SEABUCKTHORN OIL EXTRACTION, A MODEL FOR SOLID-LIQUID EXTRACTION PROCESS

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The study intends to establish the equilibrium data concerning the oil extraction from seabuckthorn berries powder. A laboratory Soxhlet apparatus and n-hexane as solvent were used. The oil concentration and carotene derivative contents were determined by UV-VIS spectrophotometry in the extract and in the extracted solid in order to obtain the equilibrium data.

Keywords: solid-liquid cocurrent multiple extraction, equilibrium data, Soxhlet, seabuckthorn oil

1. Introduction

Seabuckthorn (Hippophae rhamnoides) a thorny bush plant, grown in Europe and Asia is considered in the last decades, a real hope for improving the human health, due to its nourishing, vitaminic, revitalising and even healing action.

A large variety of natural compounds, salutary for human beings, are present in seabuckthorn berries, seeds, leaves, and even in its branches. Recent reviews [1-2] underline the importance of seabuckthorn oil obtained from the berries or seeds, very rich in polyunsaturated fatty acids (PUFA, ω3 and ω6), carotene derivatives, tocopherols, tocotrienols and sterols, besides various minerals (Ca, Mg, Zn, Se, I). The presence of these compounds in oil justifies its usage in prevention and treatment of present and severe disorders as: cardiovascular, dermatologic, nervous, geriatric, immunological, gastric, hepatic diseases, cancer and diabetes [3-6].

Object of numerous and present studies, the technologies for obtaining seabucthorn oil imply extraction proceedings, using aqueous or nonaqueous solvents, supercritical fluid extraction with CO_2, and the screw pressing of berries [11].

From the comparative study of the technologies, the extraction using non aqueous hydrocarbonated solvents is considered to be superior as quality of the obtained oil [7].

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In connection to these, the present work studies the seabuckthorn berries extraction using a hydrocarbonated solvent, n-hexane, selected based on the criterions mentioned in academic treaties [8-11] and recent works [12].

It was assumed that in the industrial extractors working in fix-bed contacting, as in a laboratory Soxhlet apparatus, the extraction process can be equivalent to a multiple cocurrent extraction process, represented in fig. 1.

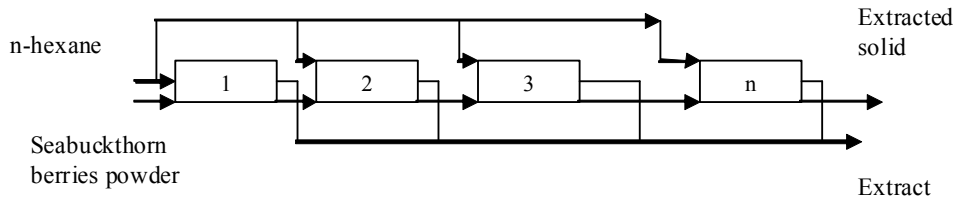

Fig. 1. A multiple cocurrent extraction process

In the fix-bed extractors working at the boiling temperature of the solvent, as well as in the Soxhlet apparatus, the solid particles are several times contacted with fresh solvent, in each stage of operation, and it can be assimilated to a multiple cocurrent extraction. That is why the equilibrium data concerning the oil extraction from seabuckthorn berries powder were established on a laboratory Soxhlet apparatus.

Given to the experimental difficulties, in order to appreciate the oil quantity after each stage of operation in the extract and in extracted solid as well, a control parameter was the carotene content (expressed in β-carotene), easy to be determined by UV-VIS spectrophotometry.

The obtained equilibrium data can be used in the extraction calculation using graphic or analytical methods presented in the available literature [10; 13-15].

2. Experimental part

Materials and methods

n-Hexane (Sigma, Germany), β-carotene (Sigma, Germany) were used in experimental determination.

The seabuckthorn berries powder was obtained from fresh fruits harvested from organic cultures, and processed by drying at 40°C and milling to a Φ=1-2 mm.

The extraction was performed in a Soxhlet apparatus, represented in fig. 2.

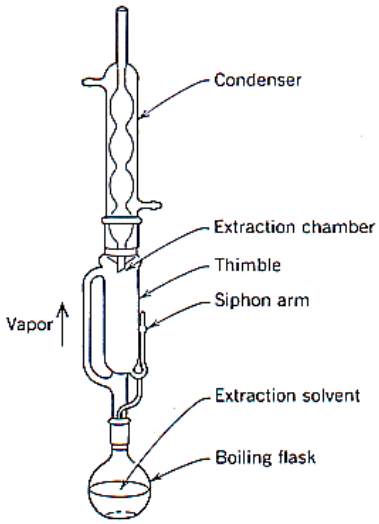

Fig. 2. Soxhlet apparatus

The extraction was carried out by inserting quantities of 20 g seabuckthorn powder in the cartridge of Soxhlet and pouring 400 ml n-hexane in the roundbottom flask. The temperature in the in extraction space was measured by introducing a thermometer in this space; thus it can be read through the glass wall of extractor. During the extraction, the temperature varied from 50 –60°C, in time of 30 minutes, necessary for a stage of operation. In this interval, the equilibrium data varied in a small extent [12]. It was assumed that these data are valuable for a mean temperature of 54°C.

The experimental determinations aim to:

1. Observe the increase of oil concentration and carotene content in hexane extracts and their decrease in the solid exposed to extraction on the account of rising the extraction time and number of operation stages.

2. Realize the equilibrium curve for oil extraction from seabuckthorn berries-powder with n-hexane in Soxhlet apparatus, at a medium temperature of 54°C, using a solvent: powder rate of 5:1 v/w.

A stable distribution between solid and liquid phase was verified by determination of the carotene content in the liquid surrounding the cartridge. The samples were taken by manual stirring, just before the first operation stage. It was established that the carotene content in the liquid remains constant after 10 minutes, and the equilibrium is achieved. The carotene contents of the extracts were determined using a JASCO 530 UV-VIS spectrophotometer, reading the solution absorbance at 460 nm. The results were expressed in β-carotene.

In order to build the equilibrium curve, the quantity of oil in the extracts and in extracted solid were also determined in every operation stage. Certain

powders of seabuckthorn berries enriched in exhausted powder, considered as inert solid, were extracted.

A gravimetric method was used in order to establish the oil content in the extracts and solid extract. The primary extracts and those obtained from the previously extracted solids were evaporated to dry state, using an Rotavapor, and then they were weighted.

3. Results and discussions

The increase of the oil concentration and the carotene content and the decrease of these values in the extracted solid with the time of extraction and the rising of the number of operation stagesare presented in the table 1, as in the fig. 3 and 4.

Table 1

The variation of oil concentration and carotene content in the extracts and extracted solids, versus time of extraction and number of operation stages

Nr. of operation stages	0	1	2	3	4	5	6
Extraction time [min]	0	30	60	95	130	160	195
Mass (mg carotene/100g extract)	0.00	5.64	12.70	15.00	16.30	16.70	16.30
% oil in extract	0.00	0.34	0.77	0.92	0.95	1.00	1.02
Mass (mg carotene/100g solid)	41.40	4.27	3.45	3.38	3.07	2.52	1.44
% oil in solid	3.56	0.36	0.30	0.29	0.26	0.21	0.12

From Table 1, an increase of oil and carotene derivatives in the extract, and a decrease of these values in the extracted solid with extraction time can be observed. Figs. 3, 4 present these observations.

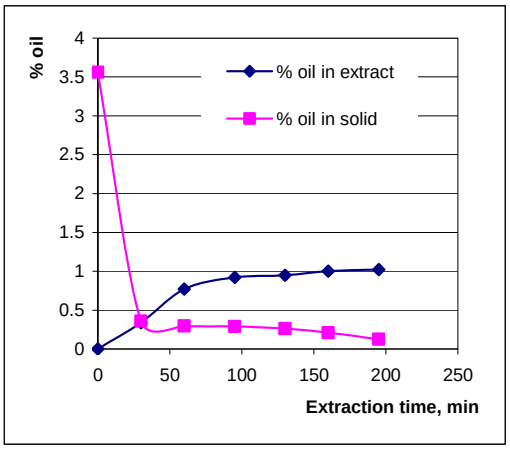

Fig. 3. Variation of the oil concentration in the extract and in the extracted solid vs. the extraction time of the seabuckthorn berries using n-hexane at 54°C

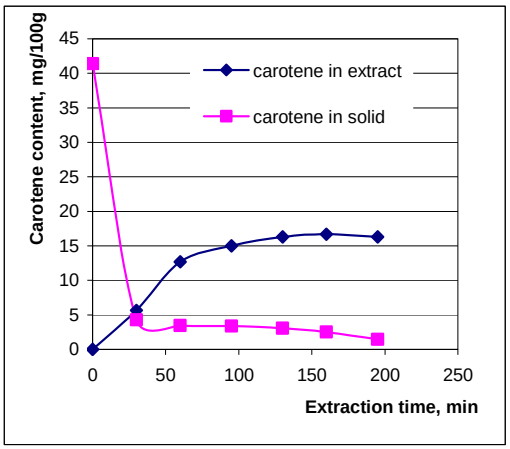

Fig. 4. Variation of carotenes concentration in the extract and in the remaining solid vs. the extraction time of seabuckthorn berries using n-hexane at 54°C

A remarkable similitude can be noticed between plots 3 and 4, both emphasizing that the 4 operation stages, and 2 hours are enough for an efficient extraction. The extension of the extraction time is not justified, as long as oil and carotenes concentration in the extract and in the solid remain invariable.

Table 2 and 3 and Figs. 5, 6 organize the determinations realized to establish the equilibrium curve for the extraction of seabuckthorn berries using n-hexane in a Soxhlet apparatus at a medium temperature of 54°C.

Table 2

Mass balance for seabuckthorn oil extraction from berries using n-hexane at 54°C

	ENTERED				ISSUED							
	Oil		Carotene		Oil in				Carotene in			
	g	%	mg	%	extract		extracted solid		extract		extracted solid	
					g	%	g	%	mg	%	mg	%
IV	1.17	11.70	2.10	2.00	0.81	0.38	0.36	3.75	1.45	0.67	0.65	6.70
III	0.58	5.80	1.04	10.40	0.40	0.21	0.18	1.90	0.72	0.39	0.32	3.40
II	0.29	2.90	3.20	3.20	0.20	0.11	0.09	0.91	0.36	0.20	0.16	1.67
I	0.18	1.75	0.31	0.31	0.12	0.06	0.06	0.58	0.21	0.12	0.10	1.04

Table 3

Equilibrium data for seabuckthorn oil extraction from berries using n-hexane at 54°C

	0	I	II	III	IV
Oil [g] / 100ml extract	0.00	0.06	0.11	0.21	0.38
Carotene [mg] / 100 ml extract	0.00	0.12	0.20	0.39	0.67
Retained solution [g]/insoluble solid [g]	1.30	1.36	1.38	1.45	1.58

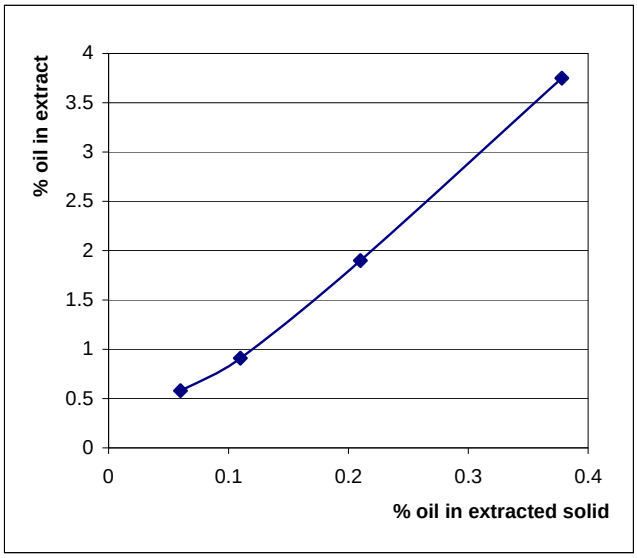

Fig. 5. Plot of oil percent in extract vs. oil percent in the extracted solid

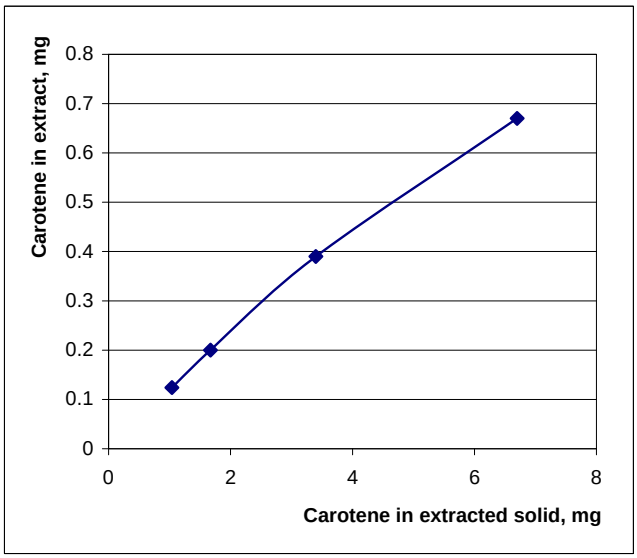

Fig. 6. Plot of carotene [mg]/100 ml extract vs. carotene [mg]/ 100 g extracted solid

In order to obtain a graphical solution of the extraction, the functions N_x - as a mass report of the solid in the extract vs. "x" mass report of the oil in the extract (having no solid) - and N_y - as the mass report of the insoluble solid in the restrained extract vs. "y" mass report of the oil in the restrained extract - were defined. The obtained data are presented in the Table 4.

Table 4

Equilibrium data in the seabuckthorn oil extraction from berries (n-hexane, 54°C)

Nr. crt.	Mass report y = oil [g] / restrained extract [g]	Mass report $1/N_y$ = restrained extract [g] /insoluble solid [g]	Mass report N_y = insoluble solid [g]/ restrained extract [g]	Mass report N_x = insoluble solid in extract [g] /extract [g]*	Mass report x = oil [g] / extract (having no solid)[g]**
1	0	1.30	0.75	0.00	0
2	0.7×10^{-3}	1.36	0.73	0.00	0.7×10^{-3}
3	1.3×10^{-3}	1.38	0.72	0.00	1.3×10^{-3}
4	2.5×10^{-3}	1.45	0.68	0.00	2.5×10^{-3}
5	4.6×10^{-3}	1.58	0.69	0.00	4.6×10^{-3}

* the extract does not contain a solid;

** the total extract and the restrained extract have the same composition.

Fig. 7 represents the plots of N_x, N_y vs. x, y for the seabuckthorn oil extraction from berries using n-hexane at 54°C.

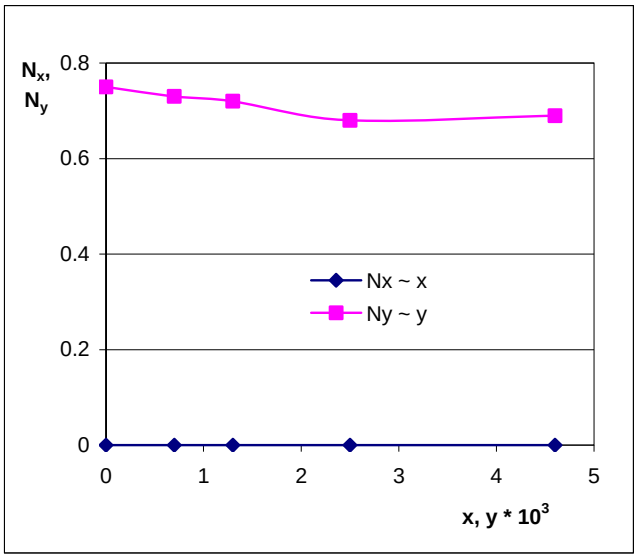

Fig.7. Plot of N_x , N_y vs. x,y for the extraction of seabuckthorn oil

6. Conclusions

Extraction of oil from seabuckthorn (Hyppophae rhamnoides) berries with n-hexane, at medium temperature of 54°C, was studied using a Soxhlet apparatus, as a model for a multiple cocurrent extraction.

The determinations aimed to establish the proper number of operation stages (the time of extraction) necessary for an efficient extraction, and to obtain the equilibrium data of the extraction.

The experiments implied the determination of the oil content in the extract, and in the extracted solid. The data were verified by establishing the carotene content by UV-VIS spectrophotometry.

It was proved that 4 operation stages or 2 hours of extraction are enough for an efficient extraction in the given working conditions.

Using seabuckthorn berry powder having various oil content in the first operation stage, the mass balance, and the equilibrium data, as well as the functions N_{xy} were represented in view of a graphic calculation of the extraction.

R E F E R E N C E S

[1] *Abdel-Salam, A.M.*, "Functional foods hopefulness to good health", Am. J. Food 5, 2010, pp. 86-99.

[2] *Raj – Kumar, G., Phani Kumar, O.P.*, "Phytochemical and Pharmacological Profile of Seabuckthorn oil – A Review", Res. Journ. Med. Plant, **5**, (5) 2011, pp. 491-499.

[3] *Zeb, A.*, "Important therapeutic uses of seabuckthorn (Hyppophae). A review", J. Biological, Sci., **4**, 2004, pp. 687-693.

[4] *Rongsen, L.*, "Hyppophae, and its general chemical composition, a resource for health, a challenge to modern technology", Proc. Cong. Int. Seabuckthorn Assoc. Germany, **4**, 2004, pp. 26-35.

[5] *Parimelazhagan, T., Chaurasia, O.P., Ahmed, Z.*, "Seabuckthorn oil with promising medicinal value", Curs. Sci., **88**, 2005, pp. 8-9.

[6] *Pelton, R.*, "Alternative in cancer therapy", Ed. Simon & Schuster New York, 2004.

[7] *Cenkowski, S., Yakimisnen, R., Przybylski, R., Muir, W.E.*, "Quality of extracted seabuckthorn seed and pulp oil", Canadian Biosystems Engineering, **48**, 2006, pp. 3.9-3.16.

[8] *Perry's* "Chemical Engineer's Handbook", Seventh edition, Mc. Grow Hill, 1997, pp. 18.55-18.59.

[9] *Kirk-Othmer*, "Encyclopedia of chemical technology", fourth edit., **10**, John Wiley & Sons New York, 1997.

[10] *Gamse, F.*, "Liquid-Liquid and Solid-Liquid extraction", Technische Universitat, Graz, 2002.

[11] *Dobre, T., Floarea, O.*, "Separarea produsilor chimici din produse naturale", Ed. MatrixROM, Bucuresti, 1997.

[12] *Sturzoiu, A., Stroescu, M., Dobre, T.*, "Betanine extraction from betavulgaris experimental research and statistical modeling", U.P.B. Sci. Bull Ser. **B 73**, Iss 1, 2011, pp. 145-156.

[13] *Schmighelski, O., Jinescu, G.J., Balaban,C., Dima, R., Vasilescu, P.*, "Modelarea procesului de extractie solid lichid", Rev. Chim. (Bucharest), **32**, (2), 1981, pp. 150-155.

[14] *** Enciclopedia de chimie, **Vol. 6**, Extracte vegetale, Ed. Stiintifica si Enciclopedica Bucuresti, 1989, pp. 475-481.

[15] *Bratu, E.*, "Operatii si utilaje in industria chimica", Editia a II, vol. II, Ed. Tehnica, Bucuresti, 1970.

[16] *Pavlov, C.F., Romancov, P,C., Noskov,A,A.*, "Procese si aparate in industria chimica. Exercitii si probleme" (traducere din limba rusa), Ed. Tehnica, Bucuresti, 1981.