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Optimization of Oil Extraction from *Pistacia atlantica* Desf. Seeds Using Hydraulic Press

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ABSTRACT

This study was carried out to determine the effects of pressure, temperature, pressing time and moisture content on the yield of oil mechanically expressed from *Pistacia atlantica* Desf. seeds using a hydraulic press conceived at our laboratory and made in Algeria. The ground sample is compressed at various pressures (50-90 and 120 bars) and temperatures (25-40 and 60°C). The effect of thickness cake, pressing time and moisture content was determined at 120 bars and 25°C. The oil expressed was collected and the oil yield was estimated as a percentage of the raw sample before expression. Statistical analysis of effect of these parameters on yield oil was carried out by student's t-test. Effect combined of pressure and temperature was evaluated using Response Surface Methodology. Chemical proprieties of *Pistacia atlantica* Desf. seeds were determined. Physicochemical analysis of the oil was also carried out to determine its quality potential. The optimum parameter values for maximum oil yield (44, 17%) from seeds were the following: moisture content during pressing = 3.95%, pressing time = 60 min, pressing pressure = 120 bars and temperature during pressing = 40°C.

Key words: *Pistacia atlantica* Desf., fatty acids, optimization, response surface methodology, hydraulic press, oil yield

INTRODUCTION

Pistacia belonging to the family of Anacardiaceae grows in many regions in Algeria. It is represented by four species: *Pistacia lentiscus, Pistacia terebinthus, Pistacia atlantica* and *Pistacia vera*. *Pistacia atlantica* Desf. is widely grown in arid regions. Al-Saghir (2010) have recently revised in detail the phylogeny of *Pistacia* based on morphological data and have concluded that the genus is monophyletic and is divided into two sections : section *Pistacia* and section *Lentiscella*.

The fruit of *Pistacia atlantica*, commonly called El khodiri and its oil are used by natives in traditional medicine and also as food. The seeds are often ground, mixed with sugar solution, dried and consumed like peanuts and also mixed with ground dates. Benamar *et al*. (2010) have reported that the leaves of *Pistacia atlantica* Desf. grown in Algeria are rich in total phenolic compounds and in total flavonoids. Some studies have been carried out to determine the physical and chemical proprieties of fruits and seed oil (Daneshrad and Aynehchi, 1980; Yousfi *et al*., 2003; Ghalem and Benhassaini, 2007; Ghalem and Mohamed, 2009). There is no reports of oil extraction by pressing from *Pistacia atlantica* Desf. seeds, the purpose of this study was to investigate the optimization of oil extraction using hydraulic pressure, the chemical properties of *Pistacia atlantica* Desf. seeds and the physical and chemical properties of its oil.

MATERIALS AND METHODS

Vegetable material: The seeds of *Pistacia atlantica* Desf. were collected from the region of Laghouat (Algeria).

Analytical methods: All analytical determinations were performed in triplicate for each sample with the standard deviation. Response Surface Methodology (RSM) was used to estimate the main effects of pressure and temperature on oil extraction from *Pistacia atlantica* Desf. seeds. A central composite design was used with independent variables temperature (25-40-60°C) and pressure (50-90-120 bars) (Table 1). For the generated 13 experiments, RSM was applied to the experimental data using design expert 8.0.4.1

Oil extraction by hydraulic press: The hydraulic press is equipped with a hydro-electric power connected to a jack screw which compresses the sample inside a metallic cylinder surrounded by a resistance controlled by a thermostat (Fig. 1). A thermocouple is installed in this press for the recording of the temperature value inside the ground seeds. Oil extraction is carried out from 600 g of ground seeds packed in cloth; steel plates are placed between each pair of cloth. The ground sample is compressed at various pressures (50-90 and 120 bars) and temperatures (25-40 and 60°C). The residual oil is extracted from the cake, obtained after pressing in the Soxhlet using hexane.

The oil yield is the ratio between the mass of oil extracted and the mass of the sample. The efficiency of hydraulic press is the ratio between the mass of extracted oil and the initial mass of

Table 1: The levels of different process variables in coded and un-coded form for the oil extraction from *Pistacia atlantica* Desf.

Levels and range	-1	0	+1
Temperature (A, °C)	25	40	60
Pressure (B, bars)	50	90	120

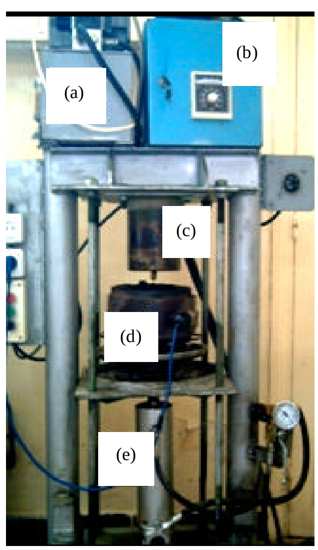

Fig. 1: Hydraulic press: (a) Hydro-electric power, (b) Thermostat, (c) Full cylinder, (d) Sample cell and (e) Jack screw

the seed oil. The effect of thickness cake (2.2±0,1; 3.2±0.3; 3.4±0,1; 4.4±0.2 cm) was determined at 120 bars, 120 min, 25°C and 7.2% of moisture content. The effect of pressing time (15-30-45-60-90 and 120 min) was determined at 120 bars, 25°C with a thickness cake of 3.2 cm. The effect of moisture content (7,83±0,03; 3,97±0,23 and 2,36±0,04%) was determined at 120 bars, 40°C, with a thickness cake of 3.2 cm during 60 min. Statistical analysis of effect of these parameters on yield oil was carried out by student's t-test.

Oil content: The oil content was determined on three samples of whole fruits, kernels and outer skin. Oil is extracted from 10 g of ground seeds in a Soxhlet extractor using hexane as a solvent. The result is expressed as the percentage of lipids in the dry matter of seed.

Determination of chemical proprieties of *Pistacia atlantica* seeds: Moisture was determined using Infra-humid meter (Sartorius). Crude proteins, lipids, ash and cellulose were determined by the method described by AFNOR (2000). The total carbohydrates represents the fraction of available carbohydrates that can be digested by human enzymes. The available carbohydrates were estimated by difference using the following formula (FAO, 1998) 100-(weight in gram (protein+fat +water+ ash+dietary fibre) in 100 g of food.

Determination of physical and chemical proprieties of seeds oil of *Pistacia atlantica*: The extracted oil, after removing hexane is immediately analyzed for iodine, saponification value, acid and peroxide number, unsaponifiable matter, specific gravity and refractive index by the standard methods recommended by AFNOR (2000).

Determination of fatty acid composition: The fatty acids methyl esters derived from triglycerides by the method described by AFNOR (1977) which involves the saponification of glycerides of fatty substances and esterification of fatty acids released in the presence of boron trifluoride. The fatty acids methyl esters derived from whole fruits, kernels and outer skin were analyzed in Thermo Finnigan gas chromatograph equipped with flame ionization detector. The analysis of the fatty acids methyl ester was realized by the means of 30 m×0.32 mm capillary packed with the polar stationary phase nonafluoropentanoic acid (NFPA). The temperature of the detector was 230°C and the injection block was recorded as 210°C and the temperature of the column as 190°C. The velocity of the carrier gas was recorded as 1 mL min^{-1} N2 and injection volume 0.4 µL. peaks were identified by comparing the retention times with those of a mixture of standard methyl esters (Sigma L 9405). Peroxidizability Index (PI) was calculated according to equation of Song *et al.* (2000) as given below: PI = (%monoenoic×0.025)+(%dienoic×1)+ (% trienoic×2)+(%tetraenoic ×4)+(%pentaenoic×6)+(%hexaenoic ×8).

RESULTS AND DISCUSSION

Chemical proprieties of *Pistacia atlantica* seeds: Table 2 show the chemical composition of *Pistacia atlantica* seeds. Moisture content of whole seeds was 7.83%. On dry weight basis, oil content of pistacia seeds was 52.08%, this reflects the importance of using such seeds for oil production. Crude protein level was 14.8±0.11%, show that the *Pistacia atlantica* seeds have an important nutritional value. The other components were in the following decreased order : cellulose (12.11±0.03), carbohydrates total (9.95±0.17) and ash content (3.24%±0.23). The cellulose content indicates that the of *Pistacia atlantica* seeds are rich in dietary fiber.

Table 2: Chemical characteristics of *Pistacia atlantica* Desf.

Constituents	Value
Moisture (%)	7.83±0.03
Crude fat (%)	52.08±0.07
Crude protein (%)	14.8±0.11
Carbohydrate (%) (by difference)	9.95±0.17
Ash (%)	3.24±0.23
Cellulose	12.11±0.03

Table 3: Effect of thickness cake on the oil yield

	Thickness cake (cm)			
Parameters	2.2±0.1	3.2±0.3	3.4±0.1	4.4±0.2
Oil yield (%)	29.32±0.95	36.17±0.89	28.32±0.81	18.83±0.75
Residual oil (%)	22.71±0.44	14.09±1.09	22.92±1.61	29.02±0.54

Table 4: Effect of pressing time on the oil yield

	Time pressing (min)					
Parameters	15	30	45	60	90	120
Oil yield (%)	24.52±1.42	27.97±0.66	31.15±0.60	33.4±0.64	33.74±0.93	33.86±0.86
Residual oil (%)	23.82±1.03	20.79±0.62	18.18±0.74	14.09±1.09	12.29±1.66	12.21±1.27

Effect of some parameters on oil yield: The parameters investigated for their effects on the oil yield were: thickness cake, pressing time, moisture content, pressure and temperature. The analysis of variance revealed that all these factors have a significant effect on oil yield ($p<0.05$).

Effect of thickness cake: The results of effect of thickness on the oil yield are given in Table 3. The effect of thickness cake on the yield of oil was studied by keeping pressure, temperature and pressing time constants, respectively at 120 bars, 25°C and 60 min. The maximum oil yield was 36, 17% with a thickness cake of 3.2 cm. For a thickness cake of 4.4±0.2 cm the oil yield decreases from 36.17 to 18.83±0.75% and the residual oil content increases clearly to 29.02±0.54%. This result indicates that the maximum oil yield was obtained with a small cake thickness. These observations are in agreement with those reported by Hickox (1953) and Lanoiselle (1994).

Effect of pressing time: The results of influence of pressing time are shown in Table 4. The effect of this parameter on the oil yield was studied at 120 bars, 25°C and a thickness of 3.2 cm. Oil yield increased progressively from 24.52 to 33.86%, when the pressing time was increased from 15 to 120 min and the residual oil content decrease respectively from 23.82 to 12.21%. This result show that the influence of pressing time in oil yields was significant. Indeed Singh *et al*. (1984), by studying the effect of some parameters on the oil extraction from sunflower seeds by pressing, have reported that the pressing time has a very significant impact on the oil yield.

Effect of moisture content: The results of effect of this parameter on oil yield are presented in Table 5. The effect of moisture content on the oil yield was studied by fixing the temperature, pressure and time, respectively at 40°C, 120 bars and 60 min. The oil yield increase from 40.88 to 44.17% when the moisture content decrease from 7.83 to 3.97%. However, it was observed that the reduction of moisture content from 3.97 to 2.36% decreases the oil yield from 44.17 to 39.08%. This

Table 5: Effect of moisture content on the oil yield and residual oil content

	Moisture content (%)		
Parameters	7.83±0.03	3.97±0.23	2.36±0.04
Oil yield (%)	40.88±0.56	44.17±0.62	39.08±0.84
Residual oil (%)	5.42±0.54	2.81±0.96	10.70±0.63

Table 6: Effect of temperature on the oil yield and the oil residual content

Pressure (bars)	Temperature (°C)	Oil yield (%)	Residual oil(%)	Efficiency of hydraulic press (%)
50	25	14.89±0.63	32.09±1.54	28.63±0.05
50	40	30.43±0.91	17.17±0.83	54.82±0.11
50	60	25.32±1.17	20.97±1.66	64.23±0.23
90	25	28.51±0.93	20.60±1.09	58.51±0.09
90	40	37.02±092	7.60±1.10	71.19±0.18
90	60	32.27±0.89	20.97±1.09	62.05±0.21
120	25	33.4±0.64	14.09±1.09	48.69±0.13
120	40	40.87±0.56	5.45±0.53	78.59±0.09
120	60	36.19±1.2	9.75±1.09	69.59±0.10

result showed that the value 3.97% can be considered as the optimal moisture content to obtain a maximum oil yield from *Pistacia atlantica* seeds. This result is in agreement with that reported by Koo (1942) and Singh *et al.* (2002). Lanoiselle (1994) have reported that the temperature and moisture content have a significant combined effect on oil extraction from sunflower seeds. The authors explained that the increase of oil yield at low moisture content would be due to the weakening of the lipid globules.

Effect of pressure and temperature: The values of content oil extracted, using hydraulic press, under different pressures of crushed *Pistacia atlantica* seeds are presented in Table 6. For 50 bars, the oil yield obtained was 14.89%. When the pressure increase to 90 bars and 120 bars the oil yield increased, respectively to 28.51 and 33.4%. The oil rate led to an asymptotic value between 90 and 120 bars. These results revealed clearly that increase of pressure improves the oil extraction. These results are similar to those reported by Smith *et al.* (1993) who announced the existence of optimal pressure mechanical expelling for soybeans. For the effect of the temperature it was observed that oil yield which is 14.89±0.63% at 25°C increases to 30.43±0.91% at 40°C. However, the increase of the temperature from 40 to 60°C decreases the oil yield from 40.87±0.56 to 36.19±1.20% with an increase of the residual oil content in cake from 5.45±0.53 to 9.75%±1.09. Thus, the efficiency of hydraulic press decreases from 78.59 to 69% (Table 6). These results are in accordance with those reported by Adeeko and Ajibola (1990), Hamzat and Clarke (1993), Tchiegang *et al.* (2005) and Kartika (2005). Wiesenborn *et al.* (2001) have explained that the reduction of the oil yield would be due to a reduction of the cake plasticity caused by the water loss at high temperature. An increase of temperature reduces the oil viscosity with an increasing mobility of biopolymers in cellular walls which stopped the pores and consequently causes a deceleration of the oil flow during pressing (Lanoiselle, 1994).

Combined effects of pressure and temperature on oil yield using response surface methodology: Experiments were performed according to the CCD experimental design given in Table 7 in order to search for the optimum combination of pressure and temperature for oil extraction rate. The Model F-value of 83.86 implies that this model is significant. Values of "Prob>

Table 7: Experimental conditions and observed response values of CCD

Run	Temperature	Pressure	Oil yield (%)
1	0	1	40.87
2	0	-1	30.14
3	-1	-1	14.89
4	-1	1	33.34
5	0	0	37.02
6	0	0	37.02
7	1	1	36.19
8	0	0	37.02
9	0	0	37.02
10	-1	0	28.51
11	1	-1	25.32
12	1	0	32.27
13	0	0	37.02

Table 8: Analysis of variance (ANOVA) for response surface quadratic model for effect of temperature and pressure on oil yield by press hydraulic

Source	Coefficients	Sum of squares	df	Mean of squares	F-value	Prob.>F
Constant	37.22	552.16	5	111.83	83.86	<0.0001
A (temperature)	2.83	48.05	1	48.05	36.05	<0.0005
B (pressure)	6.88	268.14	1	268.14	201.06	<0.0001
AB	-1.91	14.59	1	14.59	10.94	<0.0130
A^2	-7.32	147.91	1	14.91	110.91	<0.0001
B^2	-2.28	13.40	1	13.40	10.05	<0.0157
Residual		9.34	7			
Lack of fit		9.34	3			
R^2		0.9836				
Adj R^2		0.9718				
Predicted R^2		0.8411				
Adeq. precision		32.411				
SD		1.15				
CV (%)		3.52				
Press		90.33				

F" less than 0.050 indicate model terms are significant. In this case A, B, AB, A2, B2 are significant on the oil yield. The results of response are shown in Table 8. The mathematical expression of relationship to the response with variables A (temperature) and B (pressure) is shown following: Oil rate% = $+37.222+2.83\times A+6.88\times B-1.91\times AB-7.32\times A^2-2.20\times B^2$. The relative magnitude of the coefficients values indicates the maximum positive effect of pressure (6, 88), followed by temperature (2.83) and quadratic of temperature (-7.32) (Table 8). These results are in agreement with the findings of effect of pressing characteristics on oil extraction from sunflower seeds reported by Singh *et al.* (1984). These results indicate an increased oil yield with increase of pressure and temperature. The quadratic of pressure and the interaction temperature- pressure have negligible effects on oil yield, compared to the linear terms. The Fig. 2 and 3 which depicts the 3D plot and its corresponding contour plot, shows the effects of temperature and pressure on the oil yield. The elliptical shape of the curve indicates that the relative interactions between temperature and pressure are significant. The Fig. 2 shows that the yield oil increased with the increase of temperature then decreased, indicating the existence of optimal temperature. However, the pressure increased continually the expelled oil with a possible trend towards an optimal value.

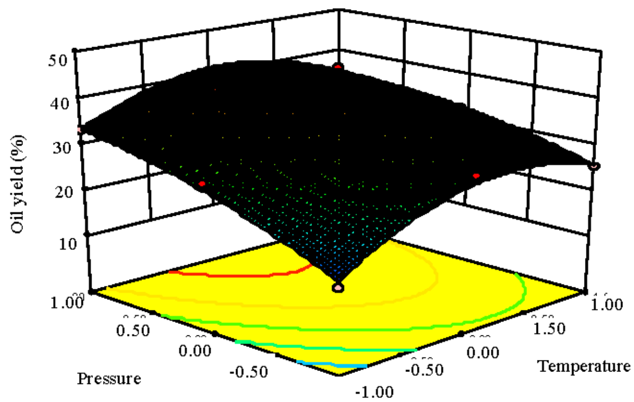

Fig. 2: Response surface plot of the combined effects of pressure and temperature on the oil yield

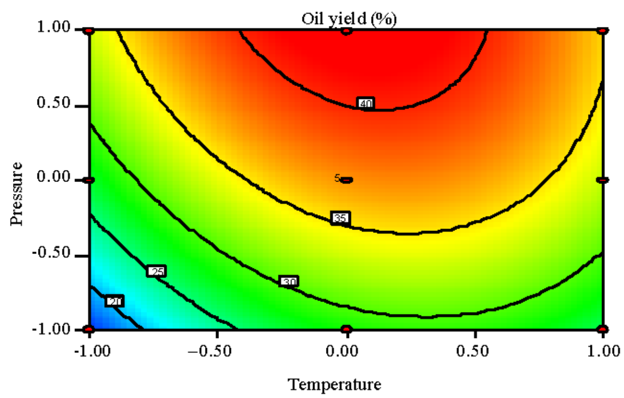

Fig. 3: Contour plot of the combined effects of pressure and temperature on the oil yield 31.5%

Physical and chemical proprieties of oil *Pistacia atlantica* seeds: The physical and chemical proprieties of oil *Pistacia atlantica* Desf. seeds are shown in Table 9. The crude oil obtained by hydraulic press and the oil extracted by hexane are the same proprieties. The values of the density, refractive index, saponification value, iodine number, unsaponifiable matter (%), peroxide index and acidity (%) are in agreements with those found by Yousfi *et al*. (2003). The iodine number of the oil was 88, thus, this oil could be categorized as a no drying oil. The acid value is considered high compared to that of an oil of good quality whose content of 'oleic acid should not exceed 2% corresponding to 4 mg of KOH per gram of oil. The high value of acidity would be related to the intrinsic nature of seeds, whereas, many oleaginous seeds such as olive and palm, contain high acidity oils (Patterson, 1989). This high acidity would be due also to the presence of fatty-acids released by the action of lipase on triglycerides during the crushing of seeds (Alibert *et al*., 2001). Ustun *et al*. (1990) have reported that the presence of lipase in seeds could increase the free fatty acids rate to 40% and higher.

Fatty acids composition of oil *Pistacia atlantica* seeds: The fatty acid composition of whole fruits, kernels and outer skin of *Pistacia atlantica* and their percentages is presented in order of

Table 9: Physico- chemical characteristics of oil *Pistacia atlantica* seeds

Properties	Oil press (25°C)	Oil solvent
Density (20°C)	0.919±0.002	0.917±0.002
Refractive index (20 °C)	1.472±0.001	1.472±0.001
Viscosity 20°C (Cp)	87.000±0.550	85.000±0.750
Absorbance ('UV-268 nm)	0.652±0.027	0.650±0.081
Acid value ((mg KOH g^{-1})	9.950±0.050	8.350±0.120
Peroxide index		
(meq O_2 kg^{-1})	10.490±0.023	9.950±0.950
Iodine value (g 100 g)	88.000±0.050	88.000±0.010
Saponification value (mg KOH g^{-1})	204.300±0.010	204.490±0.040
Unsaponifiable matter (%)	1.720±0.020	1.740±0.010

Table 10: Fatty acids composition of whole fruits, kernels, outer skin oil of *Pistacia atlantica* Desf.

Fatty acids	Whole fruits	Kernels	Outer skin
C16:0	20.42±0.05	12.50±0.02	28.63±0.54
C16:1	1.02±0.01	0.56±0.66	1.49±0.88
C18:0	1.93±0.02	2.64±0.06	1.48±0.01
C18:1	52.68±0.05	55.80±0.85	48.62±0.79
C18:2	22.81±0.07	27.15±0.90	18.23±0.23
C18:3	0.60±0.23	0.52±0.02	0.88±0.42
C20:0	0.13±0.11	0.19±0.01	0.11±0.02
C20:1	0.38±0.15	0.61±0.03	0.25±0.05
∑SFA's	22.48±0.18	15.33±0.09	30.22±0.57
∑UFA's	77.49±0.51	84.64±0.90	69.47±0.48
∑ MUFA's	54.08±0.21	56.36±0.23	50.36±0.67
∑ PUFA's	23.41±0.30	27.67±0.33	19.11±0.65
UFA's /∑SFA's	3.44±0.61	5.52±0.21	2.29±0.61
Peroxidizability Index* (%)	25.35	29.6	21.24

SFA's: Saturated fatty Acids, MUFA's: Monounsaturated fatty acids, PUFA's: Polyunsaturated fatty acids

their elution in the column in Table 10. Palmitic and stearic acids were the major saturated, whereas, oleic and linoleic were the major unsaturated acids. The content of oleic acid was much higher than that of linoleic acid. Kernel oil is rich in linoleic acid and the concentration of this acid in outer skin oil is lower. These results are in accordance with those of Daneshrad and Aynehchi (1980), Yousfi *et al.* (2002), Ghalem and Benhassaini (2007) and Farhoosh *et al.* (2008). The presence of high amounts of the saturated palmitic and stearic acids, shows that the oil of *Pistacia atlantica* is nutritionally unfavourable, since these acids are involved in coronary heart disease. The PI for the whole fruits, kernels and outer skin oil was, respectively 25.35, 29.6 and 21.24% and ∑UFA's / ∑SFA's ratio was 3.44, 5.52 and 2.29. It indicated that the crude oil extracted is stable to auto-oxidation rancidity during storage.

Effect of temperature during optimization of oil yield by press on the physical and chemical proprieties of *Pistacia atlantica* seeds oil and on fatty acid composition: The results of effect of temperature, at 120 bars during optimization of oil extraction by press, on the physical and chemical proprieties of oil and on the composition of fatty acids are presented, respectively in Table 11 and 12. It was observed that at 60°C the viscosity and absorbance (UV-268 nm) increased, respectively to 95±0.15 and 1.259±0.02. Concerning the chemical characteristics at 60°C the peroxide and acid index increased, respectively to 11.62±0.12 and 11.31. For the fatty acids the effect of the temperature at 60°C is observed on the oleic acid content which

Table 11: Effect of temperature during oil extraction by press hydraulic on the physical and chemical proprieties of oil

	Temperature (°C)		
Proprieties	25	40	60
Density at 20°C	0.919±0.001	0.918±0.002	0.920±0.001
Refractive index at 20°C	1.472±0.001	1.471±0.001	1.469±0.002
Viscosity at 20°C (Cp)	87.000±0.550	92.000±0.500	95.000±0.130
Absorbance UV	0.652±0.027	0.655±0.023	1.259±0.020
Acid index (mg KOH g^{-1})	8.740±0.060	9.950±0.050	11.310±0.010
Peroxide index (meq O_2 Kg^{-1})	9.850±0.130	10.490±0.230	11.620±0.120
Iodine value (g I 100 g)	88.030±0.050	87.150±0.320	87.960±0.140
Saponification value	204.000±0.010	202.000±0.250	199.000±0.920
Unsaponifiable matter	1.720±0.010	1.580±0.020	1.490±0.070

Table 12: Effect of temperature during oil extraction by press hydraulic on the fatty acids composition

	Temperature (°C)		
Fatty acids	25	40	60
C16:0	21.68±0.42	22.05±0.01	20.66±0.45
C16:1	0.97±0.13	1.05±0.02	0.97±0.12
C18:0	1.96±0.12	2.09±0.01	2.13±0.01
C18:1	50.90±0.01	50.61±0.02	52.17±0.01
C18:2	23.30±0.01	22.85±0.06	22.69±0.09
C18:3	0.70±0.11	0.83±0.16	0.79±0.12
C20:0	0.11±0.02	0.12±0.11	0.14±0.01
C20:1	0.35±0.12	0.35±0.18	0.40±0.05

increase to 52.17%. These results are in agreements with those of Wolf (1991) who reported that the heat and the pressure causes the hydrolysis of triglycerides which increase the acidity of oil. In order to avoid the alteration of oil during pressing it would be preferable to carry out the oil extraction at temperature below 40°C.

CONCLUSION

The results of this study shows that the oil *Pistacia atlantica* seeds is rich in linoleic acid (22, 86%) and palmitic acid (22, 05%). Analysis of variance has shown that the effects of temperature and pressure were statistically significant. Second order polynomial models were obtained for predicting the amount of extracted oil. The optimum parameter values for maximum amount of expelled oil (44, 17%) from *Pistacia atlantica* seeds were the following: moisture content during pressing = 3.95%, pressing time = 60 min, pressing pressure = 120 bars and temperature during pressing = 40°C.

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