladzad et al., 2021) on date fruit processing waste discussed various DFP valorization approaches, highlighting the need to conduct detailed characterization studies of pomace from date fruit varieties. The objective of this study was to obtain a full characterization of the physicochemical properties of DFP of Emirati varieties, in order to identify suitable valorization strategies such as the recovery and utilization of its high content of dietary fiber and phenolic compounds. Chemical characterization of the DFP samples was performed by analyzing their composition in terms of moisture content, soluble sugars, ash, total solids, protein, fat, fiber, pectin, and phenolic compounds. Pectin is a structural heterogeneous complex polysaccharide found in the cell walls of fruits and vegetables (Chua et al., 2020). Owing to its gelling properties, pectin is used as a thickening, stabilizing, and emulsifying agent in the food industry (Mohamed et al., 2020). Pectin is also applied as biomaterial for tissue engineering (Cui et al., 2020), drug delivery compound (Sun et al., 2020), and nutraceutical antitumor agent (Naqash et al., 2017; Reichembach and Petkowicz, 2021). These applications highlight the need to explore various alternative sources of pectin. The morphology of the DFP samples was studied using scanning electron microscopy (SEM) and their thermal characteristics were measured by thermogravimetric analysis. The total antioxidant activity of the DFPs was determined using the Folin–Ciocalteu, 2,2'-azino-bis-3-ethylbenzothiazoline-6-sulfonic acid (ABTS^{•+}), ferric ion reducing antioxidant power assay (FRAP), and 2,2-diphenyl-1-picrylhydrazyl (DPPH) methods.

2. Experimental section

2.1. Materials

2.1.1. Date fruit pomace samples

The DFP samples analyzed in this study were obtained from the Al Foah date processing industry (Al Saad, Abu Dhabi Emirate, UAE) as a by-product of date syrup (dibs) production. In the industry, date fruits were washed, mixed with water (1:3, w/w), and heated at 90 °C for one hour to extract the syrup. The DFP was separated from the syrup via industrial filtration. We characterized DFP samples from three single date varieties (namely Kastawi, Khenazi, and Lulu) and a sample of mixed varieties (Khalas, Sayer, Lulu, and Kastawi). Prior to analysis, the DFP samples were stored in a freezer at −18 °C.

2.1.2. Chemicals and reagents

Folin–Ciocalteu phenol reagent, ABTS^{•+}, DPPH, ferric chloride (FeCl₃), 2,4,6-tris(2-pyridyl)-s-triazine (TPTZ), potassium persulfate (K₂S₂O₈), acetate buffer, sodium carbonate (Na₂CO₃), sodium hydroxide (NaOH), potassium bromide (KBr), citric acid, gallic acid, and 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) were purchased from Sigma Chemical Company (St. Louis, MO, USA). Analytical-grade methanol, ethanol, ethyl acetate, petroleum ether (boiling point 40–60 °C), sulfuric acid, and nitric acid were purchased from Honeywell (Seelze, Hanover, Germany).

2.2. Characterization analyses

2.2.1. Pomace color and morphology

The color of the DFP samples was measured using a Hunter Lab colorimeter (Hunter Association laboratory, Inc., Reston, VA, USA). The instrument was calibrated with black and white standard tiles before each experiment. The results were expressed as Hunter color parameters *L*^{*}, *a*^{*}, and *b*^{*}, where *L*^{*} ranges from 0 (black) to +100 (white), *a*^{*} from −100 (green) to +100 (red), and *b*^{*} from −100 (blue) to +100 (yellow) (Manickavasagan et al., 2015). Chroma (*C*^{*}), indicating color intensity, and hue angle (*h*_{ab}) were calculated from the *L*^{*}, *a*^{*} and *b*^{*} values using equations (1) and (2) respectively. The hue angle values range from 0° (pure red color), 90° (pure yellow color), 180° (pure green color) to 270° (pure blue color). To interpret the color difference between the DFP varieties, the total color change, Δ*E*, is calculated using equation (3). To calculate Δ*E*, the Lulu DFP was taken as reference, as it presented the darkest shade with the lowest *L* value among the DFP varieties tested.

$$\text{Chroma}(C^*) = \sqrt{a^{*2} + b^{*2}} \quad (1)$$

$$\text{Hue angle}(h_{ab}) = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (2)$$

$$\text{Colour change } \Delta E = \sqrt{(L_0 - L)^2 + (a_0 - a)^2 + (b_0 - b)^2} \quad (3)$$

where *L*₀, *a*₀ and *b*₀ are the color values of DFP from Lulu variety.

Morphological analyses were carried out on all the pomace varieties with a scanning electron microscope (JSM-6610 JEOL PLUS / LA, Tokyo, Japan), operated at 10 kV and a working distance of 20 mm in high vacuum mode (Suresh et al., 2013). All samples were placed on adhesive carbon tape, fixed in the SEM stub, and coated with gold in a sputter coater under vacuum in the presence of inert gas. The gold-coated samples were then observed under high vacuum conditions.

2.2.2. Analysis of proximate components and major elements

Total solids and ash content determinations were performed according to the National Renewable Energy Laboratory (NREL) protocols (NREL, 2020). The total solid content was determined at a temperature of 105 °C overnight, whereas the ash content was determined in a muffle

furnace at 575 °C for 3 h (Sluiter et al., 2008). The protein content was analyzed using the Kjeldahl method with a nitrogen-to-protein conversion factor of 6.25 (AOAC, 2000), whereas the fat content was analyzed in an automated Soxhlet extractor with petroleum ether as solvent (Shea et al., 2015). Major elements such as potassium, calcium, magnesium, sodium, phosphorus, cadmium, cobalt, chromium, copper, iron, manganese, nickel, lead, and zinc were also determined. For this analysis, the samples were ashed at 575 °C for 3 h; then, the ash was dissolved using an aqua regia solution [3:1 (v/v) concentrated HCl to concentrated HNO₃] and the main elements were determined directly from the solution. The analysis was carried out by inductively coupled plasma optical emission spectrometry (ICP-OES, Varian 710-ES, CA, USA), as discussed by Ajaj et al. (2018).

2.2.3. High-performance liquid chromatography analysis of soluble sugars

Sugars were extracted by mixing DFP (25 g) with water (50 mL) with continuous stirring at 80 °C for 2 h, followed by centrifugation at 4500 rpm for 15 min (Rabetafika et al., 2013). The supernatant was removed, and the remaining solids were subjected to four consecutive sugar extraction steps. The supernatant from each extraction was filtered using 0.2 µm syringe filter and analyzed for glucose, fructose, and sucrose using high performance liquid chromatography (HPLC). HPLC analysis of the samples was performed with a Dionex Ultimate 3000 UHPLC instrument (ThermoScientific, Kansas, USA) using a Rezex RCM Monosaccharide LC column [Ca²⁺ (8%), 300 × 7.8 mm, Phenomenex, California, USA] and Millipore water as mobile phase at a flow rate of 0.6 mL/min. The sugar concentrations were determined using a refractive index detector (ERC RefractoMax 520, ERC, Inc., Japan) at 75 °C.

2.2.4. Determination of total dietary fiber and pectin extraction

Fiber extraction from date fruit was performed following the method of Ali et al. (2022). Date fruit pomace fiber was recovered via removal of the sugars with hot water (50 mL) (100 °C for 15 min, five times), followed by centrifugation (6500 g, 15 min). The residue obtained was air-dried and stored as fiber concentrate or desugared DFP. The content of crude protein (obtained as total nitrogen content × 6.25) and ash in the desugared DFP sample were determined by the AOAC (2000) method and used to correct the fiber content of the DFP. Dietary fiber was expressed as grams per 100 g of DFP on a dry matter basis.

To determine the pectin content of DFP, we used ultrasonic-aided extraction, as described by Chua et al. (2020). Fresh and dried (60 °C, overnight) DFP samples of each variety were mixed with 0.1 M citric acid (in a ratio of 1:20, w/v); then, the mixture was heated in an ultrasonic water bath at 65 °C for 25 min, cooled, and subjected to vacuum filtration using Whatman No 1 filter paper. The obtained filtrate was mixed with 95% ethanol employing a filtrate to ethanol ratio of 1:2 (v/v), kept at room temperature for 120 min, and centrifuged to achieve the precipitation of pectin. After centrifugation, the supernatant was removed, and the pellet was rinsed three times with 95% ethanol. After drying at 50 °C overnight, the yield of pectin was determined gravimetrically using Equation (4):

$$\text{Pectin yield(\%)} = \frac{\text{weight of dried pectin(g)}}{\text{weight of DFP(g)}} \times 100 \quad (4)$$

Fourier Transform infrared spectroscopy (FTIR) analysis of the DFP and extracted pectin samples was performed using a Spectrum Two™ spectrometer (Perkin-Elmer, Massachusetts, USA) in transmission mode, using KBr pastilles prepared with 0.8 mg sample and 80 mg KBr (Varma and Mondal, 2016). The spectra were obtained in the spectral range of 400 to 4000 cm⁻¹ using the Spectrum software.

2.2.5. Determination of total antioxidant activity

Soluble and hydrolyzable phenolic compounds were extracted from DFP following the method of Alam et al. (2021). Soluble phenols were extracted with a methanol solution (80% v/v), while hydrolyzable phenols were extracted from the pellet remaining after centrifugation

Table 1

Color parameters of the analyzed date fruit pomace samples.

Color parameter	Kastawi	Khenaizi	Lulu	Mixed	Mean*
<i>L</i>	24.7 ± 0.01 ^c	27.9 ± 0.01 ^a	21.6 ± 0.05 ^d	26.5 ± 0.01 ^b	25.2 ± 2.7 ^c
<i>A</i>	8.3 ± 0.02 ^{cd}	10.7 ± 0.01 ^b	7.9 ± 0.02 ^d	11.2 ± 0.02 ^a	9.5 ± 1.7 ^c
<i>B</i>	14.7 ± 0.04 ^c	17.8 ± 0.03 ^a	15.0 ± 0.0 ^c	16.7 ± 0.01 ^b	16.1 ± 1.5 ^b
<i>C*</i>	16.9 ± 0.03 ^c	20.8 ± 0.02 ^a	16.9 ± 0.01 ^c	20.2 ± 0.01 ^a	18.7 ± 1.4 ^b
Hue angle (°)	60.9 ± 0.03 ^b	59.5 ± 0.02 ^c	62.2 ± 0.01 ^a	56.3 ± 0.01 ^d	59.7 ± 1.4 ^c

L-brightness; a-redness, b-yellowness; C*-color intensity. *All values represent the mean ± SD of three determinations per variety. Mean* represents the mean ± SD value of the four samples (SD: standard deviation). The values with different superscript letters in each row are statistically different (P < 0.05).

with methanol, by treatment with 3 M sodium hydroxide using ethyl acetate. The total phenolic content and antioxidant activities were determined using Folin-Ciocalteu, ABTS⁺, FRAP, and DPPH methods (Alam et al., 2021). The Folin-Ciocalteu total phenolic content was calculated as milligrams of gallic acid equivalents (mg GAE) per 100 g sample, while the ABTS⁺ and FRAP antioxidant activities were expressed as micromoles of Trolox equivalents (µmol TE) per 100 g sample. The radical scavenging activity obtained by the DPPH method was expressed as the half-maximal inhibitory concentration (IC₅₀, in mg/mL).

2.2.6. Thermogravimetric analysis

Thermogravimetric analysis (TGA) was performed using a Netzsch STA409 EP (Selb, Germany) instrument. Approximately 8 mg of samples were heated in a TGA pan from 20 to 800 °C under a nitrogen atmosphere at a heating rate of 10 °C min⁻¹. Differential thermogravimetry (DTG) curves were obtained from the first derivative of the weight loss rate (Rabetafika et al., 2013).

2.2.7. Water- and oil-holding capacities

The water-holding (WHC) and oil-holding (OHC) capacities were determined using a modified method described by Rabetafika et al. (2013) for both the dried whole pomace and dried desugared pomace varieties. To determine the WHC, 1 g of sample was stirred with 15 mL of distilled water in a 50 mL centrifuge tube that was left overnight at room temperature. Then, the mixture was centrifuged at 4500 rpm for 15 min. The supernatant was discarded and the absorbed water (final residue) weighted. The WHC was calculated using Equation (5) and expressed as grams of water per gram of dry powder. For the OHC determination, 5 g of sample were added to 30 mL of sunflower oil in a 50 mL centrifuge tube. The content was left overnight at room temperature; after that, the tubes were centrifuged at 4500 rpm for 15 min. The supernatant was decanted, and the absorbed oil (final residue) was weighted. The OHC was calculated using Equation (6) and expressed as grams of oil per gram of dry powder (Rabetafika et al., 2013).

$$\text{WHC} = \frac{\text{Final residue weight} - \text{Initial sample weight}}{\text{Initial sample weight}} \quad (5)$$

$$\text{OHC} = \frac{\text{Final residue weight} - \text{Initial sample weight}}{\text{Initial sample weight}} \quad (6)$$

2.2.8. Statistical analysis

All analyses were performed in triplicate and the results were expressed as mean ± standard deviation. The physicochemical properties of each of the DFP variety studied were analyzed and compared using one-way ANOVA and Tukey's comparison method with 95% confidence with the SPSS 23.0 software (SPSS Inc., Chicago, IL, USA).

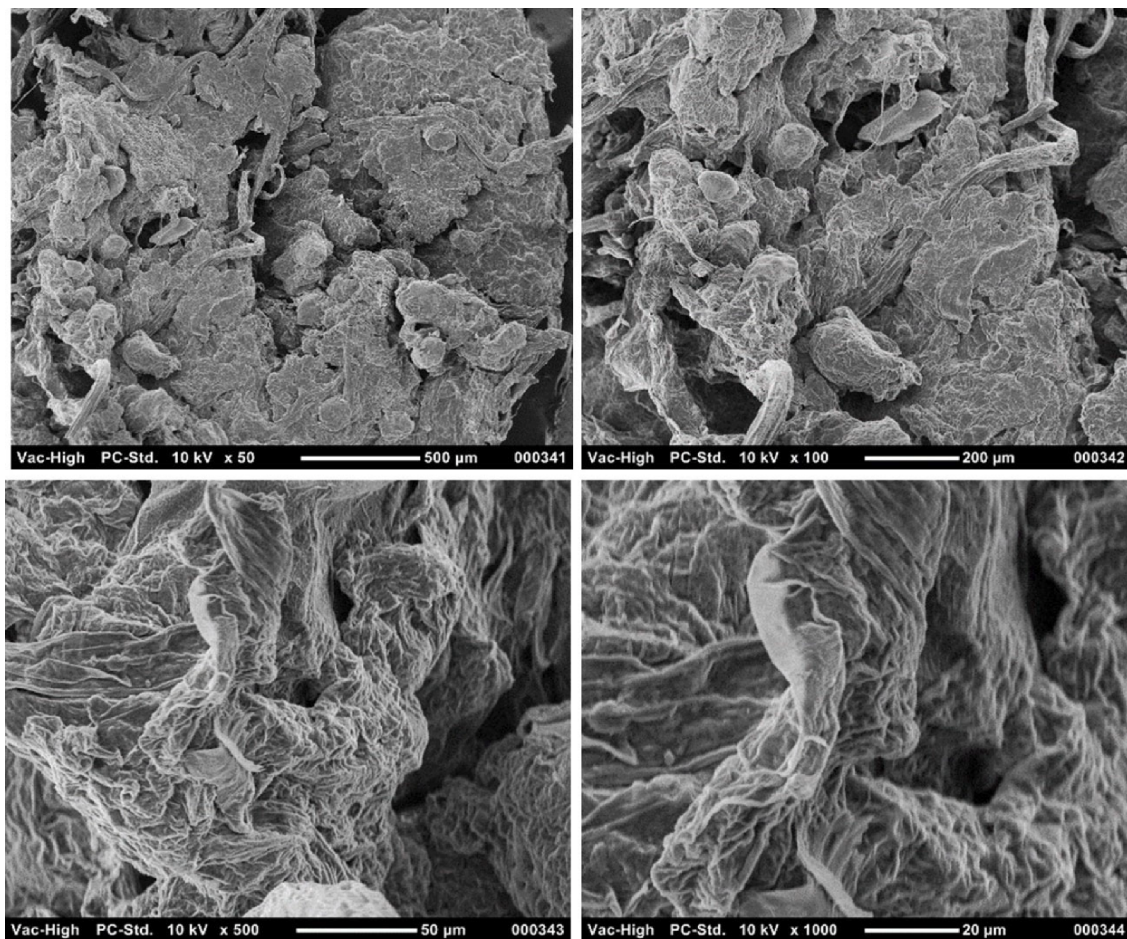


Fig. 1. Scanning electron microscopy images showing morphology of date fruit pomace (DFP) at 50, 100, 500, and 1000 $\times$ magnifications. Lulu DFP is presented as a typical example.

Differences were considered significant for $p \leq 0.05$.

3. Results and discussion

3.1. Pomace color and morphology

The studied DFP samples mainly consisted of the fruit pulp and skin. Their color values are given in Table 1. The mean color values (L^* , a^* , b^* , C^* , hue angle) obtained for all the studied samples were (i.e. 25.2, 9.5, 16.1, 18.7 and 59.7 $^\circ$, respectively), were lower than those reported by Majzoobi et al. (2019) (L^* , a^* , b^* values of 70.62, 14.25 and 36.5), which may be due to differences in the color of the date fruits and/or the processing conditions of the date syrup. The color values indicate that all the samples were brownish in color because of the pigments present in the date fruit flesh. In reference to the DFP of the Lulu variety, the ΔE values of Kastawi, Khenazi, and mixed DFP were 3.2, 7.5 and 6.1 respectively. The highest ΔE value resulted for Khenazi and the lowest for Kastawi, indicating that Khenazi had the lightest color among the four samples.

Similar SEM images were obtained for all the studied samples. As all the samples studied had a similar morphology, only the image of the Lulu variety, selected as a representative sample, is provided. The SEM image of Lulu pomace (Fig. 1), at lower magnification shows a clump-like formation mainly because of the sugars in the DFP and at higher magnification, bundle-like fibrous structure can be seen, consistent with the reported predominance of insoluble dietary fiber in dates (George et al., 2020; Kamal-Eldin et al., 2020). The SEM image shows that the analyzed DFP samples mainly consisted of flesh of the date fruit.

Table 2

Composition of the analyzed date fruit pomace varieties.

Component (g/100 g)	Kastawi	Khenazi	Lulu	Mixed	Mean*
Ash	3.5 $\pm$ 0.1 ^d	4.6 $\pm$ 0.03 ^b	3.5 $\pm$ 2.1 ^d	4.8 $\pm$ 0.1 ^a	4.1 $\pm$ 0.7 ^c
Protein	10.5 $\pm$ 0.4 ^b	9.0 $\pm$ 0.0 ^d	13.0 $\pm$ 1.7 ^a	10.0 $\pm$ 3.4 ^c	10.6 $\pm$ 1.7 ^b
Fat	n.d.	0.02 $\pm$ 5.1 ^c	0.02 $\pm$ 1.0 ^c	5.3 $\pm$ 2.0 ^a	1.3 $\pm$ 3.0 ^b
Total sugar	33.5 $\pm$ 4.8 ^d	40.7 $\pm$ 8.5 ^a	38.5 $\pm$ 2.4 ^b	28.5 $\pm$ 6.0 ^e	35.3 $\pm$ 5.4 ^c
Glucose	13 $\pm$ 1.9 ^d	18.0 $\pm$ 4.6 ^a	15.2 $\pm$ 0.6 ^b	12.1 $\pm$ 2 ^c	14.6 $\pm$ 2.6 ^c
Fructose	20.5 $\pm$ 2.4 ^c	22.5 $\pm$ 0.9 ^b	23.2 $\pm$ 0.9 ^a	16.2 $\pm$ 0.9 ^d	20.6 $\pm$ 1.5 ^c
Sucrose	n.d.	0.2 $\pm$ 0.1 ^a	0.1 $\pm$ 0.5 ^b	0.2 $\pm$ 0.07 ^a	0.1 $\pm$ 0.06 ^b
Total dietary fiber	49.0 $\pm$ 1.1 ^a	42.0 $\pm$ 1.8	43.5 $\pm$ 5.2 ^d	47.7 $\pm$ 5.2 ^b	45.5 $\pm$ 3.3 ^c
Pectin	3.5 $\pm$ 1.1 ^b	3.7 $\pm$ 1.4 ^a	1.4 $\pm$ 0.6 ^d	3.7 $\pm$ 1.4 ^a	3.2 $\pm$ 1.1 ^c

* All values represent the mean $\pm$ SD of three determinations per variety. Mean* represents the mean $\pm$ SD of the four samples (SD: standard deviation; n.d: not detected). The values with different superscript letters in each row are statistically different ($P < 0.05$). All values are reported on a dry matter basis.

Table 3

Major elements in the analyzed date fruit pomace varieties.

Elements (ppm)	Kastawi	Khenaizi	Lulu	Mixed	Mean*
Ca	49 ± 1.4 ^a	44.7 ± 1.5 ^{bc}	47.5 ± 4.3 ^{ab}	42.5 ± 1.4 ^c	46.0 ± 2.9 ^b
K	152.7 ± 9.2 ^b	196.7 ± 12.6 ^a	120.7 ± 6.2 ^d	112.2 ± 7.5 ^c	145.7 ± 38.3 ^c
Mg	24.5 ± 2.2 ^c	29.0 ± 2.2 ^a	28.2 ± 1.5 ^{ab}	25.2 ± 3.7 ^{bc}	26.7 ± 2.2 ^b
Na	7.5 ± 2.1 ^c	10.7 ± 2.6 ^a	10.7 ± 2.1 ^a	4.2 ± 2.1 ^d	8.2 ± 3.1 ^b
P	33.2 ± 2.5 ^a	29.0 ± 3.8 ^b	27.2 ± 2.6 ^{bc}	26.0 ± 4.5 ^c	28.7 ± 3.2 ^b
Cd	<0.002 ^a	<0.002 ^a	<0.002 ^a	<0.002 ^a	<0.002 ^a
Co	<0.007 ^a	<0.007 ^a	<0.007 ^a	<0.007 ^a	<0.007 ^a
Cr	0.02 ± 0.1 ^a	0.02 ± 0.1 ^a	0.02 ± 0.08 ^a	0.02 ± 0.1 ^a	0.02 ± 0.0 ^a
Cu	0.5 ± 0.3 ^a	0.2 ± 0.3 ^b	0.2 ± 0.5 ^b	0.2 ± 0.4 ^b	0.2 ± 0.15 ^b
Fe	3.2 ± 0.2 ^a	1.2 ± 0.6 ^d	1.5 ± 0.9 ^c	2.0 ± 0.4 ^b	2.0 ± 0.9 ^b
Mn	0.5 ± 0.1 ^a	0.2 ± 0.2 ^b	0.2 ± 0.2 ^b	0.2 ± 0.1 ^b	0.2 ± 0.2 ^b
Ni	0.05 ± 0.1 ^a	0.02 ± 0.2 ^b	0.02 ± 0.1 ^b	0.02 ± 0.1 ^b	0.02 ± 0.02 ^b
Pb	0.05 ± 0.1 ^a	<0.02 ^b	<0.02 ^b	<0.02 ^b	<0.02 ^b
Zn	0.5 ± 0.3 ^a	0.5 ± 0.1 ^a	0.5 ± 0.3 ^a	0.5 ± 0.1 ^a	0.5 ± 0.0 ^a

* All values represent the mean ± SD of three determinations per variety. Mean* represents the mean ± SD of the four samples (SD: standard deviation). The values with different superscript letters in each row are statistically different ($P < 0.05$). All values are reported on a dry matter basis.

3.2. Proximate components and main elements

The moisture content of the DFP samples was of 56.3–63.0 g/100 g. On a dry weight basis, the DFP samples presented the following composition: ash (3.5–4.8 g/100 g), soluble sugars (28.5–40.7 g/100 g), protein (9–13 g/100 g), fat (0.02–5.3 g/100 g), and total dietary fiber (42–49 g/100 g) (Table 2). The residual sugar content of the studied DFP samples was higher than that reported for apple (10.2 g/100 g) and pear (7.6 g/100 g) (Rabetafika et al., 2013), as well as orange (9 g/100 g) (Dong et al., 2016) pomaces. A glucose content (dry basis) of 0.24–62.7 mol % was reported in different varieties of grape pomaces, a byproduct of the winemaking industry (Beres et al., 2017). The studied DFP was within the range of sugar content reported by Antonić et al. (2020) of 12.20–40.53 g/100 g for different grape pomace varieties, with glucose contents ranging from 0.21 to 26.34 g/100 g and fructose contents in the range of 0.38–8.9 g/100 g. Date fruits can be of soft, semi-dry and dry varieties. Moisture content of soft date varieties are more than 30% and primarily store sugars as glucose and fructose, whereas the moisture content of dry varieties are 20% or less and have variable levels of sucrose (Kamal-Eldin et al., 2020). As the DFP varieties analyzed were mainly from the soft type date fruits, the content of sucrose was negligible, and the average ratio of fructose to glucose was 1.4. A higher fructose/glucose ratio was also reported for the DFP of Shahani varieties (Majzoobi et al., 2019). The high moisture and residual sugar content of the DFP samples indicated that the fresh pomace was prone to fermentation and spoilage and had to be stored at low temperature such as -18°C . The preservation of DFP may be achieved by extracting and utilizing the soluble sugars in bacterial fermentation biotechnologies, while drying the dietary fibers and phenolic compounds and using them in functional food applications. The protein content of the studied DFP samples was higher than that reported by Al Farsi et al. (2007) for DFP from date varieties in Oman (3.6–5.2 g/100 g) and that of Shahani variety (6.4 g/100 g) (Majzoobi et al., 2019). The protein content of the studied DFPs was higher than that previously found in orange (0.81 g/100 g) (Dong et al., 2016) and apple (2.4 g/100 g) pomace (Rabetafika

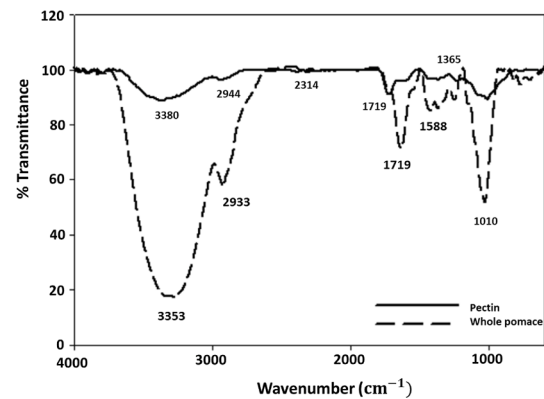


Fig. 2. Typical FTIR spectra of whole date fruit pomace (DFP) and pectin extracted from DFP of Lulu variety (DFP from the other samples showed similar profiles).

et al., 2013). The fat content of the DFP samples was very low, in agreement with previous results reported for DFP from Mabseeli, Um-sellah, and Shahal Omani date fruit varieties (Al-Farsi et al., 2007). A higher fat content (4.9 g/100 g) was reported for DFP with seeds (Majzoobi et al., 2019), which was attributed to the inclusion of the fat-rich seeds (Chandrasekaran and Bahkali, 2013).

The ash content, which reflects the mineral content, was higher than apple pomace (1.6 g/100 g) and orange pomace (3.7 g/100 g) (Shea et al., 2015). The results reported in Table 3 suggest that DFP is a good source of elements such as potassium (~145.7 ppm), calcium (~46 ppm), phosphorus (~28.7 ppm), and magnesium (~26.7 ppm). Other elements such as iron, copper, and manganese were present at lower concentrations, while the levels of chromium, cobalt, lead, and cadmium were negligible, indicating the absence of heavy metal contamination. Al-Hamdani (2019) showed that consuming DFP led to significantly increased levels of blood hemoglobin, which can be attributed to the presence of iron, as well as decreased levels of blood glucose serum and uric acid.

3.3. Dietary fiber

The total dietary fiber content of the studied DFP samples ranged from 42 to 49 g/100 g; Al-Farsi et al. (2007) reported lower levels (25–39 g/100 g) in three laboratory-prepared Omani samples, possibly due to differences in moisture and/or soluble sugar content. The fiber content of the studied DFPs was higher than the 14.3 g/100 g value measured for cabbage waste (Nikmatia and Wijaya Setia Ningrat, 2021), but lower than that reported for apple pomace (51 g/100 g) (Sudha et al., 2007). As observed in the SEM images (Fig. 1), the date fruit dietary fiber was mainly composed of fibrous bundles, consistent with a higher content of insoluble fibers (cellulose, hemicellulose, and lignin) compared to soluble (fructans and pectin) ones (George et al., 2020; Kamal-Eldin et al., 2020). Depending on the variety, date fruit dietary fiber may contain considerable amounts of lignin and of other fiber-linked phenolic compounds (George et al., 2020), which can be differentiated by comparing the results of gravimetric determinations and chromatographic analysis of polysaccharide sugars provided by Ali et al. (2022).

The levels of pectin in DFP depend on the processing conditions and on its partial extractability from the syrup. In this study, 1.4 g/100 g pectin was extracted from the fresh DFP, but the yield increased to 3.2 g/100 g (Table 2) when pectin was extracted from DFP dried at 60°C overnight. The FTIR spectra of the DFP of the Lulu variety, selected as a representative sample, and of the extracted pectin are shown in Fig. 2. The peak at 3353 cm^{-1} corresponded to the $-\text{OH}$ stretching of hydroxyl and carboxyl groups (Petrović et al., 2016), while that at 2933 cm^{-1} was attributed to the C–H bond stretching of hydrocarbon and aldehyde

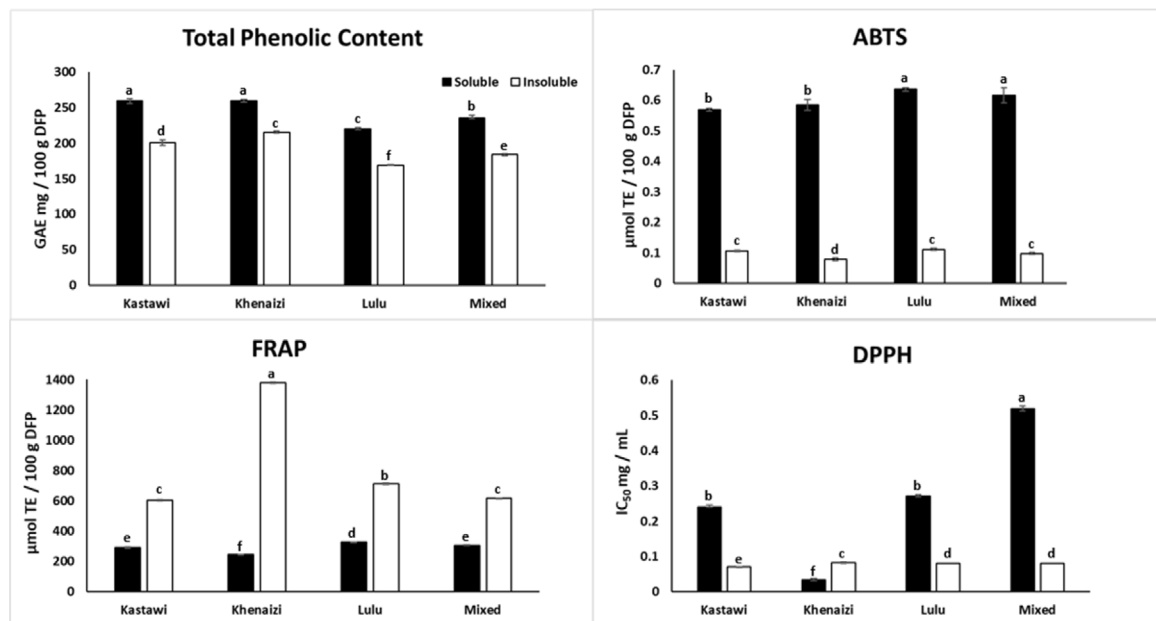


Fig. 3. Total phenolic content and antioxidant activity of Date Fruit Pomace samples, measured by (a) Folin–Ciocalteu, (b) ABTS (2,2'-azino-bis-3-ethylbenzothiazoline-6-sulfonic acid), (c) FRAP (ferric ion reducing antioxidant power assay), and (d) DPPH (2,2-diphenyl-1-picrylhydrazyl) methods. The different superscript letters in the column indicate significant difference ($P < 0.05$). The values are based on dry matter basis.

groups in the sugar backbone (Liu et al., 2011; Kozłowicz et al., 2020); the 1719 cm^{-1} peak corresponded to flexible $\text{C}=\text{O}$ bond vibrations (Liu et al., 2011), while the multiple peaks around 1588 cm^{-1} were due to the $\text{C}-\text{H}$ and $\text{O}-\text{H}$ bending vibrations from glucose and fructose (Bureau et al., 2019); finally, the 1010 cm^{-1} peak was due to the $\text{C}-\text{O}$ and $\text{C}-\text{C}$ stretching of primary alcohols and the $\text{C}-\text{O}-\text{H}$ bending in glucose (Petronić et al., 2016; Bureau et al., 2019). The absence of a distinct peak around 995 cm^{-1} confirms the low concentration of sucrose in the samples (Leopold et al., 2011). The reduction of the intensity of the broad $-\text{OH}$ stretching peak at 3380 cm^{-1} can be attributed to moisture removal during pectin extraction (Sucheta et al., 2020). Furthermore, the peak intensity reduction can also be attributed to intra- and inter-molecular hydrogen bonding of galacturonic acid in pectin, as reported for pectin extracted from carrot (Jafari et al., 2017; Lara-Espinoza et al., 2018; Sucheta et al., 2020). The $-\text{CH}$ stretching vibration peaks between 2944 and 2314 cm^{-1} , assigned to CH , CH_2 and CH_3 groups, also showed a reduced intensity after pectin removal (Sucheta et al., 2020). The band around 1719 cm^{-1} shows the presence of $\text{C}-\text{O}$ bonds from methyl-esterified carboxyl groups in pectin, while the peaks around 1365 cm^{-1} originate from free and esterified carboxyl groups (Raji et al., 2017). It is worth noting that the DFP-extracted pectin showed similar

functional groups to those found in commercial pectin, such as the $-\text{OH}$, $\text{C}-\text{H}$, $\text{C}-\text{O}$ from methyl-esterified carboxyl groups and free carboxyl groups, with corresponding signals at 3389 , 2940 , 1753 , and 1585 cm^{-1} , respectively (Chua et al., 2020).

3.4. Soluble and hydrolyzable phenolic constituents

The phenolic constituents of date fruits can broadly be classified into (i) soluble phenolic compounds that can be extracted, partially or fully, in solvents such as methanol and hot water, and (ii) insoluble phenolic compounds that are bound to cell wall fiber constituents mainly through ester bonds and that can be extracted after hydrolysis by alkalis (Alam et al., 2021). In this study, the soluble and insoluble phenolic compounds in date pomace were extracted using 80% aqueous methanol and ethyl acetate after treatment with 3 M NaOH to release the esterified phenolic compounds, as described by Alam et al. (2021). To the best of our knowledge, no previous studies have reported the antioxidant activity associated with the hydrolyzable phenolic fraction of DFP. The total phenolic compounds in the DFP extracts were determined by the Folin–Ciocalteu, ABTS⁺, FRAP, and DPPH methods (Fig. 3).

The Folin–Ciocalteu phenolic content of the soluble plus insoluble fractions ranged from 170 to 260 mg GAE/100 g on a dry matter basis, in agreement with the results of Al-Farsi et al. (2007) for DFP from Omani varieties (149–396 mg GAE/100 g). The total phenolic content of DFP samples was lower than that of apple pomace (1016 mg GAE /100 g) (Sudha et al., 2007) and grape pomace (3642 mg GAE /100 g) (Yu and Ahmedna, 2013). The higher values of antioxidants in apple and grapes can be attributed to the inclusion of seeds in the pomace. In olive oil pomace, including flesh, skin and pits, Cioffi et al. (2010) reported a gallic acid content of 114–126 mg / 100 g, 20–21 mg Oleuropein equivalents/100 g and a DPPH value of 0.1 mg/ml. The DPPH value of the studied DFP samples ranged from 0.025 mg/ml for the soluble fraction in Khenazi to 0.2 mg/ml for the mixed variety, indicating a slightly higher antioxidant activity in date fruit pomace compared to olive oil pomace. The Folin–Ciocalteu, ABTS⁺, and DPPH methods showed a higher total antioxidant activity for the soluble compared to the insoluble fraction, while the FRAP method gave the opposite result. The content and composition of phenolic compounds in DFP is expected

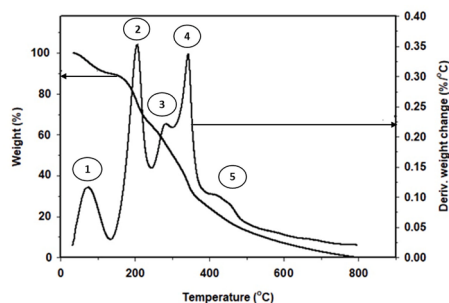


Fig. 4. Thermogravimetric and Differential thermogravimetry (DTG) curves of date fruit pomace of Lulu variety. Peaks: (1) evaporation of water around $100 \text{ }^{\circ}\text{C}$; (2) degradation of soluble sugars around $200 \text{ }^{\circ}\text{C}$; (3) degradation of hemicellulose around $300 \text{ }^{\circ}\text{C}$; (4) degradation of cellulose around $350 \text{ }^{\circ}\text{C}$; (5) degradation of lignin above $400 \text{ }^{\circ}\text{C}$.

Table 4

Water- and oil-holding capacities of the analyzed date fruit pomace (DFP) varieties and desugared DFP.

	Sample	Kastawi	Khenaizi	Lulu	Mixed	Mean*
Water-holding capacity (g/g)	DFP	2.4 ± 0.10 ^c	2.4 ± 0.00 ^c	3.3 ± 0.01 ^a	3.1 ± 0.05 ^a	2.8 ± 0.50 ^b
	Desugared DFP	5.9 ± 0.40 ^a	4.2 ± 0.30 ^d	4.6 ± 0.40 ^c	4.8 ± 0.40 ^b	4.9 ± 0.70 ^b
Oil-holding capacity (g/g)	DFP	1.0 ± 0.01 ^b	1.0 ± 0.04 ^b	0.9 ± 0.04 ^c	1.1 ± 0.12 ^a	1.0 ± 0.10 ^b
	Desugared DFP	1.7 ± 0.05 ^c	3.3 ± 0.10 ^a	2.5 ± 0.04 ^b	3.4 ± 0.06 ^a	2.7 ± 0.80 ^b

* All values represent the mean ± SD of three determinations per variety. Mean* represents the mean ± SD of the four samples (SD: standard deviation). The values with different superscript letters in each row are statistically different ($P < 0.05$). All values are reported on a dry matter basis.

to be different from that of the fruit, because a considerable amount of these phenols is extracted from the date syrup and affected by thermal treatment (Rambabu et al., 2020). The different results of the four assays can be attributed to the different reactivities of the reagents with the wide range of structurally different phenolic compounds present at different concentrations (Alam et al., 2021). Further studies are needed to investigate the partitioning of phenolic compounds between syrup and pomace and its relation with the molecular structures of the different phenols.

3.5. Thermal stability

The thermogravimetric analysis illustrated the thermal behavior of the DFP samples in terms of weight loss corresponding to the degradation temperature of the components of DFP. The DTG curves of the DFP samples showed five peaks at around 100, 200, 300, and 350 °C, with a broad peak at 400 °C (Fig. 4), representing the stages of thermal degradation of the main components (Garcia-Maraver et al., 2013). The DTG profile is consistent with the typical features of moist sugar-containing lignocellulosic materials, where water evaporates at around 100 °C, while soluble sugars, hemicellulose, cellulose, and lignin degrade at 200, 325, 350, and >400 °C, respectively (Brebú and Vasile, 2010; Liu et al., 2011; Rabetafika et al., 2013). The thermogravimetric curves show that the $T_{\max}$ (temperature of maximum weight loss) of the DFP fiber was around 200 and 350 °C, demonstrating their thermal stability. This property suggests the possibility of employing DFP fiber as potential fillers in biocomposites or as packaging material (Gouw et al., 2017; Difonzo et al., 2021). The $T_{\max}$ value indicates that the DFP fiber can withstand a processing temperature up to 350 °C (Coelho et al., 2018). For developing biocomposites, DFP fiber can be used as a filler material and processing parameters can be tailored based on their thermal properties obtained through thermogravimetric analysis (Zlatanović et al., 2019).

3.6. Water- and oil-holding capacities

Table 4 shows the water- and oil-holding capacities of the DFP and desugared pomace samples. The water holding capacity of Kastawi, Khenaizi, Lulu, and mixed varieties ranged from 2.4 to 3.3 g/g (dry matter) and increased upon desugaring and drying. DFP from Shahani variety (dried but not desugared) exhibited a slightly higher water holding capacity of 4.06 g/g (Majzoobi et al., 2019). Desugared pomace mainly consists of fiber, and the increased water holding capacity was likely due to the increased water binding to the hydroxyl group-rich fiber (Rabetafika et al., 2013). The WHC values are comparable to those of blackcurrant (3.2 g/g), chokeberry (3.85 g/g), (Reißner et al., 2019), and pear (3.2 g/g) (Rabetafika et al., 2013) pomace, but lower than that of apple pomace (8.39 g/g) (Sudha et al., 2007). The oil holding capacity of the DFP samples ranged between 0.9 and 1.1 g/g, and increased to 1.7–3.4 g/g in the desugared and dried samples. The higher oil holding capacity observed upon desugaring could be attributed to the exposure of the nonpolar side chains of the fiber (Rabetafika et al., 2013). The OHC values are comparable to those of redcurrant (1.94 g/g) and chokeberry (1.91 g/g) pomace (Reißner et al., 2019), but

lower than those of pear and apple pomace (2.1 g/g) (Rabetafika et al., 2013). Functional properties such as the water- and oil-holding capacities show that DFP is a potential source of dietary fiber, with possible applications as fortifying agent in food and promoter of intestinal health (Gouw et al., 2017).

4. Conclusions

In this study, the valorization potential of date fruit pomace was analyzed through a comprehensive physicochemical characterization. The obtained results highlight that DFP is not just a by-product but a potential source of functional ingredients exploitable to obtain value added products. Based on the observed composition, numerous valorization strategies and engineering possibilities can be proposed with the aim of developing sustainable biorefineries based on DFP. The major components of DFP are soluble sugars, fiber and phenolic compounds, which together with the elemental composition suggest a high nutritional value, hence DFP can be used as a dietary supplement. By removing the residual sugars, this dietary fiber complex can be used for fortifying various food products such as bread, bakery products, cereals and so on. Recovered sugars from the DFP can be used as a potential feedstock for various biorefinery applications based on fermentation routes such as bioethanol, organic acids (eg: lactic acid, citric acid, itaconic acid), and volatile fatty acids production. The color of the DFP indicates the presence of pigments that may provide another valorization strategy. By further analyzing its antimicrobial activity, it could be assessed if DFP could be used as microbial agent in various industrial applications or for cosmetic formulations. It is advised to dry the DFP (at 60 °C overnight) and use it for the various valorization processes to prevent the spoilage of DFP owing to fermentation because of its high moisture content. Further exploitation and addition of value to the date fruit waste generated by the industry today will improve the economic value and sustainability of the date palm farming and date fruit processing industries.

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.

Data availability

Data will be made available on request.

Acknowledgments

Funding: This research work was funded by the ASPIRE Award for Research Excellence (AARE-2020) grant number: G00003595, United Arab Emirates University (UAEU) PhD fund (grant number: 31N326) and UAEU Program for Advanced Research (UPAR) fund (grant number: 31F125).

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