Hazelnut oil production using pressing and supercritical CO_2 extraction

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Abstract
In the hazelnut oil production it is very important to find an appropriate method to recover the oil from kernels. The objective of this study was to evaluate the oil extraction process from hazelnuts by screw pressing followed by extraction with supercritical CO_2. The effects of temperature head presses, frequency and nozzle size in pressing experiments on oil temperature and recovery were monitored. The optimal pressing condition using response surface methodology was determined. In obtained hazelnut oil the following quality parameters were determined: peroxide value 0 mmol O_2/kg, free fatty acids 0.23%, insoluble impurities 0.42%, moisture content 0.045%, iodine value 91.55 g I_2/100 g, saponification value 191.46 mg KOH/g and *p*-anisidine value 0.19. Rosemary extract was the most effective in protecting the oil from oxidative deterioration. The residual oil that remained in the cake after pressing was extracted totally with supercritical CO_2 and such defatted cake, free of toxic solvents, can be used further in other processes.

Keywords: hazelnut oil, quality, screw pressing, supercritical CO_2 extraction, response surface methodology.

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Hazelnuts (*Corylus sp.* L.) are an important commercial crop in many countries including Croatia. Hazelnut has a high nutritional value, containing, generally, 65% oil, 14% protein, and 16% carbohydrates. More than 90% of its oil consists of unsaturated fatty acids, especially oleic (80%) and linoleic (6–12%) acids. Several authors have studied the physicochemical characteristics and nutritional values of different hazelnut oils [1–5] and they concluded that hazelnut oil is appreciated as valuable edible oil for health food. It is a good source of vitamin E (α-tocopherol) and may be used as food value improving the shelf-life of the product by its antioxidant function. At the same time it belongs to the non-drying oils with an excellent stability and is used in the cosmetic industry as well as in the production of oleic acid. These unsaturated fatty acids, as well as sterols and tocopherols also present in the oil, play a preventive role in many diseases, especially cardiovascular ones, as they contribute to lower the low density of lipoprotein cholesterol. Currently, hazelnut oil is used mainly in salad dressings and cosmetic and pharmaceutical products.

Two main types of processes for obtaining oil are physical and chemical [6]. The physical process involves the use of mechanical power to remove oil from material, such as hydraulic pressing and screw pressing. Extraction is a process based on chemical characteristics of solute and solvent. Conventional solvent extraction produces low-quality oil that requires extensive purification operations while screw pressing does not require the use of organic solvent and is able to retain bioactive compounds such as essential fatty acids, phenolics, flavonoids and tocopherols in the oils [7], as well as the possibility of using cake free of toxic solvents in other processes. Unfortunately, the main disadvantage of this process is low oil extraction yield. But nowadays, technologies such as supercritical fluid extraction (SFE) are more often applied in the extraction of fatty oils [8]. All these mentioned extraction processes can be combined in commercial operations i.e. continuous mechanical pressing followed by supercritical CO_2 extraction by which is an environmentally-friendly solvent [9]. Eggers *et al*. [10] studied process of fatty oil SFE from rapeseed and soybean and explained that the oil containing cell wall had to be disrupted in order to enable successful extraction. They showed that the mechanical pre-deoiling remained the superior pretreatment in the supercritical extraction of oilseeds. Extracts obtained using CO_2 as the extraction solvent are solvent-free / without any trace of toxic extraction solvents, and are thereby highly valued [6,8]. Use of SFE as a replacement of organic solvents in fatty oil extraction was considered in early 1980's [10–13]. At present moment, SFE on a commercial scale are limited to decaffeination, production of soluble hops extracts, sesame seed oil production, extraction of certain petroleum products, high-value compounds from spices, herbs, and other vegetable material, animal tissue, and microalgae [14,15]. Despite industrial application for almost four decades, there is reluctance in some world

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regions to adopt SFE because of the wrong perception that it is not fully competitive [14].

In the production of hazelnut oil, the amount and quality of extracted oil are crucial for determining feasibility of commercial production. So, the objective of this work was to investigate the effects of process parameters during the screw pressing of hazelnuts using response surface methodology (RSM) and to check the possibility to complete recovery of the residual oil from pressed cake using green solvent-supercritical CO_2.

MATERIAL AND METHODS

Materials

Hazelnuts without shell were kindly provided from company PP Orahovica d.d. (Orahovica, Croatia) in 2014. Rosemary extract Oxy.Less CS, green tea extract, and pomegranate extract were supplied from Naturex (France). Olive leaf extract was supplied from Exxentia (Spain). Essential oils of oregano (*Origanum vulgare*), sage (*Salvia officinalis*) and winter savory (*Satureja Montana*) were produced by steam distillation according to the standard Ph. Jug. IV procedure [16].

The purity of CO_2 used for extraction was 99.97 mass% (Messer, Osijek, Croatia). *n*-hexane was provided from Merck KGaA (Darmstadt, Germany). All other chemicals and reagents were of analytical reagent grade.

Determination of initial oil and water content

The initial oil content in hazelnuts as well as the cake residual oil was measured by traditional laboratory Soxhlet-extraction with *n*-hexane as solvent [17]. The measurement was done in triplicate. Moisture content of hazelnuts was determined according to AOAC Official Method 925.40 [18].

Screw press extraction

The pressing of the hazelnuts was performed in a screw expeller (Model SPU 20, Senta, Serbia). The hazelnut oil was obtained by pressing 500 g of cleaned hazelnuts per each experiment using different process conditions. The minimum and maximum nozzle size used was 8 and 12 mm, respectively. The frequency had a minimum of 20 Hz and a maximum of 40 Hz. The temperature of head presses was between 70 and 100 °C. After pressing, the volume of screw pressed oils and their temperature were measured and after that the oil was centrifuged. The sedimented solids were recovered and solid percentage of the oils was calculated by weight difference.

Oil quality parameters

Free fatty acids, iodine value and saponification value were determined according to AOAC official methods 940.28, 920.185 and 920.160 [19]. Peroxide value (*PV*) of oil samples was determined according to ISO 3960 [20] and is expressed as mmol O_2/kg of oil. Insoluble impurities were determined according to ISO 663 [21]. *p*-Anisidine value (AV) was determined according to ISO 6885 [22]. Totox value was calculated as 2PV+AV [23]. All these determinations were carried out in triplicate.

Determination of oxidative stability

The oxidative stability was determined by rapid oil oxidation test – Schaal or Oven Test (63 °C) [24]. The influence of the addition of natural antioxidants, namely rosemary extract, green tea extract, olive leaf extract, and pomegranate extract in concentrations of 0.1 and 0.3%, and essential oils of oregano, sage and winter savory in the concentration of 0.05%, on the oxidative stability of hazelnut oil were monitored. The result of oil oxidation was expressed as peroxide value during 4 days of the test. All determinations were carried out in duplicate.

Experimental design

Box-Behnken design which includes three variables and three factorial levels was chosen in this study [25]. The ranges for the variables, namely temperature head presses (70, 85 and 100 °C), frequency (20, 30 and 40 Hz) and nozzle size (8, 10 and 12 mm) were selected to approximate the optimal conditions for screw pressing of hazelnut oil. Coded and uncoded levels of the independent variables and the experimental design are given in Table 1. Coded value 0 stands for center point of the variables and repeated for experimental error. Factorial points are coded as ±1.

Table 1. The uncoded and coded levels of independent variables used in the RSM design in pressing experiments

Independent variable	Symbol	Level		
		Low (–1)	Middle (0)	High (+1)
Nozzle, mm	X_1	8	10	12
Temperature, °C	X_2	70	85	100
Frequency, Hz	X_3	20	30	40

Second-order polynomial equation was used to express the investigated responses (Y) after pressing, namely the volume of screw press oil (mL), oil temperature (°C) and residual oil in pressed cake (%) as a function of the coded independent variables, where X_1, X_2,..., X_k are the independent variables affecting the responses Y's; β_0, β_j (i = 1, 2,..., k), β_{ii} (i = 1, 2,..., k) and β_{ij} (i = 1, 2,..., k; j = 1, 2,.. , k) are regression coefficients for intercept, linear, quadratic, and interaction terms, respectively; k is the number of variables.

$$Y = \beta_0 + \sum_{i=1}^{k}\beta_i X_i + \sum_{i=1}^{k}\beta_{ii} X_i^2 + \sum_{\substack{i=1 \\ i<j}}^{k-1}\sum_{j=2}^{k}\beta_{ij} X_i X_j \quad (1)$$

Statistical analysis was performed using RSM software Design-Expert®, v. 7 (Stat Ease, Minneapolis, MN, USA). The results were statistically tested by the analysis of variance (ANOVA) at the significance level of p = = 0.05. The adequacy of the model was evaluated by the coefficient of determination (R^2) and model p-value. Mathematical models were established to describe the influence of single process parameter and/or interaction of multiple parameters on each investigated response. Response surface plots were generated with the same software and drawn by using the function of two factors, and keeping the other constant.

Supercritical CO_2 extraction

The experiment was performed in supercritical fluid extraction system described in detail elsewhere [9,26]. The pressed cake of 100 g was placed into an extractor vessel to recover the residual cake oil using supercritical CO_2. The extracts were collected in previously weighed glass tubes. The amount of extract obtained at regular intervals of time was established by weight using a balance with a precision of ±0.0001 g. Extraction was performed at the following conditions: pressure of 30 MPa, temperature of 40 °C and a CO_2 mass flow rate of 1.73 kg/h. Separator conditions were 1.5 MPa and 25 °C.

RESULTS AND DISCUSSION

Screw pressing experiments

The cold pressing method was used to extract oil from hazelnuts. Before pressing experiments, the initial oil and moisture content of hazelnuts were determined. The average of the initial oil content was 65.17±0.38% and moisture content was 4.08±0.09%. The oil content was consistent with reported values [27–29]. The experimental design of 17 pressing experiments was carried out using the described factors and levels (Table 2). Effects of temperature head presses, nozzle size and frequency on recovery and quality parameters of hazelnut oil were studied by RSM. In all 17 experimental runs only a small percentage of moisture was found in the obtained oils (average value 0.045%) and the peroxide value of all experimental runs was 0 mmol O_2/kg. The oil temperature in all experimental runs was in the range from 30 to 54 °C.

Model for prediction the investigated responses and his coefficients are obtained by using linear regression statistical method of the experimental data, and the obtained model provides a functional dependence between the independent and dependent variables. Table 3 shows the regression coefficients obtained by fitting experimental data to the second order response models for investigated responses. The coefficients are related to coded variables. The first-order term of temperature (X_2) had significant effect ($p < 0.05$) on the oil temperature on the amount of cake residual oil. The first-order term of frequency (X_3) had significant effects on oil temperature. The second-order term of nozzle size (X_1^2) had significant effect ($p < 0.05$) on cake residual oil, while second-order term of frequency (X_3^2) had significant effect on oil content and temperature. The interactions between the pressing temperature and frequency (X_2X_3) had significant effect on the oil content. Other interactions had no significant ($p > 0.05$) effect on investigated responses.

Table 2. Experimental matrix and values of the observed response

Run	Nozzle mm	Temperature °C	Frequency Hz	Oil volume mL	Oil yield g/500 g	Oil temperature °C	Cake oil %	Recovery % of total oil
1	12	85	40	240	230.40	53	15.92	72.82
2	8	100	30	270	259.20	52	15.43	81.92
3	10	70	20	195	187.20	41	9.79	59.17
4	12	70	30	255	244.80	42	13.53	77.37
5	10	85	30	295	283.20	41	12.83	89.51
6	8	70	30	245	235.20	38	12.32	74.34
7	10	85	30	285	273.60	45	12.08	86.47
8	12	100	30	270	259.20	52	15.94	81.92
9	10	85	30	260	249.60	43	15.05	78.89
10	10	85	30	275	264.00	46	13.23	83.44
11	10	85	30	280	268.80	45	14.33	84.96
12	12	85	20	225	216.00	45	15.08	68.27
13	10	70	40	270	259.20	46	13.18	81.92
14	8	85	40	265	254.40	54	14.06	80.40
15	10	100	40	220	211.20	50	13.2	66.75
16	8	85	20	265	254.40	47	14.32	80.40
17	10	100	20	245	235.20	50	15.53	74.34

*Table 3. Estimated coefficient of the second order polynomial equation; X_1: nozzle size; X_2: temperature; X_3: frequency; *: significant at $p \leq 0.05$*

Term	Coefficient[a]	Oil volume	Oil temperature	Cake oil
Intercept	β_0	279.00*	44.00*	13.50*
X_1	β_1	-6.88	0.13	0.54
X_2	β_2	5.00	4.63*	1.41*
X_3	β_3	8.13	2.50*	0.21
X_1^2	β_{11}	-1.38	2.50	1.36*
X_2^2	β_{22}	-17.62	-0.50	-0.56
X_3^2	β_{33}	-28.88*	3.25*	-0.020
X_1X_2	β_{12}	-2.50	-1.00	-0.18
X_1X_3	β_{13}	3.75	0.25	0.28
X_2X_3	β_{23}	-25.00*	-1.25	-1.43*

[a] $y=\beta_0+\beta_1x_1+\beta_2x_2+\beta_3x_3+\beta_{11}x_1^2++\beta_{22}x_2^2+\beta_{33}x_3^2+\beta_{12}x_1x_2+$ $+\beta_{13}x_1x_3+\beta_{23}x_2x_3$

The ANOVA results for modeled responses are reported in Table 4. Joglekar and May [30] suggested that for a good fit of a model, R^2 should be at least 0.80. In our study, the R^2 values for these response variables were higher than 0.80, indicating the adequacy of the applied regression models. Table 4 shows the test statistics for the model (*F*-test and probability) of oil recovery and oil quality. The probability (*p*-value) of all regression models was below 0.05, which means that there was a statistically significant multiple regression relationship between the independent variables and the response variable.

The best way of expressing the effect of screw pressing parameters on oil recovery and oil temperature within the investigated experimental range was to generate response surfaces of the model (Fig. 1). From Fig. 1 can be seen that the amount of obtained oil significantly increased with the increase of pressing temperature, and maximum oil yield is obtained using electromotor frequency of 30 Hz. In our earlier paper [9] where the walnut oil extraction using screw pressing were investigated, oil temperature was also significantly influenced by used temperature for heating the output press head.

Furthermore, increasing the nozzle size ID the volume of oil decreases. The amount of residual oil in cake increased also with the nozzle size from 10 to 12 mm and decreased with increase of temperature. It can be seen that the oil temperature is significantly influenced by used temperature for heating the output press head and by used frequency, while nozzle size did not have significant influence on oil temperature. The ANOVA showed that the models were acceptable and could be used for optimization the pressing parameters with respect to oil recovery and temperature.

Optimization of screw pressing of hazelnut oil

The final goal of RSM is the process optimization. Thus, the developed models can be used for simulation and optimization. Optimization is an essential tool in food engineering for the efficient operation of different processes to yield a highly acceptable product [31]. During optimization of screw pressing process, several response variables describe the oil quality characteris-

Table 4. Analysis of variance (ANOVA) of the modelled responses; the recovery

Source	Sum of squares	Degree of freedom	Mean square	*F*-value	*p*-value
		Oil volume			
Model	8811.99	9	979.11	3.97	0.0413
Residual	1726.25	7	246.61		
Lack of fit	1056.25	3	352.08	2.10	0.2427
Pure error	670.00	4	167.50		
Total	10538.24	16			
		Oil temperature			
Model	306.49	9	34.05	4.79	0.0254
Residual	49.75	7	7.11		
Lack of fit	33.75	3	11.25	2.81	0.1717
Pure error	16.00	4	4.00		
Total	356.24	16			
		Cake oil			
Model	36.00	9	4.00	4.86	0.0246
Residual	5.77	7	0.82		
Lack of fit	0.14	3	0.046	0.033	0.9910
Pure error	5.63	4	1.41		
Total	41.77	16			

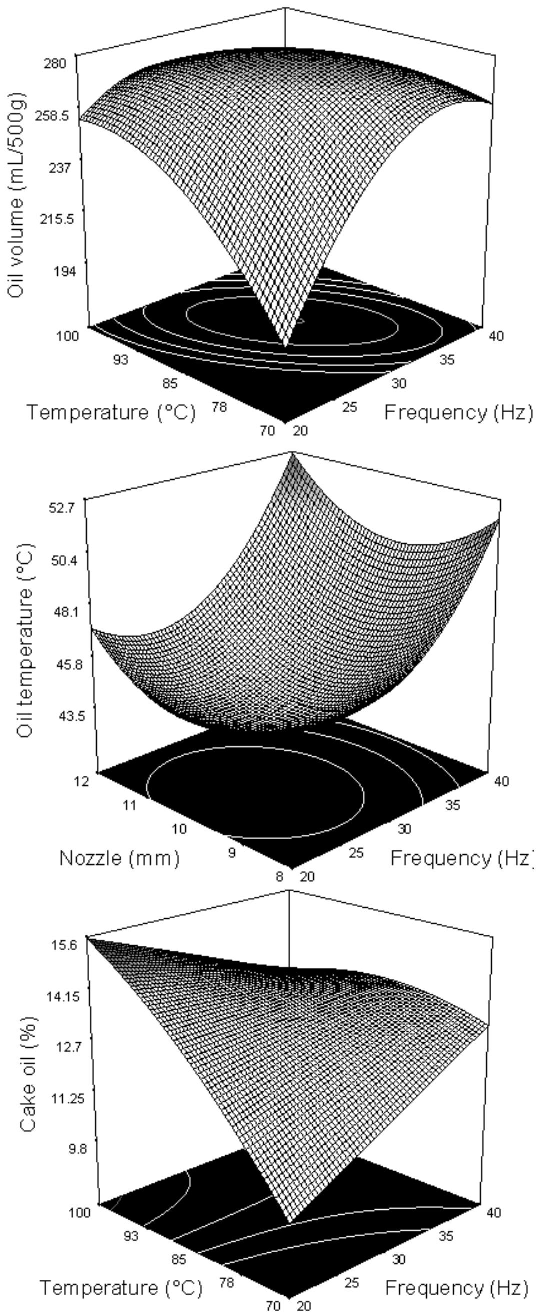

Figure 1. Response surface plots showing the effects of investigated variables on oil temperature and recovery.

tics and had influence on oil recovery [9]. Some of these variables need to be maximized, while others need to be minimized. In this study the following optimization conditions were proposed for calculations: the maximum oil yield and the minimum oil temperature. The goal of this research was to find the best settings for the screw pressing, *i.e.*, the best temperature, frequency and nozzle size. By applying desirability function method [32], the optimum screw pressing conditions were obtained: temperature of 70 °C, frequency of 30 Hz and using nozzle of ID 9 mm. Screw press oil volume was calculated to be 256.8 ml, oil temperature 41.7 °C and cake residual oil 11.4%, which is in very close agreement with experimental obtained data.

Oil quality

In obtained hazelnut oil at calculated optimal screw pressing conditions given in the text above, the oil quality parameters were determined. From Table 5 it can be seen that the produced hazelnut oil had excellent quality. Peroxide is one of the oil quality indicators and its value in obtained hazelnut oil was 0 mmol O_2/kg. Primary oxidation processes in oil mainly form hydroperoxides, which are measured by the peroxide value. In general, the lower the peroxide value the better is the oil quality [33]. This obtained peroxide value indicates that the investigated oil had good quality according to excellent quality of used raw material-hazelnuts, which is very important because such obtained oil is not necessary to pass further refining.

Table 5. Physicochemical properties of hazelnut oil

Property	Value
Iodine value, g I_2/100 g of oil	91.55
Saponification value, mg KOH/g of oil	191.46
Peroxide value, mmol O_2/kg of oil	0
Free fatty acids, %	0.23
Insoluble impurities, %	0.42
Moisture content, %	0.045
p-Anisidine value	0.19
Totox value	0.19

Moisture content (mentioned earlier) in the oil was 0.045%, free fatty acids 0.23% and insoluble impurities 0.42% which are also the another parameters of oil quality. Water contributes to the hydrolysis of oil during processing, which generates free fatty acids and glycerol products. It is very important that cold pressed oils are low in moisture content and free fatty acids to maintain the quality and shelf life of the oils [7].

Furthermore, other physicochemical properties of hazelnut oil were also determined. Iodine value was expressed as the grams of iodine absorbed per 100 g of lipid and was determined to be 91.55 g I_2/100 g. Saponification value, which is the number of milligrams of KOH required to neutralize the fatty acids resulting from complete hydrolysis of one gram of oil was determined to be 191.46 mg KOH/g. Good quality oil should have *p*-anisidine value of less than two, and Totox oxidation value, so called Totox value less than four [33]. The hazelnut oil in this study had *p*-anisidine value 0.19. The calculated Totox value was 0.19.

Oxidative stability of hazelnut oil

Oxidative stability of cold pressed hazelnut oil with and without the addition of natural antioxidants is shown in Table 6. The stability of oil is determined by the accelerated oxidation test, the Schaal oven test (63 °C). Hazelnut oil without added antioxidants (control sample) after four days of the test had a peroxide value 10.35 mmol O_2/kg. The addition of the OxyLess CS rosemary extract in concentrations of 0.1 % and 0.3% achieved the best stability of oil to oxidative deterioration (peroxide value was 0.98 and 0.74 mmol O_2/kg after four days) in relation to the use of other investigated natural antioxidants. Green tea extract leads to an increase in the stability of the hazelnut oil, in relation to hazelnut oil with addition of the pomegranate extract and olive leaf extract. Winter savory essential oil (0.05%) effectively protected this oil against oxidative deterioration compared to essential oil of oregano. However, the addition of sage essential oil did not affect the stability of hazelnut oil to oxidative deterioration; on the contrary, peroxide value was higher than the control sample after 4 days of the test.

Extraction of residual oil from pressed cake with CO_2

The cake resulting from pressing at optimal conditions (temperature of 70 °C, frequency of 30 Hz and nozzle ID 9 mm) was extracted with supercritical CO_2 and the obtained kinetic curve for this experiment is shown in Fig. 2. The amount of residual oil in pressed cake at optimal conditions was 11.81% (obtained by Soxhlet extraction). From Fig. 2 it can be seen that after 4 h of extraction the residual oil in the press cake was totally extracted by supercritical CO_2. From the shape of the extraction curve can be seen that the extraction process is divided in three periods: rapid extraction period, transition period and slow extraction period [34]. The first period is the constant extraction rate period, where the external surface of the particles is covered with solute and the convection is the dominant mass transfer mechanism. The amount of oil is ultimately limited by the solubility of the oil in the supercritical CO_2. The second is the falling extraction rate period where failures in the external surface oil layer appear and the diffusion mechanism starts combined with convection. In the third period the mass transfer occurs mainly by the diffusion. The similar shape of extraction curves was also obtained by other authors [28,29] who investigated supercritical CO_2 extraction of hazelnut oil.

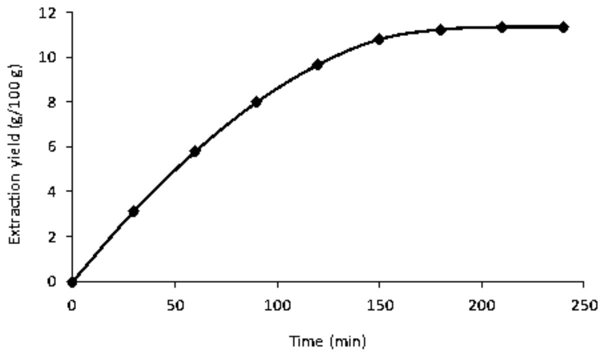

Figure 2. Extraction of cake residual oil with supercritical CO_2.

Table 6. Influence of natural antioxidants on oxidative stability of hazelnut oil

Sample	Concentration of antioxidants, %	Peroxide value, mmol O_2/kg				
		0 day	1 day	2 day	3 day	4 day
Hazelnut oil	–	0	1.97	4.44	8.46	10.35
Rosemary extract (Oxy Less CS)	0.1		0.24	0.50	0.75	0.98
	0.3		0.25	0.37	0.50	0.74
Green tea extract	0.1		1.00	1.47	1.96	2.45
	0.3		0.48	0.99	1.48	2.00
Pomegranate extract	0.1		1.46	2.91	5.88	8.50
	0.3		1.21	2.88	5.39	6.86
Olive leaf extract	0.1		1.46	4.25	7.28	9.90
	0.3		1.48	3.43	6.80	9.41
Oregano essential oil	0.05		1.98	4.41	7.84	10.00
Sage essential oil	0.05		2.02	4.41	8.01	11.38
Winter savory essential oil	0.05		0.75	2.48	6.93	8.42

CONCLUSION

The results of this study showed that the screw pressing conditions influenced the hazelnut oil extraction. Obtained hazelnut oil showed excellent quality as it had very small percentages of moisture, insoluble impurities and free fatty acids, as well as its peroxide value is 0 mmol O_2/kg. The optimal condition to obtain the best oil recovery and quality using response surface methodology was at temperature of head presses 70 °C, frequency of 30 Hz and using nozzle of ID 9 mm. The influence of different natural antioxidants on the oxidative stability of hazelnut oil was investigated, and the rosemary extract was the most efficient in protecting the hazelnut oil against oxidative deterioration. Residual oil content in pressed cake was totally extracted with supercritical CO_2. Such defatted cake, free of toxic solvents, can be used further in other processes, for example, in development of new functional and enriched products based on extrusion process which is our area of interest in future research.

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IZVOD

PROIZVODNJA LEŠNJAKOVOG ULJA PRIMJENOM METODE PREŠANJA I EKSTRAKCIJE POMOĆU SUPERKRITIČNOG CO_2

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U proizvodnji lješnjakovog ulja vrlo je važno pronaći odgovarajući postupak za što veće iskorištenje ulja iz jezgre lješnjaka. Cilj ovog istraživanja bio je ispitati utjecaj različitih procesa ekstrakcije ulja iz lješnjaka procesom hladnog prešanja, nakon čega je provedena ekstrakcija zaostalog ulja unutar pogače pomoću superkritičnog CO_2. U eksperimentima prešanja korištena je metoda odzivnih površina (RSM) pomoću koje je ispitan utjecaj temperature zagrijavanja glave preše, frekvencije elektromotora i veličine otvora pužne preše na temperaturu i iskorištenje ulja. U dobivenom lješnjakovom ulju određeni su parametri kvalitete ulja: peroksidni broj 0 mmol O_2/kg, slobodne masne kiseline 0.23%, netopljive nečistoće, 0.42%, vlaga u ulju 0.045%, jodni broj 91.55 g I_2/100 g, saponifikacijski broj 191.46 mg KOH/g i anisidinski broj 0.19. Najbolje antioksidacijsko djelovanje kod ispitivanog ulja pokazala je primjena ekstrakta ružmarina. Količina zaostalog ulja u pogači nakon hladnog prešanja potpuno je ekstrahirana pomoću superkritičnog CO_2, te tako odmašćena pogača može se dalje koristiti u drugim procesima.

Ključne reči: Lješnjakovo ulje • Kvaliteta • Hladno prešanje • Superkritična ekstrakcija • Metoda odzivnih površina