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Essential oil composition of *Cleome heratensis (Capparaceae)* at different growing stages

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Abstract

In this study, the essential oil content in the aerial parts of *Cleome heratensis* which is growing in the east of Iran was investigated. The aerial parts of the plant were collected from the late August to October 2014, for a total of four harvests (S_1-S_4). Essential oils were isolated by hydro-distillation. The oils were analyzed by GC-FID and 30 constituents, representing 84.93-92.9 % of the total compositions of the oil, were identified. The major components of the essential oil in the four harvests were hexanal (7.57%-33.96%), α-phellandrene (6.08% -13.17%), α-farnesen (7.54%-10.9%), methyl eugenol (6.74%-8.31%), eugenol (3.94%-7.4%), verbenone (3.98%-6.24%), myrcene (1.54%-5.75%), hexadecane (2.34%-4.82%), linalool (1.59% -3.53%) and α-humulene (1.01%-1.93%). The findings indicated that the main component groups of oil are monoterpenoids (11.83–19.29%), oxygenated monoterpens (10.34–16.96%), sesquiterpenoids (10.67–20%) and oxygenated sesquiterpenes (0–4.34%) which are obviously increased during the growing stages. Monoterpenoids were higher during the late flowering stage (S_3), but oxygenated monoterpens were observed to be slightly lower in this stage. Monoterpens are slightly higher during development stages.

Keywords: *Cleome heratensis*; essential oils; hydro-distillation; *Capparaceae*; different growing stages.

Introduction

Essential oils and their chemical components as secondary metabolites are important for the growth and development of plants. These compounds play a vital role in protecting plants against a broad range of organisms such as bacteria, fungi, viruses, protozoa as well as insects and plants [1]. Essential oils have been also used as therapeutic agents since ancient times. Some of them have been methodically proven to possess medicinal activities including anti-inflammatory [2], anti-viral [3], anti-tumor [4], anti-hyperglycemic [5], anti-nociceptive [6] and anti-carcinogenic activities [7]. Several studies demonstrated that plant secondary metabolites differ between species. Environmental conditions such as temperature, day length and light influence the quantity of the composition of essential oils [8]. These conditions change during the vegetative period leading to a layout of seasonal variation in plant metabolite which is generally repeated every year.

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Cleome heratensis (*C. heratensis*) as a flowering plant is a genus ofthe family *Cleomaceae*. This family has about 170 species of herbaceous annual or perennial plants and shrubs [9]. *C. heratensis* is an annual herbaceous plant growing in warm temperate areas during summer and autumn. The germinating, flowering and fruiting stage of this plant are in May, September and October, respectively [10]. Some species has been traditionally known for its different medicinal properties such as headache, earache, skin diseases and rheumatism in traditional medicine [11]. In addition, some species are commonly known as spider flowers, spider plants, spider weeds or bee plants [12-13]. To the best of our knowledge, the chemical composition of the essential oil of *C. heratensis* has not been studied previously. Therefore, we were prompted to investigate the chemical composition of the essential oils isolated from the aerial parts of *C. heratensis* at different developmental stages. These results defined the optimum harvesting time of this plant for related industries.

Experimental

Plant material

Aerial parts of *C. heratensis* (Figure 1) were harvested at four developmental stages of growth (vegetative (S_1), flowering (S_2), fruiting stages (S_3) and seeding stages (S_4), respectively) in the wild habitat in plains around Birjand, South Khorasan Province in August, early September, late September and October 2014, respectively. Specimen of the sample was identified in the herbarium of agriculture institute, Mashhad, Iran.

Figure 1. Aerial parts of *C. heratensis*

Isolation of the essential oils

Dried aerial parts (30 g) of each growing stage of *C. heratensis* were subjected to hydro-distillation method using a Clevenger-type apparatus. The essential oils were separated from water, dried over anhydrous sodium sulfate and stored in sealed amber glass vials at a temperature of -18°C for further analysis.

Gas chromatography (GC) analysis

The volatile oil samples analysis was performed on a Shimadzu GC-17A gas chromatograph equipped with a FID detector and a fused silica capillary column DB5 (5% phenyl polysiloxane, 30 m × 0.25 mm, film thickness 0.25 µm). The injector and detector operated at 240 and 280°C, respectively. The oven temperature was programmed to kept at 60 °C for 1 min (Isothermal), ramped from 60 °C to 150 °C at a rate of 3 °C min^{-1}, increased by 5 °C $min^{-1}$to 210 °C and then held constant for 10 min (Isothermal). Helium was used as a carrier gas at a flow rate of 0.9 mL/min.

Identification of components

Identification of the essential oils constituents was performed on the

basis of retention indexes (RIs) using a homologous series of n-alkanes (C_8-C_{28}) under identical experimental conditions. The RIs was calculated for each constituent and compared to the literatures. The oil compositions, retention index and their percentage are summarized in Table 1.

Results and discussion

Variations in the quality and quantity of essential oils plants are significantly influenced by primary parameters (chemo type, genotype, genetic structures, different anatomical plant parts, organogenesis) and growth stages [14]. In addition, external factors such as ecological and environmental conditions including season, climatic and soil conditions [15], could effect on the quality and quantity of essential oils. The essential oil of *C. heratensis* was extracted by hydro-distillation using a Clevenger-type apparatus and analyzed at different phonological stages. As shown in Table 1, thirty compounds were identified representing 84.93%–92.9% of the essential oils in four developing stages (S_1-S_4). The physiological stages could significantly affect on the composition of the essential oil (Table 1). The major constituents of the oils were hexanal (7.57%-33.96%), α-phellandrene (6.08%–13.17%), α-farnesen (7.54%-10.9%), methyl eugenol (6.74%-8.31%), eugenol (3.94%-7.4%), verbenone (3.98%-6.24%), myrcene (1.54%-5.75%), hexadecane (2.34%-4.82%), linalool (1.59%-3.53%), and α-humulene (1.01%-1.93%). The main components of oils are monoterpenoids (11.83–19.29%), oxygenated monoterpens (10.34–16.96%), sesquiterpenoids (10.67–20%) and oxygenated sesquiterpenes (0–4.34%) which are obviously increased during the growing stages. The amount of monoterpenoids was higher during the late flowering stage (S_3), but the amount of oxygenated monoterpens was observed to be slightly lower in this stage. Monoterpens are slightly higher during development stages. The amount of sesquiterpenoids was recorded to be higher during vegetative and early flowering stages (S_1, S_2).

The amount of constituents such as hexanal, α–phellandrene and linalool were recorded to be increased during the growing stages. However, myrcene, verbenone, eugenol, methyl eugenol, α–humulene, α-farnesen and hexadecane were decreased during the growing stages. Some components were specific to one stage of growth (S_1& S_2) including fenchone (3.84%), β-sinensal (4.34%), carotol (2.66%), elemicin (2.95%). Camphene (1.96%), heptanal (1.85%) and sabinene (1.74%) are detected only in the stages of S_3 and S_4. As shown in the results, the harvesting of *C. heratensis* at late stages of growth (S_3,S_4) could produce essential oil that is rich in hexanal (33.96%), α–phellandrene (14.65%) and linalool (4.43%). Hexanal was found to be the major aldehyde of the total volatiles of the oils that is known by its antifungal activity [16]. Linalool is used in cleaning agents including soaps, detergents, shampoos and lotions. It is also used as a chemical intermediate in the synthesis of valuable substances such as vitamin E.

The harvesting of this plant at primary stages of development (S_1, S_2) could give us essential oil which is rich in β-myrcene (5.75%), verbenone (6.24%), eugenol (7.4%), methyl eugenol (8.82%), α–humulene (1.93%), α–farnesen (14.1%) and hexadecane (4.82%). Eugenol has antiseptic and anaesthetic effect [17] while α–humulene [18] and β–Myrcene [19] have anticancer effect.

Table 1. Chemical compositions of the essential oils of *C. heratensis* at different stages

NO	RI [a]	MF [b]	Compound	Area (%)			
				Vegetative Stage	Pre-flowering	Flowering Stage	Fruiting Stage
1	800	$C_6H_{12}O$	Hexanal	7.57	15.83	21.58	33.96
2	865	$C_6H_{14}O$	1-Hexanol	t_r	t_r	t_r	0.75
3	901	$C_7H_{14}O$	Heptanal	t_r	t_r	t_r	1.85
4	953	$C_{10}H_{16}$	Camphene	t_r	t_r	1.19	1.96
5	969	$C_{10}H_{16}$	Sabinene	t_r	t_r	1.74	1.55
6	*990*	$C_{10}H_{16}$	Myrcene	5.75	1.49	1.71	1.54
7	1002	$C_{10}H_{18}$	α-phellandrene	6.08	10.8	14.65	13.17
8	1083	$C_{10}H_{16}O$	Fenchone	3.84	t_r	t_r	t_r
9	1095	$C_{10}H_{18}O$	Linalool	1.59	2.94	4.43	3.53
10	1118	$C_{10}H_{18}O$	Cis-p-menth-2-en-1-ol	2.11	t_r	t_r	t_r
11	1204	$C_{10}H_{14}O$	Verbenone	6.24	5.43	4.71	3.98
12	1360	$C_{10}H_{12}O_2$	Eugenol	7.4	5.93	5.69	3.94
13	1371	$C_9H_{10}O_3$	Methyl-4-methoxy benzoate	1.8	1.52	1.41	1.1
14	1377	$C_{12}H_{20}O_2$	Geranyl acetate	2.89	2.11	1.6	1.22
15	1393	$C_{11}H_{16}O$	Cis-jasmone	1.84	2.62	t_r	t_r
16	1404	$C_{11}H_{14}O_2$	Methyl eugenol	8.31	8.82	8.51	6.74
17	1417	$C_{14}H_{20}O_2$	cis threo-davanafuran	0.74	0.67	0.61	0.51
18	1420	$C_{15}H_{24}$	β-caryophyllene	1.41	1.5	1.17	0.51
19	1424	$C_{13}H_{20}O$	α-lonone	t_r	t_r	t_r	0.9
20	1434	$C_{13}H_{22}O$	Neryl aceton	2.4	2.41	1.93	1.61
21	1452	$C_{15}H_{24}$	α-humulene	1.93	1.8	1.34	1.01
22	1461	$C_{15}H_{24}$	β-farnesen	1.00	0.97	0.79	0.6
23	1476	$C_{15}H_{24}$	Alloaromadendrene	1.79	1.63	1.24	1.01
24	1504	$C_{15}H_{24}$	α-farnesen	10.9	14.1	9.4	7.54
25	1546	$C_{12}H_{16}O_3$	elemicin	2.95	t_r	t_r	t_r
26	1555	$C_{15}H_{24}$	Germacrene B	1.13	t_r	t_r	t_r
27	1566	$C_{15}H_{24}O_2$	Davanone B	2.67	t_r	1.47	1.58
28	1600	$C_{16}H_{34}$	Hexadecane	4.82	1.7	2.42	2.34
29	1606	$C_{15}H_{26}O$	Carotol	t_r	2.66	t_r	t_r
30	1699	$C_{15}H_{22}O$	β-sinensal	4.34	t_r	t_r	t_r
Total				**91.5**	**84.93**	**87.59**	**92.9**

t_r = trace < less than 0.5%. [a] Relative retention indices on DB5 capillary column in reference to n-alkanes; [b] molecular formula of the component.

The constituents of the essential oils divided into different classes including terpenoids, aromatics, aldehydes, ketones, alcohols and alkenes (Table 2). As shown in Table 2, the amount of terpenoids and aromatics decreased while aldehydes showed increasing in the amounts during developmental stages. Other constituents of the essential are slightly changed during the phonological stages (Table 2 & Figure 2).

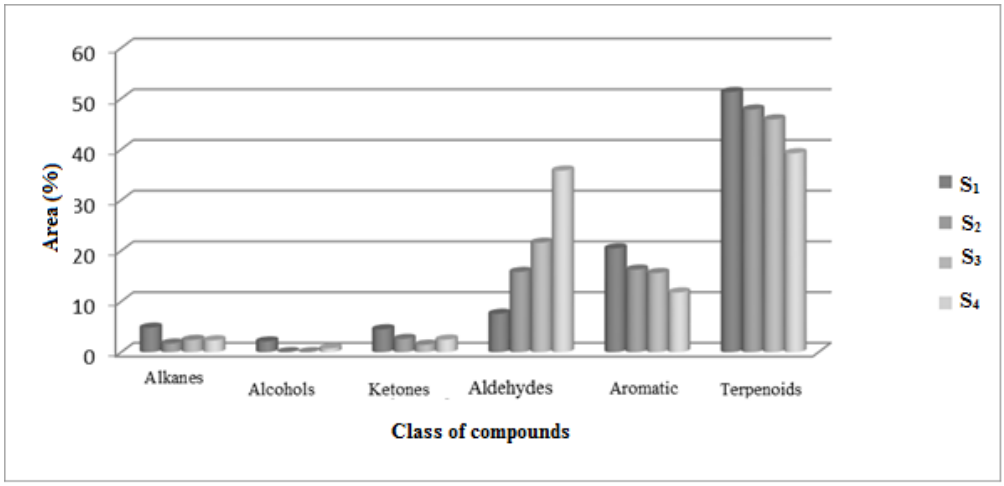

Figure 2. Repartition of compound classes in the whole aerial part of *C. heratensis* in different stages of growth

Table 2. Classification of essential oils of (*C. heratensis*) at different growth stages

Group compounds	**Area [a] (%)**			
	S_1	S_2	S_3	S_4
Hydrocarbons:				
Alkanes, Alkenes	4.82	1.7	2.42	2.34
Alcohols	2.11	-	-	0.75
Ketones	4.51	2.62	1.47	2.48
Aldehydes	7.57	15.83	21.58	35.81
Aromatic compounds	20.46	16.27	15.61	11.78
Terpenoids	51.29	47.84	45.9	39.23
Monotrrpene hydrocarbons	11.83	12.29	19.29	18.22
Oxygenated Monotrrpene	16.96	12.89	12.67	10.34
Sesquiterpene hydrocarbons	18.16	20.00	13.94	10.67
Oxygenated Sesquiterpene	4.34	2.66	-	-
Other	0.74	0.67	0.61	0.51
Total	**91.5**	**84.93**	**87.59**	**92.9**

[a]Chemical composition percent of essential oils

The chromatograms of the essential oils of *C. heratensis* with labelled major peaks are shown in Figure 3.

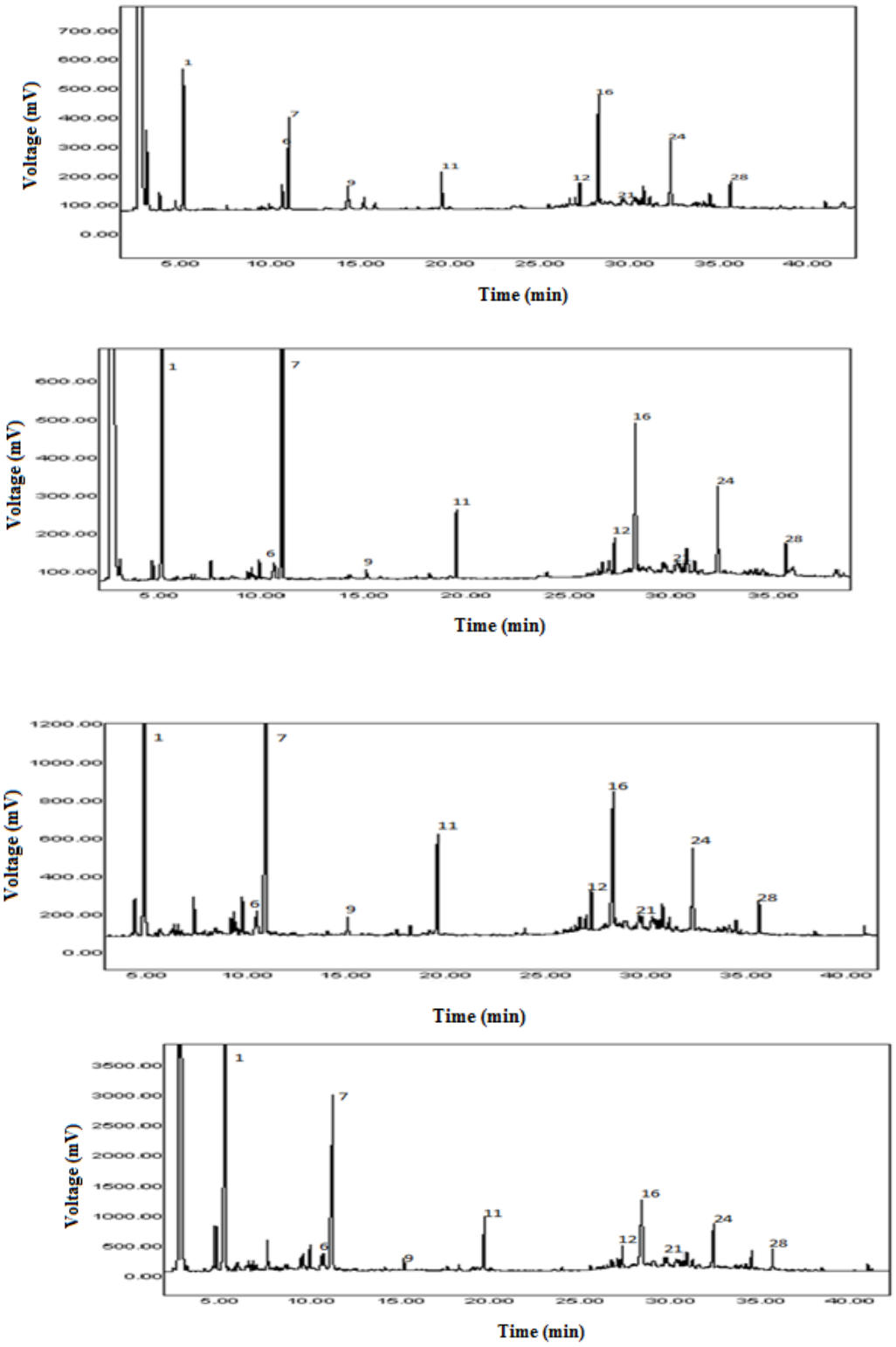

Figure 3. Chromatograms of *C. heratensis* volatile oils of whole aerial parts. (a) Aug 2014 (S_1), (b) Early Sep 2014 (S_2), (c) late Sep 2014 (S_3), (d) Oct 2014 (S_4)

Figure 3a-d clearly explained that the composition of essential oil is strongly dependent on the growth stage of the plant. Therefore, harvesting time is one of the most important factors that influence on the oils quality. This finding are in agreement with the results reported in the literature which demonstrated that the concentration of some essential oil components increases or decreases at the use of other components during the growing stage of plant's life [20-23].

Conclusion

The essential oil of aerial parts of *C. heratensis* was analyzed by GC–FID and a total 30 volatile compounds which are accounted for 84.93- 92.9% of the oils composition were identified. The essential oils are rich in terpenoids. The amount of terpenoids is decreased during developmental stages while aldehydes showed increasing in the amounts during developmental stages. Some compounds such as monoterpenoids (β–Myrcene), sesquiterpenoids (α–humulene) and hexanal have less portion in the essential oils. Due to the existence of the valuable compounds such as α–humulene and β–Myrcene, the essential oil of *C. heratensis* may have anticancer and antifungal effects.

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