Estimation of some active compounds in members from Cynareae tribe (Asteraceae) growing in central and northern Iraq

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Abstract. In this study, 8 monoterpenes compounds (α-pinene, Camphor, Cineole, Limonene, Linalool, Myrcene, Sabinen and Terpinene) were identified in volatile oils or essential oils (EOs) from leaves and flowerheads, and total carotenoids from the flowerheads of 7 Cynareae taxa (Asteraceae) growing in Iraq were studied. Linalool is the main ingredient in all leaves of the studied species (ranging 12.3 - 13.4%), while the proportion of myrcene ranged (15.0 - 26.8%) in the flowerheads in the rest of the studied species. Cineole in both plant parts exhibited the lowest amounts in all studied species. The flowerheads showed higher EOS contents than the leaves. In all species studied Centaurea Hyalolipids showed the highest EOS content compared to the other studied species. Centaurea benedicta showed the highest carotenoid content (15.96 mg/L) while Centaurea bruguieriana exhibited the lowest (8.88 mg/L).

1. Introduction

Asteraceae (Compositae) is the largest angiosperm family on earth with 13 subfamily, 45 tribes, 1600-1900 genera and more than 34000 species with worldwide distribution including Antarctica[1-5]. Its also the largest family in Iraq, represented by 123 genera and about 433 species [6-8]. Cynareae is one of the largest tribes of the 43 described in the family [9] and the subfamily Carduoideae.[10 ] Most of them are commonly known as thistles the best known genera are Carduus, Cirsium, Centaurea, Cousinia, Jurinea, and Saussurea [11]. It is one of the clans with a wide spread in central and northern Iraq [6,7]. Information on the active compounds of the most members of this tribe is still very limited [12-13]. Sesquiterpene lactones, lignin compounds, flavonoids and their glycosides are reported as the main characteristic bioactive metabolites in Cynareae [14-17]. However, Very few Reports are available on the phytochemicals of Asteraceae taxa including those of Cynareae occurring in Iraq. These Asteraceae metabolites exhibit several biological activities such as, antifungal, antibacterial, anti-protozoan parasites, anti-inflammatory, antioxidant [18-19]. This study reports for the first
time information on the essential oils and total carotenoids of seven wild Cynareae taxa in central and northern Iraq.

2. Materials and methods

2.1. Plant materials

Plant samples were collected from different districts in Iraq (Kirkuk, Salah ad Din, Sulaymaniyah) Governorate at flowering stage during November 2018 - December 2019. Plant samples were identified according to relevant literatures, keys and flora of Iraq and bordering countries[20-24] and were confirmed by the National Herbarium of Iraq, Kurdistan Botanical Foundation and Macrogen Inc / Korea. The identified samples were kept in Biology Department, College of Education for Pure Sciences, Tikrit University, Iraq.

2.2. Preparation of essential oil by hydrodistillation

Fresh leaves and flowerheads were air dried at 35-40 °C, till constant well. Each sample was distilled for 2-3 hours using a Clevenger system, samples were dried with anhydrous sodium sulfate and kept in vials at 4 °C until use. [25].

2.3. Determination of chemical content

EO, in the leaves and flowerheads of the studied species were determined by using a Gas chromatography device, and the concentration of each compound was calculated by comparing the area of the sample package with the area of the standard so that was injected under the same conditions and the process was repeated on all samples of samples, and the percentage of active compounds was estimated depending on area of the model, according to the following equation[25]:

\[
\text{Compound (\%)} = \frac{\text{standard substance concentration} \times \text{sample area}}{\text{area of a standard} \times \text{dilution factor}} / \text{sample size}
\]

2.4. Total Carotenoids

40 mg of fresh leaves were taken in 10 ml of 80% (v/v) acetone and kept in refrigerator for 4-5 days. After 5 days the supernatant was collected & the intensity of colour was measured at 480, 510 and 652 nm on UV-VIS 118 spectrophotometer [26]. mg/ l was calculated by using the following formula:

\[
\text{Total Carotenoids} = 7.6 \times (\text{O.D. at 480 nm}) - 1.49 \times (\text{O.D. at 510 nm})
\]

3. Results and Discussions

Table 1 and Figure 1 are shown The components of the EO, of 7 wild Asteraceae species studied (Carduus pycnocephalus, Carthamus lanatus, Centaurea benedicta, Centaurea bruguieriana, Centaurea hyalolepis, Cousinia wesheni, Echinops Spinosisimus) from leaves and flowerheads. These components belong to monoterpene group (α-pinene, Camphor, Cineole, Limonene, Linalool, Myrcene, Sabinen and terpinene). Monoterpenes and other secondary metabolites are predominant group in wild plants including Asteraceae taxa as compounds involved in plant protection against environmental stresses microbes and herbivores, and in other ecological functions attraction of insects for pollination [27,28]. In all studied species,
the percentages of the identified components of the EO, from the flowerheads are higher than those from the leaves and the major components of the EO, from the leaves are α-pinene (10.3-10.5%), camphor (9.5-10.3%), Cineole (0.7-0.8%), Limonene (1.1-1.3%), Linalool (12.3-13.4%), myrcene (7.3-8.4%), Sabinen (7.4-8.5%), and Terpinene (0.7-1.3%) while the main components of EO, for the flowerheads include α-pinene (12.5-14.3%), camphor (9.6-14.5%), Cineole (0.8-1.2%), Limonene (7.4-9.8%), Linalool (12.8-16.5%), myrcene (15-26.8%) and Sabinen (12.5-14.2%) and Terpinene (1.8-4.0%). These results are in agreement with previous reports that mentioned the effect of plant parts on the composition of the EO, of Asteraceae species [29-31]. Cineole from both leaves and flowerheads exhibited the lowest percentages in all plant species investigated. Except for E. Spinosisssmus, which had the lowest EO, leaves of Terpinene. Whereas, Linalool and Myrcine had the highest percentage of EO, in leaves and flowerheads, respectively, in the studied species. Carduus pycnocephalus also had the highest myrcine concentration, while Centaurea briguieriana showed the highest Linalool. While the two species, Carhamus lanatus and Centaurea hyalolepis, were the richest in volatile compounds compared to the rest of the studied species (Table 1), that the species differ at the level of a single genus in their EO content, as C. briguieriana recorded the highest total content of oils in the leaves with 52.8%, while C. benedicta recorded the lowest total content of oils in the leaves at 50.7% while C. hyalolepis the highest percentage of total oils content in flowerheads was 93.1%, while the total content of oils in C. benedicta, C. briguieriana and Cousinia wesheni was very close (87.9, 87.6 and 87.8%). C. hyalolepis had the highest total content of oils, 144.9% compared to the rest of the Cynareae tribe, while C. lanatus recorded the lowest total oil content of 133.4% compared to the rest of the species in which the concentration of oily compounds varies between the highest and lowest concentrations of these compounds. The total carotenoid content of the studied taxa ranged between 8.88 mg/ l in Centaurea briguieriana and 15.69 mg/ l in Centaurea benedicta (Table 2). However this study is the first report on essential oils and total carotenoids of some Cynareae taxa occurring in Iraq.
### Table 1. Composition of EO from leaves and flowerheads of Cynareae taxa

| Species                  | a-pinene | Camphor | Cineole | Limonene | Linalool | Myrcene | Sabinen | Terpinene | TOTAL Leaves* V C | TOTAL flower heads* V C | TOTAL Oil |
|--------------------------|----------|---------|---------|----------|----------|---------|---------|-----------|------------------|--------------------------|-----------|
| Carduus pycnocephalus    | 10.3     | 14.3    | 10.3    | 12.5     | 0.7      | 1.1     | 1.2     | 7.6       | 12.3            | 12.8                     | 8.4       | 26.8     | 8.2     | 13.8    | 11.1    | 19       | 52.5 | 90.8 | 143.3 |
| Carthamus lanatus        | 10.5     | 13.5    | 9.5     | 14.5     | 0.9      | 1.2     | 1.2     | 7.6       | 12.8            | 14.5                     | 7.4       | 15.0     | 8.4     | 13.4    | 1.2     | 1.8      | 51.9 | 81.5 | 133.4 |
| Centaurea benedicta      | 10.3     | 13.2    | 9.7     | 9.7      | 0.8      | 0.8     | 1.3     | 8.2       | 12.7            | 15.8                     | 7.4       | 23.8     | 7.4     | 13.2    | 1.1     | 3.2      | 50.7 | 87.9 | 138.6 |
| Centaurea bruguieriana   | 10.4     | 13.5    | 9.6     | 9.6      | 0.8      | 0.8     | 1.2     | 7.4       | 13.4            | 16.9                     | 8.3       | 24.1     | 7.8     | 12.5    | 1.3     | 2.8      | 52.8 | 87.6 | 140.4 |
| Centaurea hyalolepis     | 10.4     | 12.5    | 9.5     | 13.4     | 0.8      | 1.1     | 1.1     | 7.7       | 13.0            | 14.8                     | 8.2       | 25.8     | 7.6     | 14.2    | 1.2     | 3.6      | 51.8 | 93.1 | 144.9 |
| Cousinia wescheni        | 10.3     | 13.8    | 10.3    | 10.3     | 0.7      | 1.1     | 1.3     | 9.8       | 12.6            | 16.5                     | 7.3       | 20.3     | 7.6     | 12.5    | 0.8     | 3.5      | 50.9 | 87.8 | 138.7 |
| Echinops Spinosismus     | 10.5     | 14.2    | 10.2    | 10.2     | 0.8      | 1.0     | 1.1     | 7.6       | 12.4            | 14.8                     | 7.5       | 19.1     | 8.5     | 13.3    | 0.7     | 4.0      | 51.7 | 84.2 | 135.9 |
Carduus pycnocephalus - leaves
Carduus pycnocephalus - flowerheads

Carthamus lanatus - leaves
Carthamus lanatus - flowerheads

Centaurea benedicta - leaves
Centaurea benedicta - flowerheads
Centaurea bruguieriana - leaves
Centaurea hyalolepis - leaves
Cousinia wesheni - leaves

flowerheads - Centaurea bruguieriana
leaves - Centaurea bruguieriana
flowerheads - Centaurea hyalolepis
leaves - Centaurea hyalolepis
flowerheads - Cousinia wesheni
leaves - Cousinia wesheni
Echinops spinosissmus - leaves
Echinops spinosissmus - flowerheads

Fig1: compounds EO, from leaves and flowerheads of Cynareae taxa

Table2: Total carotenoids in flowerheads of Cynareae taxa

| No. | Species                  | Total carotenoids. mg/l |
|-----|--------------------------|-------------------------|
| 1   | Carduus pycnocephalus    | 13.08                   |
| 2   | Carthamus lanatus        | 14.69                   |
| 3   | Centaurea benedicta      | 15.69                   |
| 4   | Centaurea bruguieriana   | 8.88                    |
| 5   | Centaurea hyalolepis     | 11.41                   |
| 6   | Cousinia wesheni         | 13.06                   |
| 7   | Echinops Spinosisssmus   | 14.59                   |

4. Conclusion

This study showed for the first time the presence of 8 monoterpe compounds in EO, from leaves and flowerheads of 7 wild Cynareae taxa (Asteraceae) growing in central and northern Iraq. Linalool and myrcene are the predominant volatile constituents in EO of these taxa. Total carotenoids in studied plant species ranged between 8.88 mg/l in Centaurea bruguieriana and 15.69 mg/l in Centaurea benedicta.
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