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Optimization of ultrasonic assisted extraction of oil from *Moringa oleifera* shelled and unshelled seeds

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SUMMARY

This study aims to improve the extraction of oil from shelled and unshelled *Moringa oleifera* seeds using ultrasound-assisted extraction (UAE). The extraction parameters optimized to obtain the highest yields are the ratio of 61.5 and 58.7 ml/g petroleum ether, extraction time 20–10 min, temperature 30°C - 25 °C for shelled and unshelled seeds, respectively. Under these optimum conditions, the oil yield was in the range of 43.4 % and 36.6 %, respectively, which is in line with the prediction range of the BBD's prediction range of 42.9 %, 35.2 %. The optimized oil was analyzed to determine its properties. This study shows that the physical-chemical parameters such as acidity 1mgKOH/g - 1.75 mgKOH/g, and peroxide value 1.45 meqO2/Kg - 1.15 meqO2/Kg for, refractive index 1.46, Specific extinctions coefficients (K232) were 0.177 and 0.222, (k270) were 0.165 and 0.222, each of the shelled and unshelled seed oils, respectively.

1. Introduction

Oilseeds are part of the human diet. They contain plant proteins and steroids, unsaturated fatty acids and fibers, and important photochemical properties (Kornsteiner et al., 2006). These oil-rich seeds have recently become available in certain regions of Algeria (Tamanrasset, Bechar, Sidi Belabès, jijel and Bejaïa). In these regions, farmers are increasingly interested in growing *Moringa oleifera* Lam. The MO seeds whose oil is suitable for consumption and currently considered a very attractive product for commercial uses worldwide (Saa et al., 2019).

Moringa oleifera is native to India and Pakistan and belongs to Moringaceae family. It is characterized by a long drumstick-shaped pod containing 12 to 15 seeds, dark brown in color, and round, surrounded by a balsa wood shell and three fines wings. It is a multipurpose plant, cultivated in tropical and subtropical regions (Garcia et al., 2019). The MO is referred to as a "miracle tree" due to its richness in several nutritional supplements (Gopalakrishnan et al., 2016), and antihypertensive properties (Dangi et al., 2002). Moreover, all these plant parts: seeds, leaves, roots and even flowers are suitable for human and animal consumption (Anwar and Bhanger, 2003). The seeds have particularly attracted scientists' attention because of their significant oil content, ranging from 19 to 47 % (Anwar et al., 2007). Interestingly, this oil has many uses due to its beneficial ingredients and interesting properties, such as it contains high concentrations of oleic acid, a potentially anti-inflammatory fatty acid. It affects and enhances the proliferation and lipogenesis of human sebocytes (Zouboulis et al., 2023). The properties of moringa seed oil make it an interesting nutrient and a promising ingredient for skin care products. The oils has also anti-cancer properties and MUFAs have interesting anti-thrombotic effects (Abd-Rabou et al., 2016). The oil contains several antioxidant compounds that have therapeutic properties, such as reducing the risk of cardiovascular disease and lowering blood sugar levels, making moringa oil a nutraceutical food (Seifu and Teketay, 2020). *Moringa* seeds contain α-, γ- and δ-tocopherols, the α-tocopherol (vitamin E) is the most abundant, which can prevent certain cardiovascular disease.

It can also prevent lipid oxidation in the body. *Moringa oleifera* seed oil has the potential to become a green source of fat-soluble vitamins and bioproducts of industrial interest (Gharsallah et al., 2021). *Moringa oleifera* oil can be used both for biodiesel production and as a raw material for the cosmetics, food, medicinal and pharmaceutical industries (Pereira et al., 2016).

In order to guarantee a high quality of MO seed oil, highly efficient extraction techniques are needed. Indeed, several techniques have been employed for moringa seeds oil extraction, including: solvent extraction, Soxhlet (Bhutada et al., 2016), cold press (Özcan et al.,

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2019), supercritical fluid extraction (Dzuvor et al., 2018) ultrasonic assisted extraction (UAE) (Zhong et al., 2018). In food processing, ultrasound is a new and frequently technology.

It enabels faster mass and energy transfer, extraction selectivity, faster responses to extraction process control and increased productivity (Chemat et al., 2011). Interestingly, this latter is a potential approach to extract valuable substances from plant raw materials such as oil extraction from various types of seeds, including *Sekaki papaya* (Samaram et al., 2013). In fact, this method is shown to be useful in improving yield and mass transfer in many solid-liquid extraction processes (Vetal et al., 2013).

In order to efficiently optimize extraction parameters, the response surface method (RMS) can be used to evaluate oil extraction from shelled and unshelled MO seeds.

However, there are limited studies on oil extraction from unshelled MO seeds (Arguelles-Peña et al., 2021). The originality of this study lies in the differences in extraction time, temperature and liquid/solid ratio; its main objective is to optimize oil extraction conditions from shelled and unshelled MO seeds harvested in Algeria using the surface response method (RMS). The obtained MO oils have been compared in terms of physical properties and antioxidant activities.

2. Materials and methods

2.1. Sample preparation

The MO seeds have been harvested in June 2019 in the province of Tamanrasset (Southern Algeria). They have been first cleaned and the damaged ones have been discarded. We are used a quantity one kilogram of unshelled seeds (Fig. 1a) and 1 Kg of shelled seeds (Fig. 1b) was used The latter was shelled with a mortar (Fig. 1c) then dried at 40 °C in a forced-air oven until a constant weight is reached. Then, they were ground using an electric mill (IKA model-A11, Staufen, Baden-, Germany) and sieved using a 500 mm sieve (Fig. 1d) to obtain a powder which was later used for oil extraction.

2.2. Ultrasonic assisted extraction of Moringa oleifera seed oil

Extraction of *Moringa oleifera* oil was performed by the method adopted by Zhong et al. (2018) with, however, applying some changes have been applied. For each extraction, an amount of 10 g of MO seed powder was mixed with 80 ml of petroleum ether (boiling point: 60–90 °C). The mixture was treated for varying ultrasound duration (5, 10, 15, 20, 25, 30, 35 min). The mixture was set in an ultrasonic bath (Branson Ultrasonics Baths, Danbury, CT 06813, USA), with a power of 100 W at a frequency of 42 kHz under various temperatures (20 °C, 25 °C, 30 °C, 35 °C, 40 °C, 45 °C) and for different liquid/solid ratio. The mixture was first centrifuged at 4000 rpm for 15 min. Then, the supernatant was separated from the residue, and the liquid evaporated at 40 °C under vacuum. Finally, the resulting oil sample was placed in an oven at 100 °C until a constant weight was reached.

The extraction yield of the MO seed oil was calculated using the following formula (Eq. (1)):

$$\text{Oil yield } \% = (M1/M2) * 100 \quad (1)$$

Where: M1: The extracted MO seed oil mass (g).

M2: Total seed material processed using standardized extraction (g).

2.3. BehnkenBox design (BBD)

Moringa oleifera oil was ultrasonically extracted after a preliminary study to determine the range of factors studied. Preliminary experiments were carried out to select the relevant variables to obtain maximum yield of oil from shelled and unshelled MO seeds as well as the experimental range for the independent factors. Firstly, for the choice of the nature of the solvent, the oil extraction was carried out using three solvents (methanol, petroleum ether, and the mixture of petroleum ether– methanol). Then the ratio of material to solvent (1:50, 1:75, 1:100) and the extraction time (5, 10, 15, 20, 25, 30, 35 min) and temperature (20 °C, 25 °C, 30 °C, 35 °C, 40 °C, 45 °C) were considered individually in Table 1. The extraction efficiency is used to optimize the experimental conditions. The optimization of oil extraction from shelled and unshelled MO seeds was carried out by RSM. A three-factor, three-level Behnken Box Design BBD (time X_1, temperature X_2 and ratio X_3) consisting of 15 trials was used. The generated series are represented in Table 2. Oil yield (oil yield of shelled and un-shelled seeds, Y1 and Y2, respectively) was considered as an input in this study. The data for three independent variables and one response variable were explored to obtain a second-order polynomial tracking model according to the following Eq. (2):

$$Y = \beta_0 + \sum_{(1=0)}^{K} \beta_i X_i + \sum_{(i=1)}^{K} \beta_{ii} X_i^2 + \sum_{(i>1)}^{K} \beta_{ij} X_i X_j + E \quad (2)$$

Where:

β_0, β_i, β_{ii} and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively.

X_i and X_j are the independent variables, while E is error.

2.4. Determination of quality indices

In order to check the quality and appropriate conservation of the oil samples, the acidity index (expressed as a percentage of oleic acid), peroxide value (expressed in milli-equivalent of active oxygen per Kg of oil), the refractive, the K270 and K232 extinction coefficients were measured by following the analytical methods described by the Commission of the European Communities (Commission EU, 1991). Acidity was evaluated by titration of oil samples dissolved in 50 ml of a mixture of ethanol (5 ml) and diethyl oxide (45 ml) with a potassium hydroxide solution (0.25 N). The peroxide value was evaluated by dissolving the oil in a mixture of chloroform-acetic acid and a solution of potassium iodine. In the dark, the obtained solution was titrated with a solution of sodium thiosulfate (0.002 N). The refractive index and the density are measured by following the methods

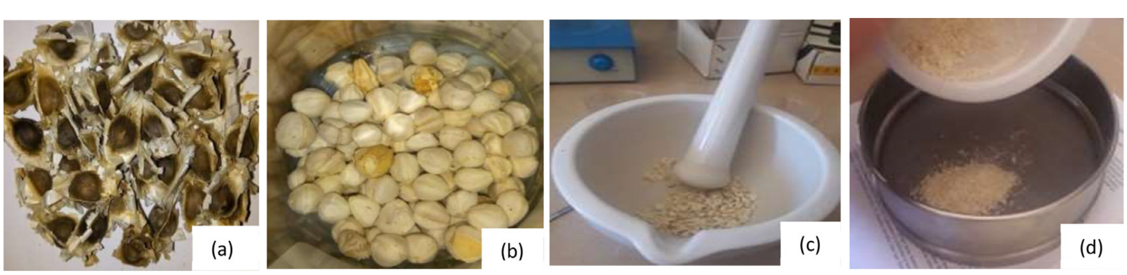

Fig. 1. Unshelled (a) and shelled (b) seeds, shelled with mortar (c) and sieved seeds (d) of *Moringa oleifera*.

Table 1
Yield of shelled and unshelled *Moringa oleifera* seed oil from single factor experiments.

Unshelled seeds								
Solvent type	Yield %	Extraction time (min)		Extraction temperature °C		Solvent to solid ratio (ml/g)		
Methanol	8,86 ± 0,8[c]	5	17,48 % ± 0,88[d]	20	21 % ± 0,5[c]	25	11,4 % ± 0,55[a]	
Ether de petrol	24,42 ± 0[a]	10	24,58 % ± 0,62[b]	25	23 % ± 0,9[b]	50	18,65 % ± 0,14[b]	
Ether-methanol	13,03 ± 0[b],02	15	30,31 % ± 0,09[a]	30	25,24 % ± 0,5[a]	75	20,61 % ± 0.39[a]	
		20	22,34 % ± 0,80[c]	35	23,53 % ± 0,2[b]	100	19,47 % ± 0,24[b]	
		25	21,52 % ± 0,59[c]	40	22,66 % ± 0,1[c]			
		30	21,52 % ± 0,59[c]					
		35	21,14 % ± 0,62[c]					

Shelled seeds							
Solvent type	Yield %	Extraction time (min)		Extraction temperature °C		Solvent to solid ratio (ml/g)	
Methanol	17 ± 0,07[c]	5	26,4 % ± 0,5[d]	20	28 % ± 0[b]	25	30,8 % ± 0,5[d]
Ether de petrol	35 ± 0,06[a]	10	28,45 % ± 0[d]	25	34,35 % ± 0,49[a]	50	32 % ± 0,15[c]
Ether-methanol	30 ± 0,4[b]	15	38 % ± 0,07[a]	30	25,24 % ± 0,5[c]	75	36 % ± 0[a]
		20	33,5 % ± 0,3[b]	35	23,53 % ± 0,2[d]	100	34 % ± 0[b]
		25	32 % ± 0,8[b]	40	22,66 % ± 0,1[d]		
		30	30,85 %± 0,02[c]				
		35	30,06 %± 0,6[c]				

One-way ANOVA analysis was determined (n = 3) by Duncan's multiple range tests. Different superscript letters in the same row indicate significant difference between values at $P\leq$ 0.05 level.

described in the official journal (AOAC, 2000). The oil density is the ratio between the mass of a certain volume of this oil and the mass of the same volume of distilled water at 20 °C (AOAC, 2000).The refractive index was measured with ABBE type refractometer AR 12. Specific extinction coefficients, K232 and K270, were measured for 1 % oil solution mixed with cyclohexane by estimating its absorption in a path of 1 cm leng that 232 and 270 nm, respectively, using a Shimadzu UV Spectrophotometer. All analyzes were performed in three repetitions for all quality indices.

2.5. Anti-radical activity DPPH

In order to evaluate the antioxidant potential by free radical scavenging, the method employed by Alam et al. (2013) was applied. For this, an amount of 2 ml 0.5 mM DPPH solution was added to the sample extract diluted in 0.2 ml methanol. Thirty minutes later, the absorbance was measured at 517 nm and the following formula (Eq. (3)) used to calculate the percentage of DPPH radical scavenging:

$$\%\ \text{inhibition of DPPH radical} = [(A_c - A_t)/A_c] * 100 \quad (3)$$

With:
A_c: Absorbance of DPPH solution control
A_t: Absorbance of the solution after incubation.

2.6. Statistical analysis

All measurements were performed in triplicate. All data collected were reported as means ± standard deviation. The influence of each factor on oil yield in the single-factor UAE experiment was evaluated by comparing quality indices, antioxidant activity of shelled and unshelled oils by analysis of variance ANOVA by JMP software (version 13.0, SAS) to highlight significant differences at the 5 % probability level. JMP was used to construct the BBD and analyze the ANOVA results.

Table 2
Adjustment and analysis of variance (ANOVA) of oil yield Y1and Y2.

Unshelled seeds Y2 source	DF*	Sum of squares	Square of means	F ratio	prob>F
Model	9	391,785	43,528	11,828	0,007**
Error	5	18,399	3680		
Total error	14	410,158			
Lack of fit	3	12,233	4077	1325	0,457
Pure error	2	6166	3083		
Total error	5	18,399			
R2 Adj	0,955				
R2	**0,874**				
Shelledseeds					
Y1 source	DF*	Sum of squares	Square of means	F ratio	prob>F
Model	9	400,366	44,485	40,0467	0,004**
Error	5	5554	1110		
Total error	14	405,920			
Lack of fit	3	3025	1008	0,797	0,597
Pure error	2	2528	1264		
Total error	5	5554			
R^2	0,986				
Adj-R^2	0,961				

* Degrees of freedom;.
** Value statistically significant at p <0.05.

3. Results and discussion

3.1. Optimization of the extraction conditions

A preliminary study using different solvents was carried out to determine the factors influencing the extraction of MO seed oil, which considerably reduced the number of experiments needed to obtain the results presented in the following section. To extract the oil from MO seed, petroleum ether, methanol, mixture methanol-petroleum are generally used. In this study, petroleum ether was an effective solvent for the extraction of shelled and unshelled MO seed oil. It gave the highest percentage of extraction, with a significant difference at $p\leq$ 0.05 compared with methanol and mixture methanol-petroleum (Table 1).

Some researchers revealed that petroleum ether is effective in extracting oil from oil seeds, because it is non-polar it easily penetrates the matrix (Zhong et al., 2018). It is used as an alternative extraction solvent because of its cost-effectiveness in extracting oil from oilseeds; it has a high solvent extraction capacity, so it does not impact the chemical properties of the oil (Masime et al., 2017). The obtained results using BBD for optimization are presented in Table 3 . Oil yield varied from 23.5 % to 43 % for shelled MO seeds and from10 % to 33.66 % for unshelled ones, suggesting the possible influence of the studied parameters, namely, the time, temperature and

Table 3
Box–Behnken design with the observed responses and predicted values for yield of *Moringa oleifera* unshelled and shelled seed oil.

Shelled seed						Unshelled seed					
N°	X1	X2	X3	Y1 Yield	Predicted value Y1	N°	X1	X2	X3	Y2 YIELD	Predicted value Y2
1	20	30	100	34.4	34.35	1	15	35	50	10	10.46
2	10	25	75	26.6	28.06	2	15	30	75	21.78	21.22
3	20	30	50	23.5	24.24	3	10	30	50	13.74	13.63
4	15	35	50	33.5	34.22	4	10	35	75	24.48	24.12
5	15	25	50	33.5	31.98	5	20	30	100	22.80	22.90
6	20	35	75	25.5	24.03	6	10	25	75	24.48	24.08
7	20	25	75	34.5	35.26	7	20	25	75	33.66	34.01
8	10	35	75	43	42.23	8	15	25	100	23.6	23.13
9	15	30	75	37	38.83	9	20	35	75	20.28	20.68
10	15	30	50	39	38.83	10	20	30	50	21.44	20.57
11	10	30	100	36.8	36.84	11	10	30	100	22.5	23.36
12	15	25	100	36.5	35.75	12	15	30	75	22.08	21.28
13	10	30	100	33.5	32.75	13	15	35	100	26.04	25.89
14	15	30	75	40.5	38.83	14	15	30	75	20.0	21.28
15	15	25	100	34.95	36.46	15	15	25	50	26	26.50

One-way analysis was determined (n = 3).

ratio on yield oil. In order to get a good response, several conditions should be verified. The F-test was used for testing to test the statistical significance of the regression equations. Both models have high f-values (11.82 and 40.04) for shelled and unshelled seed oil, respectively) and low p-values (p <0.0001), p (p <0.0004), indicating that both models produced are highly significant. The coefficients of determination (R2 = 0.96 and R2 = 0.99) for shelled and unshelled seed oil extraction are close to 1 (Fig. 2), which means that there is a good correlation between the measured and predicted results. For this and that each model can describe up to 96 % and 99 % of the fluctuation of the oil yields. In sum, the regression analyses show that the two models are both adequate and highly significant with p= 0.0071 < 0.05 and p= 0.0004 < 0.05 for shelled and unshelled seed oil, respectively. The model presented a lack of fit with an F value of 1.3225 and 0.79 giving a probability of 0.45 and 0.55, above the set significance level (p<0.05), indicating that the error on the fitted model was not significant(Table 2). Therefore, these results demonstrated the relevance and adequacy of the Y1 and Y2 model in predicting the response.

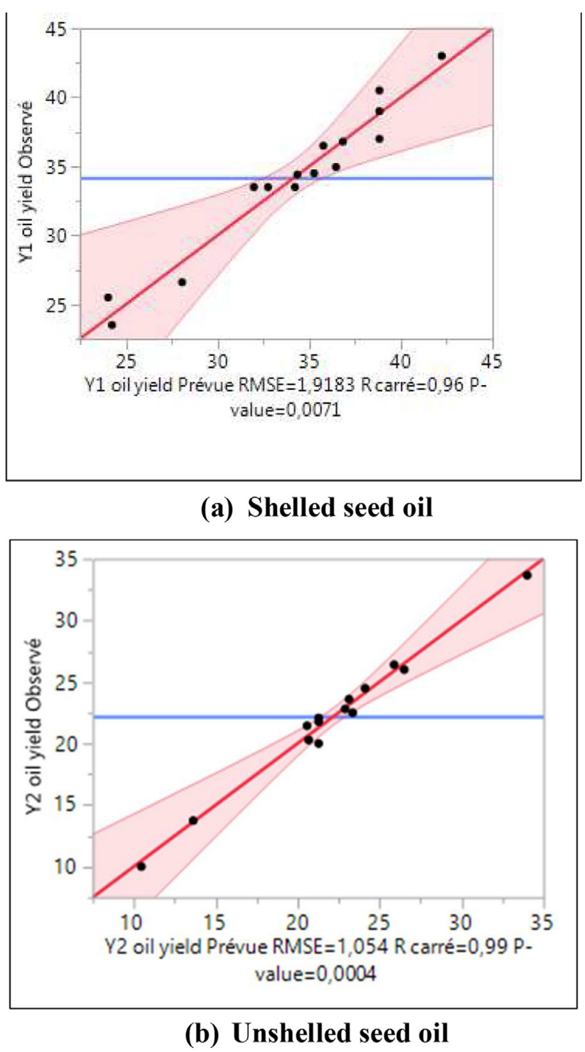

Fig. 2. Observed values graph versus predicted values for shelled and unshelled seed oil.

Results of the analysis of variance obtained during the inspection and the factors affecting the oil yield response are presented in Table 4. According to these results, a factor with a probability value less than 5 % has a significant effect. Indeed, the closer the value is to 0, the greater the effect of the factor (Table 4). Interestingly, the regression analysis of the data, presented in Table 4 revealed that X_1, X_2 and X_3 variables has a significant impact on the extraction of unshelled seed oil. Moreover, these results show that the extraction of unshelled seed oil was mainly influenced by temperature (p= 0.0003), then by solvent /solid ratio (p= 0.0005) and, finally, by time (p = 0.0073). Also, interactions X_1X_2, X_1X_3, X_2X_3, and quadratic effect X_1^2, X_2^2 and X_3^2 showed a significant impact on p-value (p <0.005) (Eq. (5)). However, the shelled seed oil is only affected by the time (p= 0.0098), the quadratic coefficient X_1^2 (p= 0.0064) and the $X_1 * X_2$ (p= 0.0012), $X_1 * X_3$ (p= 0.0141) interactions but what concerns X_2, X_3 and the interaction X_2*X_3, and the quadratic interaction, X_3^2 and X_2^2· have significant negative effects (Eq. (4)).

$$Y_1 = 38.83 - 2.75X_1 - 0.73X_2 + 1.50X_3 - 6.35X_1X_2 + 3.55X_1X_3 + 0.38X_2X_3 - 4.49X_1^2 - 1.93X_2^2 - 2.22X_3^2 \quad (4)$$

$$Y_2 = 21.28 - 1.662X_1 - 3.32X_2 + 3.015X_3 - 3.345X_1X_2 - 1.85X_1X_3 + 4.7X_2X_3 + 1.529X_1^2 + 2.09X_2^2 \quad (5)$$

The Coefficient of Variation (CV) describes the degree of dispersion in the data. In general, a small CV value, less than 10, gives better reproducibility, and a high value indicates that the variation of the mean value is large and does not allow developing satisfactorily an

Table 4
Analysis of the variance (ANOVA) for response surface quadratic model for the extraction of *Moringa oleifera* seed oil.

Parameters	Y_1 shelled yield extraction				Y_2 unshelled yield extraction			
	Estimated coefficient	Sum of Square	F value	Prob > *F*	Estimated coefficient	Sum of Square	F value	Prob > *F*
Constant Linear	38.83333	391.758554	11.82	<0.001*	21.286667	400.36679	40.04	<0.0001*
X_1	−2.75	60.50	16.44	0.0098*	1.6225	21.060050	18.958	0.0073*
X_2	−0.73125	4.27781	1.162	0.3302	−3.3225	88.312050	79.500	0.0003*
X_3	1.50625	18.15031	4.932	0.0770	3.015	72.721800	65.466	0.0005*
Quadratic								
X_1^2	−4.497917	74.700	20.29	0.0064*	1.5291667	8.633910	7.772	0.0385*
X_2^2	−1.935417	13.8307	3.758	0.1103	2.9091667	31.248926	28.131	0.0032*
X_3^2	−2.28941	19.285	5.240	0.0707	−2.695833	26.833910	24.156	0.0044*
Interaction								
$X_1 * X_2$	−6.35	161.2900	43.82	0.0012*	−3.345	44.756100	40.290	0.0014*
$X_1 * X_3$	3.55	50.41100	13.69	0.0140*	−1.85	13.690000	12.324	0.0171*
$X_2 * X_3$	0.3875	0.60062	0.163	0.7029	4.7	88.360000	79.543	0.0003*
Lack of fit		12.233125				3.0259000		
Pure error		6.166667				2.5282667		
Core total		410.15833				5.5541667		

The * means that there is a significant impact on the extraction.

adequate response model (Liyana-Pathirana and Shahidi, 2005).The Coefficient of Variation for shelled and unshelled seed oil was within the acceptable range 5.61 %, 4.74 %, respectively.

In one hand, the oil extraction from unshelled MO seeds was more efficient when using 75 ml of petroleum ether at 25 °C for 20 min. On the other hand, the efficiency of oil extraction from shelled MO seeds occurred when using 75 ml of petroleum ether at 35 °C for 10 min.

The relationships between temperature, extraction time, and solvent/solid ratio on the oil yield of shelled and unshelled seeds are illustrated by the response surface in Fig. 3.

As shown in Fig 3a' and Fig 3a, the oil yield reached its maximum at time of 10 min for shelled seed oil, and 20 min for unshelled seed oil; It is possible to state that increasing the extraction time increased the oil yield; Moghimi et al. (2018) have been detected similar results in their study promising the ultrasonic assisted extraction for black cumin oil. The oil extraction has been increased by increasing time. Goula (2013) have found that the oil yield increased by increasing the ultrasonic time exposure and remained constant when the ultrasonic pretreatment has been extended. A longer processing time increases the cavitation time, so more cell walls are destroyed, which increases the contact surface between the oil seeds and the oil. This releases more oil from the solid structure and increases the extraction rate. This is consistent with the results found by Luque-Garcıa and De Castro (2004).

As shown in Fig 3b' and Fig 3b, the maximum oil yield was obtained at a concentration of 61.56 ml/g for shelled seed oil and 58.53 ml/g for unshelled seed oil. These results are in conformity with those reported by Goula (2013). It is noteworthy that high solid to solvent ratio improved the oil yield, the higher solid to solvent ratio, the greater the concentration gradient on the diffusion of the solid to the solution and the more the swelling of the plant material increases the contact area between the solid and the liquid.

In Fig 3c' and Fig 3c, the maximum oil yield was obtained at a temperature of 35 °C for shelled seed oil and 25 °C for unshelled seed oil. Increasing the temperature has increased the extraction yield, this could be explained by the fact that the high-temperature ultrasonic extraction process can promote the transfer of oil from the seed powder to the thermal medium. These observations are in agreement with those reported by Esmaeilzadeh Kenari and Dehghan (2020). According to these authors, the increase in temperature is due to greater penetration of the solvent into the seeds following the decrease of solvent viscosity and the increase in kinetic energy, which enhances the diffusion. Brahmi et al. (2021) showed that higher temperature can break down cell walls and target compounds are more available, resulting in a higher extraction rate.

The optimal conditions have been established by RSM and the predicted results were compared with the measured values by using these conditions for experiment.

The optimal conditions for oil extraction were: ratio 61.84 g/ml, extraction time 10 min, and temperature 35 °C. Under these optimal conditions, the model predicted a maximum response of 43.43 % the optimal conditions for the unshelled seed oil were: ratio 58.68 g/ml, extraction time 20 min, temperature 25 °C that gave a maximum yield of 35.51 %.

3.2. Physical and chemical parameters

In order to control the quality of the oil obtained, physical and chemical parameters were analyses. These parameters include acidity, density, refractive index, K232, K270 and peroxide value. Lists the results of the quality parameters determined (Table 5).

The acidity index is a very important factor in assessing the stability of the oil. It is a quality criterion that provides information on the conservation of an edible oil. It allows the quantification of free fatty acids present in the latter (Kandji., 2001). A significant difference ($p \leq 0.05$) was noticed between the acidity values of the two oils. The acidity of MO shelled oil 1.00 mgKOH/g± 0 and unshelled 1.57 mgKOH/g± 0 mesured in this study are very lower than those found by Moghimi et al. (2018) for nigella oil for which variation ranges from 9.68 mg/gto12.8 mg/g.

The peroxide index measures the degree of lipid oxidation in oils. It measures the peroxides contained in the oil. It is determined by measuring the iodine released by potassium iodide and it is used to measure the extent of rancidity reactions that occurred during storage (Zahir et al., 2017). A significant difference ($p \leq 0.05$) was noticed between the peroxide values of the two oils. The peroxide value of MO shelled oil 1.45 meqO_2/Kg ± 0 and unshelled 1.15 meqO_2/Kg ± 0. Our results are lower than those found by Esmaeilzadeh Kenari and Dehghan (2020) which correspond to 1.8- 9.6 meqO2/Kg. Our low peroxide value could be affected by the oxidation of the oil studied during the extraction conditions.

The density of the oil depends on the temperature and their chemical composition. Our results revealed that the shelled seed oil (0.918) was significantly higher than the unshelled seed oil (0.875).

The refractive index depends on the density, chemical composition of the oil and temperature (Boukeloua et al., 2012).There is no significant difference between shelled and unshelled seed oil of MO. The values were the same (1.46 for the two oils). These values are in

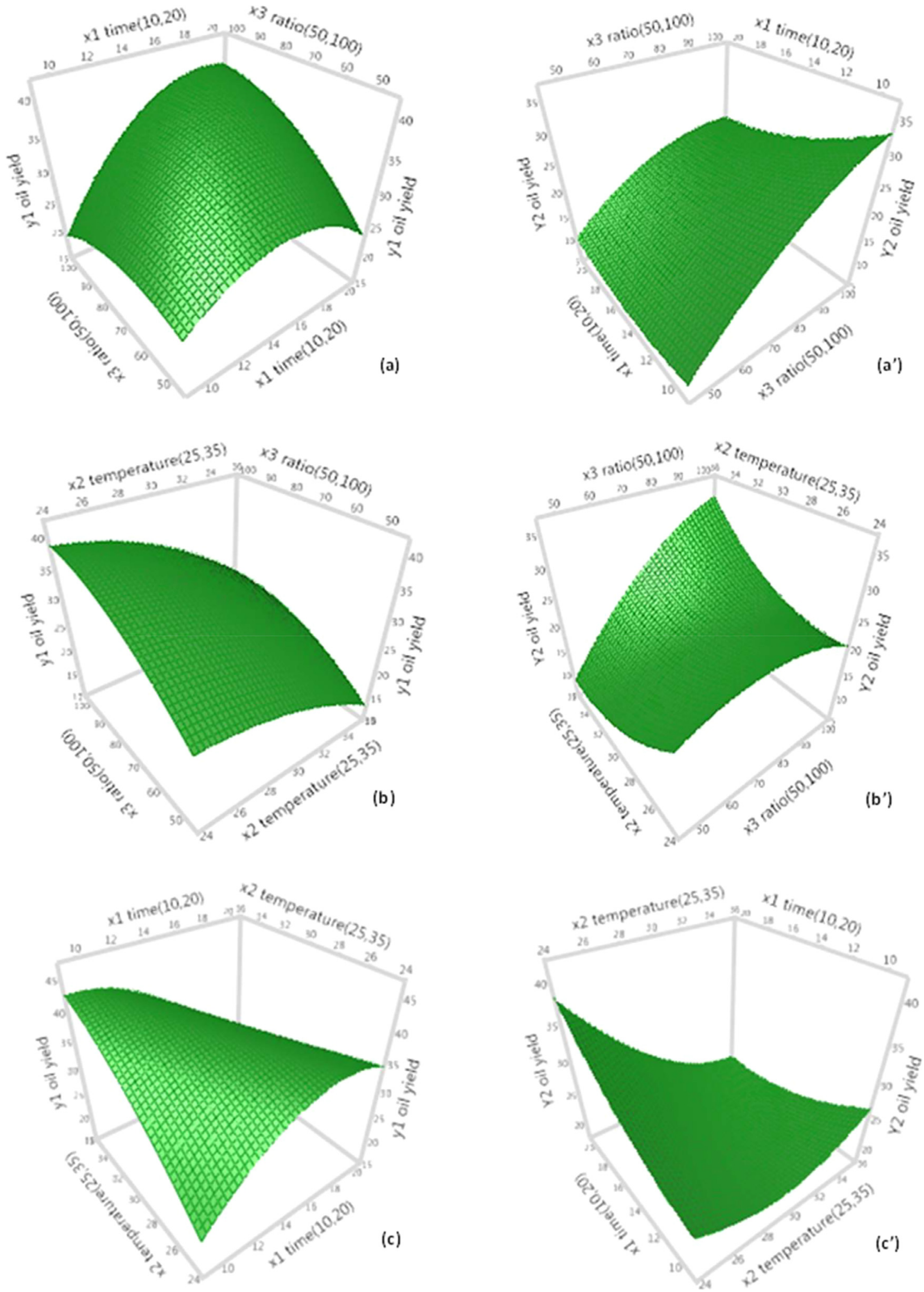

Fig. 3. Responses surface plots for the yield shelled oil (a, b, c) and unshelled oil (a', b', c').

agreement with that reported in the literature for vegetable oils: olive oils (1.468 - 1.471), sunflower oils (1.466–1.477) as well as nigella seed oil (1.469–1.473).

The values of K232 and K270 for dehulled seed oil were 0.177 and 0.222, respectively and, for unshelled seed oil 0.165 and 0.222, respectively. No significant difference between the both oils. Our results are lower than those reported by Taticchi et al. (2019), who found that the K232 of olive oil extracted by KAU ranged from 1.025 to 1.98 but the value of K270 was higher than the range found by the author (0.070 to 0.158).

The differences between the physical and chemical parameters found in this work and those reported in the literature can be caused

Table 5
Quality indices and antioxidant of the seed oils from *Moringa oleifera* shelled and unshelled.

	Shelled	Unshelled
Acid value (mg/g)	$1{,}00 \pm 0{,}16^b$	$1{,}57 \pm 0^a$
Peroxide value (meqO$_2$ /Kg)	$1{,}45 \pm 0^a$	$1{,}15 \pm 0^b$
K 232	$0{,}177 \pm 0^a$	$0{,}165 \pm 0^b$
K270	$0{,}222 \pm 0^a$	$0{,}222 \pm 0^a$
Density	$0{,}918 \pm 0^a$	$0{,}875 \pm 0^b$
Refractive index	$1{,}46 \pm 0^a$	$1{,}46 \pm 0^a$
Antioxidant activity		
Dpph%	$33{,}67 \pm 0{,}12^a$	$10.20 \pm 0{,}19^b$

Different superscript letters in the same row indicate significant difference between values at $P \leq 0.05$ level.

by many factors, including geographical factors, seed variety and different conditions of ultrasonic assisted extraction.

3.3. Antioxidant activity

The antioxidant activity of shelled and unshelled seed oil was evaluated by tracking the ability of free radical reduction to decrease color in the presence of antioxidant.

The anti-radical activity of shelled and unshelled seed oil is 33.67 % ± 0.12, 10.20 % ± 0.19, respectively. The shelled seed oil shows significantly higher free radical scavenging activity than the unshelled seed oil ($P < 0.05$). Quaisie et al. (2021) have found that *Allanblackia parviflora* oilshowed an antioxidant capacity of 21.32 % ± 0.6, which is lower than the obtained results found. The antioxidant activity of MO seed oil found in the present study was higher (33.67 %) than that found by Zhong et al. (2018) (9 %). This difference could be due to the extraction method, the analysis method conditions, the nature of the standard used, the geographical origin of the sample, the degree of maturity and storage.

4. Conclusions

The present study reports the application of ultrasound for the extraction of oil from Algerian MO representing a promising alternative source of vegetable oils. The seeds could obtain provide an oil, comparable in quality to olive oil, which could obtain an important place in the local vegetable oil industry, one a same level with conventional oils from European countries. This study provides scientific evidence that hulling of MO seeds before oil extraction is not mandatory and that the physicochemical and biological properties of shelled MO seed oil are almost similar to those of whole MO seed oil, which represents a significant saving time, workers and a low production cost.

Declaration of Competing Interest

The authors declare no conflicts of interest.

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