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Analytical study and physical effect of extraction oil from *Cercies Siliquastrum L.*

Basel Ibrahim

Dep. Chemistry, Faculty of Science, Damascus University, Syria.

***Corres.author: baselibrahim60@yahoo.com., phone No. 963 930009716**

Abstract: The investigation was carried out during 2013 - 2014 at the laboratories of Chemistry Dep. Damascus University. the constituents of oil extracts isolated from ripe seeds of Cercies siliquastrum L. from grown in Damascus, Syria,. Extracts were prepared from dried and powdered ripe seeds with solvent petroleum ether. The percentage of oil in the studied sample is determined and found to be 5.72%. The GC-M analysis of petroleum ether seeds extract from C.siliquastrum revealed the presence of (8) components: n-Hexadecanoic acid(1), 9,12-Octadecadienoic acid (z,z)-,2,3-dihydroxypropyl ester(2), Heneicosane(3), Octadecane(4), Phenyl-1,2-diamine,N,4,5-trimethyl-(5), 9-Octadecyne(6), 9,17 Octadecadienal,(Z)-(7) and E-11-Hexadecenal(8).
Oil from the seeds of C. siliquastrum L was extracted with petroleum ether, and was evaluated for specific gravity, SG (0.923), iodine value, IV (129.61mgI2/100g), acid value, AV (0.095mgKOH/g), free fatty acid value, FFA (0.042%), and saponification value, SV (192.65mgKOH/g).
The physical factors conclude that: Soaking for a sufficient period and when the boiling point of the solvent and Percent (dry seed : solvent) is (1:7).
Keyword: Ceries siliquastrum, petroleum ether

1. Introduction:

Cercies siliquastrum L. is native to South eastern Europe and South-west Asia and is prevalent throughout continental Greece [1,2]. The C. siliquastrum L. has a potential use for landscaping due to its ornamental features (blackish bark, heart-shaped leaves and bright purplish-rose flowers) also used for borders, erosion control, windbreaks and wildlife plantings [3,4] and as a medical plant [5]. Furthermore, the species is well adapted to semi-arid conditions and is tolerant of air pollution and nutrient deficient soils [3,6]. The chemical composition of neutral lipids from seeds of C. siliquastrumL., Sapium sebiferum, and Koelreuteria paniculata were studied. Characteristic features of their individual classes were established [7]. The essential oil of C.siliquastrum L. was isolated from the flowers of C. siliquastrum and the oil was obtained with 0.08% yield, which was analysed by GC and GC/MS. Twenty-one compounds were characterized representing 83.9% of the total content of components detected. Studies of essential oil revealed heptadecane (26.2%), nonadecane (13.7%), pentadcecane (12.6%), linalyl acetate (8.5%), eicosane (6.5%), limonene (5.9%) as the main constituents [8]. The ability of C. siliquastrum L. leaves for the adsorption of Pb(II), Cu(II) and Ni(II) ions were studied. The maximum uptake for all metal ions was obtained in pH 4. The results obtained in this study indicated the highest drption ability of C.siliquastrum L. for Pb(II), among the tested metal ions [9]. The importance of wild plants where there are approximately 121 species have many uses medical and food [10]. Been studied the fabric and installation of the seed in the fruits of C. siliquastrum L .[11]**.** Show the nutritional value of the seeds and contains fatty acids [12] **.**

Objective of the study:

The Studies interested in oil of C.siliquastrum L. are very few especially Syrian C.siliquastrum L. is non-existent. Therefore, the aim of this study is to identify the seeds essential oil`s composition of

C.siliquastrumL.plant from Syria using GC-MS, based on their retention time and retention index in order to try to find a plausible explanation for the numerous midical applications of this plant.

2. Materials and Methods

2.1 Chemicals:

The solvent and chemicals used in the study were of analytical grad and purchased from Merck Chemical Company (Darmstadt, Germany), Sigma-Aldrich (Oakville, ON, Canada) and Fisher Scientific (Ottawa, Ontario, Canada).

2.2 Plant material(C.siliquastrum Seed):

The C.siliquastrum seed used was obtained from University Damascus Garden, Syria. The ripe seeds of C.siliquastrum plant were collected during the month of November 2013, The ripe seeds of C.siliquastrum, were air-dried in a shaded area at ambient temperature.

2.3 Extraction of the C.siliquastrum seeds oil(CSO):

The ripe seeds were ground by a blender and 10 grams of their powders were soaked and stirred in 70 ml petroleum ether for 48 hours at laboratory condition followed by filtration, then through Whatman No.1. The solvent was evaporated at lower temperature under reduced pressure in rotary flash evaporator to get the oil extracts were stored in dark vials at 4°C for future uses.

2.4 Extracts Analysis:

Analysis of extracts were carried out by GC-MS chromatography (GC-agilent 7986, indictor: inert-MS) in Atomic Energy Commission(AECS)- Damascus, Syria. This instrument was fitted with HP-5MS capillary column (30cm×0.25mm i.d., film thickness 0.25μm). The temperature injector and indictor 250 °C .The oven temperature program was 60-270°C (2.5°C per min.).. The identity of components was ascertained based on the spectra and compared with library and literature data. Also, the identification of each compound was confirmed by comparison of its retention index with those of authentic compounds.

2.5 Determination of the Physicochemical Properties of CSO:

A.O.A.C.[13] standard methods were used to determine the physicochemical properties of the oil, which includes the Free Fatty Acid value (FFA), Acid Value (AV), Saponification Value (SV), Iodine Value (IV), and Specific Gravity (SG). All tests were performed in triplicate.

2.6 The effect of physical factors on the amount of oil extracted using petroleum ether(PE):

a - the proportion of the amount of dry seeds in grams to the size of the solvent (ml): Soaking different amounts of weights (5 , 10 , 15g) of C. Siliquastrum seeds powder with fixed sizes (70 ml) PE To determine the best ratio for mixing the seeds powder (g) / PE(ml).

b- Soaking temperature with Stirring electric: Soaking fixed amount (10g) of C.SiliquastrumL. seeds powder with fixed sizes (70 ml) PE at different temperatures (30 , 50 , 65^{0}C) with Stirring electric to choose the best temperature for the extraction process.

c- Soaking time: Soaking amounts (10g) of C. Siliquastrum powder with fixed sizes (70 ml) PE at different times (90 , 180 , 270 , 360 sec and 24 hours) to choose the soaking time for the extraction of the oil.

3. Results and Discussion

The lipid content of dry matter of C.siliquastrumL studied is around 5.72%.The composition of oil extracts of CSO is shown in the table 1. The major components identified were: n-Hexadecanoic acid(3.07%), 9,12-Octadecadienoic acid (z,z)-,2,3-dihydroxypropyl ester(34.22%), Heneicosane(9.89%), Octadecane (18.05%), Phenyl-1,2-diamine,N,4,5-trimethyl-(9.26%), 9-Octadecyne(6.65%), 9,17 Octadecadienal,(Z)-(6.39%) and E-11-Hexadecenal(6.72%) in petroleum ether extract.

Table 1 Identified constitutents from the Petroleum Ether seed Extract of Ceries siliquastrumL.

Name of the compounds	Molecular formula	Retention time	Area%
n-Hexadecanoic acid	$C_{16}H_{32}O_2$	88.59	3.07
9,12-Octadecadienoic acid (z,z)-, 2,3-dihydroxypropyl ester	$C_{18}H_{34}O$	98.80	34.22
Heneicosane	$CH_3(CH_2)_{19}CH_3$	103.35	9.89
Octadecane	C_{18}-H_{38}	103.68	18.05
Phenyl-1,2-diamine,N,4,5-trimethyl-	$C_9N_{14}N_2$	106.29	9.26
9-Octadecyne	$C_{18}H_{34}$	109.90	6.65
9,17-Octadecadienal,(Z)-	$C_{18}H_{32}O$	110.96	6.39
E-11-Hexadecenal	$C_{16}H_{30}O$	112.38	6.72

The physicochemical properties of CSO: it is yellowish in color. It had a specific gravity of 0.923 at 25 ^{0}C which showed that it is less dense than water . The iodine value is 129.61 mgI_2/100 g. Oils are classified into the following types according to their iodine values: drying, semi drying and non-drying oils. Since the iodine value of CSO is higher than 100 it could be classified as semi-drying or drying oil. The high iodine value indicates that the oil has a high content of unsaturated fatty acids which is evident in the acid and free fatty acid values of 0.095 mg KOH/g and 0.042% respectively.

The saponification value of the CSO was (192.65 mg KOH/g) similar to the other typical seed oil such as sunflower, and corn oil [14]. this was lower than values for some common oils like palm oil (196-205 mg KOH/g), coconut oil (253 mg KOH/g) and palm kernel oil (247 mg KOH/g) [15]. However, this saponification value fall just below the range expected of some edible oils reported by [16] .The low saponification value is an indication that the oil may not be suitable for soap making, oil-based ice-cream and shampoos.

Table 2 Physiochemical properties of CSO

Parameter	Concentration
Specific Gravity	0.923 at 25 ^{0}C
Iodine Value	129.61 mgI_2/100g
Acid Value	0.95 mg KOH/g
Free fatty acid Value	0.042%
Saponification Value	192.65 mgKOH/g

The effect of physical factors on the amount of oil extracted using the (P.E.)

1- Rate dry seeds (g) to the size of the (P.E.) (ml): The table 3. shows different quantities (g) of C.siliquastrum L. seeds soaked in sizes uniform (70 ml) of (P.E).

Table 3.

Quantity (g) of seeds / size of the (P.E.). (70 ml)	Seed powder: PE	The amount of oil extracted (g)	Percentage
5 g/70 ml	1 : 14	0,21	4,2%
10 g/70 ml	1 : 7	0,47	4,7%
15 g/70 ml	1 : 4,7	0,61	4,1%

He results shown in the table 3. ,that the amount of oil extracted is proportional to the amount of seed powder of C.siliquastrumL., this is evident through the transition from (5g/70ml) to (10g/70ml), Increasing the amount of oil extracted from 0.21 to 0.47g which means it is more than doubled , but within certain limits the Best Mixing ratio (dry seed:solvent) , is (1:7)

2-The effect of soaking temperature with stirring Supply: The table 4 shows, that the effect of soaking temperature with stirring electric, when soak fixed amounts (10g) of seeds C.siliquastrum L. with sizes in equal (70 ml) of PE.

Table 4.

Temperature soaking (^oC)	The amount of oil extracted (g)
30	0,47
50	0,49
65*	0,52

***The boiling point of the solvent**

Table 4. shows the increasing amount of oil extracted in a sample by Rising temperature, but the increase becomes more at the boiling point of the solvent.

3 **Soaking time:** Table 5. Studing the effect of the soaking time(sec) of 10 g crushed seeds in 70 ml PE and quantity of extracted oil.

Table 5.

Soaking time (sec)	The amount of oil extracted (g)
90	0,44
180	0,47
270	0,48
360	0,49
24 hours**	0,57

****First experiment**

Table 5. shows that does not affect the apparent time of the growing amount of oil extracted from the seeds of C.siliquastrum L., but the biggest amount extracted extraction when the period of soaking time 24hours.

Conclusion :

In the present study the percentage of oil 5.72% and (8) chemical compounds have been identified from the petroleum ether extract of the seeds of C.siliquastrumL. by Gas Chromatogram-mass Spectrometry (GC-MS) analysis. Also the physicochemical properties of CSO and the effect of physical factors on the amount of oil extracted using petroleum ether.

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