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ORIGINAL PAPER

Ultrasound-assisted extraction of cranberry seed oil: food waste valorization approach

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Abstract

Cranberry pomace considered as a by-product of fruit industry contains seeds which may be processed to highly nutritive oil. Conventional extraction methods may be, however, harmful to natural environment, thus alternative, ultrasound-assisted extraction method may be useful tool to reduce environmental impact. In the following study, sonication was applied to extract oil from cranberry seeds. The aim of the study was to determine the most efficient conditions of ultrasound-assisted extraction of oil and to investigate sonication influence on the properties of final product. Ultrasound amplitude and extraction time were independent variables; yield and maximum induction time of oils were responses. The most efficient conditions were amplitude of 95% and extraction time of 11.38 min. Model predicted extraction yield of 22.55 ± 0.36% (vs. actual 21.98 ± 0.08%) and induction time of 52.60 ± 0.95 min (vs. actual 61.95 ± 3.06 min). Detailed analyses of oil extracted in the most efficient conditions and the control sample were performed. Kinetic parameters of oil oxidation, fatty acid profile and distribution, melting characteristics studies were carried out. Sonication influenced activation energy of oxidation reaction, contribution of chosen fatty acids (oleic, α-linolenic and eicosenoic fatty acids) and distribution of oleic and α-linolenic fatty acids in sn-2 position of triacylglycerols. Slight changes in melting profile of oils were also recorded. Scanning electron microscopy of cranberry seeds revealed that ultrasound treatment resulted in pore enlargement and fat agglomeration damage. Additional studies of thermal properties of cranberry seeds: differential scanning calorimetry and modulated differential scanning calorimetry were performed, which confirmed that cranberry seeds may be a new source of oil with unique properties.

Keywords Ultrasound-assisted extraction · Cranberry seed oil · By-product · Response surface methodology · Oil quality

Introduction

Cranberry (*Vaccinium macrocarpon*) belongs to a family *Ericaceae* and genus *Vaccinium*. It is a berry fruit rich in vitamins, organic acids, sugar, fiber and mineral salts. Also, bioactive compounds, like polyphenols, terpenes and carotenoids are highly abundant in cranberry fruits [1]. According to Food and Agriculture Organization of the United Nations (FAO), in 2020 world production of cranberries exceeded 660,000 tons [2]. The fruits are industrially processed to obtain products such as: juice, jams, concentrates, jellies, powders. However, by-products including peel, seeds, stem cells, etc. are still a rich source of bioactive compounds. The recovery of valuable compounds which can bring financial and environmental benefits is also supported in circular economy statements [3]. Whole pomaces may be used in the preparation of value-added food enriched with fruit by-products. Mildner-Szkudlarz et al. [4] formulated muffins with raspberry and cranberry pomaces incorporation. Although, the textural properties of the muffins changed undesirably with increasing percentage of pomace, they were characterized by high bioactive phytocompounds contents. Also, Bajerska et al. [5] prepared muffins with sour cherry pomace, which turned out to be rich in polyphenols and dietary fiber.

Valorization of fruit waste is also possible by extracting valuable compounds. However, conventional extraction methods may contribute to environment being degraded and are also cost-intensive due to high organic solvents consumption and high energy input needed. That is the

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reason why novel, unconventional extraction methods, which enable to reduce both solvent and energy usage, and additionally enhance the effectiveness and selectivity of the process are increasingly applied. One of those alternative extraction methods is ultrasound-assisted extraction (UAE). According to review by Chemat et al. [6] ultrasound-assisted extraction is based on several mechanisms including sonocapilary effect, sonoporation, detexturation and intense mixing. Those physical phenomena may be responsible for extraction enhancement when ultrasound is applied. Aside from that, acoustic cavitation bubbles collapse results in exceeded chemical activity of solvent and also improved mass transfer from solid to liquid phase. One of the most crucial advantages of ultrasound-assisted process of lipid extraction mentioned by Deng et al. [7] is time and energy saving, comparing to conventional extraction methods, which involve solvent heating. The comparable yields of oil may be achieved by UAE faster by decreasing effective extraction time. To specify economic issues some studies could be recalled. Jacotet-Navarro et al. [8] introduced laboratory-scale results comparing 30 min UAE with 5 h heat reflux conventional extraction of bioactive compounds from rosemary leaves. The ultrasound-assisted process consumed almost 37-fold less energy (in kWh/kg extract) comparing to the conventional extraction. Also, conventional extraction took 10 times longer to be comparably efficient to UAE. Gharibzahedi et al. [9] concluded similarly in case of oil extraction from lesser mealworm larvae, where UAE was almost 18.5-fold less electric energy consuming (in kWh/g oil) than conventional extraction described as two-hour process with stirring and increased temperature (57–80 °C).

The UAE was applied formerly by Górnaś et al. [10] as a method of fruit seed valorization in case of apples, red currant, gooseberries, grapes, pomegranates, quinces, sea buckthorns, watermelons and melons. Previous studies revealed that UAE significantly improved extraction yield with simultaneous decrease in solvent consumption and time of the process in case of flaxseed oil [11], soybean oil [12], rapeseed oil [13] or mango kernel oil [14]. The qualitative studies of UAE oils are, however, inconclusive. The mass transfer enhancement during sonication process may result in higher antioxidant release (especially lipophilic compounds) and thus improvement of oxidative stability of oil and that was observed in case of hemp seed oil [15], canola seed oil [16], grapeseed oil [17] or raspberry seed oil [18]. On the other hand, a few undesired effects of ultrasound application in oil extraction were reported. UAE of grapeseed oil resulted in elevated free radicals' content and peroxide value [19], as well as decreased total polyphenol content [20]. Excessive ultrasound treatment, without constant temperature may result in decomposition of bioactive compounds naturally present in seeds. Moreover, within the temperature increase, higher free fatty acid concentration was observed in UAE pumpkin seed oil [21]. That indicates the triacylglycerol degradation to free fatty acids occurred during sonication due to hydrolysis or oxidation process. Aforementioned considerations force to study further the UAE effects on yield and quality of oil. To obtain reliable and the most efficient process conditions, applying response surface methodology (RSM) appears to be useful [22, 23]. In the following study, the extraction yield and oxidative stability as responses were determined by the objectives to obtain satisfying amount of oil characterized by maximum oxidative stability possible, since oxidative stability is an indicator of oil utility and shelf life. To assess the oxidative stability, accelerated oxidation study called pressure differential scanning calorimetry (PDSC) might be used [24]. RSM was successfully used to optimize oil UAE process of papaya seed oil [25], rice bran [26], apricot kernel oil [27], hemp seed oil [28], raspberry seed oil [18] or perilla oil [29]. Nevertheless, none of mentioned studies considered oxidative stability measured in PDSC study as a dependent variable of an experiment.

Apart from oxidative stability, thermal characteristics e.g., melting profile of oil may be assessed to define the quality and functionality of oil [30]. Differential scanning calorimetry (DSC) which records the heat flow associated with thermal transitions in the material in function of time or temperature is one of the methods to obtain melting characteristics of oil [31]. Fatty acid profile is another property of oil determining its nutritional value and utility. Moreover, detailed analysis of fatty acid distribution in sn-2 and sn-1,3 positions of triacylglycerols allows to assess the digestibility of fat [32]. Both features may be assessed in fatty acid methyl esters gas chromatography study.

The importance of sustainable acquisition of cranberry seed oil may be justified by the fact that oil obtained from cranberry seeds may be considered as rich in bioactive compounds. The polyphenol content in cranberry seed oil was investigated by Yu et al. [33] and it was reported at the level of 1.61 mg gallic acid equivalent (GAE) per gram of oil or at 13.68 μmol GAE/g reported by Luo et al. [34]. Other bioactive compounds were studied by Van Hoed et al. [35] total sterol content equal to 6.92 mg/g was reported with β-sitosterol as major sterol. From tocopherols and tocotrienols the most abundant one was γ − tocotrienol with the content of 1235 mg/kg oil. Comparing to other fruit sources, the cranberry seed oil has higher sterol content than gooseberry, pomegranate, apple and grape seed oils [10]. Sum of tocopherol and tocotrienol content in cranberry seed oil is higher than values reported for gooseberry, grape, watermelon and melon seed oils but not as high as for the apple, red currant, pomegranate or sea buckhorn seeds oils [36]. Taking those results into account it may be concluded that cranberry seed oil is a source of bioactive compounds with antioxidant properties.

The aim of the study was to determine the most efficient conditions of ultrasound-assisted extraction regarding cranberry seed oil yield and oxidation resistance assessed by pressure differential scanning calorimetry. Also, to identify the differences between oils extracted by applying ultrasound and oils extracted conventionally, qualitative assays such as: melting characteristics, fatty acid profile and fatty acid distribution in triacylglycerols were conducted. To analyze physical influence of ultrasound on the cranberry seed's matrix, scanning electron microscopy study was carried out. To obtain a complete picture of thermal behavior of cranberry seeds, they were examined by differential scanning calorimetry and modulated differential scanning calorimetry studies. Overall purpose was to confirm if the UAE is applicable in valorization of cranberry fruit waste.

Materials and methods

Materials

Cranberry (*Vaccinium macrocarpon*) seeds purchased on the local market (Naturini, Poznań, Poland) were used in the following study. The water activity of whole seeds examined in Rotronic Hygrolab C1 (Rotronic AG, Switzerland) hygrometer at 25±0.3 ºC was lower than 0.2. Before extracting, samples of around 2 g of the seeds were milled in the laboratory IKA Tube Mill (IKA-Werke GmbH & Co. KG, Germany) applying 20,000 rpm in 60 s time.

Chemicals

The chemicals: *n*-hexane, hydrochloric acid, calcium chloride, diethyl ether, magnesium sulfate, acetic acid, methanol, potassium hydroxide and iodine were purchased in Chempur, Poland. Bile salts solution, TRIS and pancreatic lipase were purchased in Sigma-Aldrich, USA.

Thermal properties of cranberry seeds

Differential scanning calorimetry (DSC) of milled seeds was carried out in TA DSC Q200 calorimeter (TA Instruments, New Castle, DE, USA). The procedure was applied according to the method described by Ostrowska-Ligęza et al. [37] with slight modifications. The samples were cooled down to – 70 °C and then heated to the temperature of 200 °C with a heating rate equal to 5 °C/min. The peak temperatures (T_{peak}) were determined based on the curves obtained as results of the studies.

Modulated differential scanning calorimetry (MDSC) was performed in the same apparatus to record the glass transition temperature of cranberry seeds, according to the method applied by Jakubczyk et al. [38].

Experimental design

The experimental plan was designed to assess the possible impact of extraction parameters on the yield and oxidative stability of obtained oils. Central composite design (CCD) plan with nine runs and three replications at central point, carried out in triplicate was applied. The two variable factors were ultrasound wave amplitude (X_1) and sonication time (X_2). The actual and coded values of ultrasound amplitude and sonication time are presented in Table 1. The 11 runs were performed in the triplicate and obtained results of responses were analyzed in the statistical software Design-Expert (v. 22.0.2, Stat-Ease Inc., Minneapolis, MN, USA). The model fit was carried out considering values of determination coefficient, lack of fit test, p-value and coefficient of variation. The threshold p-value was 0.05. Type of model was chosen by analyzing significance of model fit.

Ultrasound-assisted extraction

Obtained seed powder (2 g) was placed in 50 mL Falcon tubes. The extraction process was carried out using UP400S ultrasound processor supplied by Hielscher Ultrasonics (Tetlow, Germany), with adjustable wave amplitude level, the output power of 400 W and replaceable sonotrode, *n*-hexane was used in solid/liquid ratio at level 1:15. Solvent was added to the sample right before the ultrasound application. The tube with seed powder and *n*-hexane was placed in the ice bath and the sonotrode was dipped in the tube to the depth which disabled *n*-hexane spilling, additionally, tube was covered with the foil. The temperature of the process, controlled by immersion thermometer, did not exceed 45 ºC.

Table 1 Actual and coded values of independent variables of ultrasound-assisted extraction of cranberry seed oil

Run	Ultrasound wave amplitude X_1		Sonication time X_2	
	Actual (%)	Coded	Actual (min)	Coded
1	30	− 1	2	− 1
2	80	1	2	− 1
3	30	− 1	10	1
4	80	1	10	1
5	19.75	− 1.414	6	0
6	90.25	1.414	6	0
7	55	0	0.344	− 1.414
8	55	0	11.656	1.414
9	55	0	6	0
10	55	0	6	0
11	55	0	6	0

Conventional extraction

Conventional solid–liquid extraction was conducted to obtain reference, control sample. Two grams of ground cranberry seeds were placed in a Falcon tube with 30 mL of *n*-hexane and stirred for two hours [39].

Oil yield determination

After extraction, samples were centrifuged for 20 min applying 4000 rpm. Then, filtered and dried with anhydrous magnesium sulfate extracts were evaporated using Büchi R-215 Rotavapor vacuum rotary evaporator (Büchi Labortechnik AG, Flawil, Switzerland) at temperature of 40 °C and pressure at minimum level of 70 mbar. Samples of oil were then dried under nitrogen atmosphere to remove residual solvent. Oil yield was determined, gravimetrically, defined as the mass of extracted oil to the initial mass of powdered seeds using Eq. 1 [40]:

$$Y = \frac{m_o}{m_s} \times 100 \tag{1}$$

where: Y–oil yield (%); m_o–mass of oil (g); m_s–initial mass of powdered seeds (g).

Oxidative stability and kinetic parameters of oxidation reaction of cranberry seed oil

The oxidation reaction maximum induction time was determined to describe the oxidative stability of obtained cranberry seed oil samples. The procedure was described in previous studies [41, 42]. Measurements were conducted in a DSC Q20 TA Instrument (TA Instruments, New Castle, DE, USA). Briefly, aluminum pan containing 3–4 mg of oil and an empty pan that was used as a reference were placed in the measuring cell. The oxygen atmosphere at flow rate of 50 mL/min and initial pressure of 1400 kPa was provided. Experiment was carried out in isothermal conditions at 120 °C. The value of maximum induction time was obtained based on the maximum rate of oxidation recorded on the plot of heat flow in the function of time.

Additionally, oxidation processes kinetics of control oil sample (CR_USC) and sample of oil extracted using ultrasound in the most efficient conditions (CR_USO) were investigated [43]. The apparatus configuration and samples preparation were the same as for aforementioned analysis, however, experiments were carried out in isothermal conditions of 110, 115, 120, 125 and 130 °C. Parameters of oxidation reaction kinetics were calculated with the use of Ozawa-Flynn-Wall methodology [44, 45] and Arrhenius equation. Parameters of regression lines equations were calculated on the basis of the graph plotted between values of the logarithm of induction time versus reciprocal temperature, using following equations:

$$\log\tau = aT^{-1} + b \tag{2}$$

where: τ is an induction time in minutes, a and b are adjustable coefficients. Oil oxidation reaction is treated as first-order reaction, so activation energy value can be determined according to the Ozawa-Flynn-Wall methodology:

$$E_a = 2.19 \times R \times a \tag{3}$$

where: E_a is activation energy, R is gas constant and a is a coefficient from Eq. (2). According to Arrhenius Eq. (4):

$$k = Ze^{\frac{-E_a}{RT}} \tag{4}$$

where: k is a reaction rate coefficient. Based on that activation energy, pre-exponential factors and reaction rate constants for all temperatures were calculated.

Gas chromatography

Percentage share of identified fatty acids, as well as fatty acid distribution in triacylglycerols of the control sample and sample obtained by applying chosen parameters of ultrasound-assisted extraction were determined in the GC method, using YL6100 GC gas chromatograph apparatus (Young Lin Bldg., Anyang, Hogye-dong, Korea) equipped with a flame ionization detector, as described by Bryś et al. [46]. To assess the fatty acid profile the samples of fat were methylated to fatty acid methyl esters (FAME), according to the PN-EN ISO:2001 method [47]. Detected FAMEs were identified by comparing their retention times with the standard FAMEs mixture. The area normalization procedure with calculation of each fatty acid percentage was applied to obtain fatty acid profile of oils. The analysis of fatty acids distribution in sn-2 and sn-1,3 positions of triacylglycerols was preceded by pancreatic lipase hydrolysis according to the method described by Pina-Rodriguez and Akoh [48].

Melting characteristics

Melting characteristics of control cranberry seed oil and oil extracted in chosen ultrasound conditions were obtained in a differential scanning calorimetry (DSC) study, applying the methodology described by Ostrowska-Ligęza et al. [49]. A DSC Q200 calorimeter (TA Instruments, New Castle, DE, USA) was used. Samples of fat were placed in aluminum, healed hermetically pans. The analysis started with heating the oils to 80 °C to promote melting crystals and erasing thermal memory of material. Then, samples were cooled to − 80 °C and heated again to 80 °C with a heating rate of 15 °C/min.

Scanning electron microscopy analysis

Microstructure of chosen seeds which underwent the conventional extraction and ultrasound-assisted extraction was analyzed using TM-3000 (HITACHI, Japan) at magnification of 2500x.

Statistical analysis

Statistical analysis of detailed oil's studies (PDSC, DSC, GC) was carried out using Statistica (v. 13.3, StatSoft, Kraków, Poland) software. All the assays were performed in triplicate, one-way ANOVA and post-hoc Tukey's test were performed. Differences were considered as significant at a p value of 0.05.

Results and discussion

DSC and MDSC studies of cranberry seeds

The results of cranberry seeds DSC were T_{peak} of endotherm I − 34.71 ± 2.15 °C and T_{peak} of endotherm II 155.32 ± 1.33 °C. The example of DSC curve is shown on the Fig. 1. It can be observed that the DSC curve is of similar course as in the case of previously studied blackberry and raspberry seeds [41]. The endothermic peak observed at around − 35 °C indicates low melting fat fraction presence in the material, which consists of mainly poly- and monounsaturated fatty acids. Second endothermic maximum, around 150 °C is observed due to carbohydrates residues in milled seeds [50]. The results are in agreement with fatty acid profile analysis described further.

MDSC study was carried out to define the glass transition temperature of the milled cranberry seeds. It is a helpful tool to determine the storage conditions of the food products as well as to describe its quality changes during storing. Mainly, food products may be considered as stable when stored under the glass transition temperature. Above the glass transition temperature, the solid state is transformed to supercooled melt highly dependent on temperature [51]. The results of the MDSC analysis, containing mean onset, midpoint and endpoint glass transition temperatures were: −14.55 ± 0.39 °C, −13.47 ± 0.06 °C and 12.50 ± 0.16 °C, respectively. According to the results, single glass transition was noted, as pictured in example, presented on Fig. 2.

Ultrasound-assisted extraction

Extraction yield

Generally, it can be observed that applying higher amplitude of ultrasounds in longer time results in higher extraction yield, detailed results are shown in Table 2. Additionally, graphs presenting observed vs. predicted by the model values of responses can be found in supplementary material (Fig. 1). Figure 3 shows surface plot of extraction time and amplitude of ultrasounds influence on the extraction yield. Enhanced mass transfer, as well as increased cavitation phenomena intensity leads to improved release of oil from the material due to solvent penetration of the matrix [52]. The results are in agreement with previous studies for raspberry seed oil [18] and hazelnut oil [53]. The yield of extraction was predicted significantly ($p < 0.05$) by applying quadratic model. That result was supported by high determination coefficient value ($R^2 < 0.90$) and not significant p value result in lack of fit test ($p > 0.05$). Additionally, based on ANOVA results it can be concluded that extraction yield was significantly ($p < 0.05$) influenced by both ultrasound amplitude and extraction time. Multiple regressions coefficients were used to analyze the linear, quadratic and interaction terms of yield in response

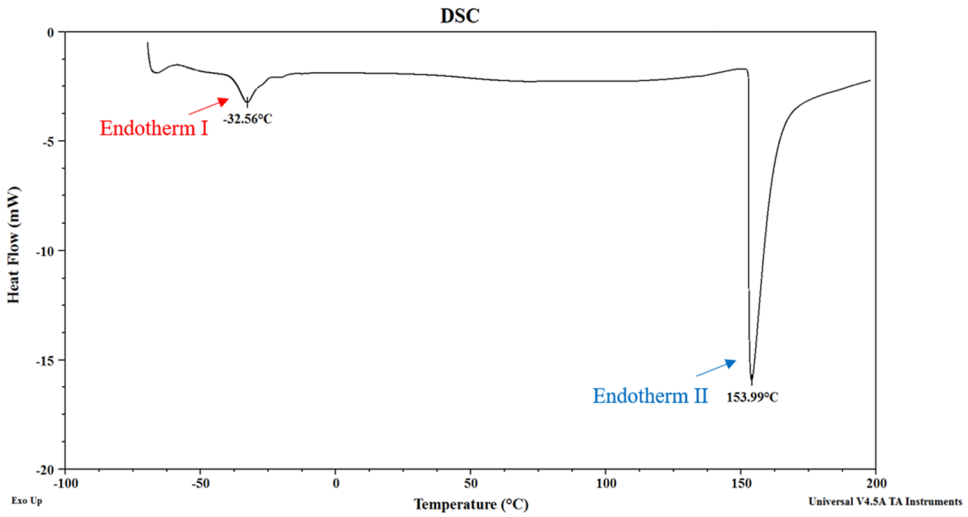

Fig. 1 The example of DSC curve recorded for cranberry seeds

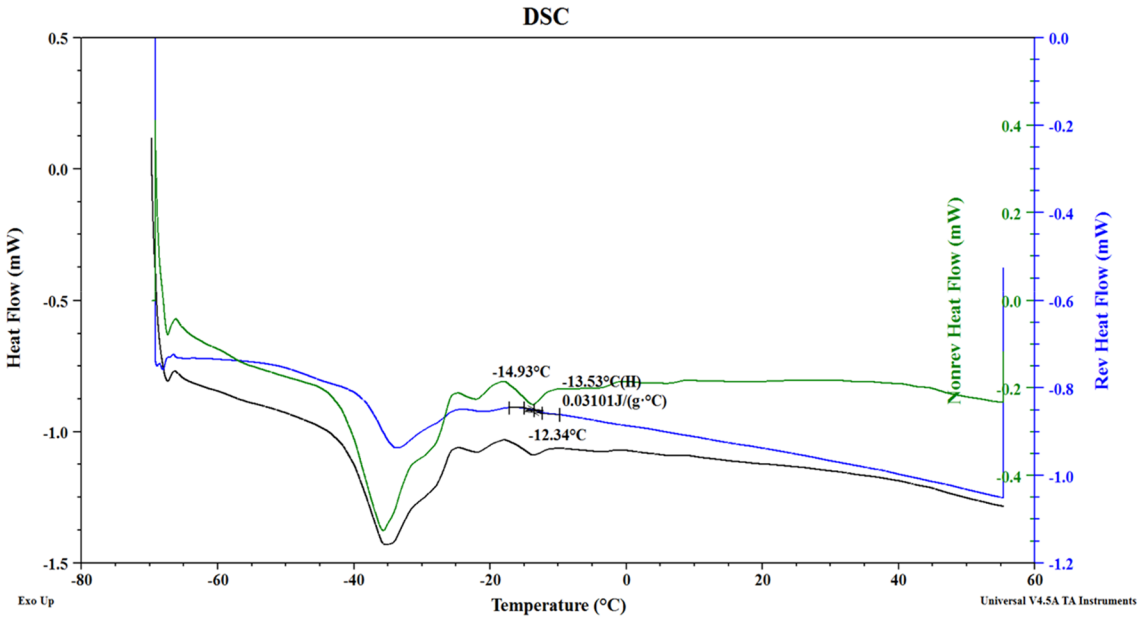

Fig. 2 Example of the MDSC graph for cranberry seeds; black line- heat flow, green line- non-reversible heat flow, blue line-reversible heat flow

Table 2 Observed mean values of yield and τ_{max} in variable conditions of amplitude wave and extraction time

Run	Variable conditions		Responses	
	Amplitude of ultrasound wave (%)	Sonication time (min)	Yield (%)	τ_{max} (min)
1	30	2	18.97 ± 0.43	45.65 ± 7.32
2	80	2	19.83 ± 0.31	49.66 ± 2.79
3	30	10	19.64 ± 1.35	47.92 ± 3.32
4	80	10	21.21 ± 2.25	50.61 ± 2.84
5	19.75	6	18.17 ± 0.44	47.27 ± 0.35
6	90.25	6	19.46 ± 0.75	52.15 ± 5.05
7	55	0.344	19.68 ± 0.86	48.08 ± 2.15
8	55	11.656	20.68 ± 0.47	49.20 ± 3.77
9	55	6	19.24 ± 1.24	49.45 ± 3.50
10	55	6	18.82 ± 1.60	48.54 ± 3.13
11	55	6	18.41 ± 1.84	50.74 ± 1.94

surface model. A second-order polynomial Eq. (5) was used to define the response of the yield the exact Eq. (6) allows to calculate the actual yield of ultrasound assisted cranberry seed oil extraction by using actual values of factors.

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \beta_{11} {X_1}^2 + \beta_{22} {X_2}^2 \quad (5)$$

where Y is the response; β_0 is the constant coefficient, β_1, and β_2 are the regression coefficients for the linear terms; β_{11} and β_{22} are the quadratic terms; β_{12} is the interaction terms;

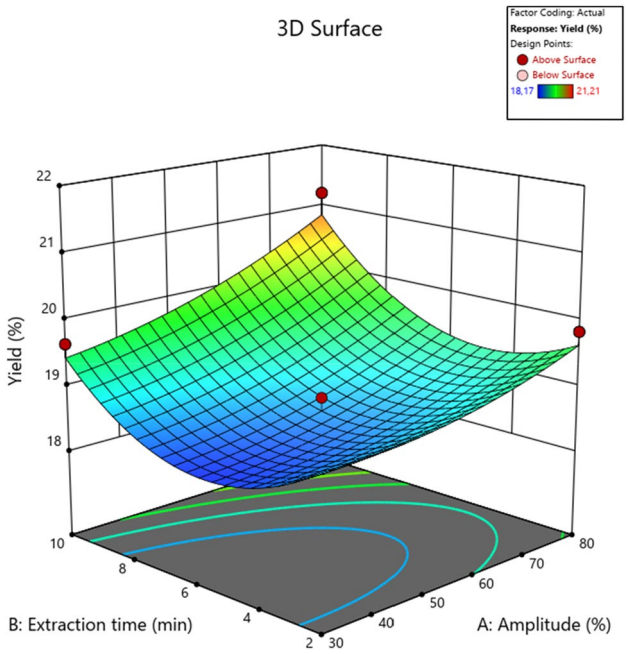

Fig. 3 Surface plot of yield (%) influenced by ultrasound extraction time (min) and ultrasound amplitude (%)

and X_1 and X_2 represent the coded values of independent variables.

$$\begin{aligned} \text{Yield} = {} & 19.83127 - 0.006905 \bullet \text{Amplitude} - 0.575931 \\ & \bullet \text{Extractiontime} + 0.001775 \bullet \text{Amplitude} \bullet \text{Extractiontime} \\ & + 0.000159 \bullet \text{Amplitude}^2 + 0.048880 \bullet \text{Extractiontime}^2 \end{aligned} \quad (6)$$

Maximum induction time of oxidation

The observed mean results of τ_{max} are shown in Table 2, the comparison of actual and predicted by the model values are shown in supplementary material (Fig. 1). Figure 4 shows the surface plot for the maximum induction time (τ_{max}) response results. Linear model of prediction was chosen as significant ($p < 0.05$), as the ANOVA results revealed that only amplitude has significantly influenced τ_{max} values. Quite high value of determination coefficient ($R^2 > 0.75$) and insignificant result of lack of fit test indicate proper model fit ($p > 0.05$). The first-order polynomial Eq. (7) was utilized to define the response of τ_{max} and exact τ_{max} values may be calculated using Eq. (8).

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 \tag{7}$$

where Y is the response; β_0 is the constant coefficient, β_1, and β_2 are the regression coefficients for the linear terms.

$$\tau_{max} = 44.38344 + 0.068007 \bullet \text{Amplitude} + 0.150122 \bullet \text{Extractiontime} \tag{8}$$

Opposite findings were described by Samaram et al. [25] for the papaya seed oil. However different oil stability test was used but the results revealed that time was the only independent variable of the experiment (among extraction time, temperature, ultrasound power and solvent to sample ratio) that influenced the oil stability measured in peroxide and p-anisidine values tests. The increased stability of oil extracted applying higher amplitude of ultrasounds may be the result of antioxidant bioactive compounds release during the extraction process. As residual particles of pomace may be found on the seeds, which was confirmed in the following study in seeds DSC analysis, they may contain bioactive compounds commonly found in cranberry pomace, such as polyphenols [54]. Those compounds may act as natural antioxidative preservatives and improve resistance to oxidation of oils.

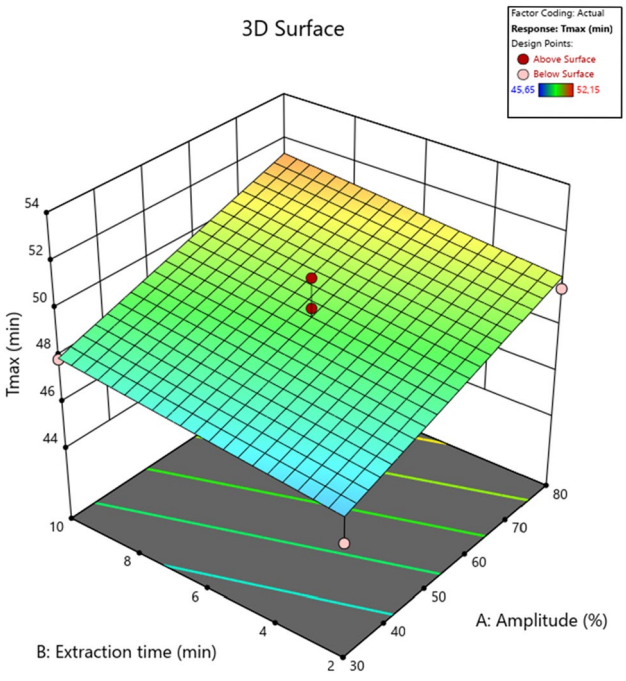

Fig. 4 Surface plot of τ_{max} value (min) influenced by ultrasound extraction time (min) and ultrasound amplitude (%)

Optimization

The most efficient parameters of cranberry seed oil ultrasound-assisted extraction were calculated using RSM. Criteria for responses i.e., extraction yield and maximum induction time were set as 'maximize' and criteria for independent variables were set as 'in range' of 20–100 for amplitude and 2–12 for extraction time. 80 solutions were suggested by the software, with one selected as the most efficient, indicating amplitude of 95% and extraction time of 11.375 min to be the most preferable. The chosen solution predicted extraction yield of $22.55 \pm 0.36\%$ (vs. actual $21.98 \pm 0.08\%$) and τ_{max} of 52.60 ± 0.95 min (vs. actual 61.95 ± 3.06 min). As the optimized parameters of extraction were outside of the applied design space, biased observed value of τ_{max} comparing to predicted by the model response was observed. However, it is in agreement with the criteria of maximizing the τ_{max} response.

Kinetic parameters of oil oxidation

Based on the results of PDSC measurements conducted at five temperatures, kinetic parameters of oil oxidation reaction were calculated. Firstly, a graph of logarithm of maximum induction time in function of reciprocal temperature was plotted (supplementary material, Fig. 2). The R^2 coefficients at a level above 0.95 meant that the data describing linear correlation could be used to further calculations.

The results of calculations are summarized in the Table 3. It was concluded that activation energy was significantly higher for oil extracted applying optimal chosen conditions of ultrasound extraction than activation energy of oil oxidation reaction for a control sample. However, activation energy values above 90 kJ/mol could be considered as high referring to the previous studies. Those results are similar as for linseed oil [55], soybean or sunflower oil [56]. Previous studies reported lowering activation energy of oxidation when ultrasound was applied [57]. However, both results are in agreement with studies by Torres et al. [58] which summarized that E_a of oil oxidation is in a range 41,842 to 104,605 kJ/mol.

Table 3 Kinetic parameters of cranberry seed oil oxidation reaction; CR_USC- control oil sample, CR_USO- sample of oil extracted using ultrasound in the most efficient conditions

Parameter	CR_USO	CR_USC
a	5.41	5.01
b	11.89	11.10
E_a (kJ/mol)	98.50 ± 1.34^b	91.22 ± 2.23^a
Z (1/min)	1.35×10^{10}	2.37×10^9
k at 110 °C	5.03×10^{-4}	8.67×10^{-4}
k at 115 °C	7.50×10^{-4}	1.25×10^{-3}
k at 120 °C	1.11×10^{-3}	1.80×10^{-3}
k at 125 °C	1.61×10^{-3}	2.55×10^{-3}
k at 130 °C	2.33×10^{-3}	3.59×10^{-3}

a, b values from line equation, E_a activation energy, *Z* pre-exponential factor, *k* reaction rate coefficient; different letters $^{a-b}$ indicate significant differences at $p < 0.05$

Fatty acid profile and distribution in triacylglycerols

Results of cranberry seed oil's fatty acid profile analysis are shown on Fig. 5. For both studied oils, linoleic acid (C18:2 n−6), followed by α-linolenic acid (C18:3 n−3) are fatty acids with the largest percentage share. It can be also concluded that cranberry seed oil was characterized by high contribution of polyunsaturated fatty acids in the studied samples. The method of extraction has significantly influenced the percentage share of oleic, α-linolenic and eicosenoic acids. Previous studies show that UAE did not affect the fatty acid composition of canola seed oil [16], grapeseed oil [17], papaya seed oil [59], flaxseed oil [11] or Moringa oleifera seed oil [60]. However, in case of pomegranate seed oil extracted using ultrasound, fatty acid profile was slightly influenced by the extraction method, especially considering SFA and PUFA [61]. Similar findings, assuming subtle changes in fatty acid composition were described for sunflower seed oil [62]. Opposite results were described for the UAE of raspberry seed oil by Teng et al. [18]. Also, Hosseini et al. [63] studied physicochemical properties of sesame, olive, sunflower and tallow olein oils treated with ultrasound. Sonication at 24 Hz at different amplitude values (25, 60 or 100%) was applied to extra virgin oils to analyze the effects of such treatment on the quality of fat. With increasing value of amplitude, a decrease in a content of linoleic fatty acid and α-linolenic fatty acid was observed. That may be connected to the higher cavitation caused by ultrasound resulting in oil degradation and as a consequence, lipid oxidation generated by free radicals formed in sonolysis process. However, overall conclusion is that UAE affects fatty acid profile to a small degree.

In the fatty acid distribution study it was found out that the share of oleic and α-linolenic fatty acids in sn-2 position of TAG differed in studied oils (supplementary material, Fig. 3). Although, the TAG content was not determined in the following study, so it remains unknown is the yield of specific TAGs also dependent on extraction method applied. The polyunsaturated fatty acids present in cranberry seed oil were located mostly in internal position of TAG, only α-linolenic fatty acid occupied rather external sn-1,3 positions than sn-2. That kind of distribution is common for vegetable oils [64]. The saturated fatty acids occupied only outer sn-1,3 positions in TAGs. The structure of TAG determines its biochemical and physical properties. Enzymatic hydrolysis of fats in the digestive system of humans cause release of fatty acids from sn-1,3 positions. So, in case of vegetable oils, mostly saturated fatty acids are being liberated during digestion process. They may, however, bind free calcium ions and be removed with feces [65]. The decrease

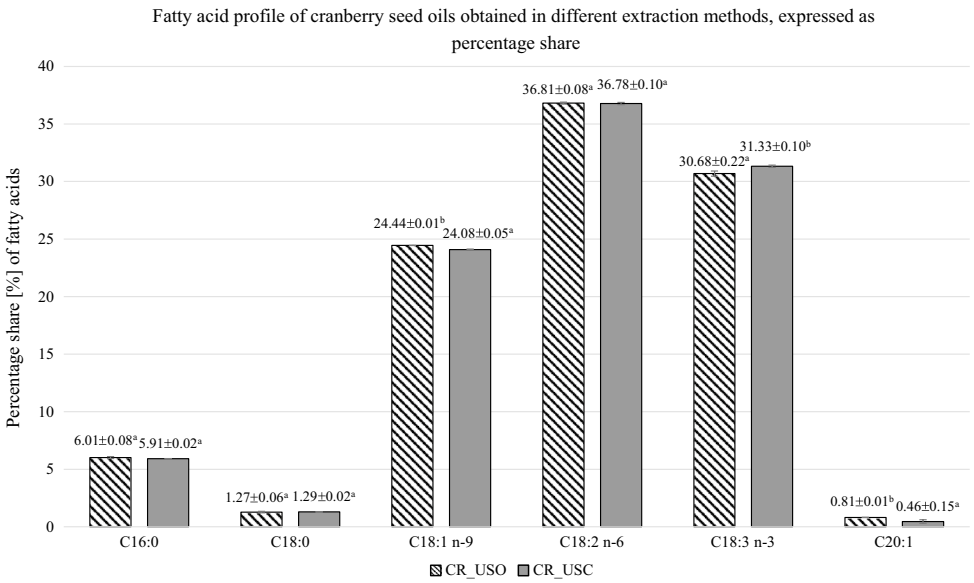

Fig. 5 Fatty acid profile of cranberry seed oils obtained in different extraction methods, expressed as percentage share (%); CR_USC- control oil sample, *CR_USO* sample of oil extracted using ultrasound in the most efficient conditions; *C16:0* palmitic acid, *C18:0* stearic acid, *C18:1* oleic acid, *C18:2* linoleic fatty acid, *C18:3* α-linolenic acid, *C20:1*- eicosenoic acid; different letters.$^{a-b}$ indicate significant differences at $p < 0.05$

of α-linolenic acid percentage share in sn-2 position in cranberry seed oil extracted using ultrasound in the most efficient conditions may be connected to the degradation of unsaturated fatty acids due to free radical's release during sonication process [66]. In other terms, a chain of chemical reactions induced particularly by hydroxyl and superoxide radicals and resulting in an autocatalytic process of fatty acid deterioration may occur during sonication [19] and it may affect the distribution of fatty acids in TAGs as well.

DSC study of oils

The melting curves of cranberry seed oils were characterized by two endothermic peaks, as presented in the example on the Fig. 6. T_{peak} for endotherm I was significantly higher for control sample comparing to oil obtained in optimal conditions and was equal to − 35.59 ± 0.19 °C in case of oil obtained in optimized conditions and − 33.59 ± 0.07 °C in case of control sample, T_{peak} values of endotherm II for cranberry seed oil extracted using sonication and control oil were measured at – 22.10 ± 0.08 °C and – 20.74 ± 0.16 °C, respectively, without any significant differences. The first peaks are connected with the presence of low-melting fractions of triacylglycerols containing mostly polyunsaturated fatty acids and second peaks indicate presence of medium-melting fractions rich in monounsaturated fatty acids [67]. The results of UAE influence on the melting characteristics of oils are lacking in the literature. Hosseini et al. [63] studied the effect of sonication treatment on extra virgin sesame, olive, sunflower and tallow olein oils' melting profiles. Higher amplitude values were related to higher crystallization enthalpies which means, ultrasound- treated oils have less ordered crystal structure than untreated ones.

Scanning electron microscopy

Pictures of milled cranberry seeds SEM analysis taken at 2500 × magnification are shown in Fig. 7. The most significant difference visible in the pictures is the pores size and their permeability. Raw cranberry seeds (1) have visible pores, but they seem to be clogged. Seeds which underwent the UAE in optimized conditions (2) have more pores which are enlarged, comparing to the pores of seeds after conventional extraction process (3). However, both extractions seem to cause release of substance present inside the pores. It can be also noticed, how extraction breaks the oil agglomerations, which are visible only in the picture A. In the pictures B and C oil residuals are present as individual particles with their higher number in case of seeds after classical extraction which proves increased efficiency of UAE comparing to conventional method. Similar findings were described in case of SEM analysis of sunflower seeds [62] and flax seeds [11] after UAE.

Conclusions

Given results of cranberry seeds thermal analysis support the hypothesis that they are a promising material for obtaining oil. The extraction optimization results show that quadratic model dependance may be applied to evaluate the impact of ultrasound amplitude and extraction time on the yield and linear model may be applied to describe maximum induction time dependance on process conditions of ultrasound-assisted extraction. The most efficient parameters of extraction were amplitude of 95% and extraction time of 11.38 min and prediction of yield was 22.55 ± 0.36% and maximum induction time 52.60 ± 0.95 min. Detailed characteristics of oil obtained in the aforementioned conditions revealed its high oxidation reaction activation energy and dominance of

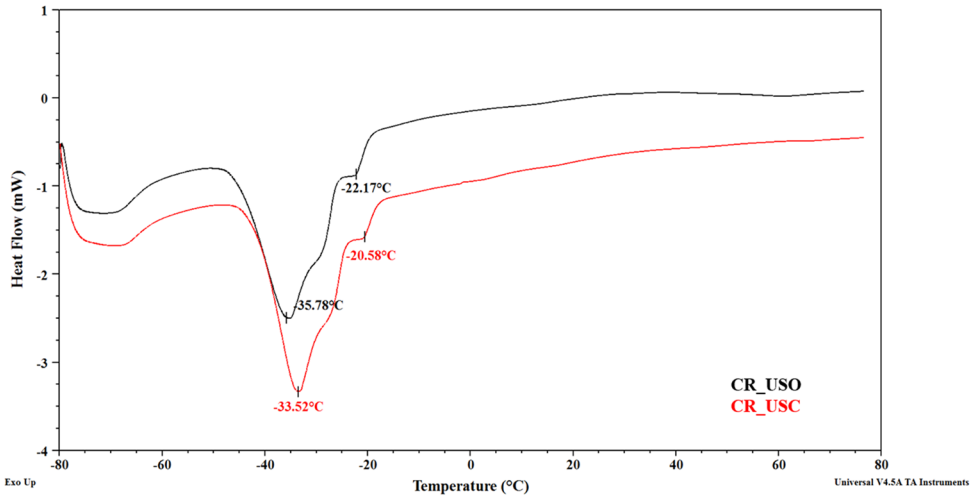

Fig. 6 Examples of cranberry seed oil's melting profile; *CR_USC* control oil sample, *CR_USO* sample of oil extracted using ultrasound in the most efficient conditions

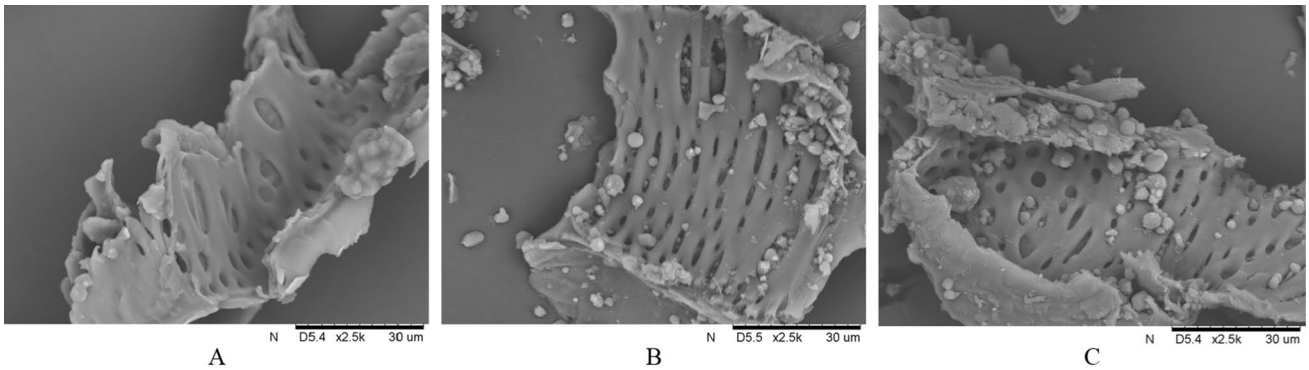

Fig. 7 SEM pictures of milled seeds: **A** raw cranberry seeds; **B** cranberry seeds treated with optimum ultrasound conditions; **C** cranberry seeds after conventional extraction

linoleic fatty acid in the fatty acid percentage share. Additional analysis in scanning electron microscopy procedure confirmed ultrasound influence on the seed matrix which is visible as the increase in pore size and oil agglomeration breakdown. To summarize, ultrasound was successfully applied to obtain oil from cranberry seeds. The process was optimized within the specified parameters of ultrasound amplitude and extraction time to achieve highest possible yield and maximum induction time. Thus, it may be concluded that ultrasound-assisted extraction is applicable in valorization of cranberry fruit waste.

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Author contributions Conceptualization of the study IP, AG, AW, methodology was prepared by IP, RB, EOL, AW and AG, formal analysis was performed by IP, statistical analysis was performed by IP and RB, manuscript was written by IP and reviewed by AG, figures were prepared by IP (Figs. 3, 4, 5 and Fig. 1 in supplementary material), EOL (Figs. 1, 2, 6 and Figs. 2, 3 in supplementary material) and AW (Fig. 7). Administration of the study and funding acquisition was performed by AG.

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Data availability The authors declare that the data supporting the findings of this study are available within the paper. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

Compliance with ethics requirements This article does not contain any studies with human or animal subjects.

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