Supercritical Fluid Extraction and Hydrodistillation of Cleosia Argentea Essential Oil

Mojtaba Sinaei Nobandegani, Tayebeh Darbandi, Bizhan Honarvar, and Mohammad Mohsen Sarafraz

***Abstract*—**The essential oil of Celosia Argentea essential oil has been extracted by supercritical fluid extraction and hydrodistillation. Celosia Argentea is an edible and medicinal plant which belongs to Amaranthaceae family. In this study, the Taguchi method has been used to design of experiments and carbon dioxide has been used as the supercritical fluid. The effect of pressure, temperature and mean particle size has been studies on the extraction yield. The supercritical fluid extraction results are compared with the hydrodistillation and the components of the essential oil has been identified by GC/MS analysis.

***Keywords*—**Celosia Argentea, Supercritical Fluid, Extraction, Amaranthaceae

I. Introduction

SUPERCRITICAL fluid extraction (SFE) has some advantages in compared with the conventional extraction methods. In supercritical fluid extraction degradation of heat sensitive materials is avoidable, which is an important issue in medical and food industrial. As the second advantage of this method, the solvent recycling is more feasible in supercritical fluid extraction. This lead the operating cost decrease. Moreover, the supercritical fluid extraction has a higher selectivity, and the extraction yield and component of production can be changed by changing the operating conditions. There are varieties kinds of solvent are used as the supercritical fluid in SFE process. Carbon dioxide is one of the most common solvent in this process because of some advantages such as non-toxicity and non-flammability. It is an environmental-friendly solvent, too. Because of the mentioned reasons, this extraction method has used widely in different processes especially in vegetal oil extraction [1]-[6].

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The essential oil of medicinal plant is also can be extracted using this process. The Celosia is a plant which consisting about 60 species and belongs to Amaranthaceae (Caryophyllales) family [7]-[8]. This plant is widely distributed in subtropical and temperature zone of Africa, South America, India, and south East Asia [8]-[9]. Celosia argentea is used as a kind of vegetable in China and other countries, and it is also used in traditional Chinese medicines. Celosia has been used for treatment of hypertension, palsy, cataract, keratitis, diabetes, iridocyclitis, caligo corneae, sarcoptidosis, eye and hepatic diseases, dysentery, diarrhea, acute abdominal pain, inflamed stomach and skin eruptions[7]-[16]. It also exhibits antibacterial activity against Bacillus subtilis, Salmonella typhi, Escherichia Coli, Agrobacterium and Mycobacterium tuberculosis [15].

In 2003, Gnanamani et al. extracted the Celosia Argentea essential oil and study its antibacterial activity [15]. Sharma et al. studied the alcoholic extract of Celosia Argentea and its antidiarrhoeal activity [11]. Rani and Raju extracted the Celosia Argentea essential oil using the soxhelt in 2014 [17]. No report has yet appeared on the supercritical fluid extraction of Celosia Argentea, but there are some studies on supercritical fluid extraction of some other medicinal plant. Darbandi et al. studied the supercritical CO_2 extraction of Ziziphora Tenuior [5]. Danh et al. studied the extraction of Zizanioides essential oil by supercritical CO_2 [18]-[19]. The extraction of Marchantia convoluta via supercritical CO_2 has been studied by Xiao et al. in 2007 [20].

In this study, the Celosia Argentea essential oil has been extracted by supercritical carbon dioxide, and the effects of operation parameters namely pressure, temperature and mean particle size on the extraction yield has been investigated. Moreover, the essential oil components have been identified using the GS/MS analysis.

II. Material and Methods

A. Plant Material

Cleosia Argentea was collected from the northern part of Shiraz, Iran, in October 2013. Then it was dried in a dark place and at the room temperature (around 25°C). The

sample was ground in a Panasonic blender (Model MX-J225G) to produce powder. The averages of particles size on the basis of ASTM E11 were 0.150, 0.212 and 0.300 mm.

B. Reagent

Carbon dioxide with the purity of 99.95% was obtained from Abu-Qaddareh Company (Shiraz). Dichloromethane with the purity of 99.99%, purchased from Merk KgaA, was used as a solute recovery.

C. Hydrodistillation

The essential oil of Celosia Argentea (50 gr material) was obtained by hydrodistillation for 3.5 hours on a Clevenger-type apparatus. The extraction yield on the basis of dry weight was 2.1%.

D. Supercritical Fluid Apparatus and Procedure

The supercritical apparatus and procedure, which has been used in this study, was the same as that one used in our previous study [5]. The SFE apparatus consisted of a CO_2 cylinder, a refrigerator to change CO_2 phase from gas to liquid, a handle reciprocating pump, a double-wall tank to load CO_2, a barometer and an extraction tank with the internal diameter of 12.5 mm and length of 40 cm. The extraction tank had two parts. In the inner part, the sample was placed; while in the outer part, water was circulating to increase the temperature of the flow. The temperatures of input and output water flow were controlled and at the end, a Joule-Thomson valve had been embedded in the outgo of the line. After the extraction, the essential oil+CO_2 mixture was expanded to atmospheric pressure. The essential oil was solved in dichloromethane, and CO_2 was released into the air (before it was released to the air, its flow was measured by a volumetric flow meter).The experimental apparatus schematic has been illustrated in Fig. 1.

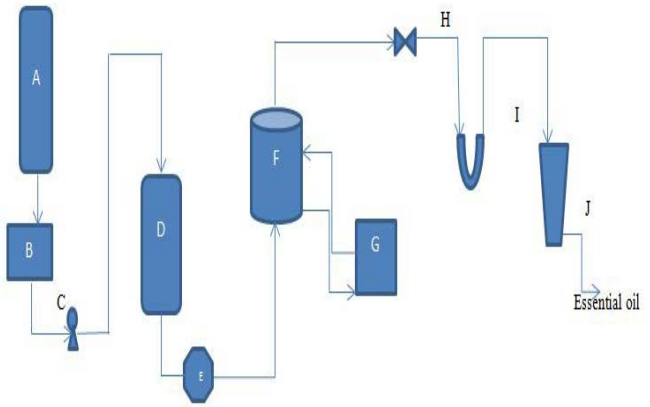

Fig. 1 The Experimental Apparatus for the Extraction of *Celosia Argentea*: A-Cylinder of CO_2, B- Refrigerator, C- Handle Reciprocating Pump, D- A Tank for CO_2 Loading, E- Barometer, F- Extraction Tank, G- Heater for Heating Water, H- Joule-Thomson Valve, I- Dichloromethane in U-Shape Tube at 0°C, J- Flow meter [5]

E. GC and GC/MS Analysis

As what has been done in our previous study [5], the GC-FID analysis was performed using Agilent GC-6890N gas chromatograph, which is made in the U.S., with nitrogen as the carrier gas with a velocity of 1.4mlit/s on HP-5 (Dimethylsiloxane, 5% phenyl) column (30×0.25 mm id, Film thickness 0.25µm). The SFE samples (1µl) were injected (without any further dilution) using the split mode with a split ratio of 1/10. The oven temperature program was 60°C, which was then increased to 230°C at a rate of 3°C/min, and it remained at 230°C for 5 minutes. The injector and detector temperature were held at 240 and 260°C, respectively. The percentages of components were calculated by the area normalization method, without considering response factors.

The GC-MS analysis was performed using a Variane 3400 equipped with a DB-5 column (30×25 mm internal diameter, film thickness 0.25 µm, and with helium as a carrier gas). The SFE samples (0.2µl) were injected (without any further dilution) using split mode with a split ratio of 1/60 and with split flow of 7.166ml/min. The oven temperature program at first was 60°C for 3 minutes, and then it was increased to 230°C at a rate of 3°C/min, and it remained at 230°C for 5 minutes. The transfer line temperature was 280°C. The ionization energy was 69.922 eV with a scan time of 1 second and mass range of 34-500 amu. The injector and detector temperatures were held at 300 and 270°C, respectively.

F. Design of Experiment

To reduce the experiment number and operating cost, it is necessary to use the design of experiment. In this purpose there are different methods such as fractional factorial, full factorial and once-at-time. In this study, one kind of fractional factorial method, namely taguchi, has been employed because it reduces the number, costs and time of experiments and it is easy to use[5], [21]-[23].

To design the experiment, the pressure, temperature and mean particle size has been used as three factors, which were variable in three different levels. As a result, the L.9 array has been used in design of experiment. Different factors of each level used in the taguchi design of experiment are demonstrated in Table I.

TABLE I
EXPERIMENTAL LEVELS OF THE FACTORS USED IN THE TAGUCHI METHOD

No.	Pressure (bar)	Temperature (°C)	Mean Particle Size (µm)
1	150	45	150
2	170	55	212
3	190	65	300

III. RESULT AND DISCUSSION

As it is mentioned in the previous sections, three parameters are considered as the most important factors on the extraction yield. Amongst the said parameters are the pressure, temperature and mean particle size, which effects has been investigated on the extraction yield. The experiment has been designed by taguchi method using the Minitab software, and its result is reported in Table II. Moreover, the component of the Celosia Argentea essential oil was

identified and quantitated in different experimental conditions.

TABLE II
THREE FACTORS, THREE LEVELS (L.9) ORTHOGONAL ARRAY DESIGN FOR SUPERCRITICAL FLUID EXTRACTION OF CELOSIA ARGENTEA

No.	Pressure (bar)	Temperature (°C)	Mean Particle Size (µm)	Yield (%)
1	150	45	150	2.74
2	170	45	212	2.77
3	190	45	300	2.89
4	150	55	212	1.80
5	170	55	300	2.93
6	190	55	150	4.80
7	150	65	300	1.70
8	170	65	150	3.65
9	190	65	212	3.39

A. Pressure Effect

According to analysis of variance (ANOVA) and as it is reported in the Table III, pressure is the most important parameter in this operation.

TABLE III
ANOVA OF THE EXPERIMENTS (AT 90% CONFIDENCE)

S.V. [a]	S.S [b]	D.F. [c]	M.S. [d]	F Value
Pressure	3.97	2	1.99	3.87
Temperature	0.22	2	0.11	0.10
Mean Particle Size	2.70	2	1.35	1.86

[a] Source of Variance
[b] Sum of Square
[c] Degree of Freedom
[d] Mean Square

Fig. 2 shows the pressure variation effect on the extraction yield. As it shown, the extraction yield increased while the pressure increased. The solvent power for solving substances increases while the fluid density and pressure increase.

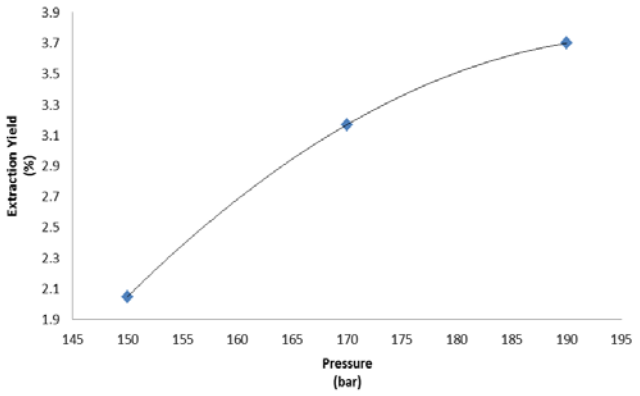

Fig. 2 The pressure effect on the extraction yield

B. Temperature Effect

As it is illustrated in Fig. 3, when the temperature was increased from 45 to 55 °C, the extraction yield rose. However, there was a reduction in the amount of extraction yield when the pressure was increased from 55 to 65 °C. Indeed, the temperature has a positive and a negative effect on the extraction yield. Increase in some transport properties like vapor pressure of essential oil, binary diffusion coefficient and volatility of essential oil in supercritical CO_2 make the positive effect, and lead the extraction yield increase. On the other hand, the extraction yield decreases while the density and solvent power of supercritical CO_2 decrease by the temperature increase, and this is the negative effect of temperature [24] -[26].

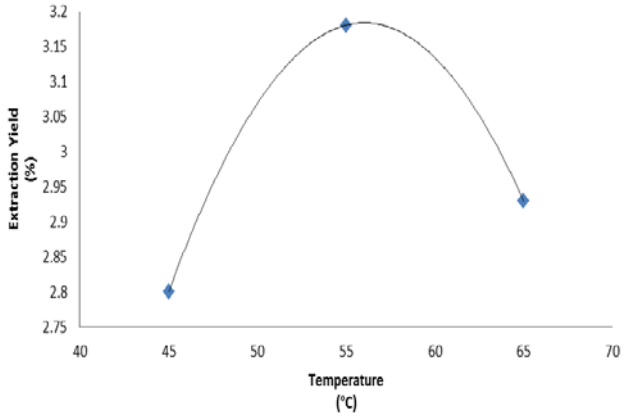

Fig. 3 The temperature effect on the extraction yield

In the first stage, the positive effect of temperature on the extraction yield is stronger than the negative one, and this causes the extraction yield increase while the temperature increases. In the second stage, there is an opposite behavior. At this stage, the negative effect is greater than the positive one, and the extraction yield decreased with the temperature increase.

C. Mean Particle Size Effect

Like the previous study, the extraction yield increased with reduction of the mean particle size [5]. This is because of decrease in mass transfer resistance and increase in interfacial area which are occurred by milling the material. Besides, this process releases more oil from the cells, and it also shortens the diffusion path in particles [6], [24], [25], [27]. The mean particle size variation effect is demonstrated in Fig. 4.

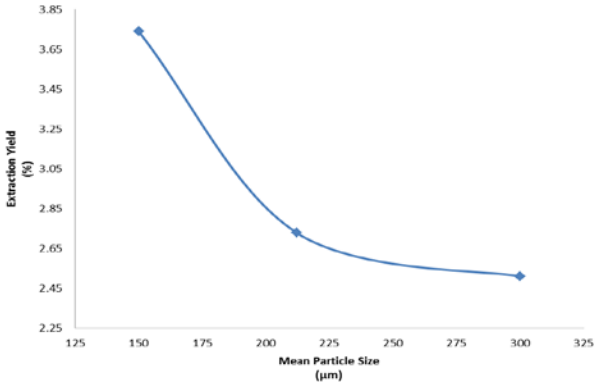

Fig. 4 The pressure effect on the extraction yield

D. Analysis Results

The main components of the Celosia Argentea essential oil are identified using GC/MS analysis and the result is reported in Table IV. As it can be seen in Table IV, the components present in higher quantities are 34.69% of cembrene, 25.075% of hexadecanoic acid, and 6.90% of 6,10,14-trimethyl-2-pentadecanone.

TABLE IV
COMPONENTS (% OF TOTAL PEAK AREA) OF CELOSIA ARGENTEA ESSENTIAL OIL OBTAINED BY SFE

Components	R.T. [a]	K.I. [b]	% In Oil
heptanal	-	902	t [c]
α-pinene	-	939	t
camphene	-	954	t
(2E)-heptenal	-	954	t
3-octanone	-	983	t
2-pentyl furan	5.237	988	1.854
n-octanal	5.555	998	0.469
limonene	-	1029	t
benzene acetaldehyde	-	1042	t
trans-linalool oxide	8.166	1086	0.881
n-undecane	-	1100	t
n-nonanal	8.814	1100	4.520
p-menth-3-en-8-ol	10.424	1150	0.931
N,N,4-trimethyl benzenamine	11.674	1183	2.894
n-dodecane	12.231	1200	1.339
n-decanal	12.610	1201	1.463
β-cyclocitral	-	1219	t
pulegone	13.943	1237	3.279
carvotanacetone	-	1247	t
(4E)-decen-1-ol	-	1262	t
cis-2-tert-butyl-cyclohexanol acetate	15.951	1293	0.490
n-tridecane	-	1300	t
undecanal	-	1306	t
(2E,4E)-decadienal	-	1316	t
n-tetradecane	20.406	1400	0.932
E-caryophyllene	21.005	1419	4.218
α-humulene	22.298	1454	0.443
geranyl acetone	22.638	1455	2.077
E-β-ionone	-	1488	t
δ-cadinene	25.215	1523	1.793
caryophyllene oxide	-	1583	t
n-hexadecane	-	1600	t
n-octadecane	-	1800	t
6,10,14-trimethyl-2-pentadecanone	37.184	1854	6.903
cembrene	39.663	1937	34.689
hexadecanoic acid	42.542	1984	25.075
n-heneicosane	45.111	2100	2.940

[a] R.T.: Retention Time
[b] K.I.: Kovats Index
[c] t: Trace (<0.05%)

Cembrene and hexadecanoic acid are the main components of Celosia Argentea essential oil with 34.689 and 25.075 percent, respectively. The other component of Celosia Argentea are 6,10,14-trimethyl-2-pentadecanone, E-caryophyllene, n-nonanal, pulegone, n-heneicosane, N,N,4-trimethyl benzenamine and geranyl acetone which are presented in the essential oil by 6.903%, 4.218%, 4.52%, 3.279%, 2.94%, 2.894% and 2.077%, respectively. The contributions of other components which present in the essential oil are lower 1%. The typical chromatogram of the SFE is given in Fig. 5.

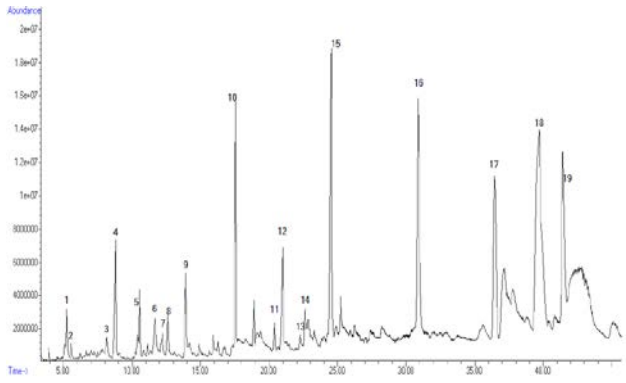

Fig. 5 Typical gas chromatogram of Celosia Argentea essential oil
The different compounds are shown by Arabic numerals; (1) 2-Pentyl furan; (2) n-Octanal; (3) trans-Linalool oxide; (4) n-Nonanal; (5) p-Mentha-3-en-8-ol; (6) N,N,4-Trimethyl Benzenamine ; (7) n-Dodecane; (8) n-Decanal; (9) Pulegone; (10) cis-2-Tert-butyl-cyclohexanol acetate; (11) n-Tetradecane; (12) E-Caryophyllene; (13) α-Humulene; (14) Geranyl acetone; (15) δ-Cadinene; (16) 6,10,14-Trimethyl-2-pentadecanone; (17) Cembrene; (18) Hexadecanoic acid; (19) n-Heneicosane.

E. Comparison of Supercritical Fluid Extraction with Hydrodistillation

The essential oil obtained by supercritical fluid extraction was deeper in color and aroma in comparison to hydrodistillation. Besides, the extraction yield in the hydrodistillation process was equal to 2.1% (w/w). On the other hand the supercritical fluid extraction yield was varied from 1.7% to 4.8%. The yield in supercritical fluid extraction could be different if the operation conditions changed. Moreover, the selectivity of supercritical fluid extraction is higher than hydrodistillation. As it is shown in Table V, δ-cadinene could be varied from 0 to 98%, in different Runs. As a result, different components can be obtained in supercritical fluid extraction.

TABLE V
GC ANALYSIS FOR CELOSIA ARGENTEA ESSENTIAL OIL IN RANDOMIZED RUNS (% IN OIL)

Components	Run 1	Run 2	Run 5
α-pinene	0.005	-	-
3-octanone	-	-	0.005
2-pentyl furan	0.008	-	-
limonene	-	-	0.005
benzene acetaldehyde	-	-	0.007
n-undecane	-	0.030	-
n-nonanal	0.008	0.900	-
p-menth-3-en-8-ol	-	0.006	-
N,N,4-trimethyl benzenamine	-	0.080	-
pulegone	0.090	-	-
cis-2-tert-butyl-cyclohexanol acetate	-	-	0.085
n-tridecane	-	0.650	-
(2E,4E)-decadienal	-	45.900	-
n-tetradecane	-	-	0.250
E-caryophyllene	-	0.230	-
α-humulene	-	-	1.090
δ-cadinene	41.020	-	98.230
n-hexadecane	-	0.090	-
6,10,14-trimethyl-2-pentadecanone	52.010	43.090	0.070
cembrene	-	-	0.040
n-heneicosane	0.900	-	-

IV. Conclusion

The Cleosia Argentea essential oil has been extracted by supercritical fluid extraction and hydrodistillation method, in this study and the results obtained from these two methods have been compared with each other. The supercritical fluid extraction has been done in three different values for temperature, pressure and mean particle size and the effect of these parameters on the extraction yield has been studied. The experiments have been designed by Taguchi method and L.9 orthogonal array. Moreover, carbon dioxide has been used as the supercritical fluid.

The pressure was the most important factor in the supercritical fluid extraction. The extraction yield increased as the pressure increased. Temperature has two different effects on the extraction yield. Firstly, the extraction yield increased by increase in temperature from 45 to 55ºC. Then, the extraction yield decreased while the temperature increased from 55 to 65 ºC. The extraction yield had an indirect proportion with mean particle size. In other words, the extraction yield decreased when the mean particle size increased. The best extraction yield was 4.8% and has been obtained at temperature of 55ºC, pressure of 190bar and mean particle size of 150µm.

The essential oil obtained from supercritical fluid extraction was deeper in color and odor in compared to that one obtained from hydrodistillation. Besides, the supercritical fluid extraction selectivity was higher than hydrodistillation. The extraction yield in hydrodistillation was 2.1%. The main components of Cleosia Argentea essential oil was cembrene and hexadecanoic acid.

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