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Research article

Conventional and eco-friendly aqueous extraction methods of date palm fruit compounds: Optimization, comparison, characterization of the date pulp extract and value-added potential

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Abstract: The extraction of the total soluble solid compounds from the pulp fruit *Phoenix dactylifera* L. is a major challenge for their valorization in the date fruit industry. However, conventional aqueous extraction methods are limited in terms of efficiency and processing time. In order to optimize this process, this study explores and compares three extraction methods: microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and conventional water bath–assisted extraction (WAE). The primary objective of this study is to maximize extraction recovery (ER) by performing an optimization using a Box–Behnken design (BBD). The second objective is to analyze the impact of the three extraction methods on extraction recovery, functional attributes, biochemical characteristics, antioxidant and antibacterial activities of the optimized date pulp aqueous extract (DPAE). Results obtained using mathematical models showed significant differences ($p < 0.05$) between the three methods tested. Optimum extraction conditions were determined as follows: for WAE, a solid/liquid (S/L) ratio of 1/4.68 g/mL, a temperature of 80 °C,

and a duration of 38.63 minutes; for MAE, an S/L ratio of 1/6 g/mL, an irradiation power of 480 W, and a duration of 11 minutes; for UAE, an S/L ratio of 1/5 g/mL, a sonication amplitude of 40%, and a duration of 60 minutes. The MAE method stood out for its extraction efficiency, with ER = 79.90% ± 1.54% and being three times faster than WAE and MAE. It also provided the highest concentrations of total soluble solids (14.33% ± 0.11% FW), total sugars (22.23 ± 0.23 g/100 mL DPAE), and total polyphenols (1.69 ± 0.04 mg GAE/mL DPAE). The antioxidant activity of DPAE was high, with an IC_{50} of 729.80 ± 12.87 µg/mL of the methanolic extract. These results suggest that the optimization of extraction processes, particularly using microwave technology, could offer promising prospects for the valorization of date fruit products by improving the production efficiency of a natural and nutritious DPAE, potentially beneficial to the date fruit industry in terms of product quality and cost reduction.

Keywords: date pulp; optimization; extraction; water bath; microwave; ultrasound; characterization; GC-MS; antioxidant; antibacterial; activity

Abbreviations: S/L ratio: solid/liquid ratio; DE: dry extract; FW: fresh weight; GAE: gallic acid equivalent; CE: catechin equivalent; QE: quercetin equivalent; IC_{50}: half maximal inhibitory concentration; µm: micrometer; µL: microliter; GC-MS: gas chromatography–mass spectrometry; eV: electron volt; M/z: mass to charge ratio; Exp: experimental; Pred: predicted; RT: retention time

1. Introduction

For centuries, the date palm has been a valuable resource in arid regions. In 2023, Algerian date production reached 1,324,767.01 tons [1], all cultivars combined. Dates are a rich source of natural sugars (65.7%–88.02%). They also contain proteins (1.22%–3.30%), lipids (0.11%–7.33%), and ash (1.43%–6.20%). In addition, they are a valuable source of bioactive compounds, including dietary fiber (1.9%–16.95%), phenolic antioxidants (1%–2%), tannin-derived pigments, and epicatechin oligomers [2]. Results also suggest the presence of other beneficial compounds as well as tannins (21.93–53.95 mg EC per 100 g dry weight) [3]. Vitamin A and E contents appear to vary from 10.06 to 30.19 µg of β-carotene and from 0.59 to 23.64 µg of α-tocopherol per 100 g of dry weight, respectively [4].

Previous studies of Hinkaew et al. (2021) and Khalil al. (2023) [5,6], showed that nutritional and bioactive compounds can be extracted by mixing date pulp with hot water (95 °C) for 15 min. In this context, given the growing demand for natural, affordable, and nutritious foods, it would be beneficial to consider ways to better utilize underutilized cultivars, both for direct consumption and for processing, to increase their market value and nutritional benefits [7]. In this regard, it has been observed that aqueous date extract has interesting nutritional and biological properties that could make it a promising semi-finished product for a wide range of date-based products [8,9]. Date syrup, obtained after the concentration of the aqueous extract of the pulp, is probably one of the most popular products derived from the date palm. In their work, Djaoud et al. [10] used an Algerian cultivar, while Mahdi et al. [11] studied Egyptian and Iraqi cultivars. On the other hand, liquid sugar, a natural sweetener, has production potential from cultivars in the United Arab Emirates, as described by AlYammahi et al. [12] and Pal et al. [13]. Aqueous extract of date pulp offers remarkable nutritional and functional potential, but optimization of the extraction process is essential

to improve the quality of the derived products.

Ganbi [14] compared conventional water bath extraction with two unconventional and environmentally friendly extraction methods, microwave-assisted extraction and ultrasonic-assisted extraction, for the extraction of total soluble solids from a Saudi cultivar. However, although his study evaluated the physicochemical properties of the extracts obtained, it relied solely on one-off experimental trials, without recourse to systematic optimization based on a design of experiments, thus limiting the statistical robustness and reproducibility of his results. More recently, Djaoud et al. [10] presented significant advances in the extraction of total sugars from date pulp using the same methods. Their main objective was to improve the yield of total sugar extraction by determining the sugar profile by HPLC technique only, without physicochemical characterization.

Therefore, the aim of the present study is to go beyond this approach by seeking to maximize the extraction recovery (ER) of total soluble matter from date pulp while integrating a complete characterization of the date pulp extract obtained. To this end, this study proposes to investigate different extraction methods, while building on previous research. However, the optimization of an extraction process cannot be limited to the analysis of the individual effects of factors; it is crucial to study the interactions between parameters and the quadratic effects that influence the overall response. These complex relationships can only be properly assessed using an advanced statistical approach such as the Box-Behnken design (BBD). We are comparing the effects of three extraction methods—water bath, microwave, and ultrasound—not only on the extraction recovery but also on the functional properties, biochemical characteristics, and antioxidant and antibacterial activities of each optimized extract. This integrated approach aims not only to improve the efficiency of the extraction process but also to ensure the production of natural date pulp extracts with optimal functional and nutritional properties.

2. Materials and methods

2.1. Plant material

The *Mech Degla* date cultivar (*Phoenix dactylifera* L.), harvested at the ripe *T'mar* stage, was selected from the local market in Biskra (Southeast Algeria). After cleaning and pitting, 20 kg of fresh pulp was dried at 40 °C in a ventilated oven (Memmert UN 260, Memmert GmbH, Schwabach, Germany) to 18.31 kg, i.e., 91.55% of the initial fresh weight. The dried pulp, with a moisture content of 4.98% ± 0.02% of fresh weight post-drying, was ground to increase the exchange surface and facilitate extraction and then sieved to 200 μm. The powder obtained was stored at −18 °C until use.

2.2. Chemical reagents

The following items were purchased from Sigma-Aldrich (St. Louis, MO, USA): ethanol, phenyl beta glucoside, hydroxylamine chloride in pyridine, 2.2-diphenyl-1-picrylhydrazyl radical (DPPH), Folin–Ciocalteu reagent, gallic acid, sodium carbonate, sodium hydroxide, hexamethyldisilazane (HMDS), trifluoroacetic acid (TFA), acetone, and heptane. Sulfuric acid (96%), phenol, 3.5-dinitrosalicylic acid, quercetin, aluminum trichloride (anhydrous), and potassium sodium tetrahydrate were acquired from Merck Millipore (Burlington, MA, USA). All utilized chemicals and reagents were of analytical grade.

2.3. Box–Behnken design (BBD)

We sought to improve the extraction approach by applying a three-factor, three-level BBD of response surface methodology (RSM), using the three extraction methods (WAE, MAE, and UAE). BBD effectively maximized response (Y), represented by extraction recovery (ER %), by adjusting the combination of three independent factors. We optimized the X_1 [S/L ratio (g/mL)], X_2 [energy treatment level, which represents the temperature (°C) for WAE, irradiation power (W) for MAE, and sonication amplitude (%) for UAE], and X_3 [extraction time (min)]. Using a one-factor experiment, preliminary essays established the optimal range values, coded as inferior (−1), median (0), and superior (+1) levels of these factors, as presented in Table 1.

Table 1. Factors and their level values used in the Box–Behnken design matrix to optimize extraction using the three methods (WAE, MAE, and UAE).

Extraction method	Factors (units)	Coded factors	Levels		
			−1	0	+1
WAE	S/L ratio (g/mL)	X_1	1/7	1/5	1/3
	Temperature (°C)	X_2	70	75	80
	Extraction time (min)	X_3	35	40	45
MAE	S/L ratio (g/mL)	X_1	1/6	1/5	1/4
	Irradiation power (W)	X_2	480	560	640
	Extraction time (min)	X_3	9	10	11
UAE	S/L ratio (g/mL)	X_1	1/5	1/4	1/3
	Sonication amplitude (%)	X_2	30	35	40
	Extraction time (min)	X_3	50	55	60

In view of the aforementioned considerations, the BBD matrix was conducted with 15 trials, comprising 12 factorial points and the central points in 3 replicates. Equation (1) was employed to fit the experimental results for a second-order polynomial model.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_{ii}^{\ 2} + \sum_{ij}^{k} \beta_{ij} X_i X_j + \varepsilon, \text{ with } k = 3 \quad (1)$$

Where Y is the measured response variable, which in our case is ER, $ß_0$ is a constant, $ß_i$ is the linear coefficient (main effect), $ß_{ii}$ is the quadratic coefficient, $ß_{ij}$ is the two coefficients of factor interaction, X_i, X_j are the independent variables, and ε is the error. The Box–Behnken matrix displaying the experimental and predicted ER values is shown in Table 2.

In this study, and as outlined by Beg and Akhter [15], we found that using quadratic response surface designs proved effective in identifying the critical effects of certain variables in a limited number of trials (15 in our case). The optimization of extraction conditions for all three methods was performed using the *optimizer* prediction function in Minitab software (version 17.1.0).

Table 2. Box–Behnken design matrix for the three extraction methods (WAE, MAE, and UAE).

	WAE					MAE					UAE				
Run	X_1	X_2	X_3	ER (%)		X_1	X_2	X_3	ER (%)		X_1	X_2	X_3	ER (%)	
				Exp.	Pred.				Exp.	Pred.				Exp.	Pred.
1	1/7	70	40	35.42	35.20	1/6	480	10	62.36	62.42	1/5	30	55	32.03	31.89
2	1/3	70	40	16.39	17.11	1/4	480	10	47.16	47.18	1/3	30	55	27.24	27.27
3	1/7	80	40	33.89	33.16	1/6	640	10	42.84	42.81	1/5	40	55	32.43	32.39
4	1/3	80	40	28.41	28.62	1/4	640	10	49.58	49.51	1/3	40	55	27.78	27.91
5	1/7	75	35	33.32	34.08	1/6	560	9	61.36	61.32	1/5	35	50	31.90	32.00
6	1/3	75	35	20.10	19.92	1/4	560	9	57.50	57.50	1/3	35	50	28.15	28.08
7	1/7	75	45	31.79	31.96	1/6	560	11	67.80	67.79	1/5	35	60	32.43	32.50
8	1/3	75	45	24.26	23.49	1/4	560	11	63.06	63.09	1/3	35	60	27.43	27.32
9	1/5	70	35	28.64	28.09	1/5	480	9	63.38	63.35	1/4	30	50	30.90	30.93
10	1/5	80	35	34.35	34.31	1/5	640	9	55.61	55.66	1/4	40	50	31.41	31.34
11	1/5	70	45	30.26	30.30	1/5	480	11	70.39	70.33	1/4	30	60	30.59	30.65
12	1/5	80	45	33.03	33.57	1/5	640	11	60.71	60.74	1/4	40	60	31.40	31.36
13	1/5	75	40	33.46	33.34	1/5	560	10	44.68	44.60	1/4	35	55	30.45	30.73
14	1/5	75	40	33.55	33.34	1/5	560	10	44.71	44.60	1/4	35	55	30.71	30.73
15	1/5	75	40	33.01	33.34	1/5	560	10	44.42	44.60	1/4	35	55	31.04	30.73

2.4. Preparation of DPAEs

2.4.1. Water bath–assisted extraction (WAE)

The S/L ratio, temperature, and time affected conventional aqueous date extraction [16–18]). According to Nishad et al. [19], the conventional extraction was carried out in a thermostatic shaking water bath (WNB 10, Memmert, Germany, 1200 W), equipped with a shaking device (type SV 2945, 230 V, 0.1 A, 50/60 Hz, 12 W, Memmert, Germany). Mixtures of 40 g of date pulp powder with 280, 200, and 120 mL of distilled water were heated at 70, 75, or 80 °C for 35, 40, and 45 min, with horizontal shaking movements adjustable at 150 strokes per minute.

2.4.2. Microwave-assisted extraction (MAE)

The S/L ratio, irradiation power, and time affected MAE [20]. Extraction was carried out as outlined by Weremfo et al. [21] in a domestic microwave oven (MAXIMOS23S, Maxipower, China) operating at 800 W maximum power and a frequency of 2450 MHz. The internal cavity dimensions of the microwave oven were 48.3 cm in width, 28.1 cm in height, and 38.7 cm in depth. The microwave was fitted with a water condenser at the top. Suspensions of 40 g of date pulp powder in 240, 200, and 160 mL of distilled water were subjected to 480, 560, or 640 W for 9, 10, and 11 min. The treatment was applied in cycles of 10 s of irradiation and 5 s of pause to prevent overheating. At the end of each run, the mixture's temperature was measured using an electronic thermometer (TP 300, 1.5 V) with a digital temperature indicator with a range of -10 to 300 °C with a precision of ± 0.1 °C. The values obtained were 94.5, 93.3, 98.1, 94.3, 96.9, 93.6, 97.3, 93.7, 95.6, 96.5, 96.4, 97.2, 96.2, 96.4, and 96 °C for runs 1–15, respectively.

2.4.3. Ultrasound-assisted extraction (UAE)

UAE was carried out as reported by López et al. [22], with slight modifications, in an ultrasonic device (Vibracell, VCX 500,115 PB, SONICS, Newtown, Connecticut, USA), with a 500 W maximum net power output at 20 kHz. The power output was connected with a standard probe (13 mm × 139 mm, 115 μm), immersed in a mixture of 40 g of date pulp powder with 200, 160, and 120 mL of distilled water, extracted for 50, 55, and 60 min, applying continuous ultrasonic waves at three sonication amplitudes (30%, 35%, and 40%), with the probe tip positioned approximately 2 cm above the sample. To prevent overheating, the treatment was set on pulse mode (10 s ON and 10 s OFF); an ice bath stabilized at 40 °C (± 1 °C) was also used.

After each experiment, the extract was filtered under vacuum using Whatman filter paper (No. 4), and then the DPAE samples were kept in storage at 4 °C.

2.5. Extraction recovery determination

We used the equation described by Ganbi [14] to determine the ER of all DPAE samples:

$$\mathrm{ER}(\%) = \left(\frac{\mathrm{W_{extrtact}X\%\ of\ TSS_{extract}}}{\mathrm{W_{pulp}}}\right) \mathrm{x}\ 100 \quad (2)$$

Where ER is the extraction recovery (%) of total soluble solids, $W_{extract}$ is the weight of the DPAE (g), and % of $TSS_{extract}$ is the total soluble solids content in the DPAE as expressed in percentage of sucrose in fresh weight (% FW).

2.6. Characterization of DPAEs

2.6.1. Functional attributes

The three optimized DPAE samples were analyzed for their physicochemical properties, namely pH, total soluble solids (TSS), and ash content, as well as titratable acidity according to AFNOR [23]. On the other hand, color intensity was calculated as reported by Vickers et al. [24]. The non-enzymatic browning index (NEBI) was fixed by measuring the alcoholic DPAE absorbance at 420 nm, whereas clarity was measured in terms of the percentage of transmittance (T) at 660 nm. Both parameters were measured using a UV-Vis spectrophotometer, as described by Muñoz et al. [25] and Rai et al. [26], respectively.

2.6.2. Biochemical features

According to Dubois et al. [27] and Miller [28], the amount of total and reducing sugars in the extracts, expressed in mg/mL, was measured with colorimetric techniques utilizing an absorption UV-Vis spectrophotometer (Schimadzu, 2600 model, Japan). The Kjeldahl method, described in AFNOR [19], was used to determine the total nitrogen matter. Following the protocol described by Amira et al. [29], the fat content was determined using the Soxhlet method with petroleum ether as solvent. Total phenolic and flavonoid contents were determined using the protocols outlined by Assadi et al. [30], with some adjustments. Pectin was measured (as calcium pectate) in accordance with Begum et al. [31]. Sodium and potassium contents were determined from ashed samples using a

flame absorption spectrophotometer system (Jenway, PFP7 model, England) according to AFNOR [19] for sodium and potassium, whereas calcium and magnesium were quantified by complexometric titration with EDTA, as in the study by Subedi [32].

2.6.3. Sugar profile determination by GC-MS method

As previously described by Ruiz-Matute et al. [33], the diluted solution of the DPAE samples (1 µL) was analyzed by GC-MS for the determination of sucrose, glucose, and fructose. After reactions with 350 µL of 2.5% hydroxylamine chloride in pyridine (30 min at 75 °C), 350 µL of hexamethyldisilazane, and 35 µL of trifluoroacetic acid (45 °C for 30 min), carbohydrates were transformed into their trimethylsilyl oximes (OTMS). Helium was used as the carrier gas at 1 mL/min for the GC-MS analyses, and a Hewlett-Packard 6890 gas chromatograph and a 5973 quadrupole mass detector (both from Agilent, Palo Alto, CA, USA) were used. An SPB-1 (cross-linked methyl silicone)-coated fused silica column from Supelco (Bellefonte, PA, USA) with a 25 m column length, 0.25 mm inner diameter, and 0.25 µm film thickness was employed. The oven temperature was programmed as described by Ruiz-Matute et al. [33], with slight modifications: the temperature was held at 200 °C for 5 min, then programmed to 270 °C at a heating ramp of 15 °C/min, then programmed to 290 °C at 1 °C/min, and finally increased to 310 °C and held for 15 min. The injector was kept at 300 °C, and injections were done in split mode with a split ratio of 1:60. With an electron ionization mode of 70 + eV and a scan mode range of m/z 50–650, the mass spectrometer's transfer line was set to 280 °C. For quantitative purposes, to determine the response factor (RF) in relation to phenyl-β-D-glucoside (internal standard), carbohydrate standard solutions over the expected concentration range in DPAEs were prepared. Every analysis was performed twice. Identifications were done by contrasting retention times of OTMS derivatives of carbohydrates found in DPAEs with those of reference compounds and verified using mass spectral data.

2.6.4. Antiradical scavenging activity

Phenolic compounds from DPAE were extracted as previously described by Saleh et al. [34], with minor amendments: 90 mL of concentrated methanol was added to 10 mL of DPAE, mixed on a magnetic stir plate at room temperature for 30 min, filtered through Whatman No. 4 filter paper, centrifuged at 2800 rpm, and then lyophilized to dryness. Following a slightly modified version of the procedure described by Braca et al. [35], the antioxidant activities of DPAE samples that were prepared at various concentrations of the dry methanolic extract (50–1600 g/mL) were assessed using the DPPH free radical scavenging assay. After 30 min, the absorbance was measured at 517 nm and was computed using Equation 3.

$$\text{DPPH scavenging effect}(\%) = \left[\frac{(A_0 - A_t)}{A_0}\right] \text{x } 100 \quad (3)$$

Where A_0 is the negative control absorbance, and A_t is the sample absorbance. A linear regression analysis was used to determine the IC_{50}, or the sample's mass concentration (in µg/mL) required to reduce the initial DPPH concentration by 50%, as a measure of the samples' ability to scavenge free radicals on DPPH.

2.6.5. Antibacterial activity

DPAE samples were tested against five bacterial strains: *E. coli* ATCC 25922, *Bacillus cereus* ATCC 14579, *Salmonella Typhimurium* ATCC 14028, *Pseudomonas aeruginosa* ATCC 27853, and *Listeria monocytogenes* ATCC 35152. According to the method outlined in Hernández-Pérez et al. [36], with slight modifications, the surface of each Petri plate containing Muller–Hinton agar (MH) was inoculated with a standardized suspension (0.5 McFarland) of one of the five bacterial strains using a sterile swab. Then, sterile 6 mm discs containing 50 µL of one of the tested extracts (100 mg/mL of 2.5% DMSO) were deposited on the MH surface. Discs containing gentamicin (positive control) and 2.5% DMSO (negative control) were placed with the other discs. The Petri plates were then incubated at 37 °C for 18–24 h. Three Petri plates per test per bacterium were used, and the experiment was repeated three times. After incubation, the antibacterial effect of the extracts was evaluated by measuring the clear zone surrounding the discs.

2.7. Statistical analysis

All data were analyzed using Minitab Software (Inc., version 17.1.0). We used analysis of variance (ANOVA) followed by the Tukey's HSD (honestly significant difference) test of multiple comparisons to check if there were any statistically significant differences relative to the effect of extraction methods (WAE, MAE, and UAE) on i) extraction recovery (ER, %), ii) functional characteristics, and iii) biochemical features of the three DPAEs samples obtained by applying the optimum factors values. A *p*-value lower than 0.05, was used to determine the statistical significance level. The accuracy and validity of the three mathematical models were checked using the coefficient of determination R^2 (%), the adjusted coefficient of determination R^2 adj. (%), and the lack-of-fit tests.

3. Results and discussion

3.1. Fitting model

A second-order polynomial model was developed to determine optimal process conditions and the actual relationship between the response and process variables, represented by coded factors (X_1, X_2, and X_3) for the response Y (ER), as illustrated in equations 4, 5, and 6 below for WAE, MAE, and UAE, respectively. The objective was to optimize ER through multiple regressions on experimental results.

$$\mathrm{ER\,exp}\ (\%) = -114 - 475{,}8\,X_1 + 1{,}79\,X_2 + 6{,}26\,X_3 - 496{,}8\,X_1^2 - 0{,}0122\,X_2^2 - 0{,}0586\,X_3^2 + 7{,}113\,X_1X_2 + 2{,}987\,X_1X_3 - 0{,}0294\,X_2X_3 \quad (4)$$

$$\mathrm{ER\,exp}\ (\%) = 1914{,}53 - 1612{,}6\,X_1 - 0{,}8595\,X_2 - 291{,}19\,X_3 + 1664{,}6\,X_1^2 + 0{,}000467\,X_2^2 + 14{,}9321\,X_3^2 + 1{,}6442\,X_1X_2 - 5{,}28\,X_1X_3 - 0{,}005969\,X_2X_3 \quad (5)$$

$$\mathrm{ER\,exp}\ (\%) = 48{,}1 + 137{,}1\,X_1 - 0{,}465\,X_2 - 0{,}851\,X_3 - 232{,}1\,X_1^2 + 0{,}00468\,X_2^2 + 0{,}00898\,X_3^2 + 0{,}108\,X_1X_2 - 0{,}962\,X_1X_3 + 0{,}00300\,X_2X_3 \quad (6)$$

3.2. Verification of the three statistical models

Table 3 displays the ANOVA results for fitting models of ER for the three extraction methods (WAE, MAE, and UAE). Each independent variable's significance was evaluated using *p*-values.

Table 3. Results of ANOVA for fitting models ER (%) for the three extraction methods (WAE, MAE, and UAE).

WAE			MAE			UAE		
Source	F-value	*P*-value	Source	F-value	*P*-value	Source	F-value	*P*-value
Model	78.03	0.0001	Model	9704.58	0.0001	Model	101.36	0.0001
Linear	161.48	0.0001	Linear	6136.50	0.0001	Linear	275.20	0.0001
X_1	410.58	0.0001	X_1	2593.36	0.0001	X_1	812.43	0.0001
X_2	72.13	0.0001	X_2	10636.53	0.0001	X_2	12.54	0.017
X_3	1.72	0.247	X_3	5179.63	0.0001	X_3	0.64	0.460
Square	42.61	0.001	Square	20091.49	0.0001	Square	26.14	0.002
X_1^2	120.29	0.0001	X_1^2	2205.28	0.0001	X_1^2	69.72	0.0001
X_2^2	0.55	0.491	X_2^2	2348.48	0.0001	X_2^2	0.99	0.364
X_3^2	12.71	0.016	X_3^2	58685.74	0.0001	X_3^2	3.66	0.114
2-way Interaction	30.01	0.001	2-way Interaction	2885.74	0.0001	2-way Interaction	2.74	0.153
X_1X_2	73.60	0.0001	X_1X_2	8578.42	0.0001	X_1X_2	0.10	0.769
X_1X_3	12.98	0.016	X_1X_3	13.80	0.014	X_1X_3	7.67	0.039
X_2X_3	3.46	0.122	X_2X_3	65.01	0.0001	X_2X_3	0.44	0.536
Lack-of-fit	11.75	0.079	Lack-of-fit	0.25	0.856	Lack-of-fit	0.30	0.825
R^2 (%)		99.45	R^2 (%)		99.99	R^2 (%)		99.45
R^2 adj. (%)		98.47	R^2 adj. (%)		99.98	R^2 adj. (%)		98.47
S (%)		0.22	S (%)		0.11	S (%)		0.22

The statistical analysis revealed promising results. The three polynomial models, as presented by equations 4, 5, and 6, show low *p*-values ($p < 0.05$) and high F-values (78.03, 9704.58, and 101.36, respectively). According to Shill et al. [37], this shows that the models are highly significant and suitable to improve the ER using the WAE, MAE, and UAE methods. Regarding the single effect, S/L ratio and energy treatment level significantly influenced ER for all extraction methods ($p < 0.05$), whereas extraction time demonstrated a significant effect only during MAE treatment. Upon further analysis of the ANOVA results, we also identified a significant effect of the quadratic terms for S/L ratio (X_1^2) across all methods, the irradiation power (X_2^2) for MAE only, and the extraction time (X_3^2) for WAE and MAE ($p < 0.05$). Furthermore, it is worth noting that there were also some notable interaction effects ($p < 0.05$): X_1X_2 and X_1X_3 for WAE; X_1X_2, X_1X_3, and X_2X_3 for MAE; and X_1X_3 only for UAE (see Table 3).

Additionally, the R^2 coefficient values for WAE (99.45%), MAE (99.99%), and UAE (99.45%) were all close to 100% (Table 3), indicating that the models may be effective in accounting for the observed variance in the experimental data. Also, the adjusted R^2 coefficient values further support the potential accuracy of the model adjustments: 98.47%, 99.98%, and 98.47% for WAE, MAE, and

UAE methods, respectively.

Ultimately, the insignificant lack-of-fit tests indicate that the models are suitable for the experimental data, as evidenced by their high *p*-values (0.079, 0.856, and 0.825, all higher than 0.05, for WAE, MAE, and UAE, respectively). The created fit models were validated using the optimized independent variables to ensure their predictability. This allowed the prediction of the optimal extraction conditions to maximize ER using BBD.

3.3. Extraction optimization

Figure 1 (a, b, and c) presents the optimization plot of the BBD for the three studied methods (WAE, MAE, and UAE) and displays the acceptability of the predicted values, denoted by the letter d.

In order to maximize ER, the optimal parameters values predicted were: X_1 = 1/4.68 g/mL, X_2 = 80 °C, and X_3 = 38.63 min for WAE, X_1 = 1/6 g/mL, X_2 = 480 W, and X_3 = 11 min for MAE, and X_1 = 1/5 g/mL, X_2 = 40%, and X_3 = 60 min for UAE. Figure 1(d) shows the maximum experimental ER values obtained, expressed as mean ± SD in triplicates: 34.66% ± 1.11%, 79.90% ± 1.54%, and 31.43% ± 1.21%. It is clear that ER differs significantly between the three methods ($p < 0.05$); these values appear to be in close alignment (R^2 = 99.8%) with those predicted (35.79% with d = 0.93, 81.06% with d = 1, and 32.94% with d = 1) using WAE, MAE, and UAE methods, respectively (Figure 1a, 1b, and 1c).

3.4. Effect of extraction parameters on recovery

The analysis of the results presented in Figure 1 highlights the variability of ER as a function of the extraction method employed and the configuration of experimental parameters. This predictive modeling enables dynamic analysis: the translation of the vertical red line along the x-axis corresponds to the level's adjustment of the parameter studied, resulting in automatic reconfiguration of the diagrams and instant recalculation of predicted responses on the blue y-axis and desirability values. In the rest of the analysis, we will examine the single effect of each parameter on the ER, keeping the two other extraction variables at their optimal level predicted.

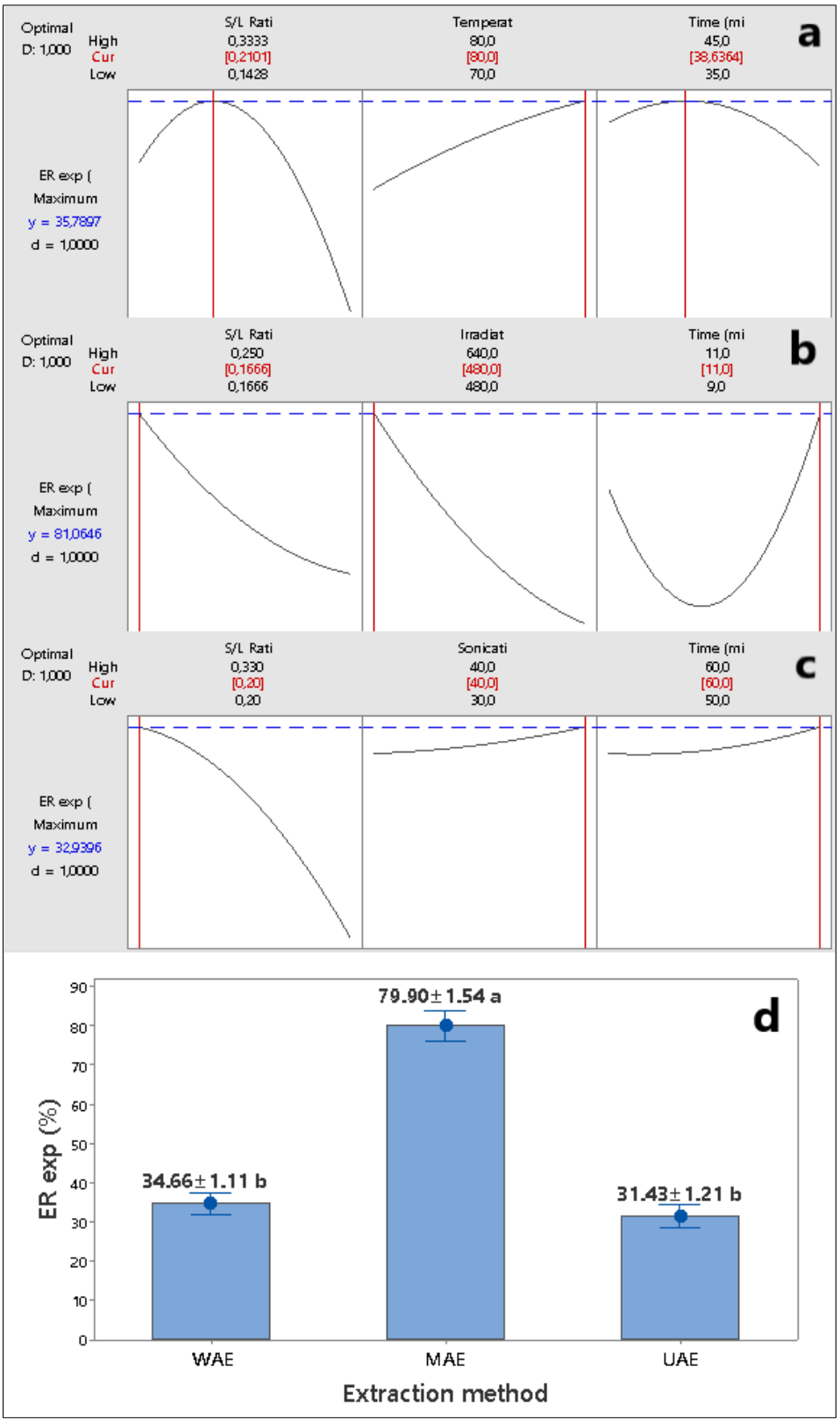

Figure 1. Optimization plot values of ER (%) derived from date pulp extracted by (a) WAE, (b) MAE, and (c) UAE, and (d) their mean experimental ER (%) values obtained by applying the optimum conditions predicted.

3.4.1. Solid/liquid ratio

The impact of the S/L ratio on the efficiency of the WAE method was significant. ER increased parabolically from 33.55% to 35.79% when the S/L ratio increased from 1/7 to 1/4.68 g/mL, while the other extraction parameters remained constant (80 °C and 38.63 min). A solvent volume of less than 280 mL for 40 g of date pulp powder may increase the powder analyte transfer in the extraction medium and generate more pulp aqueous-soluble solids. As affirmed by Mgoma et al. [38], this observation may be explained by Fick's law: the flow rate of a compound is directly proportional to the concentration gradient. However, when S/L ratio increased to 1/3 g/mL, ER decreased progressively from 35.79% to 28.23%, suggesting that insufficient solvents may also affect component extraction recoveries (incomplete extraction). In the same manner, using microwaving, increasing this ratio over 1/6 g/mL to 1/4 g/mL led to an increase in the viscosity of the date pulp-water powder mixture, which in turn reduced diffusion rate and significantly decreased ER from 81.06% to 65.39%. This phenomenon elucidates the negative correlation observed between the S/L ratio and ER, corroborating the results reported by El-Sharnouby et al. [39]. The same was true for the UAE method: a low S/L ratio favored better ultrasound transmission, releasing more compounds and increasing ER. At a higher solid/liquid ratio (1/3 g/mL), there was rapid solvent saturation and limited solubilization of extracted compounds, resulting in a decrease in ER from 32.94% to 27.83%. Furthermore, the significant quadratic term (X_1^2) for all methods supports our arguments (Table 3), indicating that the impact of the S/L ratio on ER follows a parabolic curve (Figure 1 a, b, and c).

3.4.2. Energy treatment level

Using the water bath method, ER increased slightly from 32.35% to 35.78% with increasing temperature from 70 to 80 °C. Probably, increasing temperatures cause structure disruption and increase diffusion, which could make soluble chemicals in date pulp powder easier to extract, increasing the ER. Our suggestions are supported by Kadlezir et al. [40]; when applying the water bath method, temperature significatively affects vitamin C, total polyphenol, and free amino acid contents of the aqueous date pulp extract. Those three responses increased with increasing temperature, from 73.5 mg/L, 3.61 g GA/100 g, and 497.2 mg/L at 25 °C, to values of 88.26 mg/L, 4.78 g GA/100 g, and 565.42 mg/L at 95 °C, respectively. Perhaps those constituents present in our samples, also increase, increasing ER. In contrast, this then decreased progressively from 81.06% to 60.5% when irradiation was increased from 480 to 640 W. This can be explained by microwaves rapidly heating water due to its dielectric characteristics (high constant and dissipation factor) [41]. Water absorbs microwaves powerfully at high power, causing an internal pressure that damages plant cells and promotes evaporation, disturbing the ideal solvent-to-solid ratio and limiting solute mobility [20,42]. Furthermore, the decrease in ER can be explained by the observations of Rocha et al. [43], which indicate that excessive overheating leads to thermal degradation of sugars and other sensitive compounds, reducing their recovery in the final extract. With regard to UAE, ER increased slightly from 32.29% to 32.94% with an increase in amplitude from 30% to 40%. This result is in line with Entezari et al. [44], who extracted juice from date pulp and observed that a higher intensity resulted in a higher extraction rate compared to a lower one.

3.4.3. Extraction time

Time was revealed to be not significant for WAE ($p > 0.05$). Under operating conditions set at a solid/liquid ratio of 1/4.68 g/mL and a temperature of 80°C, ER showed a slight increase from 34.95% to 35.78% over the time interval from 35 to 38.63 minutes. This slight improvement is due to a little continuous interaction between the matrix and the water. However, the extension of the extraction time (from 38.63 to 45 min) also resulted in a degradation of certain water-soluble compounds, as approved by Hasni et al. [45]. During sonication treatment, time was revealed to be not significant ($p > 0.05$). A boost in extraction time from 50 to 60 min led to a minor increase in RE from 32.29% to 32.94%. It is likely that, at around 50 min, the extraction process reaches its maximum efficiency, and that the extended time is mainly used to extract compounds that are more difficult to dissolve. The conventional extraction method achieved a maximum recovery of 35.78% in just 38.63 min at a temperature of 80 °C, approximately twice as fast as UAE. Indeed, with a sonication intensity of 40%, sonication produced a comparable recovery of 32.94% but in 60 minutes, despite a S/L ratio held constant at approximatively 1/5. This result contradicts the observations of Vinatoru et al. [46] who, in their literature review, discussed advances in extraction methods for plant compounds and highlighted the advantages of UAE in significantly reducing the time required to reach extraction equilibrium. It is likely that the level of ultrasonic power applied in this study (40%) was insufficient to induce rapid optimal cavitation, thus limiting the acceleration of extraction by sonication. Microwave irradiation shows a significant influence of time on ER ($p < 0.05$). Figure 1b reveals a parabolic curve-shaped relationship between time and ER ($p < 0.05$ for quadratic term X_3^2), indicating that either too little or too much extraction time is not optimal. Insufficient time limits compound extraction, while excessive time (over 11 min) may lead to degradation or limit release. A duration of approximately 11 min seems ideal for maximizing ER. Thus, the best extraction time must optimize ER while preserving nutritional content.

Finally, the data presented in Table 3 highlight the multifactorial nature of the extraction mechanism and the complexity of 2-way interactions (already mentioned above) between operating parameters. As highlighted by Sridhar et al. [47], these interactions, specific to each extraction technique, significantly influence compound flow in the mixture. For the conventional method, temperature and time, combined with an appropriate S/L ratio, dynamically modulate analyte solubility and diffusion. In the case of MAE, irradiation power must be fine-tuned according to the S/L ratio and exposure time, finely determining heat transfer and extraction selectivity to avoid thermal degradation while maximizing extraction efficiency. Finally, for UAE, maximum cavitation depends on choosing the right S/L ratio combined with optimal extraction time, irrespective of amplitude level. These results confirm the multifactorial nature of extraction processes and underline the importance of rigorous optimization of operating conditions for each method.

As cited above, the maximum ER value of the water bath and ultrasonic methods is significantly lower than that of microwave extraction. This result can be explained by the limitations of convective heat transfer in conventional heating and the formation of a layer of material around the pulp powder during sonication, which hinders the diffusion of constituents. Microwaves, on the other hand, disrupt this protective layer around the pulp powder and interact directly with the new water molecules present, thereby increasing extraction recovery. These observations corroborate the work of Vinatoru et al. and Flórez et al. [46,20], who showed that a rapid increase in temperature due to microwaves promotes more efficient extraction. Even when the extraction parameters were set at

minimum levels for extraction recovery (1/4 g/mL, 640 W, and 10 minutes), ER obtained with the microwave method still amounted to 49.58% (trial 4 in Table 2), significantly higher than those of the conventional and ultrasonic methods. It is worth noting that microwave heating could be considered in the beverage sector as an alternative to conventional techniques [48,49].

3.5. Effect of extraction methods on DPAEs' quality

Table 4 summarizes the functional characteristics, biochemical features, and antiradical scavenging activity of the DPAEs obtained by applying the optimum experimental conditions for each extraction method (WAE, MAE, and UAE).

3.5.1. pH and titratable acidity

Statistical analysis reveals significant variations ($p < 0.05$) in pH and titratable acidity values between extraction methods. DPAEs show a mean pH ranging from 5.44 ± 0.01 to 5.78 ± 0.05 and titratable acidity from 0.19 to 0.34 ± 0.016 g of citric acid equivalent/100 mL, in agreement with the values reported by Ganbi et al. [14]. However, our titratable acidity levels are significantly higher than those of Ogblechi et al. [50] (0.048 g of citric acid equivalent/100 mL), whose extraction process was based on a pulverizer and screw press. Although high-temperature sterilization is integrated, Ogblechi's mechanical extraction, without intensification techniques (microwaves or ultrasound), limits acid extraction, while prolonged sterilization can degrade certain sensitive acids. This optimization of modern methods thus favors extracts that are more stable and better suited to functional applications.

3.5.2. Total ash and minerals

Analysis of total ash and minerals revealed significant variations between extraction methods ($p < 0.05$), particularly for K, Ca, and Mg concentrations, while Na content remained stable. These differences suggest variable extraction efficiencies for different minerals, reflecting variable extraction efficiencies depending on the elements considered. Meanwhile, Samsalee and Sothornvit [51] evaluated the mineral extraction from used coffee grounds and demonstrated that methods such as autoclaving and ultrasonic favor increased concentrations of phosphorus, potassium, calcium, and magnesium, indicating that these easily soluble elements are preferentially extracted under intensification conditions.

Table 4. The most important functional characteristics, biochemical features, and antiradical scavenging activity of the three optimized DPAE samples as affected by the three extraction methods.

Extraction method / Characteristics	WAE	MAE	UAE
Functional characteristics			
pH	5.54 ± 0.08[b]	5.44 ± 0.02[b]	5.78 ± 0.05[a]
Total soluble solids (TSS) (% FW)	14.06 ± 0.11[a]	14.33 ± 0.11[a]	11.73 ± 0.3[b]
Clarity (T_{660}%)	47.46 ± 5.58[b]	28.58 ± 5.65[c]	64.55 ± 5.65[a]
Color intensity (index x 10^{-3})	73.45 ± 0.54[b]	76.85 ± 0.46[a]	68.82 ± 0.48[c]
Non-enzymatic browning index (NEBI)	0.35 ± 0.01[b]	0.46 ± 0.07[a]	0.27[b]
Biochemical features and antioxidant activity			
Total ash (% FW)	0.23 ± 0.01[a,b]	0.28 ± 0.03[a]	0.17 ± 0.03[b]
Titratable acidity (g of citric acid eq. /100 mL of DPAE)	0.27 ± 0.01[b]	0.34 ± 0.01[a]	0.19[c]
Total phenolic content (TPC) (mg GAE/mL of DPAE)	1.42 ± 0.03 [b]	1.69 ± 0.04[a]	1.01[c]
Total flavonoid content (TFC) (mg QE/mL of DPAE)	0.16[b]	0.18[a]	0.15[b]
IC_{50} (μg/mL of methanolic extract)	ND	729.80 ± 12.87	ND
Total sugar (g/100 mL of DPAE)	19.76 ± 0.11[b]	22.23 ± 0.23[a]	18.2 ± 0.43[c]
Reducing sugar (g/100 mL of DPAE)	5.03 ± 0.95[c]	9.03 ± 0.05[b]	11.73 ± 0.55[a]
Sucrose (mg/mL of DPAE)	139.12 ± 7.71[a]	117.34 ± 5.27[b]	56.01 ± 0.84[c]
Glucose (mg/mL of DPAE)	20.35 ± 0.83[c]	32.86 ± 0.35[b]	50.86 ± 3.68[a]
Fructose (mg/mL of DPAE)	22.08 ± 0.99[c]	35.04 ± 1.41[b]	53.57 ± 4.16[a]
Protein (as total nitrogen matter) (g/100 mL of DPAE)	0.23 ± 0.01[a,b]	0.24[a]	0.20 ± 0.01[b]
Fat (g/100 mL of DPAE)	0.13 ± 0.05[a]	0.18 ± 0.09[a]	0.16 ± 0.05[a]
Pectin amount (as % calcium pectate of FW)	0.26[b]	0.4 ± 0.01[a]	0.2 ± 0.01[c]
K (mg/100 mL of DPAE)	112.18 ± 6.8[a]	105.23 ± 12.88[a]	76.87 ± 6.67[b]
Na (mg/100 mL of DPAE)	33.49 ± 4.13[a]	31.68 ± 2.7[a]	36.19 ± 3.12[a]
Ca (mg/100 mL of DPAE)	53.85 ± 3.34[a]	49.10 ± 5.98[b]	23.33 ± 5.77[a]
Mg (mg/100 mL of DPAE)	55.33 ± 5.03[a]	51.33 ± 4.16[a]	23.33 ± 5.77[b]

Data shown are mean ± SD (n = 3); superscripts a, b, and c indicate significant differences according to Tukey's test. Values with different superscripts within the same line are significantly different at $p < 0.05$.

The apparent properties of DPAE were significantly affected by the extraction method used.

3.5.3. Pectin amount and clarity

DPAE obtained by MAE showed higher pectin recovery compared to WAE and UAE (Table 4). The heat generated quickly by microwave irradiation can enhance the release of polysaccharide compounds such as pectin, even though no previous studies have specifically explored microwaving for dates. The high pectin content of the DPAE could contribute to its potential retention and stabilization properties, as pectin is known for its ability to form gels in the presence of acids and sugars. In this respect, Masmoudi et al. [52] demonstrated in a low-sugar (45%) date jelly formulation that the interaction between lemon pectin and *Deglet Nour* date pulp promoted gelling by thermo-controlled methylation, suggesting a synergistic stabilization mechanism via sucrose-calcium gel interactions. Clarity is an essential attribute for ready-to-serve beverages based on date fruit. Thus, the removal of suspended particles such as pectin is crucial, as stated by Kulkarni et al. [53]. Thus, a higher transmittance indicates a clearer extract [54]. In this study, DPAE obtained by sonication had the highest clarity (64.55% ± 5.65%) at the lowest pectin content (0.2% ± 0.01% FW) (Table 4). These results suggest that ultrasonic methods target specific compounds with distinct solubility, making aqueous date palm pulp extract suitable for the production of light-colored energy drinks [55].

3.5.4. Non-enzymatic browning index and color intensity

NEBI and color intensity of our DPAEs were found to be significantly affected by the three extraction methods ($p < 0.05$); MAE's extract had the highest NEBI (00.46 ± 0.07), with an improvement in color intensity ($76.85 \pm 0.46 \times 10^{-3}$), followed by conventional and ultrasonic methods (Table 4). Previous studies of Patrignani et al. [56] and El-Nagga EA and Abd El–Tawab YA [57], have linked microwave quick heating to accelerated non-enzymatic processes and brown pigment formation. This accumulation then contributes to the increase in color intensity. In this context, Yilmaz and Toledo [58] agreed that heat-induced non-enzymatic browning as Maillard reaction leads to the chemical transformation of macronutrients, resulting in the generation and accumulation of compounds with strong antioxidant capacity (in this study, IC_{50} was determined at 729.80 ± 12.87 μg/mL for the methanolic extract of DPAE extracted by MAE).

3.5.5. Phenolic compounds and antioxidant activity

Our results show that MAE achieved significantly higher contents of total phenolic compounds and flavonoids under optimal conditions ($p < 0.05$), with 1.69 ± 0.04 mg GAE/mL for TPC and 0.18 mg QE/mL for TFC, highlighting the increased efficiency of MAE in extracting these bioactive compounds.

Microwave-assisted extraction enables the rapid and efficient isolation of polyphenols with minimal use of solvents. Solvents that effectively absorb microwaves, such as water, are particularly recommended for their efficient heat transfer and preservation of thermolabile compounds [59]. MAE influences both the yield and the integrity of bioactive compounds. By accelerating the process, microwave-assisted extraction minimizes the degradation of sensitive molecules. However, if not properly optimized, it may lead to the formation of undesirable by-products or partial degradation of some compounds [60]. The phenolic structure of these compounds gives them enhanced antioxidant

properties, including scavenging DPPH radical's capacity. Our optimized DPAE presented an IC_{50} of 729.80 ± 12.87 µg/mL of methanolic extract (Figure 2), whereas, according to Nariya et al. [61], the IC_{50} of ascorbic acid is 6.1 µg/mL. This indicates that ascorbic acid is approximately 120 times more effective at neutralizing free radicals, meaning that a significantly higher concentration of our DPAE is required to achieve a comparable antioxidant effect. These results confirm that microwave irradiation is the most effective method for extracting natural bioactive compounds from date pulp. Djaoud et al. [62] support our recommendation. Besides these properties, the natural date pulp extract obtained by microwave irradiation could constitute an ideal functional by-product as an adjuvant in food formulations, contributing effectively to the extension of product shelf-life [63].

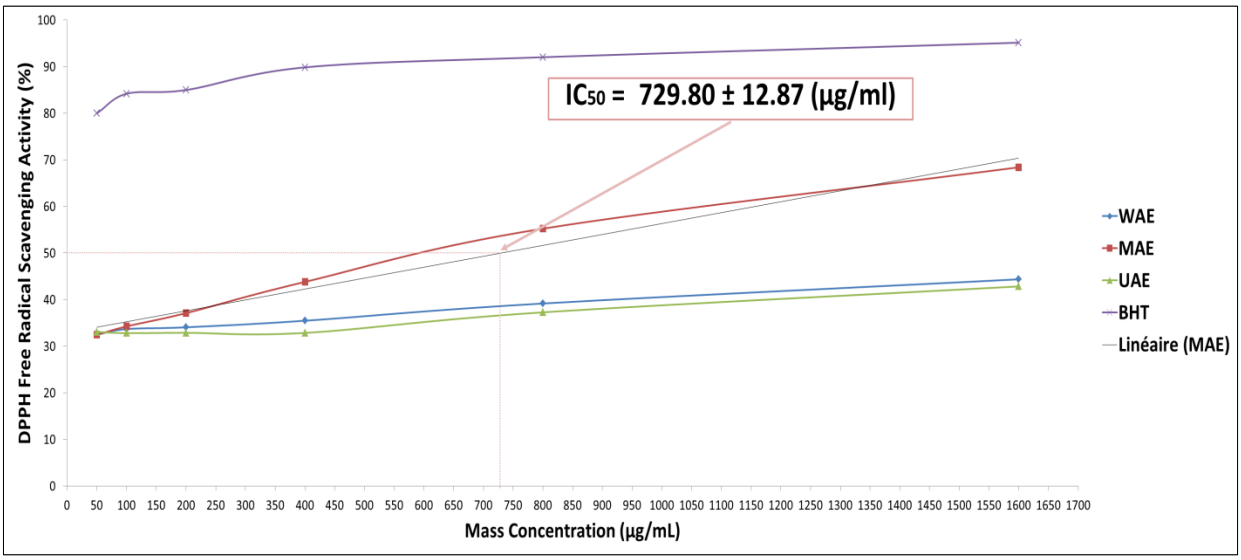

Figure 2. The antiradical scavenging activity curves of the three optimized DPAE.

3.5.6. Total soluble solids and sugar content

The TSS in our extracts varied substantially ($p < 0.05$), with MAE extract exhibiting the highest amount (14.33% ± 0.11% FW). Date pulp mostly consists of simple sugars and disaccharides, mainly glucose, fructose, and sucrose, which are readily absorbed by the body, accompanied by low levels of cellulose and starch [64]. Figure 3 shows the sugar chromatograms for the three extract samples obtained under the optimum conditions for each extraction method, using the GC-MS method.

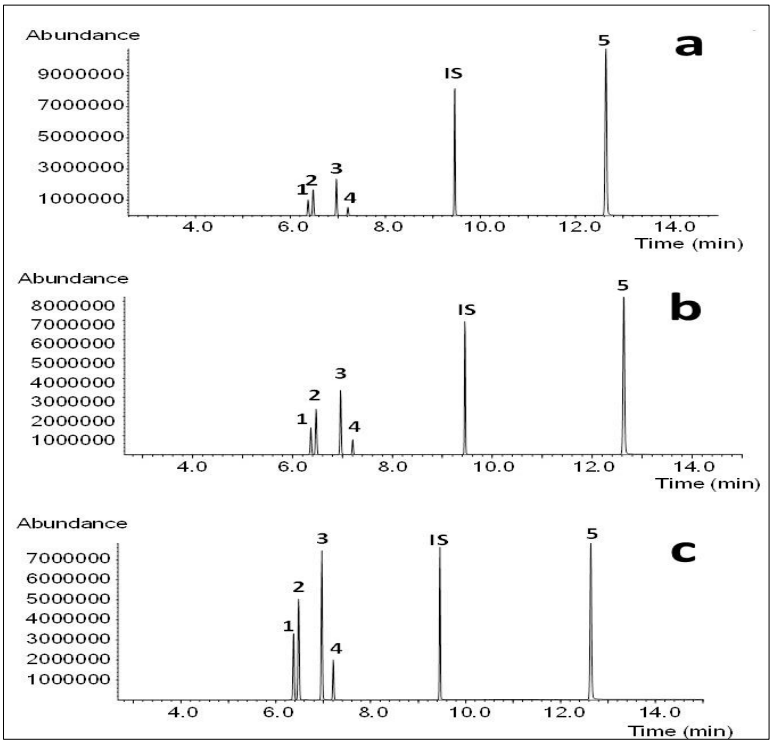

Figure 3. GC-MS chromatograms of the three DPAE samples derived from date pulp obtained by applying the optimum values of the three extraction methods: (a) WAE, (b) MAE, and (c) UAE. 1: D-Fructose, 1,3,4,5,6-pentakis-O-(trimethylsilyl)-,O-methyloxime(1); RT = 6.36 min. 2: D-Fructose, 1,3,4,5,6-pentakis-O-(trimethylsilyl)-,O-methyloxime(2); RT = 6.47 min. 3: D-Glucose, 2,3,4,5,6-pentakis-O-(trimethylsilyl)-,O-methyloxime(syn); RT = 6.96 min. 4: D-Glucose, 2,3,4,5,6-pentakis-O-(trimethylsilyl)-,O-methyloxime(anti); RT = 7.20 min. 5: Sucrose, α-D-Glucopyranosid,1,3,4,6-tetrakis-O-(trimethylsilyl)—β-D fructofuranosyl—2,3,4,6- tetrakis-O-(trimethylsilyl); RT = 12.63 min. IS: Internal standard: phenyl-β-glucopyranoside; RT = 9.45 min.

The five methylated sugar compounds, corresponding to peaks numbered 1–5, include the pyranose and furanose forms of fructose (*syn* and *anti* isomers numbered 1 and 2), the E and Z isomers of glucose, and sucrose. In addition, a blank pyridine sample was used as an internal standard. The RT in GC-MS analysis is influenced by sample treatment, injection conditions, and the type of compound analyzed [65], as well as by the stereo-chemical configuration of sugars (α and β isomers for monosaccharides and disaccharides) [66].

According to the data in Table 4, carbohydrates differed significatively ($p < 0.05$) between extraction methods, with the microwave method yielding the highest sugar extract (22.23 ± 0.23 g/100 mL of DPAE). More specifically, taking into account the inherent properties of the extracted carbohydrates, it was found that the amounts of reducing sugars (glucose and fructose) varied significantly between methods, with the UAE method offering the highest extraction yield (Figure 3). With regard to conventional methods, Messadi et al. [18] attempted to optimize the extraction of sugars from the Tunisian cultivar *Kentichi*, obtaining a total sugar content of 16.01 ± 0.03 g/100 mL aqueous extract, lower than our WAE sample (19.76 ± 0.11 g/100 mL). This difference can be attributed to sugar diffusivity coefficients of the pulp matrix, which vary according to the date pulp

texture between the two cultivars [17]. In this context, Gabsi et al. [67] developed a CFD (computational fluid dynamics) model to predict and compare the yield of natural sugar compounds extracted from three Tunisian cultivars: *Menakhe*r, *Lemsi*, and *Alligue*. The model takes into account the sugar diffusivity as a function of internal fruit matrix parameters, date/water ratio, and other process-specific characteristics. They found that optimal diffusion conditions were achieved at 50 rpm and 0.75, for stirring speed and date/water ratio, respectively, using the *Lemsi* date cultivar. This theoretical framework also explains the apparent differences between our results and those of Chaira et al. [16], who applied the conventional extraction method and found that the mean values of glucose and fructose amounts of the three date extracts from *Deglet Nour*'s cultivar were 2.31 ± 0.34 g/100 mL and 3.41 ± 0.68 g/100 mL, respectively, slightly higher than our samples (20.35 ± 0.83 and 22.08 ± 0.99 mg/mL of DPAE, respectively). These results could be explained by the fact that initial glucose and fructose amounts of the pulp of *Deglet Nour* cultivar with a semi-soft consistency were higher than those of *Mech Degla*, a dry one. Our proposals were based on the classification of 93 cultivars of Algerian date palm fruits into several consistency classes based on their sucrose and reducing sugar content established by Belguedj et al. [68].

3.5.7. Protein and fat contents

All extracts contained traces of proteins and lipids, with the highest being that extracted by MAE (0.24 and 0.18 ± 0.09 g/100 mL of DPAE, respectively), followed by the water bath method (0.23 ± 0.01 and 0.13 ± 0.05 g/100 mL of DPAE, respectively). In their study, Masmoudi et al. [69] found that date juice obtained using the conventional method (hot plate with continuous stirring) resulted in low protein content (1.79 ± 0.10 g/100 d dry matter).

3.5.8. Antibacterial activity

Figure 4 and Table 5 display the findings of the antibacterial activity of the three optimized DPAE.

The variable bacterial inhibition seems to be the result of the action of the different compounds present in the three DPAEs. On the other hand, it has been shown that the extracts studied do not show antibacterial activity, except for the one observed against *E. coli*, due to the inappropriateness of the diffusion method for poorly water-soluble agents. This proposal is supported by the results obtained by Hassim et al. [70].

In fact, only aqueous date extract obtained by the water bath method containing 1.42 mg GAE/mL polyphenols showed significant antibacterial activity against *E. coli* ($p < 0.05$) with an inhibition diameter of 18 ± 2 mm. However, the antibacterial activity of date extracts cannot be attributed only to their total polyphenol content but also to the presence of specific phenolic compounds, as demonstrated by several studies, including that of El Sohaimy et al. [71], which identified, by HPLC, several phenolic compounds present in aqueous date extracts obtained by the conventional method (date and distilled water stirred for 60 min at 25 °C) with antibacterial properties, namely phenolic acids (gallic acid: 7.51 ± 0.123 µg/g, itaconic acid: 6.40 ± 0.113 µg/g, and ferulic acid: 0.15 ± 0.194 µg/g), coumarins (esculetin: 15.11 ± 0.213 µg/g), and tannins (tannic acid: 2.85 ± 0.097 µg/g).

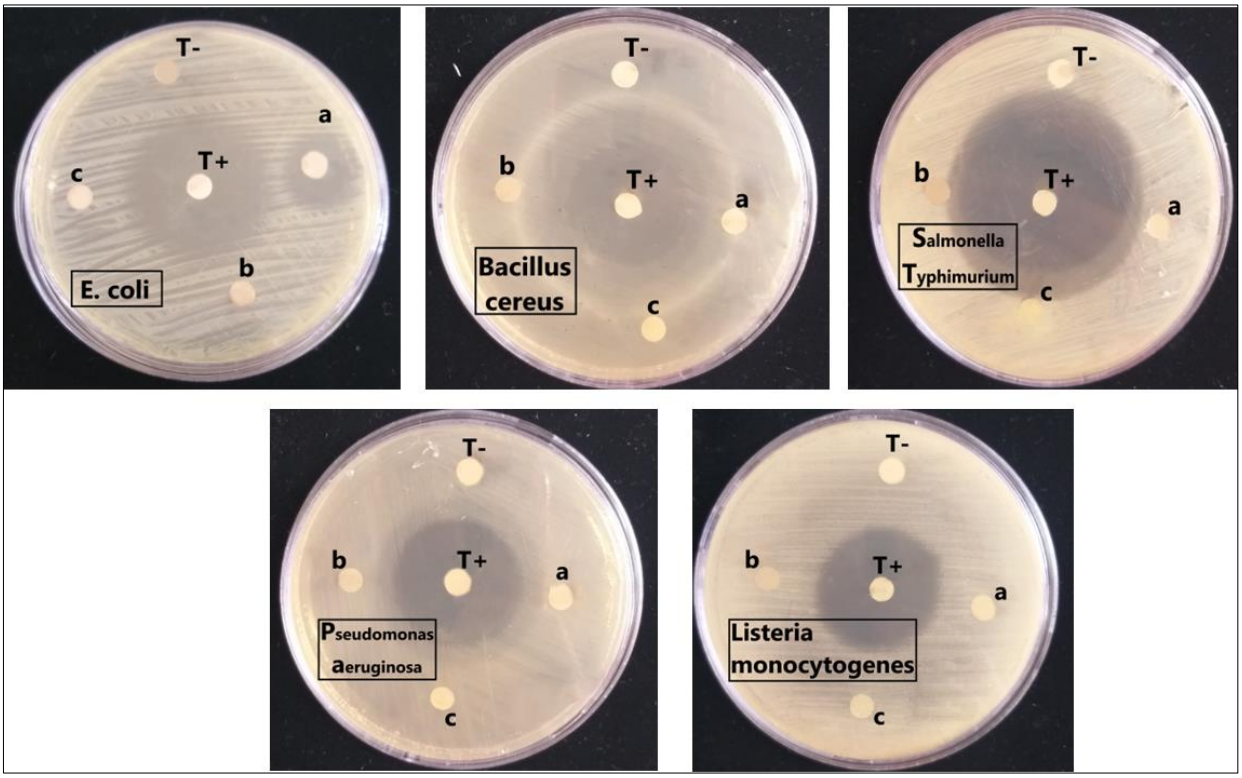

Figure 4. Antibacterial activity of the three optimized DPAE obtained using (a) WAE, (b) MAE, and (c) UAE. T- and T+ designate the positive (gentamicin) and the negative (2.5% DMSO) controls, respectively.

Table 5. Antibacterial activity of the three DPAE obtained using WAE, MAE, and UAE (diameter of inhibition zone per mm).

Strains / Extraction method	*E. coli* ATCC 25922	*B. cereus* ATCC 14579	*S. Typhimurium* ATCC 14028	*P. aeruginosa* ATCC 27853	*L. Monocytogenes* ATCC 35152
T^+	42.33 ± 2.52^a	41.67 ± 2.08^a	55.67 ± 4.04^a	34.66 ± 1.52^a	32 ± 1^a
WAE	18 ± 2^b	6^b	6^b	6^b	6^b
MAE	6^c	6^b	6^b	6^b	6^b
UAE	6^c	6^b	6^b	6^b	6^b
T^-	6^c	6^b	6^b	6^b	6^b

Data shown are mean ± SD (n = 3); superscripts a, b, and c indicate significant differences according to Tukey's test. Values with different superscripts within the same column are significantly different at $p < 0.05$.

However, no antibacterial effect was recorded for this extract against the other bacteria and for the two other extracts against all the bacteria tested ($p > 0.05$). In Saleh and Otaibi's study [72], no antibacterial effect against *E. coli* was detected for any of the aqueous date pulp extracts of the three Saudi cultivars studied (*Khulase*, *Sheshi*, and *Rezaza*). On the other hand, our results are in concordance with a previous study by Al-daihan and Shafi Bhat [73], in which date aqueous extracts from *Mosaifah* Saudi cultivar were proven to have anti-*E. coli* effect, with an inhibition zone of 11 ± 0.88 mm. Although this was effective, its effect was lower than that of our WAE extract. Also, this

extract produced a zone of inhibition of 11 ± 0.57 mm against *P. aeruginosa*, indicating a higher antibacterial efficacy than our extracts (6 mm). This shows that DPAE is less effective than some other alternative extraction methods, and that extraction conditions and date cultivars greatly affect antibacterial effectiveness, as confirmed by Bhaskaracharya et al. [74]. Furthermore, we can conclude that more intensive methods (such as those using water or thermal processes) seem to favor a better release of active antibacterial compounds. It is noteworthy that *Escherichia coli* is a Gram-negative bacterium recognized as one of the most common bacterial species encountered in nosocomial infections in humans, including urinary and enteric infections. This bacterium is also known for the development of bio resistance against some antibiotics [75]. The effectiveness of the DPAE obtained by the water bath method seems promising because it may constitute a natural alternative to antibiotics. Additionally, this efficiency suggests the presence of some specific anti-*E. coli* bioactive compounds like polyphenols (1.42 ± 0.03 mg GAE/mL of DPAE).

The MAE and UAE treatments offer rapid and efficient ways to extract bioactive compounds. However, the lack of antibacterial activity could be explained by the fact that the inhibitory effect of the targeted bioactive compound depends on its concentration in our UAE/MAE extract or by bacterial outer membrane resistance, as reported by Gowda et al. [76], i.e., under the optimal extraction conditions applied in the present study, they may inadvertently lead to the degradation of these compounds. In this context, as reported by Chan et al. [77], high microwave power raises the temperature of the solvent and matrix, which can degrade heat-sensitive compounds. For example, in the study of Lasunon et al. [78] on the effect of MAE on bioactive compounds from industrial tomato waste, an increase in power from 300 to 450 W resulted in a reduction of around 15% in trans-lycopene, highlighting the vulnerability of carotenoids and phenolic acids to excessive heat. Accordingly, it may be that the phenolic compounds of interest in our DPAE obtained in the optimal microwaving condition (480 W for 11 min) were degraded. In addition, thermogravimetric analysis (TGA) shows that temperatures in excess of 200 °C trigger the decomposition of organic compounds such as polyphenols and condensed tannins, diminishing antibacterial effects [79]. Regarding sonication treatment, as reported by Peng et al. [80], acoustic cavitation disrupts cell walls but can also degrade bioactive compounds. Collapsing bubbles generate hydrodynamic shear that fragments large molecules, as illustrated by a 67% drop in TFC in propolis after 40 min. In pomegranate skin extracts, this phenomenon is reflected in a 20% reduction in punicalagin with ultrasounds for 30–50 min. Accordingly, it is probable that the phenolic compounds that were the subject of our DPAE, which were extracted under optimal sonication conditions (40% amplitude for 60 min), were subjected to degradation. Our findings are in accordance with those of Abdennabi et al. [81] on MAE of phenolic compounds from date saps and antibacterial activity evaluation against five strains: *Pseudomonas aeruginosa*, *S. aureus*, *E. faecalis*, *Bacillus cereus*, and *Bacillus subtilis*. The sap extract (1 mg/mL) of the *Beser* cultivar showed no bacterial activity. Our recommendations are substantiated by the findings of Sandeep et al. [82], who discovered that at low concentrations (40 and 60 µL) of *Moringa* extract, the conventional soaking method exhibits a greater inhibition zone compared to the ultrasonic method, as well as the microwave method at higher concentrations (80 and 100 µL).

From a mechanistic point of view, the antibacterial effect observed with the WAE extract and the absence of this effect with extracts obtained by MAE/UAE depend closely on how these techniques modify the stability, solubility, and structural integrity of bioactive compounds. Rapid, intense microwave heating (often > 80 °C) can break hydrogen bonds and degrade heat-sensitive

compounds such as flavonoids and polysaccharides, impairing their ability to inhibit bacterial adhesion by altering interactions with microbial cell walls [83,84]. As for sonication, the shear forces generated by cavitation and localized hot spots fragment macromolecules. This process can oxidize certain phenolic acids, reducing their antimicrobial activity [85,86]. Aqueous WAE has been shown to be effective in inhibiting *E. coli* growth, although this depends on the plant species and the nature of the bioactive compounds. Its main mechanism of action is to disrupt the bacterial membrane, resulting in the release of intracellular components (nucleic acids and proteins) [87]. Thus, as highlighted in the study by Bessalah et al. [88], eight extracts obtained by WAE (50 °C for 30 min) disrupted the membrane of *E. coli* F17, causing the release of nucleic acids after 12 h of incubation. In addition, the hydrophobic surface of this strain was modified, and its motility was also altered in the presence of these extracts.

In the specific context, the absence of antibacterial activity of a plant extract is desirable or even imperative. Case in point: in a prebiotic formulation, a supplement containing the concentrated aqueous extract of date obtained by MAE and UAE can be useful to promote the growth of beneficial bacteria in the gut microbiota without inhibiting some probiotic strains of *E. coli*, such as Nissle 1917 (Mutaflor) and O83:K24:H31 (Colinfant) [89]. On the other hand, the product obtained from WAE may serve as a natural food preservative to hinder the propagation of *E. coli*. This can help extend the shelf life of perishable food products, such as meat and dairy products, by reducing the risk of bacterial contamination.

4. Conclusions

The current study highlights that the choice of aqueous extraction method of total soluble solids from date pulp is determined by the specific objective of the application, with each extract presenting distinct characteristics that influence the quality and properties of the final product. As such, it is essential to evaluate the specific attributes of each method and their potential impact. For instance, the significantly higher concentration of sucrose in the DPAE produced by WAE (139.12 ± 7.71 mg/mL of DPAE) indicates that it could be a potential source of refined sugar. Also, both non-conventional methods produced date extracts that are suitable for probiotic formulations against *E. coli* strains. The microwave method stands out for its efficiency in extracting total soluble solids, including total sugars (22.23 ± 0.23 g/100 mL of DPAE), while maintaining a high colorimetric intensity index (76.85 ± 0.46 × 10^{-3}) and an increased concentration of phenolic compounds (1.69 ± 0.04 mg GAE/mL of DPAE), reflecting a remarkable antioxidant capacity (with an IC_{50} of 729.80 ± 12.87 µg/mL for the methanolic extract). This makes microwave irradiation particularly suitable for the production of antioxidant-enriched date products for consumers seeking health benefits. In addition, this method offers a higher extraction recovery in a shorter time; MAE is three times faster than that required by conventional techniques, making it a promising option for pulp pretreatment or for use in combination with other techniques to improve material and energy transfer. The aqueous date extract obtained by sonication, rich in glucose (50.86 ± 3.68 mg/mL) and fructose (53.57 ± 4.16 mg/mL), enhances the perception of sweetness in foods. Thanks to the high sweetness intensity of fructose (1.2–1.7 times that of sucrose) and the moderate but persistent sweetness of glucose (0.7–0.8 times that of sucrose), this extract offers a superior sweetness to sucrose alone. Its balance of glucose and fructose allows less sugar to be added while maintaining optimal sweetness perception, making it an ideal ingredient for dietary products. In addition, ultrasound-assisted extraction is limited in its ability to recover all solid matter from date pulp. Therefore, we recommend further research aimed at

optimizing ultrasonic-assisted aqueous extraction (AUAE) to improve the recovery of reducing sugars from this pulp and to evaluate its impact. Regarding the antibacterial effect, since extraction is the first step in obtaining the constituents of medicinal plants, this requires that many factors are taken into account when choosing the most appropriate techniques. The use of appropriate methods guarantees the maximum production of plant compounds in sufficient quantities to perform the required antibacterial tests. We suggest further studies on water bath–assisted aqueous extraction (AWAE) to obtain a natural DPAE with high antibacterial activity (inhibition zone of 18 ± 2 mm) against *E. coli*. These results encourage the optimization of the production of natural extracts derived from date pulp, promoting their application as both functional and nutritious ingredients and strengthening the development of the date palm industry.

Use of AI tools declaration

The author(s) declare(s) they have not used generative Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of interest

The authors declare that we have no conflict of interest.

Author contributions

NB: Formal analysis, Experimental tasks, Data curation, Writing—original version, Writing—revision and editing; GR and RBS: Methodology, Technical support; KM: Conceptualization, Supervision, Project planning.

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