Optimization of supercritical fluid extraction of essential oils and fatty acids from flaxweed (*Descurainia Sophia* L.) seed using response surface methodology and central composite design

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Abstract Essential oils and fatty acids of *Descurainia sophia* L. seed were obtained by supercritical CO₂ extraction and steam distillation methods. The effect of different parameters such as pressure, temperature, modifier volume, dynamic and static extraction time on the extraction yield were optimized using a central composite design after a 2ⁿ⁻¹ fractional factorial design. The results showed that under the pressure of 355 bar, temperature of 65 °C, methanol volume of 150 µL, dynamic and static extraction times of 35 and 10 min, respectively, the major components were methyl linoleate (18.2 %), camphor (12.32 %), cis-thujone (11.3 %) and trans-caryophyllene (9.17 %). The results indicated that by using the proper conditions, the supercritical fluid extraction is more selective than the steam distillation method. Extraction yields based on supercritical fluid extraction varied in the range of 0.68 to 17.1 % (w/w), and the extraction yield based on the steam distillation was 0.25 % (v/w).

Keywords Flaxweed (*Descurainia Sophia* L.) seed · Essential oil · Supercritical Fluid Extraction · Steam distillation · Experimental design · Response surface methodology

Introduction

Descurainia Sophia L. (Flaxweed) is a member of family Brassicaceae and one of the most annual dicot weeds and a prolific seeder (Li et al. 2010), which is native to Asia, South Africa, South America, southern Europe, and New Zealand

(Aghaabasi and Baghizadeh 2012). It's widely distributed in dominant regions of Iran especially in northeast regions. Flaxweed seed is very small and dark yellow or brown bearing an uneven surface in a stretched oval form, one end of which is cut and maintains a transparent yellowish ring (Amin 2000). Seeds have long been used as a traditional laxative, appetizer and stomach medicine (Fluck 1988). According to the Iranian traditional medicine, flaxweed is of a warm and moist nature and has been used for some of the medical properties such as antipyretic, laxative, ascarid excretion, alleviating of hoarseness, lightening complexion, promote urination, excretion of renal calculus, alleviation of skin inflammations such as hives, as well as appetizer and as a treatment for measles and scarlatina (Daryaei 2007; Mirhidar 1996). According to some reports the seeds can also be used in the treatment of some kinds of cancer but anti-tumor constituents in the seeds have not yet been explored thoroughly (Sun et al. 2005). As medicinal materials, this plant is very important for its chemical composition. Up to now various preparation techniques have been used to isolate and finally identify flaxweed seed constituents. Consequently, several phytochemical studies have identified the presence of cardiac glycosides and glucosinolate degradation products such as nitriles, isothiocyanates, and epithiobutane derivatives in its seeds and dried aerial parts of the plant (Chen et al. 1981), and some new lactones and aryldihydronaphthoic acid in the seeds were also reported (Wang et al. 2004; Sun et al. 2004, 2006; Afsharypour and Lockwood 1985).

Essential oils of plants have been usually isolated by either conventional distillation techniques (steam and hydro-distillation) or solvent extraction. The traditional methods for isolation and purification of chemical components from plants tissues, present some disadvantages. Overall, they require long extraction time, great solvent amounts, and sometimes have low efficiency. Furthermore, many natural

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products are thermally unstable and may degrade during thermal extraction or distillation (Vinatoru and Mason 1997; Vinatoru 2001). In steam distillation technique elevated temperatures and water can cause chemical modifications of essential oils. Steam distillation usually results in the loss of highly volatile components and some water-soluble constituents. Alternatively, when using solvent extraction it is virtually impossible to obtain a solvent-free product. Also existence of a few adjustable parameters for hydro-distillation, steam distillation and solvent extraction to control the selectivity of the process is another disadvantage of mentioned methods (Hawthorne 1990; Ondarzat and Sanchez 1990; Langenfeld et al. 1993; Ghasemi et al. 2011; Khajeh et al. 2010). Therefore, the necessity of desirable extraction technique with high efficiency and selectivity is advised. Recently, the application of supercritical fluid extraction (SFE) has been widely studied for extracting oil from plant materials (Daneshvand et al. 2012; Ara et al. 2014). Extraction processes using supercritical carbon dioxide are potential alternative methods for essential oil extraction to replace conventional extraction processes of expeller pressing and solvent extraction. Supercritical carbon dioxide not only has the higher extraction rate but also is non-toxic, non-explosive, non-flammable, cost-efficient, readily available, and easy to remove from the extracted materials. Despite the rapid developments in the applications of SFE, the supercritical extraction of volatiles from Flixweed seed has not been reported. Therefore, the objective of this study was to optimize the process conditions by conducting response surface methodology (RSM) as a powerful tool for determining the factors effects and their interactions, which allow process optimization effectively (Bas and Boyaci 2007). This method is the preferred experimental design for fitting polynomial model to analysis the response surface of multi-factor combinations (Liu et al. 2009). To the best of our knowledge, this is the first attempt to compare the volatiles of flixweed seed obtained by steam distillation and SFE.

Materials and methods

Plant material and reagents

Descurainia Sophia L. seeds were collected from Mashhad (Khorasan, Iran), in the middle of May 2012. The mature and brown seeds were manually separated and cleaned from dust and then stored in a dark place and dried at room temperature for 1 week. Cleaned seeds were ground using a domestic grinder (Myson, China) producing particles with a mean diameter of 0.5 mm and stored at 4 °C. HPLC grade n-hexane and methanol was purchased from Caledon (Georgetown, Ont., Canada). Analytical grade toluene, ethanol and acetonitrile were purchased from Merck (Darmstadt, Germany). Carbon dioxide (99.99 % purity), contained in a cylinder

with an eductor tube, was obtained from Roham Co. (Tehran, Iran).

Steam distillation (SD)

A simple laboratory quickfit apparatus was used to perform the steam distillation. A 100 g shade-dried seed was subjected to steam distillation and the volatile components were collected into the receiving flask during 2 h of steam distillation. The yield of the oil was 0.25 % (v/w) based on the dry plant weight.

Supercritical fluid extraction (SFE)

A laboratory-scale Suprex MPS/225 system (Pittsburgh, PA) in the SFE mode was used in this study. Flixweed seed powder (0.5 g) mixed well with 1 mm diameter glass beads, was loaded into a 3-mL high-pressure stainless steel extraction vessel. Liquid CO₂ was pumped into the vessel with a flow rate of approximately (0.3±0.05 mL min⁻¹). A Duraflow manual variable restrictor (Suprex) was used in the SFE system to collect the extracted analytes. The restrictor temperature was set at 75 °C. The extracts were collected continuously from the outlet of the restrictor in a 3-mL volumetric flask containing n-hexane. To improve the collection efficiency, the 3-mL volumetric flask was placed in an ice bath during the dynamic extraction stage. The final volume of the extract was adjusted to 3.0 mL with n-hexane at the end of the extraction. For all the modifier studies, the modifier (methanol) was spiked directly onto the sample in the extraction vessel before the extraction cell was attached to the SFE system. After the completion of each run, the oil recovery percentage was determined gravimetrically by weighing the collected solution after bubbling of the solution by using nitrogen gas to evaporate the solvent and the extraction yield was calculated based on the ratio of the extracted oil's weight to the dry mass plant powder×100/g of seeds. Figure 1 shows a graphical summery of SFE and SD extraction procedures.

Gas chromatography–mass spectrometry (GC-MS)

The extracts obtained were analyzed with a Thermoquest-Finnigan Trace GC-MS instrument equipped with a 30 m cross-linked DB-5 (5 % biphenyl + 95 % methylpolysiloxane) fused-silica capillary column (0.25 mm i.d. and 0.25 µm film thickness), (HP 5-MS). The oven temperature was programmed 60 °C for 5 min, then increased by 5 °C min⁻¹ from 60 to 250 °C and held for 5 min. The analysis was performed using the MS scanning mode. Helium was used as the carrier gas at a flow rate of 1.5 mL min⁻¹. Split ratio was adjusted at 1/10. The mass spectra were obtained by electron impact ionization with an ionizing voltage of 70 eV and an ionizing current of 150 A. The components of oil were identified by

Fig. 1 Graphic of SFE and SD extraction procedures

comparing their mass spectra with those in the NIST, Wiley and Adams mass spectra libraries. The data obtained were confirmed by comparing their retention indices, either with those of authentic compounds or with the data published in the literature (Sandra and Bicchi 1987; McLaferty and Stauffer 1989).

Results and discussion

The aim of this work was to find the conditions providing the highest SFE (static–dynamic approach) recoveries of *Descurainia Sophia* L. seed oil inside the experimental domain explored, and the results were compared with essential oil composition obtained through steam distillation. For this approach, response surface methodology (RSM) was applied to optimize the processing parameters for supercritical fluid extraction of flixweed seed.

Optimization of SFE conditions

For optimization of SFE conditions a half fractional factorial design followed with a face centered central composite design (FCCD) with $\alpha=1$ was applied. STATGRAPHICS plus 5.1, statistical and graphical analysis software, was used for the experimental table generation and analysis of the results. Based on the preliminary studies, five factors including pressure (A), temperature (B), modifier (methanol) volume (C), dynamic and static extraction time (D) and (E), respectively, were considered. Firstly, a half fractional factorial design with two center points ($2^{5-1}+2$) and 18 experiments was used. Table 1 shows the main factors, their notations, and levels for half fractional factorial design. The design matrix is shown in Table 2. Normal probability plots were used to find the most important effects and interactions. In normal probability plots, the negligible effects fall on a straight line, whereas significant effects would be located off the line. The normal probability plot for the fractional factorial design is shown in Fig. 2. The

Table 1 Factors, factor notation, and their levels in half fractional factorial design

Factor	Notation	Level		
		−1	0	+1
Pressure (bar)	A	100	228	355
Temperature (°C)	B	35	50	65
Modifier volume (methanol) (μL)	C	0	50	100
Dynamic extraction time (min)	D	10	25	40
Static extraction time (min)	E	10	25	40

analysis of the results also visualized using standardized main effect and two factor interaction Pareto charts ($P=95\%$) as are shown in Fig. 3. The vertical line on the plot judges the effects that are statistically significant. Moreover, the positive or negative sign (corresponding to a purple or red) response can be enhanced or reduced, respectively, when passing from the lowest to the highest level set for the specific factor.

The results illustrated in Figs. 2 and 3 confirm that pressure (A), modifier volume (C), dynamic extraction time (D) and the interactions of pressure and dynamic extraction time (AD) and pressure and modifier volume (AC) are the most effective parameters at 95 % confidence level. Accordingly, all the other variables and their interactions are not significant factors in the studied range. Thus, the central composite design for these factors (A, C and D) was applied to determine the

Fig. 2 Normal probability plot of effects for the half fractional factorial design

optimum condition of SFE. Although it seems that the influence of temperature on the extraction yield was negative but according to Fig. 3 the interactions of temperature with other factors had positive effects on the extraction yield so according to the software suggestion two other variables (temperature and static extraction time) were fixed at their optimum amounts, 65 °C and 10 min, respectively. At second step of data analysis central composite design (CCD) has been carried out on 16 randomized runs ($2^3 + (2 \times 3) + 2$), consist of a (2^3) factorial design augmented with (2×3) star points and 2 central points. The values of responses were transformed to square root values. This was performed in accordance to the Box–Cox plot recommendation to achieve a normal

Table 2 Design matrix and the responses for half fractional factorial design

Experiment	Pressure (bar)	Temperature (°C)	Modifier volume (methanol) (μL)	Dynamic extraction time (min)	Static extraction time (min)	Recovery (%)
Run 1	+1	+1	−1	+1	−1	10.89
Run 2	+1	−1	−1	+1	+1	10.8
Run 3	+1	+1	+1	+1	+1	16.78
Run 4	+1	+1	−1	−1	+1	0.53
Run 5	+1	−1	+1	−1	+1	9.4
Run 6	+1	−1	+1	+1	−1	16.2
Run 7	−1	−1	−1	−1	−1	0.5
Run 8	−1	+1	+1	−1	−1	0.2
Run 9	+1	+1	+1	−1	−1	9.31
Run 10	−1	+1	+1	−1	+1	0.98
Run 11	−1	+1	+1	+1	+1	0.35
Run 12	0	0	0	0	0	4.9
Run 13	−1	−1	−1	+1	+1	1.7
Run 14	−1	+1	+1	+1	−1	1.21
Run 15	0	0	0	0	0	4.9
Run 16	−1	−1	+1	+1	−1	1.2
Run 17	−1	−1	+1	−1	−1	0.98
Run 18	−1	−1	−1	−1	+1	1.1

Fig. 3 Standardized ($P=0.05$) Pareto chart obtained from the half fractional factorial design, representing the estimated effects of parameters and parameter interactions on extraction recovery

distribution of results. Table 3 shows the main factors, their notations, and levels for central composite design. The design matrix is shown in Table 4. This design permitted the response to be modeled by fitting a second-order polynomial, which can be expressed in Eq. (1) in terms of coded factors:

$$\begin{aligned} \text{Sqrt}(Y) = & +3.30 + 1.22 A + 0.27 B + 0.22 C \\ & + 0.086 AB + 0.095 AC - 0.12 BC - 0.60A^2 \\ & + 0.051B^2 - 0.23C^2 \end{aligned}$$

Where A , B and C are the independent variables pressure, modifier volume and dynamic extraction time, respectively. Analysis of variance (ANOVA) was used to estimate the significances of the main effects and interactions. The ANOVA results of the model (Table 5) indicated a good performance with R^2 and R^2 -adjusted of 0.99 and 0.98, respectively. The R^2 -adjusted value is one of the measures of degree of fit and it is more suitable than R^2 for comparing models with different numbers of independent variables. The model R^2 -adjusted value of 0.98 implies that 98 % of the variations associated with extraction yield are attributed to the selected independent variables (pressure, modifier volume and dynamic extraction time). Based on the central composite design, pressure has the largest influence on the extraction

Table 3 The experimental variables and levels of central composite design (CCD)

Factor	Notation	Level		
		−1	0	+1
Pressure (bar)	A	100	228	355
Modifier volume (μL)	B	50	100	150
Dynamic extraction time (min)	C	10	25	40

$\alpha=1$

Table 4 The central composite design (CCD) program and results

Experiment	Pressure (bar)	Modifier volume (methanol) (μL)	Dynamic extraction time (min)	Recovery (%)
Run 1	+1	+1	+1	18.48
Run 2	0	+1	0	12.72
Run 3	0	0	+1	10.6
Run 4	+1	+1	−1	15.42
Run 5	−1	0	0	2.07
Run 6	−1	+1	−1	2.29
Run 7	0	0	0	10.40
Run 8	0	−1	0	9.24
Run 9	−1	−1	+1	1.88
Run 10	0	0	−1	7.72
Run 11	−1	+1	+1	2.26
Run 12	+1	−1	−1	8.81
Run 13	+1	−1	+1	14.6
Run 14	+1	0	0	14.99
Run 15	−1	−1	−1	0.8
Run 16	0	0	0	12.9

yield. The extraction yields were increased with an increase in pressure, because raising the extraction pressure leads to a higher fluid density, which increases the solubility of the analytes. The solubility of the extract in supercritical fluid directly affects the extraction yield. Due to the limited solubility of polar organic compounds in the supercritical carbon dioxide, quantitative extraction of these compounds with pure supercritical CO_2 is not possible. Therefore, the addition of a polar modifier such as methanol to supercritical carbon dioxide has been shown to offer tremendous increases in the extraction yield of polar organic compounds. In the dynamic extraction time, the supercritical fluid flows continuously through the sample and cause to enhance the analyte's solubility and the extraction yield. The obtained regression models were used to calculate the response surface for each variable separately. Estimated response surface for the pressure and modifier volume and pressure and dynamic extraction time versus extraction yield (%) and their related contours are shown in Fig. 4(a, b), respectively. After the analysis of results, the following conditions were selected to evaluate the performance of the extraction procedure: pressure of 355 bar, temperature of 65 °C, modifier volume of 150 μL, static and dynamic extraction time of 10 and 35 min, respectively.

Comparison of methods

Table 6 shows the components and their peak area ratios, obtained by SFE and SD. The major components of extracted

Table 5 Analysis of variance (ANOVA) table of the quadratic response surface model

Source	SS	df	MS	<i>f</i> -value	Prob>F	
Model	18.15	9	2.02	111.95	<0.0001	(Significant)
A-P	14.80	1	14.80	821.75	<0.0001	
B-B	0.74	1	0.74	40.91	0.0007	
C-C	0.47	1	0.47	26.12	0.0022	
AB	0.059	1	0.059	3.26	0.1210	
AC	0.072	1	0.072	3.99	0.0928	
BC	0.12	1	0.12	6.50	0.0436	
A ²	0.94	1	0.94	52.09	0.0004	
B ²	6.95E-003	1	6.95E-003	0.39	0.5573	
C ²	0.15	1	0.15	8.06	0.0296	
Residual	0.11	6	0.018			
Lack of Fit	0.041	5	8.162E-003	0.12	0.9650	(Not significant)
Pure Error	0.067	1	0.067			
Corrected total	18.25	15				

obtained by SFE were methyl linoleate (18.2 %), camphor (12.32 %), cis-thujone (11.3 %), trans-caryophyllene (9.17 %), α -humulene (7.6 %), epi-glubolol (7.3 %), borneol (4.46 %), linoleic acid (3.8 %), bornyl acetate (3.67 %), trans-

thujone (3.18 %), ethyl linoleate (3.13 %), hexadecanoic acid (2.5 %), stearic acid (2.2 %), α -bisaboene (1.7 %) and 1,8-cineol (1.64 %) and acorenone (1.5 %). Figure 5 shows GC–MS chromatogram of the essential oils and fatty acids obtained

Fig. 4 Three-dimensional (3D) estimated response surfaces for SFE of the *D. Sophia* seeds by plotting **a** extraction yield (%) versus pressure (atm) and modifier volume (μ L) at dynamic extraction time of 25 min; and **b** extraction yield (%) versus pressure (atm) and dynamic extraction time (min) at modifier volume of 100 μ L

Table 6 Percentage of the volatile components of *D. sophia* seed obtained by different extraction process (Steam distillation: 2 h; supercritical CO₂ extraction: 65 °C, 355 bar, 35 min, (methanol) 150 µL)

No	Component	RI ^a (iu)	% ^b SFE	% ^c SD	RSD (%) SFE	RSD (%) SD
1	α-pinene	960	—	0.3	—	3.94
2	Camphene	975	—	0.15	—	2.9
3	β-pinene	990	—	0.13	—	5.32
4	Myrcene	1008	—	0.4	—	3.4
5	α-Terpinene	1020	0.3	0.57	7.43	1.89
6	1,8-cineol	1034	1.64	1.5	4.3	2.32
7	cis- β-Ocimene	1049	—	19.8	—	4.2
8	trans- β-Ocimene	1057	—	0.96	—	7.3
9	Terpinolene	1087	—	0.48	—	3.6
10	trans-allo-Ocimene	1126	—	0.75	—	2.43
11	cis-allo-Ocimene	1140	—	0.15	—	5.6
12	isoborneol	1151	—	0.4	—	3.2
13	Linalool	1094	0.41	—	5.97	—
14	cis-thujone	1106	11.3	6.2	3.1	3.5
15	trans-thujone	1117	3.18	1.6	4.12	4
16	Camphor	1148	12.32	7.5	4.17	1.9
17	Menthol	1161	—	11.58	—	2.6
18	isomenthol	1170	—	0.35	—	5.8
19	Borneol	1178	4.46	1.1	2.15	1.8
20	Nerol	1225	—	1.42	—	3.5
21	Thymol	1283	0.38	0.9	7.24	—
22	Bornyl acetate	1286	3.67	0.85	1.3	—
23	Terpinyl acetate	1340	—	2.4	—	6.3
24	α-longipinene	1365	—	1.3	—	3.2
25	Longicyclene	1390	—	2	—	2.9
26	Cyperene	1402	—	1.2	—	4.2
27	trans-caryophyllene	1432	9.17	4.1	1.48	6.1
28	Trans, trans-farnesol	1440	—	0.65	—	4.16
29	Aromadendrene	1452	0.22	—	6.7	—
30	α-Humulene	1466	7.6	—	3.3	—
31	allo-Aromadendrene	1473	0.48	1.89	5.32	3.4
32	α-Bisabolene	1505	1.7	0.8	5.1	4.27
33	β-curcumene	1514	—	0.78	—	3.25
34	α-Selinene	1528	—	0.5	—	4.7
35	Cis-nerolidol	1551	—	0.65	—	3.78
36	Caryophellene	1598	0.4	—	6.8	—
37	Epi-glubolol	1606	7.3	—	2.3	—
38	α-Cedrol	1618	—	1.1	—	3.68
39	Humullene epoxide	1624	0.33	—	—	—
40	Heptadecane	1700	—	1.5	—	2.45
41	Acorenone	1712	1.5	1.8	5.3	3.84
42	Nonadecane	1905	0.3	—	3.2	—
43	Hexadecanoic acid	1945	2.5	1.2	3.7	5.32
44	Ethyl hexadecanoate	1980	0.5	0.8	—	7.2
45	Linoleic acid	2058	3.8	2.5	4.2	2.11
46	Methyl linoleate	2063	18.2	5.8	2.8	2.5
47	Stearic acid	2071	2.2	0.7	3.19	1.8

Table 6 (continued)

No	Component	RI ^a (iu)	% ^b SFE	% ^c SD	RSD (%) SFE	RSD (%) SD
48	Ethyl linoleate	2076	3.13	4.4	5.28	1.56
	unknown components	—	3.01	6.84	—	—

^a RI, Kovat's retention index using a DB-5 MS-column

^b %, Relative area percent (peak area relative to the total peak area) for Supercritical Fluid Extraction

^c %, Relative area percent (peak area relative to the total peak area) for Steam Distillation

by the optimum condition of SFE (pressure of 355 bar, temperature of 65 °C, static extraction time of 10 min, dynamic extraction time of 35 min and modifier volume of 150 μ L). The major components based on steam distillation were cis- β -ocimene (19.8 %), menthol (11.58 %), camphor (7.5 %), cis-thujone (6.2 %), methyl linoleate (5.8 %), trans-caryophyllene (4.1 %) and ethyl linoleate (4.4 %). As can be concluded from Table 6, the obtained components by steam distillation and SFE were different. Table 6 indicates that the number of the essential oil and fatty acid components

extracted by the SFE (25 components) is lower than those obtained by steam distillation method (41 components). These results showed that by changing the SFE conditions, the supercritical fluid extraction is more selective than the steam distillation method and it requires a shorter extraction time. Additionally, the flexibility in the management of the variables involved in the SFE process permitted us to optimize the experimental conditions, considering the selectivity of a substance or classes of substances of interest. The selectivity of supercritical CO₂ was allowed to maximization the

Fig. 5 GC-MS chromatogram obtained by SFE in optimized conditions. Numbers are related to the components in Table 6

concentration of selected compounds. Therefore, SFE is more advantageous than the steam distillation for the extraction of essential oils and fatty acids from *Descurainia sophia* L. seed.

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