Influence of sea buckthorn pomace pre-treatment and drying conditions on the drying kinetics, quantity and quality of seed oil

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Summary

Sea buckthorn (*Hippophae rhamnoides* L.) pomace was pre-treated by sonication or freezing and a thin layer of pre-treated or untreated pomace was dried under various conditions. The seeds separated from dried pomace were ground and subjected to batch extraction using hexane as a solvent. Effects of process factors, i.e. drying temperature (40 °C and 50 °C), pomace layer thickness (3 mm and 10 mm), ultrasound amplitude (0 % and 20 %) and pre-treatment temperature (–20 °C and 20 °C), on the drying kinetics, quantity and quality of seed oil were evaluated. Performance of air drying and oil extraction were correlated with the process factors by multiple regression equations, which highlighted a significant effect of pomace layer thickness. A thicker layer led to higher levels of effective diffusivity ($(0.141–1.484)\times10^{-9}$ $m^2 \cdot s^{-1}$), oil yield (13.1–14.5 %) and extraction efficiency (90.5–93.5 %) as well as to lower values of mean drying rate (0.144–0.354 $kg \cdot kg^{-1} \cdot h^{-1}$). Mean mass percentages of fatty acids found in the oils extracted from the seeds separated from the pomace dried at 50 °C, i.e. 26.8 % α-linolenic, 38.9 % linoleic, 22.7 % oleic, 0.9 % palmitoleic, 7.9 % palmitic and 2.8 % stearic acid, were almost invariant with the process factors.

Keywords

sea buckthorn; pomace; seed oil; air drying; effective diffusivity; fatty acids

Sea buckthorn has recently attracted considerable attention for its therapeutic, prophylactic and nutritional properties. All parts of this plant, i.e. berries (including the seeds), leaves, twig, bark and root, are sources of a large number of bioactive compounds with antioxidant, anti-inflammatory, anti-cancer, antimutagenic, antimicrobial, anti-stress, anti-atherogenic, anti-ulcerative, immunomodulatory, blood cholesterol-lowering, hepatoprotective, neuroprotective, cardioprotective, radioprotective, skin regenerating, wound- or burn-healing effects [1–16]. The berries are rich in carbohydrates, proteins, organic acids, essential fatty acids (FAs) and aminoacids, antioxidants (e.g. ascorbic acid, tocopherols, carotenoids, flavonoids), phytosterols and chemical elements (e.g. Ca, Mg, Na, P) [1, 2, 9, 10, 17].

Sea buckthorn berries are commonly processed by pressing, resulting in a juice and a pomace consisting of seeds, peel and residual pulp [1, 2, 9, 13, 14, 17, 18]. The pomace is usually dried in order to extend its shelf life for further use, either as a feed supplement or a source of valuable products, e.g. oils extracted from the seeds and the fraction of peel and pulp [2, 13–16, 18, 19]. Seeds as well as peel and pulp oils are valuable products with desirable properties for use as ingredients in functional food, nutraceuticals, dietary supplements, pharmaceuticals and cosmetics due to their high content of essential FAs and fat-soluble bioactive components, including tocopherols, carotenoids and phytosterols [2, 6, 9–11, 13–16, 18].

The pomace is generally dried by forced-air convection in a dehydrator or by lyophilization in a freeze-dryer [13, 14, 18]. Solvent extraction using hexane, petroleum ether or supercritical CO_2 are

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Tab. 1. Yield and composition of oils extracted from various fractions.

Yield/composition		Seeds	Peel and pulp	References
Oil extraction yield [%]		5–20	2–36	[1–3, 11, 14, 17]
Fatty acids [%]	α-Linolenic (C18:3n-3)	17–40	1–14	[1–3, 5, 11, 13, 14, 17, 18]
	Linoleic (C18:2n-6)	28–44	1–27	
	Oleic (C18:1n-9)	13–26	3–54	
	Palmitoleic (C16:1n-7)	0.2–10	11–54	
	Palmitic (C16:0)	7–14	17–47	
Tocopherols [g·kg^{-1}]		1.40–4.21	0.95–2.21	[1–3, 11, 13, 18]
Carotenoids [g·kg^{-1}]		0.04–2.50	1.18–10	[2, 3, 13, 15, 16, 18]
Phytosterols [g·kg^{-1}]		6.60–23	5.20–29	[3, 6, 15, 16, 18]

Fatty acids are expressed as percentage (w/w) of total fatty acids. Content of tocopherols, carotenoids and phytosterols is expressed per kilogram of oil.

the most common methods used to extract the oil from dried seeds as well as peel and pulp fraction [6, 13, 17, 18, 20]. The yield, composition and properties of seed as well as peel and pulp oils heavily depend on: (i) sea buckthorn subspecies, origin and harvesting time, (ii) pre-treatment before drying, (iii) drying technique and conditions, (iv) characteristic parameters of solvent extraction (solvent type, solid-to-liquid ratio, size of dried plant material, extraction time and temperature).

Tab. 1 presents data reported in literature on the yield and composition of seed as well as peel and pulp oils. Seed oil contains a large percentage of ω-3 and ω-6 polyunsaturated FAs (PUFAs), i.e. 17–40 % α-linolenic (C18:3n-3) and 28–44 % linoleic (C18:2n-6) acids, as well as ω-6/ω-3 ratio lower than 2, whereas peel and pulp oil is rich in saturated and monounsaturated FAs (MUFAs), i.e. 17–47 % palmitic (C16:0) and 11–54 % palmitoleic (C16:1n-7) acids [1–3, 5, 11, 13, 14, 17, 18]. Due to its high content of α-linolenic ω-3 fatty acid and low ω-6/ω-3 ratio, sea buckthorn seed oil can be used as an ingredient of pharmaceuticals, nutraceuticals and dietary supplements with cardioprotective, neuroprotective, blood cholesterol-lowering, anti-inflamatory and anti-cancer effects [1, 2, 10, 11, 13, 14, 18]. Antioxidant, anti-atherogenic, anti-ulcerative, antimutagenic, hepatoprotective, sun protective, skin regenerating, wound- and burn-healing effects of seed oil have been also reported [1, 2, 10, 13].

This study aimed at the effects of the pre-treatment procedure and air-drying conditions on the drying kinetics as well as on the yield, extraction efficiency and FA composition of sea buckthorn seed oil. Sea buckthorn pomace was pre-treated by sonication or freezing and, further, a thin layer of pre-treated or untreated pomace was dried at different values of layer thickness (3 mm, 10 mm) and temperature (40 °C, 50 °C). The seeds separated from dried pomace were ground and then subjected to batch extraction using hexane as a solvent. FA composition of the oil extracted from sea buckthorn seeds was determined by gas chromatography (GC).

MATERIALS AND METHODS

Plant material and cold pressing

Sea buckthorn (*Hippophae rhamnoides* L. ssp. *carpatica*) wild berries collected in Prahova region (Romania) in October–November 2016 were used as the plant material. They were manually cleaned, washed and then stored at (4 ± 1) °C until their pressing. Sea buckthorn juice was extracted from the berries by cold pressing (240 W power, 1 Hz speed) using a Kuvings-Slow juicer (NUC Electronics, Daegu, South Korea), obtaining a pomace consisting of seeds together with peel and pulp fraction as a by-product.

Pomace pre-treatment

Sea buckthorn pomace was divided into 3 portions. The first was pre-treated by sonication, the second by freezing at (–20 ± 2) °C for 24 h and the third was untreated. Before the pre-treatment by sonication, the pomace was mixed with water at a mass ratio of 1 : 5, then the mixture was subjected to ultrasound in an Ultrasonic Processor VCX 500 (500 W, 20 kHz; Sonics and Materials, Newtown, Connecticut, USA) for 5 min at 20% amplitude. The sonicated pomace was separated by vacuum filtration using a Duran Büchner funnel (Merck, Darmstadt, Germany).

Air drying of untreated and pre-treated pomace

Thin layer drying of sea buckthorn pomace was

performed using a Tribest Sedona Express SDE-P6280 food dehydrator (Tribest, Anaheim, California, USA). Air drying experiments of untreated, sonicated, and frozen pomaces were conducted at different levels of layer thickness (3 mm, 10 mm) and process temperature (40 °C, 50 °C) until the equilibrium state was attained.

The moisture content (X) of sea buckthorn pomace, expressed as kilograms per kilogram of dry material, was made dimensionless using Eq. 1

$$MR = \frac{X - X_e}{X_0 - X_e} \quad (1)$$

where MR represents the moisture ratio, X_0 and X_e are initial and equilibrium moisture contents, respectively.

After drying, the seeds were manually separated from dried peel and pulp fraction and then stored at (4 ± 1) °C in sealed plastic bags until the beginning of solvent extraction experiments. Initial and equilibrium moisture contents of sea buckthorn pomace and seeds were determined based on measurements performed using a MB23-Ohaus thermobalance (Ohaus, Parsippany, New Jersey, USA).

Solvent extraction of seed oil

A food grinder (Krups, Solingen, Germany) was used to reduce the size of sea buckthorn seeds to a medium dimension of (0.6 ± 0.2) mm. The oil was extracted from ground seeds using *n*-hexane (Merck) of analytical reagent grade as a solvent.

Amounts of 10 g of ground seeds were subjected to batch extraction with 150 ml *n*-hexane at 60 °C for 4 h in a glass vessel [21, 22]. Then, the mixture was filtered under vacuum using a Duran Büchner funnel, the solvent was distilled at low pressure, the extracted oil was dried until a constant weight and stored at (4 ± 1) °C until GC analysis. The maximum oil content of sea buckthorn seeds was determined by Soxhlet extraction. An amount of 10 g of ground seeds was subjected to extraction with 150 ml *n*-hexane in a conventional Soxhlet apparatus for 8 h [23–25]. Oil yield (Y) and extraction efficiency (E) were calculated using Eq. 2 and Eq. 3

$$Y = \frac{M_O}{M_S} \times 100 \quad (2)$$

$$E = \frac{M_O}{M_{Omax}} \times 100 \quad (3)$$

where M_S is the mass of dried seeds, M_O the mass of extracted oil, and M_{Omax} the maximum mass of extracted oil obtained by Soxhlet extraction.

The morphology of dried seeds before and after solvent extraction was inspected by FEI Quanta Inspect F scanning electron microscope (Thermo Fisher Scientific, Waltham, Massachusetts, USA). All samples were gold-coated prior to microscopic examination.

Analysis of fatty acid composition of seed oil

FA methyl esters (FAME) were prepared from sea buckthorn seed oil by transesterification with methanol, using 14% boron trifluoride-methanol solution as a catalyst, following a standard protocol [26]. Supelco 37 Component FAME Mix and dichloromethane (HPLC purity grade) were purchased from Merck, methanol (HPLC isocratic grade) was from J. T. Baker (Center Valley, Pennsylvania, USA) and boron trifluoride-methanol complex was from Alfa Aesar (Haverhill, Massachusetts, USA). An Agilent 7890B gas chromatograph (Agilent Technologies, Santa Clara, California, USA) equipped with auto sampler, 5975 C VL MSD triple-axis MS detector was used, with a Supelco SP 2560 capillary column (100 m length, 0.25 mm inner diameter, 0.2 μm film thickness; Sigma-Aldrich, St. Louis, Missouri, USA). Helium (1.0 ml·min^{-1}) was the carrier gas and a split ratio of 1 : 100 was used. The oven temperature was programmed as follows: 140 °C (5 min), 140–240 °C (4 °C·min^{-1}), and 240 °C (20 min). Volumes of 1 µl of FAME solutions (FAME/dichloromethane ratio of 1 : 99 v/v) were injected into the column and FAs were identified by comparing the retention times corresponding to the peaks with those of a certified standard mixture (Supelco 37 Component FAME Mix).

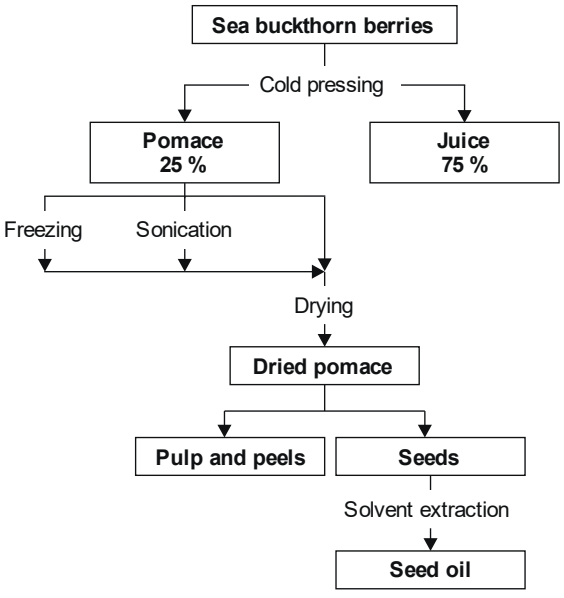

Fig. 1. Steps of processing sea buckthorn berries.

RESULTS AND DISCUSSION

A mean mass percentage of 25 % of sea buckthorn pomace was obtained by cold pressing. Untreated and pre-treated pomaces were dried under various conditions. The seeds separated from dried pomace were ground and then subjected to batch extraction. Seed oil was produced from sea buckthorn berries according to the steps presented in Fig. 1. The effects of the pre-treatment procedure and air drying conditions on the drying kinetics, extraction performance in terms of oil yield and extraction efficiency, and FA composition of sea buckthorn seed oil were analysed.

Air drying of untreated and pre-treated pomaces

Time variation of dimensionless moisture ratio (*MR*) for untreated, sonicated and frozen pomaces during drying under different conditions is presented in Fig. 2. The depicted data highlight a decrease in *MR* and in the final drying time (τ_f; 5–13 h) with a decrease in the layer thickness (L), and an increase in the process temperature (t) for untreated and pre-treated pomaces, the effect of L being significant. Moreover, the values of *MR* and τ_f for the same values of L and t were larger for frozen than for untreated pomace.

Experimental results presented in Fig. 3 and Fig. 4, expressing the variation of drying rate (r in kilograms per kilogram of dry mater per hour) defined by Eq. 4, depending on time (τ) and moisture ratio (*MR*), respectively, highlight a falling rate period, indicating that the drying process was controlled by the internal mass transfer. Moreover, a linear dependence (R^2 of 0.979–0.996) between r and *MR* was revealed by data depicted in Fig. 4, r being defined as follows:

$$r = \frac{X_{\tau+\Delta\tau} - X_\tau}{\Delta\tau} \quad (4)$$

Drying dynamics of sea buckthorn pomace were predicted using Fick's second law given by Eq. 5

$$\frac{\partial X}{\partial \tau} = \nabla \cdot (D_{\text{eff}} \nabla X) \quad (5)$$

where D_{eff} (in square metres per second) is the effective diffusivity and τ is time (in seconds).

In order to solve Eq. 5, the following simplifying assumptions were considered [27–30]: (i) the layer of plant material is an infinite flat plate with a very low thickness (L); (ii) material layer volume and temperature, as well as air relative humidity and temperature, are constant; (iii) initial moisture content is uniformly distributed; (iv) external resistance to mass and heat transfer is negligible; (v) heat transfer is more rapid than mass transfer; (vi) mass transport is unidirectional (Ox) and symmetric with respect to the centre of the material layer; (vii) D_{eff} is constant.

Accordingly, the solution of Eq. 6, which represents the unidirectional (Ox) form of Eq. 5, with initial and boundary conditions expressed by Eqs. 7–9, is given by Eq. 10 [31].

$$\frac{\partial X}{\partial \tau} = D_{\text{eff}} \frac{\partial^2 \text{X}}{\partial \text{x}^2} \quad (6)$$

$$\tau = 0: \quad X = X_0 \quad (7)$$

$$x = 0: \quad \frac{\partial X}{\partial x} = 0 \quad (8)$$

$$x = \frac{L}{2}: \quad X = X_e \quad (9)$$

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left[-\frac{(2n+1)^2 \pi^2}{L^2} D_{\text{eff}} \tau\right] \quad (10)$$

For a long drying time, only the first term ($n = 0$) of series given by Eq. 10 is significant resulting in Eq. 11.

$$MR = \frac{8}{\pi^2} \exp\left[-\frac{\pi^2}{L^2} D_{\text{eff}} \tau\right] \quad (11)$$

Eq. 12 was further obtained by taking natural logarithms on both sides of Eq. 11.

$$\ln MR = \ln\left(\frac{8}{\pi^2}\right) - \frac{\pi^2}{L^2} D_{\text{eff}} \tau = \ln\left(\frac{8}{\pi^2}\right) - k\tau \quad (12)$$

Values of D_{eff} ($(0.141–1.484) \times 10^{-9}$ m^2·s^{-1}), which were determined from the slope (k) of the straight line given by plotting ln*MR* vs τ (Fig. 5), are within the range that is generally reported for air drying of a thin layer of food materials, i.e. 10^{-11}–10^{-9} m^2·s^{-1} [29, 30, 32, 33]. Results summarized in Tab. 2 emphasize lower values of D_{eff} for inferior levels of thickness L (from 4.0 to 7.1 times) and drying temperature t (from 1.2 to 1.4 times) as well as for frozen pomace (up to 1.4 times). Moreover, data presented in Fig. 5 and Tab. 2 indicate a good agreement between experimental and predicted values of *MR* (R^2 of 0.983–0.999). Tab. 2 contains also values of mean drying rate (r_m; 0.144–0.354 kg·kg^{-1}·h^{-1}) defined by Eq. 13, where X_0 is 1.45–1.98 and X_e is 0.04–0.13. The tabulated results highlight lower values of r_m for thicker layers (up to 2.4 times), unsonicated and frozen pomace (up to 2 times) as well as a negligible effect of drying temperature.

$$r_m = \frac{X_0 - X_e}{\tau_f} \quad (13)$$

Regression equations were obtained between the drying performance, i.e. D_{eff} and r_m, and the process factors in terms of drying temperature

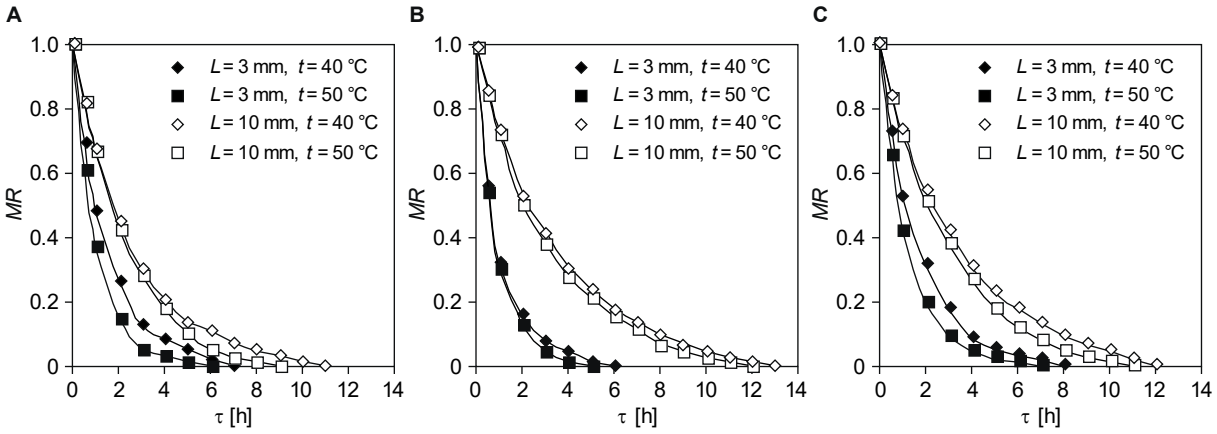

Fig. 2. Variation of pomace moisture ratio over time during drying under various conditions.

A – untreated pomace, B – sonicated pomace, C – frozen pomace.
MR – moisture ratio, τ – time, *L* – pomace layer thickness, *t* – drying temperature.

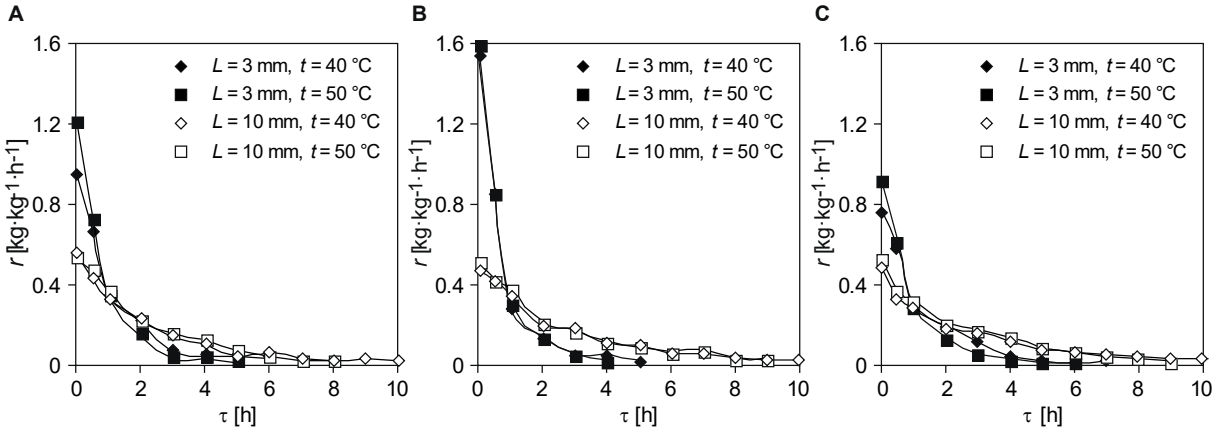

Fig. 3. Variation of pomace drying rate over time under various conditions.

A – untreated pomace, B – sonicated pomace, C – frozen pomace.
r – drying rate, τ – time, *L* – pomace layer thickness, *t* – drying temperature.

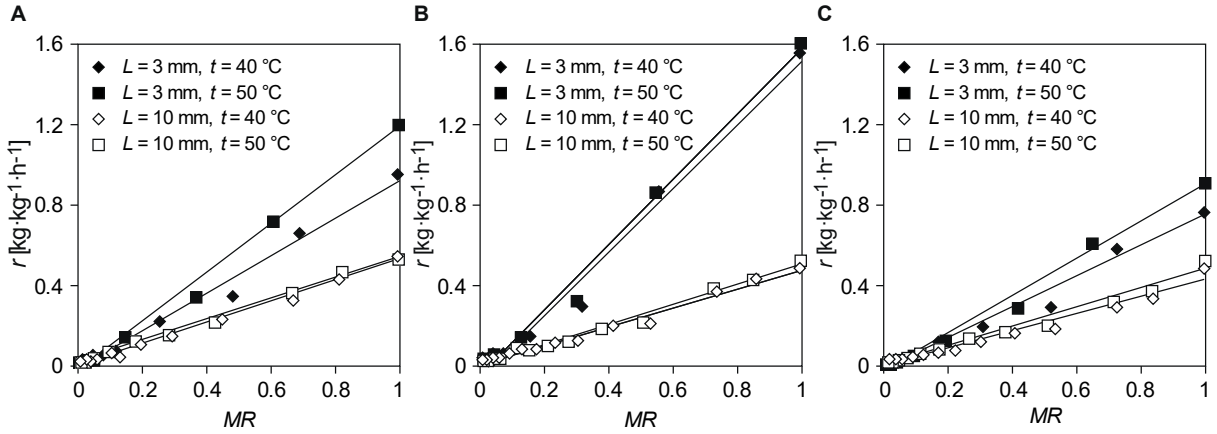

Fig. 4. Variation of pomace drying rate with moisture ratio under various conditions.

A – untreated pomace, B – sonicated pomace, C – frozen pomace.
r – drying rate, *MR* – moisture ratio, *L* – pomace layer thickness, *t* – drying temperature.
Straight lines represent the dependence calculated by Eq. 4.

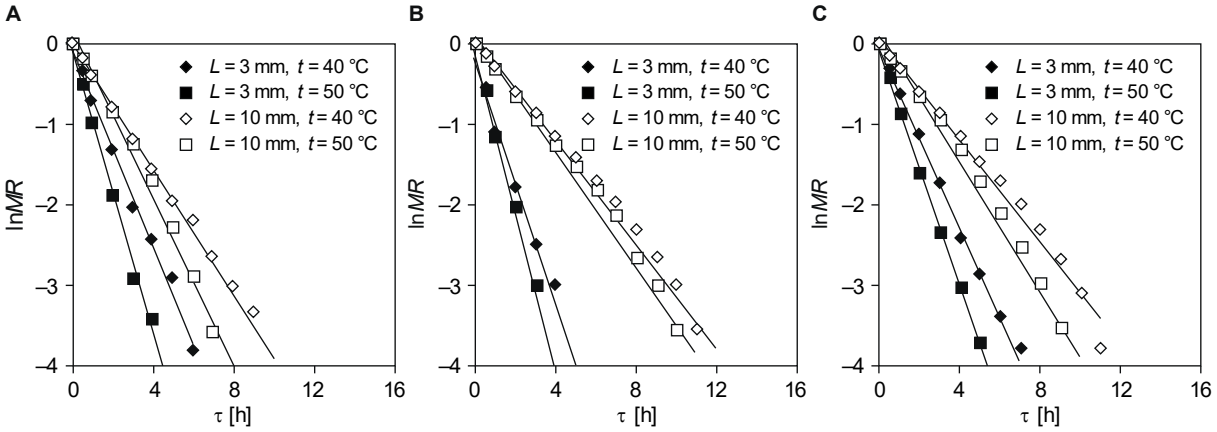

Fig. 5. Variation of natural logarithm of pomace moisture ratio over time during drying under various conditions.

A – untreated pomace, B – sonicated pomace, C – frozen pomace.
MR – moisture ratio, τ – time, *L* – pomace layer thickness, *t* – drying temperature.
Straight lines represent the dependence calculated by Eq. 12.

Tab. 2. Experimental values of drying performance under various process conditions.

No.	Pre-treatment	*t* [°C]	*L* [mm]	D_{eff} [$\times 10^9$ m^2·s^{-1}]	R^2	r_m [kg·kg^{-1}·h^{-1}]
1	No pre-treatment	40	3	0.156	0.993	0.222
2		50	3	0.226	0.995	0.256
3		40	10	1.115	0.991	0.135
4		50	10	1.484	0.988	0.167
5	Sonication	40	3	0.195	0.989	0.295
6		50	3	0.255	0.998	0.354
7		40	10	0.869	0.983	0.134
8		50	10	1.021	0.983	0.145
9	Freezing	40	3	0.141	0.997	0.174
10		50	3	0.190	0.999	0.176
11		40	10	0.850	0.988	0.127
12		50	10	1.043	0.987	0.144

t – drying temperature, *L* – pomace layer thickness, D_{eff} – effective diffusivitiy, r_m – mean drying rate, R^2 – determination coefficient.

Tab. 3. Drying performance depending on dimensionless process factors.

No.	Pre-treatment	x_1	x_2	x_3	x_4	D_{eff} [$\times 10^9$ m^2·s^{-1}]		r_m [kg·kg^{-1}·h^{-1}]	
						Experimental	Predicted	Experimental	Predicted
1	No pre-treatment	–1	–1	–1	1	0.156	0.161	0.222	0.219
2		1	–1	–1	1	0.226	0.221	0.256	0.259
3		–1	1	–1	1	1.115	1.181	0.135	0.137
4		1	1	–1	1	1.484	1.419	0.167	0.165
5	Sonication	–1	–1	1	1	0.195	0.195	0.295	0.305
6		1	–1	1	1	0.255	0.255	0.354	0.344
7		–1	1	1	1	0.869	0.826	0.134	0.125
8		1	1	1	1	1.021	1.064	0.145	0.154
9	Freezing	–1	–1	–1	–1	0.141	0.136	0.174	0.167
10		1	–1	–1	–1	0.190	0.195	0.176	0.183
11		–1	1	–1	–1	0.850	0.828	0.127	0.134
12		1	1	–1	–1	1.043	1.066	0.144	0.137

x_1 – dimensionless temperature, x_2 – dimensionless layer thickness, x_3 – dimensionless ultrasound amplitude, x_4 – dimensionless pre-treatment temperature, D_{eff} – effective diffusivitiy, r_m – mean drying rate.

(*t;* 40 °C, 50 °C), pomace layer thickness (*L;* 3 mm, 10 mm), ultrasound amplitude (*A;* 0 %, 20 %), and pre-treatment temperature (t_p; –20 °C, 20 °C). Dimensionless values of process factors (x_1, x_2, x_3, and x_4), which were determined by Eqs. 14–17, are summarized in Tab. 3.

$$x_1 = \frac{t - 45}{5} \quad (14)$$

$$x_2 = \frac{L - 6.5}{3.5} \quad (15)$$

$$x_3 = \frac{A - 10}{10} \quad (16)$$

$$x_4 = \frac{t_p}{20} \quad (17)$$

Experimental data were processed by multiple regression analysis [34], resulting in Eq. 18 and Eq. 19.

$$D_{eff} = (0.571 + 0.074x_1 + 0.375x_2 - \\ - 0.080x_3 + 0.095x_4 - 0.097x_2x_3 + \\ + 0.082x_2x_4) \times 10^{-9} \quad (18)$$

$$r_m = 0.194 - 0.056x_2 + 0.019x_3 + \\ + 0.020x_4 - 0.024x_2x_3 \quad (19)$$

These two equations highlight the following issues: (i) values of D_{eff} are larger for high levels of x_1 (drying temperature), x_2 (layer thickness), x_4 (pre-treatment temperature) and x_2x_4 interaction, as well as for low levels of x_3 (ultrasound amplitude) and x_2x_3 interaction; (ii) values of r_m are larger for high levels of x_3 and x_4, as well as for low levels of x_2 and x_2x_3 interaction; (iii) x_2 factor has the most significant effect on D_{eff} and r_m.

Values of drying performance predicted by Eq. 18 and Eq. 19 as well as those determined from experimental data using Eq. 12 and Eq. 13 are presented in Tab. 3. Tabulated results revealed a very good agreement between experimental and predicted values of effective diffusivity (root mean square error, *RMSE* of 0.0031 $m^2 \cdot s^{-1}$) and mean drying rate (*RMSE* of 0.0004 $kg \cdot kg^{-1} \cdot h^{-1}$).

Yield, extraction efficiency and fatty acid composition of seed oil

The yield, extraction efficiency and FA composition of oil obtained from sea buckthorn seeds separated from untreated or pre-treated pomaces dried at 50 °C are summarized in Tab. 4. The results corresponding to the pomaces dried at 40 °C were similar (data not shown).

Tabulated values of seed oil yield (*Y*; 13.1–14.5 %) and extraction efficiency (*E*; 90.5–93.5 %) were within the ranges reported in literature [1–3, 11, 14, 17]. In order to determine the effect of process factors on *Y* and *E*, experimental data were processed by multiple linear regression analysis [34], resulting in Eq. 20 and Eq. 21.

$$Y = 13.83 + 0.517x_2 + 0.150x_3 - 0.175x_4 \quad (20)$$

$$E = 92.75 + 0.767x_2 + 0.525x_3 - 0.625x_4 \quad (21)$$

These regression equations highlight superior extraction performance for the oil extracted from seeds separated from pre-treated pomaces (larger values of x_3 and lower values of x_4) and from those corresponding to a thicker layer (higher levels of x_2), the effect of x_2 (pomace layer thickness) factor being significant.

FA composition of oil samples, data on which are presented in Tab. 4 in a format of mass per-

Tab. 4. Yield, extraction efficiency and fatty acid mass percentage of seed oil.

Pre-treatment		No pre-treatment		Ultrasonication (A = 20 %)		Freezing (t_p = –20 °C)		Mean value [%]	Standard deviation [%]
Pomace layer thickness *L* [mm]		3	10	3	10	3	10		
Oil yield *Y* [%]		13.1	13.9	13.3	14.3	13.2	14.5	13.7	0.6
Extraction efficiency *E* [%]		90.5	92.7	92.1	93.2	92.2	93.5	92.4	1.1
Fatty acids [%]	Palmitic (C16:0)	8.0	8.0	8.0	7.9	7.7	7.7	7.9	0.1
	Palmitoleic (C16:1n-7)	0.9	1.0	0.9	0.9	1.0	0.9	0.9	0.1
	Stearic (C18:0)	2.8	2.7	2.8	2.8	2.9	2.9	2.8	0.1
	Oleic (C18:1n-9)	22.6	22.6	22.7	22.8	22.7	22.8	22.7	0.1
	Linoleic (C18:2n-6)	38.9	39.2	38.9	38.6	39.1	38.9	38.9	0.2
	α-Linolenic (C18:3n-3)	26.9	26.5	26.9	27.1	26.6	26.8	26.8	0.2
	MUFAs	23.5	23.6	23.6	23.7	23.7	23.7	23.6	0.1
	PUFAs	65.8	65.7	65.8	65.7	65.7	65.7	65.7	0.1

A drying temperature of 50 °C was applied.
A – ultrasonication amplitude, t_p – pre-treatment temperature, MUFA – monounsaturated fatty acids, PUFA – polyunsaturated fatty acids.

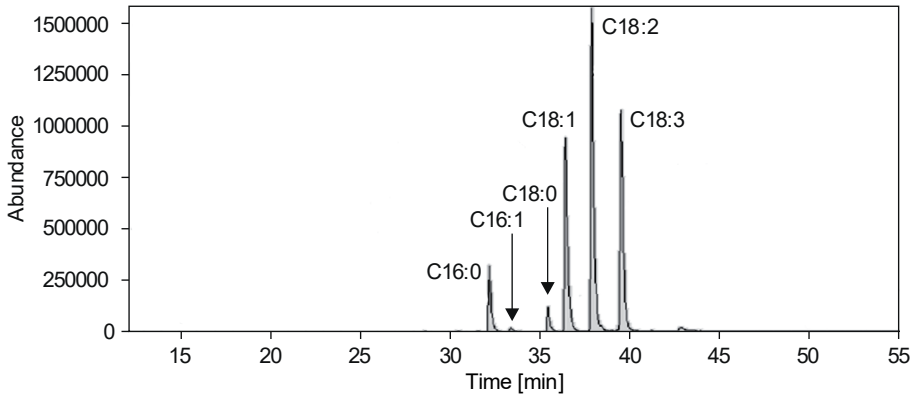

Fig. 6. Chromatogram of fatty acids extracted from dried sea buckthorn seeds.

Drying conditions: untreated pomace, drying temperature t = 50 °C, pomace layer thickness L = 10 mm.

centages of the total FA content, demonstrate a negligible effect of process factors. Tabulated data reveal a high mass percentage of PUFAs (65.7 %, i.e. 26.8 % α-linolenic and 38.9 % linoleic acids), a ω-6/ω-3 ratio of 1.4, 23.6 % MUFAs and 10.7 % saturated fatty acids. These findings are consistent with those reported by other studies referring to the composition of *Hippophaae rhamnoides* L. ssp. *caucasia* [3, 11]. A chromatogram of FAs extracted from sea buckthorn seeds is given in Fig. 6.

Experimental and predicted values of extraction performance, which are presented in Tab. 5, are in a very good agreement (*RMSE* less than 0.1 %).

Scanning electron micrographs of dried seeds before and after solvent extraction are shown in Fig. 7. The surface of a seed sample that had not

Tab. 5. Oil extraction performance depending on dimensionless process factors.

No.	x_1	x_2	x_3	x_4	Oil yield Y [%]		Extraction efficiency E [%]	
					Experimental	Predicted	Experimental	Predicted
2	1	–1	–1	1	13.1	13.0	90.5	90.8
4	1	1	–1	1	13.9	14.0	92.7	92.4
6	1	–1	1	1	13.3	13.3	92.1	91.9
8	1	1	1	1	14.3	14.3	93.2	93.4
10	1	–1	–1	–1	13.2	13.3	92.2	92.1
12	1	1	–1	–1	14.5	14.4	93.5	93.6

x_1 – dimensionless temperature, x_2 – dimensionless layer thickness, x_3 – dimensionless ultrasound amplitude, x_4 – dimensionless pre-treatment temperature.

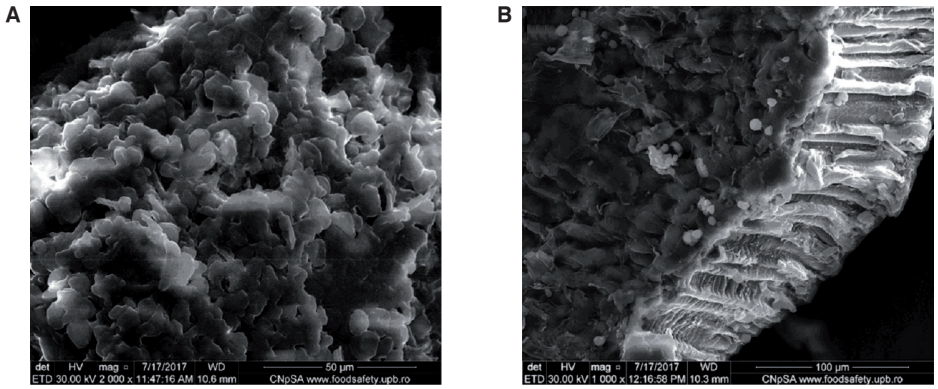

Fig. 7. Scanning electron microscopic images of dried sea buckthorn seeds.

A – before solvent extraction, B – after solvent extraction.
Drying conditions: untreated pomace, drying temperature t = 50 °C, pomace layer thickness L = 10 mm.

undergone oil extraction was rough and intact oil cells could be observed (Fig. 7a). After extraction (Fig. 7b), there were many free cells from which oil had been released to the extraction solvent as well as some intact oil cells.

CONCLUSIONS

The influence of the pre-treatment procedure and drying conditions of sea buckthorn pomace on the performance of air drying and seed oil extraction as well as on the oil composition was evaluated. Sea buckthorn pomace was pre-treated by sonication or freezing and further a thin layer of pre-treated or untreated pomace was dried under various conditions. Drying temperature (40 °C and 50 °C), pomace layer thickness (3 mm and 10 mm), ultrasound amplitude (0 % and 20 %) and pre-treatment temperature (–20 °C and 20 °C) were selected as process factors. Their effects on the drying and extraction performance were found to be as follows: (i) effective diffusivity values were higher, $(0.141–1.484)\times10^{-9}$ $m^2 \cdot s^{-1}$, for superior levels of layer thickness, higher drying temperature and higher pre-treatment temperature; (ii) mean drying rate (0.144–0.354 $kg \cdot kg^{-1} \cdot h^{-1}$) was not affected by drying temperature and its values were lower for thicker layers, unsonicated and frozen pomace; (iii) higher levels of oil yield (13.1–14.5 %) and extraction efficiency (90.5–93.5 %) were determined for oil extracted from seeds separated from pre-treated pomaces and from those corresponding to a thicker layer.

Drying performance and extraction performance were correlated with the process factors by multiple regression equations, which revealed a significant influence of pomace layer thickness. FA composition of the oil extracted from sea buckthorn seeds separated from untreated and pre-treated pomaces dried at 50 °C was similar. A high percentage of PUFAs (65.7 %) and a low value of ω-6/ω-3 ratio (1.4) of sea buckthorn seed oil were determined under the conditions considered in the experimental studies.

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