

Chemical compositions of the essential oil and calculation the biophysicochemical coefficients of the components of *Hymenocrater longiflorus Benth.* of Iran

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Received 8 December 2010; revised 10 January 2011; accepted 12 January 2011.

ABSTRACT

The volatile constituents of the essential oil of *Hymenocrater longiflorus Benth.* growing wild in Kurdistan-Iran were investigated using the GC and GC/MS techniques. Fifteen compounds, representing twenty (97.03%) of the total oil were identified. The main components were: α -Pinene (22.47%), β -Caryophyllene (18.05%), β -Eudesmol (14.92%), α -Copaene (9.84%), γ -Elemene (6.79%), δ -Cadine-ne (6.13%), (-)Bornyl acetate (5.61%), α -Amorphene (3.84%), α -Fenchyl acetate (2.35%) and β -Pinene (2.07%). Some of the physicochemical properties like the logarithm of calculated Octanol-Water partitioning coefficients ($\log K_{ow}$), total biodegradation and (TB_d in mol/h and gr/h), water solubility (S_w , mg.L⁻¹ at 25°C) and median lethal concentration 50 (LC₅₀) were calculated for the 15 components of *Hymenocrater longiflorus Benth.*

Keywords: *Hymenocrater longiflorus Benth.*; Essential Oil Compounds; Hydro Distillation; α -Pinene; β -Caryophyllene; β -Eudesmol; α -Copaene; Octanol-Water Partitioning; Total Biodegradation; LC₅₀; Gas Chromatography; Mass Spectroscopy

1. INTRODUCTION

Hymenocrater genus has over 21 species in the world. [1,2] The *Hymenocrater longiflorus Benth.* materials of this study were collected from west of Iran (from the mountains area, altitude 1550-1800 m, in front of the Dezlie village-Marivan-Kurdistan province of Iran) at the

Jun. 2008. A voucher specimen has been deposited in herbarium of Research Center of Agriculture & Natural Resources, Sanandaj-Kurdistan, Iran (Herbarium ID: 447898-1).

This herb was firstly nominated by G. Bentham in 1848. The local name of *Hymenocrater longiflorus Benth.*, is *Soor-Halale (Sóór-HALALE)*. The other names of this herb are: *Gole Arvaneh-Avarmani* and *SoorSanduo*. The *Hymenocrater longiflorus Benth.* was utilized as a medicinal herb in local and traditional medicine (in Kurdistan). From the aerial parts of this herb in crude or baked form was utilized as an anti-inflammatory, sedative, anti-skin allergic reaction (for skin diseases and insect bite) by folks in local medicine.

The octanol-water partition coefficient (K_{ow}) is a measure of the equilibrium concentration of a compound between octanol and water that indicates the potential for partitioning in to soil organic matter (*i.e.*, a high K_{ow} indicates a compound which will preferentially partition into soil organic matter rather than water). This coefficient is inversely related to the solubility of a compound in water. The $\log K_{ow}$ is used in models to estimate plant and soil invertebrate bioaccumulation factors. This parameter is also used in many environmental studies to help determine the environmental fate of chemicals [3-5].

Biodegradation is usually quantified by incubating a chemical compound in presence of a degrader, and measuring some factors like oxygen or production of CO₂. The biodegradation studies demonstrate that microbial biosensors are a viable alternative means of reporting on potential biotransformation. However, a few chemicals are tested and large data sets for different chemicals need for quantitative structural relationship studies [6].

An LC₅₀ value is the concentration of a material in air

that will kill 50% of the test subjects (animals, typically mice or rats) when administered as a single exposure (typically 1 or 4 hours). Also called the median lethal concentration and lethal concentration 50, this value gives an idea of the relative acute toxicity of an inhalable material. Typical units for LC₅₀ values are parts per million (ppm) of material in air, µg (micrograms, 10⁻⁶ gram) per liter of air and milligrams (10⁻³ gram) per cubic meter of air [7].

2. ANALYTICAL METHODS

Dried aerial parts of *Hymenocrater longiflorus Benth.* were subjected to hydrodistillation for 5 hours using Clevenger-type apparatus to produce a yellow oil in 0.28% (w/w) yield. The essential oil of the aerial parts of *Hymenocrater longiflorus Benth.* was examined by GC/MS (GC: HP 6890, MS: HP 5973), column (HP5-MS, 30 m × 0.25 mm fused silica capillary column, film thickness 0.32 µm) by temperature program 60°C (3 min)-210°C (2 min) at the rate of 6°C/min (injection temperature 250°C, carrier gas: helium (with purity 99.999%), detector temperature 150°C, ionization energy in mass was 70 eV, mass range 10-300 amu, and scan time was 1 sec.

The list of identified components is presented in **Table 1**. The constituents were identified by comparing their MS spectra with those in computer library or with authentic compounds. The identifications were confirmed by comparison of their retention indices either with

those of authentic compounds or with data in the literature [8-10]. In the aerial parts of *Hymenocrater longiflorus Benth.* the major identified components and the relative amounts based on peak area were: α-Pinene (22.47%), β-Caryophyllene (18.05%), β-Eudesmol (14.92%), α-Copaene (9.84%), γ-Elementene (6.79%), δ-Cadinene (6.13%), (-) Bornyl acetate (5.61%), α-Amorphene (3.84%), α-Fenchyl acetate (2.35%) and β-Pinene (2.07%).

The calculated data of the Octanol-Water partitioning coefficients ($\log K_{ow}$) and the total biodegradation TB_d (in mol/h and gr/h), water solubility (S_w , mg.L⁻¹ at 25°C) and median lethal concentration 50 (LC₅₀) were calculated by the EPI-suit v4.00 package [11].

3. DISCUSSION

Some of the plants from genus *Hymenocrater* have been previously studied, but there are rare studies of the chemical composition of essential oil of plants from genus *Hymenocrater*. This study elaborates upon the volatile constituents of the essential oil of *Hymenocrater longiflorus Benth.* growing wild in Kurdistan-Iran were investigated by GC and GC/MS technique. As could see in **Table 1**, α-Pinene (22.47%), β-Caryophyllene (18.05%) (*trans*-Caryophyllene) and β-Eudesmol (14.92%) have the high percentages (about 55.44%) among the fifteen components that were identified. Some other components, *i.e.* α-Copaene (9.84%), γ-Elementene (6.79%), δ-

Table 1. Essential oil constituents, logarithm of calculated Octanol-Water partitioning coefficients ($\log K_{ow}$), total biodegradation and (TB_d in mol/h and gr./h), water solubility at 25°C (mg/L) and median lethal concentration 50 (LC₅₀) of *Hymenocrater longiflorus Benth.* of Iran.

No	Name of Compound	K. I.*	RT**	%	$\log K_{ow}$ ^a	Water Solubility at 25°C (mg/L)	LC ₅₀ (in mg/L or ppm)	Total Biodegradation mol/h × 10 ⁻⁵	gr./h × 10 ⁻²
1	α-Pinene	923	5.42	22.47	4.27 (4.44) ^a	4.071	1.928	7.70	1.05
2	β-Pinene	962	6.38	2.07	4.35 (4.16) ^a	7.061	1.642	6.00	0.81
3	α-Fenchyl acetate	1136	10.79	2.35	3.86	23.230	6.322	13.00	2.64
4	(-)Bornyl acetate	1236	13.21	5.61	3.86 (4.30) ^a	42.514	2.316	22.00	4.28
5	γ-Elementene	1243	13.37	6.79	7.09	0.575	0.005	18.00	3.71
6	α-Copaene	1314	14.96	9.84	5.71	0.161	0.086	12.00	2.47
7	<i>trans</i> -Caryophyllene	1351	15.81	18.05	1.54	302.6	40.5874	7.80	0.94
8	Valencene	1366	16.16	1.81	6.30	0.543	0.026	13.00	2.56
9	α-Humulene	1378	16.44	1.15	6.95	0.014	0.013	15.00	3.13
10	α-Amorphene	1394	16.81	3.84	6.19	0.512	0.033	11.00	2.30
11	β-Maaliene	1411	17.18	1.14	6.21	0.067	0.031	17.00	3.41
12	Germacrene-D	1426	17.50	0.85	6.99	0.819	0.006	18.00	3.68
13	δ-Cadinene	1433	17.63	6.13	6.32	0.808	0.025	12.00	2.38
14	unknown	1523	19.51	2.97	-	-	-	-	-
15	β-Eudesmol	1542	19.87	14.92	4.88	7.289	0.496	29.00	6.46

* Kovats index ** Retention time (min.)^a The values in parentheses are the experimental values for logarithm of Octanol-Water partitioning coefficients ($\log K_{ow}$). The other values were calculated by the EPI-suit v4.00 package.

Cadinene (6.13%), (-) Bornyl acetate (5.61%) are located in the second level of the concentration in the essential oil. Although, in accordance with the data in **Table 1**, some components *i.e.*, α -Amorphene (3.84%), α -Fenchyl acetate (2.35%), β -Pinene (2.07%), Valencene (1.81%), α -Humulene (1.15%), β -Maaliene (1.14%) and Germacrene-D (0.85%) have the medium up to low relative percentages, could see some important compounds with effects like mold and mildew preventive, microscopy, preservative and antioxidant. Biological and aroma effects of the main and minor compounds of the essential oil of *Hymenocrater longiflorus Benth.* are discussable in terms of their possible use in medicine, cosmetics and foods.

α -Pinene has the highest percentage (22.47%) in this herb. α -Pinene, is a natural bicyclic sesquiterpene that is a constituent of many essential oils. α -Pinene and β -Pinene. As the name suggests, both forms are important constituents of pine resin; they are also found in the resins of many other conifers, and more widely in other plants. Both are also used by many insects in their chemical communication system. α -Pinene and β -pinene are both produced from geranyl pyrophosphate, via cyclisation of linaloyl pyrophosphate followed by loss of a proton from the carbocation equivalent [12].

β -Pinene with 12.06% in this herb was utilized as an intermediate for perfumes and flavorings. It also occurs naturally in rosemary, parsley, dill, basil, yarrow, and rose. α -Pinene with 9.94% in this herb was utilized as solvent for protective coatings, polishes and waxes, synthesis of camphene, comphor, geraniol, terpin hydrate, terpineol, synthetic pine oil, terpene esters and ethers, lube oil additives, flavoring and odorant. It is also found in the essential oil of rosemary (*Rosmarinus officinalis*).

β -Caryophyllene or *trans*-Caryophyllene is one of the chemical compounds that contribute to the spiciness of black pepper. β -Caryophyllene, is a natural bicyclic sesquiterpene that is a constituent of many essential oils, especially clove oil, the oil from the stems and flowers of *Syzygium aromaticum* (cloves), the essential oil of hemp *Cannabis sativa*, and rosemary *Rosmarinus officinalis*. It is usually found as a mixture with isocaryophyllene and α -humulene, a ring-opened isomer. Caryophyllene is notable for having a cyclobutane ring, a rarity in nature [12,13].

It was found that β -eudesmol, a sesquiterpenol constituent of Chinese herb antagonized organophosphate-induced lethal toxicity by reversing the neuromuscular failure and reducing the occurrence of convulsions [13].

Its possible antiepileptic action was further explored in electroshock seizure mice *in vivo* and in high potassium treated rat hippocampal slices *in vitro*. At a dose with little effect on the motor activity, β -eudesmol pre-

vented the convulsions and lethality induced by maximal electroshock but not those by pentylenetetrazol or picrotoxin. At sub effective doses, β -eudesmol and phenytoin showed additive effect in preventing electroshock seizures. Extracellular recording of field potentials in CA1 pyramidal layer of hippocampal slices showed that β -eudesmol reduced the high potassium (8.5 μ M)-induced electrographic seizure activity. The potential of β -eudesmol to serve as an antiepileptic or a conjunctant in phenytoin therapy is suggested [14].

α -Copaene, a potent attractant for male Mediterranean fruit flies. *Ceratitis capitata* is found as a minor component in the essential oils of various plant species, including its hosts such as orange, guava, and mango. Despite the specific attraction of male flies and the wide distribution of the compound in host plants, the biological significance of α -copaene remains unknown [15]. Chemically, the copaenes are tricyclic sesquiterpenes. The molecules are chiral, and the α -copaene enantiomer most commonly found in higher plants exhibits a negative optical rotation of about -6° . The rare (+) α -copaene is also found in small amounts in some plants. It is of economic significance because it is strongly attracting to an agricultural pest, the Mediterranean fruit fly *Ceratitis capitata* [15]. Bornyl acetate is a constituent of some essential oils. It has been used in aromatic preparations in the treatment of coughs.

Chemically, the cadinenes are bicyclic sesquiterpenes. The term "cadinene" has sometimes been used in a broad sense to refer to any sesquiterpene with the so-called cadalane (4-isopropyl-1, 6-dimethyldecahydronaphthalene) carbon skeleton. Because of the large number of known double-bond and stereochemical isomers, this class of compounds has been subdivided into four subclasses based on the relative stereochemistry at the isopropyl group and the two bridgehead carbon atoms. [13-15].

The acid-catalyzed cyclization of Germacrene-D to give cadinane and selinane sesquiterpenes has been computationally investigated using both density functional (B3LYP/6-31G*) and post Hartree-Fock (MP2/6-31G**) *ab initio* methods by Setzer in 2008 [16]. It is generally observed that essential oils containing large concentrations of the sesquiterpene germacrene D are typically accompanied by cadinane and muurolane sesquiterpenoids [16-22] and germacrene-D has been suggested to serve as biogenetic precursor to a number of different sesquiterpenoid skeletons [16,23,24]. Bülow and König have demonstrated that germacrene-D readily undergoes acid-catalyzed cyclization to give cadinane, muurolane, and amorphane sesquiterpenes [16,25]. In addition, there is concern that skeletal rearrangements may occur during the hydrodistillation of plant materials

to obtain essential oils [16,26-29]. Bülow and König [16,24] had found that acid-catalyzed cyclizations of germacrene D generally give a preponderance of δ -cadinene, followed by γ -cadinene, and lesser amounts of α -cadinene. The abundant δ -cadinene is consistent with the *ab initio* calculations, but the calculated energies of α -cadinene and γ -cadinene are not in agreement with the experimental results, and would predict α -cadinene to be more abundant than γ -cadinene. In addition, γ -cadinene is more abundant in these essential oils than α -cadinene. α -Cadinene has been shown to undergo acid-catalyzed rearrangement to give β -cadinene [16,30], which in turn, has been found to isomerize to ω -cadinene [16,30-32], in agreement with the calculated energies; β -cadinene and ω -cadinene are lower in energy than α -cadinene by 1.88 kcal/mole and 2.90 kcal/mol, respectively. Bülow and König [16,24] reported that ω -cadinene can be formed from δ -cadinene in a 1.4:1 ratio, consistent with the nearly equal calculated energies.

It is reported and accepted that the toxicity property of organic compounds can be predicted on the basis of the $\log K_{ow}$ [7]. Total biodegradation (TB_d) is another useful and important factors in chemical and biochemical studies [33]. The LC_{50} value is called the median lethal concentration and lethal concentration 50, this value gives an idea of the relative acute toxicity of an inhalable material. One of the other important physicochemical factors of compounds is water solubility (S_w , $mg.L^{-1}$ at $25^\circ C$). In accordance with the calculated data of the components 1-15 (see **Table 1**) γ -Elemene, Germacrene-D and α -Humulene have the highest $\log K_{ow}$, among the components 1-15 (7.09, 6.99 and 6.95, respectively). α -Humulene and α -Copaene have the lowest water solubility (S_w , $mg.L^{-1}$ at $25^\circ C$). β -Caryophyllene has the highest LC_{50} (mg/L) and γ -Elemene, Germacrene-D and α -Humulene are three components with lowest LC_{50} (0.005, 0.006 and 0.013, respectively). The total biodegradation (TB_d) of β -Eudesmol among 1-15 is the highest and for β -Pinene is the lowest amount (in mol/h and gr/h). The total biodegradation (TB_d) for α -Pinene (22.47%) (The highest percentage component) is calculated: 7.70 (mol/h) and 1.05 (gr/h). In **Table 1**, the values in parentheses are the experimental values for logarithm of Octanol-Water partitioning coefficients ($\log K_{ow}$). The other values were calculated by the EPI-suit v 4.00 package. For the other items there were not available values from data base [11].

Perhaps, the high densities of the main compounds give some biological activities to the essential oil or to this herb. Although no records of toxicity have been found for this plant, it belongs to a family that includes many poisonous plants so some caution is advised [34,35].

4. CONCLUSIONS

Hymenocrater longiflorus Benth., one of the *Hymenocrater* genus, was collected from Kurdistan area in Iran. It is utilized as the medicinal herb for the various purposes in local and traditional medicine by folks in Kurdistan-Iran. Fifteen components in the essential oil of *Hymenocrater longiflorus Benth.* representing fourteen of the total oil were identified by GC and GC/MS technique. In this herb, α -Pinene, *trans*-Caryophyllene (β -Caryophyllene) and β -Eudesmol have the most percentages among compounds of the essential oil. Some other components α -Copaene (9.84%), γ -Elemene (6.79%), δ -Cadinene (6.13%), (-) Bornyl acetate (5.61%) are located in the second level of the concentration in the essential oil. α -Amorphene (3.84%), α -Fenchyl acetate (2.35%), β -Pinene (2.07%), Valencene (1.81%), α -Humulene (1.15%), β -Maaliene (1.14%) and Germacrene-D (0.85%) have the medium up to low relative percentages among 1-15. Some of the other physicochemical data, *i.e.* octanol-Water partitioning coefficients ($\log K_{ow}$), the total biodegradation TB_d (in mol/h and g/h), water solubility (S_w , $mg.L^{-1}$ at $25^\circ C$) and median lethal concentration 50 (LC_{50}) were calculated by the EPI-suit v4.00 package.

5. ACKNOWLEDGEMENTS

The authors gratefully acknowledge the colleagues in Chemistry Department of The University of Queensland-Australia, for their useful suggestions. We are also thankful from the Research Council of Science and Research Campus of Islamic Azad University and Arak branch of I. A. University.

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