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ORIGINAL ARTICLE

Pomegranate seed oil extraction by cold pressing, microwave and ultrasound treatments

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Abstract

In this study, pomegranate seed oil was extracted by microwave-assisted extraction, ultrasound-assisted extraction, and cold pressing techniques. Dimethyl succinate was the solvent of microwave and ultrasound-assisted extraction methods. The optimum conditions were determined by using response surface methodology (RSM, Design Expert software version 7.0 and Box-Behnken design). The ultrasound-assisted extraction technique was found to be superior to the microwave-assisted extraction technique in terms of extraction efficiency. The maximum extraction efficiency of microwave-assisted extraction was 22.01% under the optimized conditions (liquid/solid ratio, 5/1; time, 3 min; and microwave power, 300 W). The maximum extraction efficiency obtained by ultrasound-assisted extraction was 26.31% under the optimized conditions (liquid/solid ratio, 10/1; pulse duration/pulse interval ratio, 1; temperature 60 °C; and time, 20 min). The extraction efficiencies were compared at the optimum conditions with hexane, which is the most used solvent for pomegranate seed oil extraction, and the difference was insignificant. The results of this study are very important, especially in the field of green chemistry and chemical engineering.

Keywords Pomegranate seed oil · Microwave-assisted extraction · Ultrasound-assisted extraction · Cold pressing

1 Introduction

Pomegranate (*Punica granatum L.*), belonging to Punicaceae's family, has been widely grown in the Mediterranean region. Pomegranate seeds are by-products of the pomegranate juice industry. Since the seeds are rich in essential oils with several health benefits, they deserve to be used in the food and pharmaceutical industry instead of as animal feed [1]. When extracted by conventional or modern techniques, pomegranate seeds are known to have around 20% oil content by weight, depending on fruit genotypes, cultivation, geographical locations, harvesting, and climate conditions. Pomegranate seed oil (PSO) is a yellow-colored product with a slight odor. It is chiefly used in biodiesel, lubricant, and paint formulations [1]. Later on, its applications have been expanded to cosmetics, pharmaceuticals, and nutrition [2–4]. PSO is rich in essential fatty acids. When the oil content of the pomegranate seed is examined, it is seen that it consists of 65–80% by weight of conjugated fatty acids. [3]. Punicic acid (PA) (also called an omega-5 conjugated linolenic acid) is the most abundant conjugated fatty acid in the pomegranate seed [5]. PA has several health benefits that help to prevent many diseases. PA has antioxidant, antimicrobial, anti-inflammatory, anti-atherosclerotic, anticancer, anti-estrogen, immunomodulatory, nephroprotective, hepatoprotective, and neuroprotective properties [6].

Due to the increasing trend towards natural ingredients in recent years, how these ingredients are produced has begun to be scrutinized, and environmentally friendly

Highlights
- In this study, pomegranate seed oil was extracted by microwave-assisted extraction, ultrasound-assisted extraction, and cold pressing techniques.
- In this study, dimethyl succinate was used as a green solvent in microwave-assisted extraction and ultrasound-assisted extraction systems, and the efficiencies were compared with hexane at the optimum operating conditions.
- The main goal of this study is to optimize the process conditions for pomegranate seed oil extraction to achieve the highest efficiency.
- Gas chromatography with a flame ionization detector (GC-FID) was used to analyze the oil content in pomegranate seeds.

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production methods have gained importance along with the concept of sustainable environmental management. At this point, researchers are working on new and environmentally friendly solvents and production methods [7–9]. The selection of an accurate method is the most important step in the extraction to obtain the target of interest effectively and efficiently. The most popular methods for oil extraction are microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), soxhlet extraction, cold pressing, and supercritical fluid extraction. However, some of these methods can be either time-consuming or costly and might destroy the structure of thermally sensitive compounds. Cold pressing is a mechanical technique that relies on pressure. The cold pressing method is environmentally friendly because it does not require any organic solvent. Besides, cold-pressed oils have better physiochemical properties like low peroxide value, high phenolic content, and good aroma [10]. Even so, the oil efficiency is low. Soxhlet is one of the most used oil extraction methods, but this method is outdated due to the hazardous solvent usage and low oil recovery [11]. Supercritical fluid extraction is a selective, rapid, and clean process. It does not require any solvent; therefore, no residue is left in the oil extract. Nevertheless, the capital and investment costs are the obstacles to further commercialization [12].

MAE is an effective oil extraction method from plant seeds [13–16]. The ion transmission and dipole rotation are the keys to microwave heating. Microwaves are high-frequency electromagnetic waves, the frequency of which changes between 300 MHz and 300 GHz. At this frequency range, the waves are absorbed by the polar oxygen group of water in the plant material. Although seeds are dry, they have moisture, which is the target for microwave heating. When the plant is heated, the moisture evaporates and applies pressure to the cell membrane. Ultimately, the cell membrane explodes, and consequently, bioactive compounds move out of the pores and dissolve in the solvent. MAE is a promising technology because it reduces extraction time and saves energy due to the electromagnetic field conveying heat throughout the material [17]. Factors affecting the selection of the proper solvent are the ability of the solvent to absorb microwave radiation, the interaction between the solvent and the plant, and the solubility of the sample in the solvent. Solvents with high dipole moments with a polar characteristic absorb the microwave energy better and may get heated faster. On the other hand, non-polar solvents having low dipole moments may not heat the sample effectively [18].

UAE is another alternative technique to the traditional ones. Several bioactive compounds have been extracted from plant matrixes by UAE [19–23]. The main advantages of UAE are lower operation temperatures, less solvent consumption, and lower extraction times [5]. These moderate operating conditions reduce the energy input and protect the structural properties of thermally sensitive bioactive compounds. The mechanical vibrations induced by ultrasound waves create cavitation bubbles during the expansion and contraction cycle. The formed bubbles weaken due to the continued expansion and contraction cycle. Ultimately, the bubbles implode, bringing high energy through heat, pressure, and mechanical shear [24]. The implosion near the solid surface causes plant tissue rupture and particle breakdown, releasing intercellular molecules into the solvent [25].

Hexane is chiefly used in vegetable oil extractions due to its apolar characteristic, low vapor pressure, low cost, high stability, and high solvation ability [17, 26–28]. Today, hexane still remains a popular option. However, hexane is produced from non-renewable petroleum products. It is toxic and flammable [29]. Hexane consumption is questioned due to some possible residues in the extract. Because of global concerns about environmental pollution, there is a growing demand to use environmentally friendly solvents. In this context, dimethyl succinate can be considered as a great option. Dimethyl succinate has several properties that make it profitable to use as a solvent instead of hexane. It is less toxic, less flammable, and more biodegradable than hexane [30].

The main goal of this study is to optimize the process conditions for PSO extraction to achieve the highest efficiency. In this study, dimethyl succinate was used as a solvent in MAE and UAE systems, and the efficiencies were compared with hexane at the optimum operating conditions. For MAE, the optimum operating conditions (liquid/solid ratio, time and microwave power) were determined using Box-Behnken design of response surface methodology (RSM). Similarly, for UAE, the optimum operating conditions (liquid/solid ratio, pulse duration/pulse interval ratio, temperature, and time) were determined by using Box-Behnken design of RSM. Cold pressing is used as a reference method to compare the results obtained by MAE and UAE methods. Gas chromatography with a flame ionization detector (GC-FID) was used to analyze the oil content in pomegranate seeds at the optimum extraction conditions. At the end of the paper, the oil contents and efficiencies were compared in detail with other studies reported in the literature. Here we propose a novel and green approach to obtain essential oils from PSO.

2 Materials and methods

2.1 Pomegranate seed preparation

Pomegranate seeds were purchased from a local market (Istanbul, Turkey). All the seed samples were washed with tap water and rinsed with distilled water to remove dust and impurities. Further, the seed samples were dried naturally in

the air. The dried seed samples were stored in polyethylene bags at −4 °C until analysis. Before the experiments, the material was ground with a mixer mill and passed through a 60-mesh sieve to obtain homogeneous particle sizes.

2.2 MAE procedure

PSO microwave-assisted extraction was performed in a microwave laboratory oven (Milestone, NEOS-GR Bergamo, Italy) equipped with a time controller, a temperature sensor, a power regulator, and a circulating water-cooling system. The input microwave power was altered from 200 to 300 W at 2450 MHz frequency. Efforts were made to create a synergistic combination of the process variables namely microwave power, liquid/solid ratio and microwave time. Therefore, the input variables were selected based on the preliminary trial tests. During the treatments, power, temperature and time were controlled by a control panel. In the beginning, 2 g of seeds was added to the flask containing the solvent. Then, the flask was placed on the turntable plate of the microwave oven. The microwave power, time, and solid/liquid ratio entered into the Design-Expert program were determined through preliminary trials. After the extraction procedure, the mixture was centrifuged at 4000 rpm for 20 min to obtain a clear layer of solvent-oil mixture at the top of the tube. After that, the sample was filtered on a 0.45-µm filter paper. The solvent was removed from the mixture by lyophilization (Virtis Advantage, SP Scientific, Suffolk, UK). Lyophilization (freeze-drying) is a process in which solvent is sublimated from the product after freezing at a specified temperature and pressure. The oil was stored in a freezer until GC-FID analysis. The extracted oil was characterized, and the oil efficiency was determined according to the formula below. The extraction procedure was repeated thrice, and the mean value was taken for the calculations to minimize measurement errors.

$$\text{Oil yield}(\%) = \text{m(oil extracted;mL)}/\text{m(pomegranate seed;g)} \times 100 \quad (1)$$

2.3 UAE procedure

Ultrasound was applied with an ultrasonic probe (Sonics VCX500) operating at 25 kHz. First, 2 g of seed powder was placed into the flask and mixed with a specified amount of the solvent, and then the extraction was achieved by applying acoustic energy with an ultrasonic probe. Attempts were made to establish a successful combination of process variables. Thus, the variables were selected after conducting preliminary trial tests. The solid/liquid ratio, pulse duration/pulse interval ratio, temperature, and time have been determined based on the results of preliminary trials. The intervals were selected based on the possible optimal efficiencies that the extraction system can provide. The temperature of the ultrasonic-assisted extraction system was monitored through the control panel. After extraction, the mixture was centrifuged and filtered under the same conditions with microwave treatment. After that, the solvent was removed by lyophilization and stored in the freezer until the analysis. The oil efficiency was determined as described above. The extraction procedure was repeated three times to ensure consistency, and the average value was taken for the calculation.

2.4 Cold pressing procedure

Pomegranate seed was pressed by a cold pressing machine (NF 80, Turkey) that operates at room temperature. The extract was filtered and centrifuged at 4000 rpm for 20 min to separate the oil. Then, the oil was stored until analysis. Three independent experiments were done for each analysis, and the average value was taken. Similarly, the oil efficiency was determined according to the formula above.

2.5 GC-FID analysis

The fatty acid composition of extracted oils was determined by GC-FID analysis after deriving fatty acid methyl esters (FAMEs) [26]. The chromatographic analysis was performed on a gas chromatograph (Shimadzu GC 2010 plus) equipped with a flame ionization detector and a fused silica capillary column (20 m × 0.18 mm). Air was used as the carrier gas at a 1 mL/min flow rate. The samples were injected in split mode. The split ratio was 1:100; the sample injection volume was 1 µl. All samples were injected in triplicate. The injection port and detector temperature were maintained at 250 °C. The oven temperature program was operated as follows: initial temperature were 80 °C then temperature increase to 160 °C at 40 °C/min speed rate, and increase from 160 to 180 °C at 5 °C/min, finally hold isothermally constant at 185 °C for 15 min. The peaks were identified based on the retention times using commercial reference compounds (Sigma Aldrich, USA).

2.6 Statistical analyses

The Box-Behnken design of response surface methodology (Design Expert software, State-ease Inc., Minneapolis, USA, Version: 7.0) was applied for optimization purposes. RSM is used to achieve maximum response with the minimum number of experiments. RSM also examines the individual and combination effects of the factor variables. The MAE variables were liquid/solid ratio (A), time (B), and microwave power (C), while the response was the extraction yield (Y). The interactions were investigated with 16 runs. The design matrix is presented in Table 1. The UAE variables were liquid/solid ratio (A), pulse duration/pulse interval

Table 1 Symbols and levels for MAE optimization

Parameters	Units	Symbols	Levels		
Liquid/solid ratio	mL/g	A	5	7.5	10
Time	min	B	2	3	4
Microwave power	W	C	200	250	300

ratio (B), temperature (C), and time (D). The response was the extraction yield (Y). The interactions were investigated with 27 runs. The UAE design matrix is shown in Table 2. The experimental data were fitted to a second-order polynomial equation in Eq. (2).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} x_i x_j + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \varepsilon \quad (2)$$

Y is the extraction efficiency (%), X_i and X_j are the independent variables. β_o, β_i, β_{ii} and B_{ij} are the regression coefficients. k refers to the number of independent variables, and ε signifies the residual term. The statistical importance of the results was evaluated by the analyses of variance (ANOVA) test.

3 Results and discussion

3.1 Optimization of MAE conditions

In the MAE optimization study, the examined parameters were liquid/solid ratio, time, and microwave power (see Table 3). The extraction efficiency was analyzed by a second-order polynomial equation, and the data were fitted into Eq. (3) by multiple regression procedure.

$$\begin{aligned} Y = {} & 12.40 - 1.44A + 2.31B - 2.78C - 1.76AB \\ & - 2.90AC - 4.13BC - 2.89A^2 + 0.34B^2 \\ & + 0.057C^2 - 1.92A^2B + 3.48A^2C + 0.28AB^2 \end{aligned} \quad (3)$$

In the above polynomial equation, a positive coefficient implies a positive effect on the response, and a negative coefficient indicates an inverse relationship. The higher the coefficient in the absolute value is, the stronger the effect becomes.

Table 2 Symbols and levels for UAE optimization

Parameters	Units	Symbols	Levels		
Liquid/solid ratio	mL/g	A	5	7.5	10
Pulse duration/ pulse interval ratio	–	B	1	3	5
Temperature	°C	C	40	50	60
Time	min	D	20	30	40

Table 3 MAE results of different treatments

Treatment	Liquid/solid ratio (X_1)	Time (min) (X_2)	Power (Watt) (X_3)	Yield (%) (Y)
1	7.5	3	250	12.14
2	5	3	300	22.01
3	5	4	250	10.31
4	7.5	4	200	10.05
5	7.5	2	200	8.86
6	5	3	200	9.13
7	7.5	3	250	13.9
8	10	3	200	11.84
9	7.5	3	250	12.25
10	7.5	2	300	13.16
11	10	2	250	14.61
12	7.5	3	250	11.3
13	7.5	4	300	7.32
14	10	4	250	5.94
15	10	3	300	8.2
16	5	2	250	7.4

ANOVA test was conducted to evaluate the statistical suitability of the derived model equation. The coefficient of determination value converging to the unity ($R^2 = 0.98$) indicated the good fit of the model to the experimental data. The adequate precision (AP = 16.38) measures the signal to noise ratio, and a ratio value greater than 4 is desirable. The coefficient of variance (% CV = 9.76) value indicated that there are no significant differences between the actual and predicted values. As seen in Table 4, the findings indicate statistically significant differences between the treatments. It is evident that the model is significant, as suggested by the model F value (F = 15.24) and the model probability value ($p = 0.02$). The important variables ($p < 0.05$) were B and C from the linear coefficients, AB, AC, BC, and A^2C from the interaction coefficients, and A^2 from the quadratic coefficients [6].

The extraction efficiency results of different treatments can be seen in Table 3. The oil efficiency range varied from 5.94 to 22.01%. The maximum efficiency was achieved by Treatment 2, while the minimum efficiency was obtained by Treatment 14. The combined effects of microwave time and liquid/solid ratio can be seen, when the results of treatment 2 and 14 are compared. It can be seen that increasing the liquid/solid ratio together with microwave power facilitated favorable interactions [31]. Thermodynamically, elevating the temperature leads to an increase in the kinetic energy of the molecules, more rapid diffusion of molecules, and thereby enhancing the extraction efficiency [32]. However, keeping the microwave duration excessively long leads to

Table 4 ANOVA results of MAE

Factors	Sum of squares	Degrees of freedom	Mean square	*F* value	*p* value
Model	216.47	12	18.04	15.24	0.0229
A	8.29	1	8.29	7.01	0.0772
B	21.34	1	21.34	18.04	0.0239
C	30.80	1	30.80	26.03	0.0146
AB	12.36	1	12.36	10.44	0.0482
AC	33.52	1	33.52	28.33	0.0130
BC	68.23	1	68.23	57.66	0.0047
A^2	33.41	1	33.41	28.23	0.0130
B^2	0.46	1	0.46	0.39	0.5762
C^2	0.013	1	0.013	0.011	0.9225
A^2B	7.35	1	7.35	6.21	0.0883
A^2C	24.29	1	24.29	20.53	0.0201
AB^2	0.15	1	7.35	6.21	0.0833
Source	Sum of squares				
R^2	0.9839				
R_{adj}^2	0.9193				
AP	16.389				
% CV	9.76				
SD	1.09				
Lack of fit	7.4				

the degradation of value-added products and unnecessary energy use [7]. Therefore, optimizing process variables is crucial for both process cost and extraction efficiency [25].

Three-dimensional (3D) response surface plots were constructed to study the interactive effects of process variables on oil efficiency. Two of the three variables were changed, while the other was kept constant at the center point to examine the variables' binary effects.

The binary effect of microwave power and treatment time on the oil efficiency is shown in Fig. 1a. Increasing microwave power from 200 to 300 W enhanced the efficiency for 2 and 3 min extraction times. As known, increased microwave power results in higher temperatures in the extraction system. Due to the increment in temperature, the cell structure was damaged more effectively, so extracts could diffuse into the solvent. Moreover, at higher temperatures, the oil solubility increased and solvent viscosity decreased, which promoted the penetration of the solvent into the sample matrix [6]. However, for 4 min duration, increasing microwave power from 200 to 300 W was found to be unfavorable, which is quite likely due to the degradation and volatilization of oil with longer irradiation times [33]. At longer irradiation times, extreme temperatures might have caused severe cellular damage, and thus, impurities may have leached out of the cell and penetrated the solvent, thus decreasing efficiency [34].

Generally, increasing the liquid/solvent ratio promotes the mass transfer between two phases due to the higher concentration gradient, so the efficiency increases. A high amount of solvent can dissolve all the oil. On the other hand, a lower solvent amount would lead to non-uniform distribution and incomplete extraction. Surprisingly, PSO removal efficiency declined sharply as the liquid/solid ratio increased from 5:1 to 10:1 (see Fig. 1b and c). Apparently, excessive solvent usage limited solvent movement, and for this reason, the efficiency decreased. At this point, unnecessary solvent usage is not profitable regarding process economy and waste management [33].

Extraction time is also an important factor affecting oil efficiency. A longer irradiation time can be unfavorable after a point due to the possible degradation of bioactive compounds [33]. The increase in extraction time inversely affected PSO removal efficiency (see Fig. 1a, b). At the end of 4 min, the solvent might have volatilized, which made it difficult to interact with PSO. Besides, the same active compounds might have been absorbed on the sample surface because of the long irradiation time [35].

3.2 Optimization of UAE conditions

The treatments and the oil efficiency results are written in Table 5. PSO efficiency varied from 6.71 to 26.3%. The maximum oil efficiency (%26.3, Treatment 21) was achieved under the process conditions: 7.5 liquid/solid ratio, 3 pulse interval/pulse duration ratio, 50 °C temperature, and 20 min time. UAE conditions were optimized by

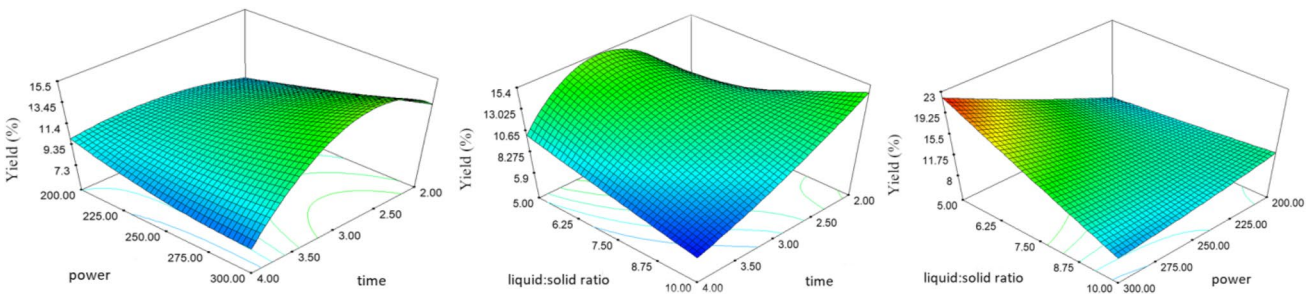

Fig. 1 Binary effects of **a** power and time, **b** liquid/solid ratio and time, and **c** liquid/solid ratio and power on the yield

Table 5 UAE results of different treatments

Treatment	Liquid/solid ratio (X_1)	The pulse duration/pulse interval ratio (X_2)	Temperature (C°) (X_3)	Time (min) (X_4)	Yield (%) (Y)
1	7.5	1	50	40	16.7
2	5	5	40	20	17.6
3	10	5	60	20	9.66
4	10	1	60	60	17.18
5	7.5	3	60	40	25.42
6	5	5	60	20	11.39
7	7.5	3	50	40	17.65
8	7.5	3	40	40	12.14
9	5	3	50	40	13.55
10	10	1	40	20	9.77
11	10	5	40	20	14.17
12	7.5	3	50	40	17.54
13	7.5	3	50	60	24.52
14	7.5	3	50	40	17.36
15	7.5	5	50	40	8.17
16	10	1	60	20	26.31
17	5	5	40	60	9.087
18	5	1	60	20	24.57
19	10	5	60	60	14.64
20	5	1	60	60	22.3
21	7.5	3	50	20	26.3
22	10	1	40	60	9.14
23	5	1	40	60	6.71
24	10	5	40	60	20.91
25	10	3	50	40	16.77
26	5	5	60	60	20.13
27	5	1	40	20	11.28

using the design of RSM (see Table 6). A second-order polynomial equation was developed to relate the response and operational variable and written in Eq. (4). The variable D was the most significant factor because it has the highest positive coefficient value in Eq. (4).

$$\begin{aligned} Y(\%) =& 18.29 + 1.61A - 4.27B + 6.64C - 0.89D + 0.23AB \\ & - 1.24AC + 0.54AD - 3.71BC + 1.78BD + 0.58CD \\ & - 3.52A^2 - 6.25B^2 + 0.098C^2 + 6.73D^2 - 0.71ABC \\ & + 0.90ABD - 1.86ACD + 1.36BCD + 3.66A^2B \\ & - 3.67A^2C + 0.60A^2D - 1.69AB^2 \end{aligned} \quad (4)$$

The data was analyzed by ANOVA test. The model's F and *p* values were 22.89 and 0.0039, respectively. Therefore, the model was found to be significant. The model's regression coefficient value (R2 = 0.99) implied that the model is potentially correct. The signal to noise ratio (AP) obtained is 16.52, which indicates an adequate signal; hence, the model can be used to navigate the design space. The low value of the coefficient of variance (% CV = 8.28) indicated a high degree of precision.

3-D surface plots were used to study the interactive effect of the operating variables on the response. The contour plots were drawn by changing two variables while keeping the other variables constant at the center points.

Obviously, increasing the temperature from 40 to 60 °C enhanced the efficiency (see Fig. 2b, d, and f). With higher temperatures, the mass transfer from solid matrix to bulk liquid is enhanced, likely due to decreased solvent viscosity and density [36]. Nevertheless, a further increase in temperature would adversely affect PSO efficiency, such as that of Rojo-Gutiérrez et al.'s study [6]. They studied the temperature effect for PSO extraction by UAE and reported that the efficiency decreased from 45 to 60 °C. They explained this outcome with the vapor pressure effect. As known, the solvent's vapor pressure directly affects the acoustic cavitation's intensity and formation. At lower temperatures, the

Table 6 ANOVA results of UAE

Factors	Sum of squares	Degrees of freedom	Mean square	*F* value	*p* value
Model	920.80	22	41.85	22.89	0.0039
A	5.18	1	5.18	2.84	0.1675
B	36.38	1	36.38	19.90	0.0112
C	88.18	1	88.18	48.23	0.0023
D	1.58	1	1.58	0.87	0.4046
AB	0.82	1	0.82	0.45	0.5386
AC	24.78	1	24.78	13.56	0.0212
AD	4.59	1	4.59	2.51	0.1881
BC	220.57	1	220.57	120.65	0.0004
BD	50.93	1	50.93	27.86	0.0062
CD	5.40	1	5.4	2.95	0.1609
A^2	31.89	1	31.89	17.44	0.0140
B^2	100.34	1	100.34	54.89	0.0018
C^2	0.025	1	0.025	0.014	0.9128
D^2	116.41	1	116.41	63.67	0.0013
ABC	8	1	8	4.38	0.1046
ABD	12.98	1	12.98	7.1	0.0561
ACD	55.55	1	55.55	30.39	0.0053
BCD	29.41	1	29.41	16.09	0.0160
A^2B	23.82	1	23.82	13.03	0.0226
A^2C	23.95	1	23.95	13.10	0.0224
A^2D	0.64	1	0.64	0.35	0.5864
AB^2	5.08	1	5.08	2.78	0.1708
Source	Sum of squares				
R^2	0.9921				
R_{adj}^2	0.9488				
AP	16.529				
% CV	8.28				
SD	1.35				
Lack of fit	7.7				

solvent has a lower vapor pressure; therefore, less number of bubbles form. Even so, the acoustic cavitation is high, and bubbles collapse with high intensity, which disrupts the cell membrane more effectively. On the other hand, at higher temperatures, the vapor pressure of the solvent increases, and thus, more bubbles form. However, the bubbles collapse with less intensity because of the small pressure difference between the internal and external medium of the bubbles, which limits the cell tissue disruption. In addition to that, Gaulo's study on PSO claims similar results [37]. The main reason why our study revealed the opposite results might be explained by the solvent type difference. Both of the above mentioned studies used hexane as the solvent. At the same temperatures, the vapor pressure of dimethyl succinate and hexane is different; expectedly, it affects the extraction efficiency differently.

The liquid/solid ratio of 7.5 gave higher efficiencies than the ratio of 5 (see Fig. 2a, b, and c), possibly due to the increased driving force for the mass transfer between solid particles and bulk liquid in the case of a higher solvent amount [25]. However, increasing the ratio to 10 caused a sharp decline in efficiency. This might be due to the fact that increasing the contact surface area between the solvent and targeted compounds enabled decreased penetration of solvent into the sample matrix and thus decreased the extraction efficiency [34].

Within the first 20 min, the oil efficiency reached the maximum value, and no significant change was observed up to 60 min. Apparently, the cell membrane was completely damaged, and the solvent sufficiently penetrated the solid matrix in 20 min. The mechanical effect of cavitation, along with the high shear force and local turbulence generated around the bubbles, leads to the disruption of the plant matrix. Impurities may have arisen due to the extended extraction time at the 40th minute. While more oil may have been obtained at the 60th minute, 20-min duration can be determined as an appropriate extraction time, as an extended period may lead to the emergence of impurities along with the oil (see Fig. 2c, e and f) [38].

Regarding the process economy, there is no need to prolong UAE time. Besides, prolonged extraction times would cause more solid matrix degradation, so impurities like pectin, cytosol, gums, and lipid suspends would limit the oil recovery.

The pulse duration/pulse interval ratio is also a significant factor affecting the oil efficiency. The efficiency decreased as the pulse duration/pulse interval ratio increased from 3 to 5. A higher duration to interval ratio means less processing time, and less processing time is not enough to complete the mass transfer. Similar results were obtained by Goulo's study [37].

Microwave-assisted extraction achieved a peak extraction efficiency of 22.01% when operating under optimized conditions, including a liquid/solid ratio of 5:1, a 3-min extraction time, and a microwave power of 300 W. Under the specified optimal conditions (liquid/solid ratio of 10:1, pulse duration/pulse interval ratio of 1, temperature of 60 °C, and a 20-min extraction time), ultrasound-assisted extraction yielded a maximum extraction efficiency of 26.31%. The optimum conditions determined by the program have been confirmed with experimental studies. When experiments were conducted under optimum conditions, it was observed that the yield of microwave-assisted extraction under optimum conditions was 22.5%, while the yield of ultrasonic-assisted extraction was 26.7%. Clearly, the predicted efficiencies by the program under optimum conditions closely matched the experimental results at optimum conditions. Consequently, it was observed that the program is adequate in predicting the yield under optimum conditions [38, 39].

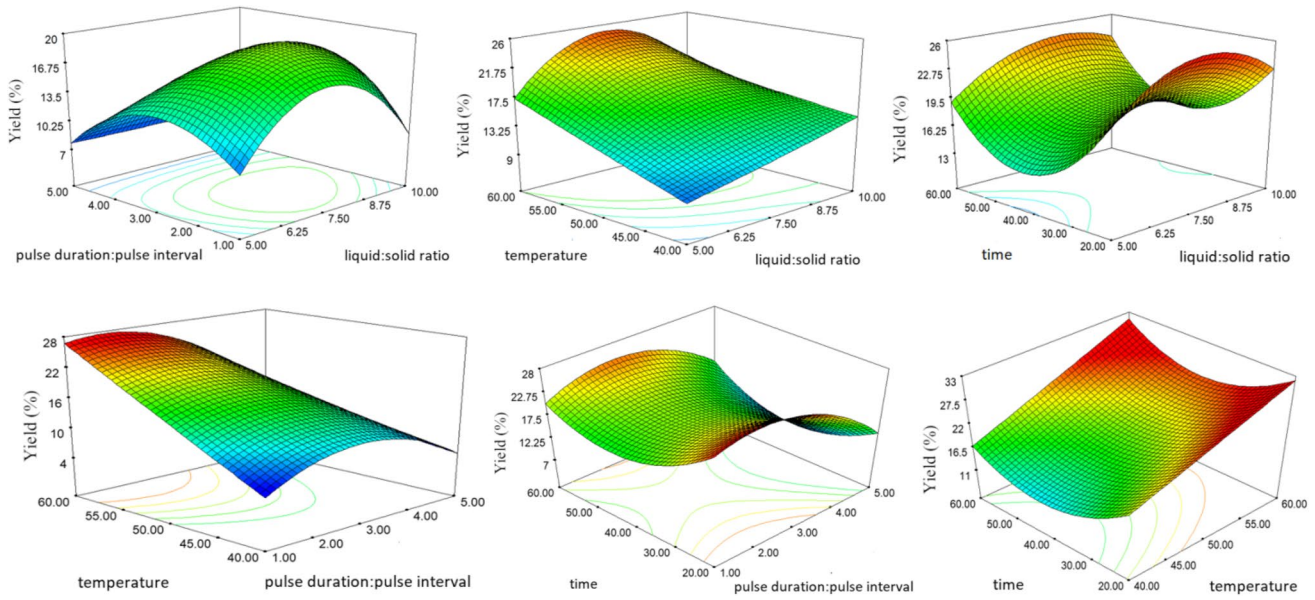

Fig. 2 Binary effects of **a** pulse duration/pulse interval ratio and liquid/solid ratio, **b** temperature and liquid/solid ratio, **c** time and liquid/solid ratio, **d** temperature and pulse duration/pulse interval ratio, **e** time and pulse duration/pulse interval ratio, and **f** time and temperature on the yield

3.3 Oil content analysis

The cold pressing method gave 5% oil efficiency. Punicic acid (88.33%) ranked first, followed by oleic acid (3.68%), linoleic acid (3.22%), palmitic acid (2.19%), stearic acid (1.56%), *cis*-11-Eicosenoic acid (0.64%), linolenic acid (0.36%), and lignoceric acid (0.02%).

MAE extraction efficiency with dimethyl succinate was between 5.94 and 22.01%. Punicic acid (87.65–88.15%) ranked first, followed by oleic acid (1.58–3.70%), linoleic acid (3.32–3.64%), palmitic acid (2.20–2.69%), stearic acid (1.55–1.66%), *cis*-11-Eicosenoic acid (0.57–0.64%), linolenic acid (0.30–0.64%), lignoceric acid (0.00–0.04%), and margo oleic acid (0.00–0.04%).

MAE extraction efficiency with hexane was 25.3%. Punicic acid (88.39%) came first, followed by oleic acid (3.55%), linoleic acid (3.27%), palmitic acid (2.19%), stearic acid (1.58%), *cis*-11-Eicosenoic acid (0.64%), linolenic acid (0.37%), and lignoceric acid (0.02%).

UAE extraction efficiency varied from 6.71 to 26.3%. Punicic acid (74.4–88.0%) was the first, followed by linoleic acid (3.4–7.5%), oleic acid (3.6–7.1%), palmitic acid (2.3–5.0%), stearic acid (1.6–3.1%), *cis*-11-Eicosenoic acid (0.6–1.1%), linolenic acid (0.3–0.8%), lignoceric acid (0.08–0.5%), arachidic acid (0.03–0.1%), and heptadecanoic acid (0.0–0.1%).

UAE extraction efficiency with hexane was 31.2%. Punicic acid (75.26%) ranked first, followed by oleic acid (8.23%), linoleic acid (8.03%), palmitic acid (3.94%), stearic acid (2.28%), *cis*-11-Eicosenoic acid (1.47%), linolenic acid (0.72%), and lignoceric acid (0.08%). All the results are written in Table 7.

MAE and UAE extraction procedures were performed with hexane under optimized conditions in order to reveal the extraction ability difference between hexane and dimethyl succinate. The maximum extraction efficiency for MAE was 22.01% under the optimum conditions (liquid/solid ratio of 5, irradiation time of 3 min, and microwave power of 300 W). For UAE, the maximum extraction efficiency was 26.3% under the optimum conditions (liquid/solid ratio, 10; pulse duration/pulse interval ratio, 5; duration, 20 min; and temperature, 60 °C).

Solvents that can dissolve the target of interest and absorb microwave radiation are more suitable for MAE. Hexane recovered more oil than dimethyl succinate from pomegranate seed, which is most likely due to the better dissolving and absorbing ability. Hexane has a higher polarity than dimethyl succinate, directly affecting the microwave radiation absorbing performance. For this reason, it is one of the most studied solvents in the literature [26–28].

Table 7 The maximum extraction efficiency obtained by different treatments

Extraction method-solvent	Oil efficiency (%)
MAE-hexane	25.3
MAE-dimethyl succinate	22.01
UAE-hexane	31.2
UAE- dimethyl succinate	26.3
Cold pressing	5

Table 8 Literature studies on PSO extraction

Ref	Growing region	Extraction method/solvent	Best conditions	Maximum yield (%)
Barizão et al. [26]	Sao Paulo, Brazil	UAE: hexane SE: hexane BDE: chloroform-methanol-water	SE: seed powder, 5 g; time, 4 h; and temperature, 68 °C	SE: 30% UAE: 27.99% BDE: 22.3%
Tian et al. [36]	Xinjiang, China	UAE: n-hexane, ethyl acetate, diethyl ether, acetone, and isopropanol SE: n-hexane SFE: liquid CO_2	UAE: petroleum ether; ultrasonic power, 140 W; temperature, 40 °C; and liquid/solid ratio, 10 mL/g	UAE: 25.11% SE: 20.50% SFE: 15.72%
Rojo-Gutiérrez et al. [6]	Hidalgo, Spain	MAE: hexane UAE: hexane	MAE: temperature, 90 °C; time, 10 min; and solid/solvent ratio, 1:20 g/mL	MAE: 18.38% UAE: 17.64%
Goula et al. [37]	Thessaloniki, Greece	UAE: hexane	UAE: seed particle size, 0.2 mm; temperature, 20 °C; solvent/solid ratio, 20/1 mL/g; amplitude level, 60%; and pulse duration/pulse interval ratio, 5/15	UAE: 59.8%
Eikani et al. [42]	Fars Province, Iran	SHHE: n-hexane SE: n-hexane Cold pressing	SHHE: temperature, 80 °C; particle size, 0.25 mm; and flow rate, 1 mL/min	SHHE: 22.18% SE: 17.94% Cold pressing: 4.29%
Silva et al. [43]	Saveh, Iran	Aqueous extraction Hexane extraction Cold pressing Hot pressing	Aqueous extraction: water/solid ratio, 2.2:1.0 mL/g; pH, 5; temperature, 63 °C; and time, 375 min	Aqueous extraction: 19.3% Hexane extraction: 26.8% Cold pressing: 7.0% Hot pressing: 8.6%
Cavdar et al. [44]	Nigde, Turkey	MAE: n-hexane SE: n-hexane Cold pressing	MAE: power, 220 W; liquid/solid ratio, 10; and extraction time, 5 min	MAE: 35.19% SE: 34.70% Cold pressing: 17.50%
Aruna et al. [45]	Navi Mumbai, India	SE: hexane, petroleum ether, chloroform, chloroform: methanol (2:1 v/v), and ethanol Cold pressing	SE: hexane; time, 8 h; and temperature, 30 °C	SE: 20% Cold pressing: 13.2%
Liu et al. [46]	Lintong, China	SE: n-hexane, petroleum ether MAE: n-hexane, petroleum ether UAE: n-hexane, petroleum ether SubE: n-hexane, petroleum ether Shaking extraction: n-butane	SE: n-hexane; time, 8 h; temperature, 40 °C	SE: n-hexane, 15.66%; petroleum ether, 14.92% MAE: n-hexane, 12.47%; petroleum ether 11.72% UAE: n-hexane, 13.63%; petroleum ether, 12.82% SubE: n-butane, 14.50% Shaking extraction: n-hexane, 12.10%; petroleum ether, 11.32%
Ahangari et al. [47]	Tehran, Iran	SC-CO_2: CO_2 SC-Propane: Propane SE: n-hexane	SE: extraction time, 1200 min; temperature, 70 °C; and seed amount; 10 g	SE: 22.31% SC-Propane: 17.12% SC-CO_2: 13.06%
Natolino et al. [48]	Giulia, Italy	SC-CO_2: CO_2 SE: hexane	SC-CO_2: pressure, 320 bar; temperature, 60 °C; and flow rate, 8 kg/h	SC-CO_2: 18%
Goula et al. [49]	Thessaloniki, Greece	UAE aqueous enzymatic extraction: cellulase, pectinase enzymes Aqueous enzymatic extraction: cellulase, pectinase enzymes	UAE aqueous enzymatic extraction: enzyme type, Peclyve V; temperature, 55 °C; liquid/solid ratio, 6/1 mL/g; enzyme concentration, 2% w/w; and time, 2 h	UAE aqueous enzymatic extraction: 18.15% Aqueous enzymatic extraction: 15.33%

The economic aspects are the most important factor affecting the method selection. Long extraction times and increased temperature result in higher costs. In this study, UAE not only gave a higher efficiency than MAE but also operated at moderate conditions while extracting the maximum oil (26.3%). Therefore, UAE seems a better option than MAE for PSO extraction [40, 41].

3.4 Comparison with literature studies

For PSO extraction, the previously applied methods are UAE, MAE, cold pressing, subcritical extraction (SubE), supercritical carbon dioxide extraction (SC-CO_2), supercritical fluid extraction (SFE), superheated hexane extraction (SHHE), bling and dyer extraction (BDE), and shaking extraction (see Table 8). Clearly, the cold pressing technique has relatively low efficiency, which is surely due to the incomplete extraction [42, 43]. Obviously, SE provides high efficiency; however, SE necessitates more time to complete the extraction and requires a high amount of solvent that must be recovered and recycled through evaporation and condensation [26]. Shaking extraction leads to residual solvents and impurities in the extract. MAE, UAE, and SubE are novel techniques. MAE uses microwave heating energy to increase efficiency. Uniform and rapid microwave energy enhances product recovery and effectively shortens extraction time. UAE applies cavitation force, which comes from the acoustic waves. Due to the cavitation force, the cell wall breaks down easily and solvent penetrates into the sample matrix. SFE is also another environmentally friendly technique, but the large-scale application of SFE is limited due to the high operational and equipment cost. SubE is a good choice for the extraction of heat-sensitive products. For the case of SubE, the target of interest is easily removed under a certain pressure by using liquefied subcritical solvent; however the solvent must be removed and evaporated, which results in high cost.

Our study showed similar efficiency results with literature studies on the PSO extraction. The slight differences between results might be due to the treatment variations, extraction conditions, growing regions, climate conditions, and fruit genotypes [33]. Table 8 revealed that hexane is superior to other solvents for all the extraction techniques studied. Cold pressing gave relatively low efficiency [42, 44, 45]. SE was found to be one of the most effective methods in terms of efficiency; however, it has taken long extraction times. Liu et al. [46] extracted PSO with SE method at 8 h. Ahangari et al. [47] extracted PSO with SE technique at 1200 min. It is clear from the results that SE takes far more time and consumes a lot more solvent compared to UAE and MAE techniques. It is clear from the results that MAE and UAE are the most effective methods in terms of efficiency. For UAE, only Barizao et al. (27. 99%) and Goula et al. (59.8%) reported better efficiency results than the results obtained by this study [26, 37]. However, there is no sharp difference between this study and Barizao et al.'s study, possibly due to the similar operating conditions. Goula et al. achieved 59.8% oil efficiency, which is quite surprising. In case of MAE, only Çavdar et al.'s study [44] achieved better extraction efficiency than this study (35.19% > 22.01%). The difference might be explained by growing region and climate conditions because the optimized conditions are similar with this study. Most importantly, the last three successful studies mentioned above used hexane as the solvent. Hexane is still one of the greatest solvents available today, but according to EU Annex II of the cosmetics regulations, it cannot be used to make cosmetic items. As a result, the finished product needs to be highly purified. On the other hand, DMS, ranked number 1 in the "Environmental Working Group" (EWG) classification, is an eco-friendly solvent that can be used in cosmetics. DMS is more sustainable than hexane, as shown by the study's findings, and the differences in extraction efficiency were not noticeably large. Therefore, for the sake of protecting the environment and public health, DMS can be considered a good substitute for hexane in extracting PSO.

4 Conclusion

After GC-FID analyses, punicic acid was the pomegranate seed's most abundant fatty acid (>80%). UAE method was more effective than MAE method for PSO extraction in terms of oil efficiency. RSM determined the best conditions of MAE and UAE treatments. The highest efficiency was 22.01% in the case of MAE, while the highest efficiency was 26.31% in the case of UAE. For PSO extraction, UAE seems to outperform MAE as the more suitable method. The optimum conditions of MAE were a liquid/solid ratio of 5, irradiation time of 3 min, and microwave power of 300 W. The optimum conditions of UAE were a liquid/solid ratio of 10, pulse duration/pulse interval ratio of 1, 60 °C temperature, and 20 min extraction time. A sustainable strategy has been the focus of this research within the use of dimethyl succinate. Dimethyl succinate is a more environmentally friendly solvent compared to hexane. The results obtained from this study are promising for the use of dimethyl succinate instead of hexane in oil extraction studies. If the oil extraction from pomegranate seeds is to be carried out under the conditions specified in this study, our recommendation is to utilize the ultrasonic-assisted extraction method with dimethyl sulfoxide as the solvent.

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Özge Demir: Investigation, Writing-Original draft.

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Declarations

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

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