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Research Article

Essential Oil Composition and Antioxidant Activity of Fresh and Air-Dried *CynaraCardunculus* L. Grown Wild in Jordan

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Abstract:
The composition of the hydro-distilled oils of *Cynara Cardunculus* L. (fresh and air-dried) at the flowering stage was evaluated by gas chromatography-mass spectrometry analysis (GC-MS). Additionally, the essential oils that were isolated from both fresh and air-dried *Cynara Cardunculus* L were analyzed by using DPPH free radical scavenging and $ABTS^{\cdot+}$cation radical scavenging to investigate the antioxidant activity. The gas chromatography-mass spectrometry analysis of fresh and dried essential oils identified 53 and 41 compounds, respectively. The main ingredient classes in the fresh aerial section were oxygenated sesquiterpenes with (37.21%) of the overall amount, followed by oxygenated monoterpenes (19.56%) and diterpenes (18.68%). On the other hand, diterpenes (28.27%) and oxygenated sesquiterpenes (23.44%) made up most of the dried aerial portion. The most prevalent component in the fresh plant was carissone (22.01%) which was followed by cubeban-11-ol (15.20%),(3Z)-cembrene A(10.11%), limonen10-ol (9.69%) andmaaliol(9.17%). The most prevalent components of the dried were (3Z)-cembrene A (18.22%), carissone (12.22%), and cubeban-11-ol (11.22%). The isolated essential oils of both fresh and dried *C. cardunculus* showed significant antioxidant activities, however, the dried plant's oil exhibited stronger antioxidant activity than the fresh one.

Keywords: $ABTS^{\cdot+}$, antioxidant, Asteracea, *CynaryaCardunculus*, DPPH, hydro distillation

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Introduction:

Essential oils are a complex combination of volatile and strong-smelling compounds derived from plants. Most essential oils are either colorless or pale yellow [1]. The Essential oils consist of a variety of aromatic and aliphatic compounds with low molecular weight, such as monoterpenes, sesquiterpenes, diterpenes, oxygenated derivatives, alcohols, ketones, esters, and aldehydes [2,3,4]. Due to their antimicrobial and antioxidant properties, essential oils, have been extensively utilized in the treatment of a variety of diseases. Traditionally, essential oils are used in food flavoring, fragrances, pharmaceutics, and cosmetics [5].

Cynara cardunculus L. is known as cardoon. It belongs to, Asteraceae, which is one of the largest families of the plant kingdom.*C. Cardunculusis* is a perennial plant that resembles thistles. It can be found in the Canary Islands, Northwest Africa, the Middle East, and the Mediterranean region [6, 7]. Furthermore, due to its high nutritional content, *C. Cardunculus* oil is safe for human

ingestion [8, 9]. Despite not having any wood, *C. Cardunculus* stems can be used to produce paper since they contain 17% lignin. Cardoon leaves' high polyphenol content makes them useful for medicinal purposes [8]. The leaves often have a diuretic and hepatoprotective effect, enhance gallbladder function, increase the production of digestive fluids, particularly bile, and limit cholesterol synthesis. Significant antioxidant and antibacterial qualities of root extracts make them useful for medical and pharmaceutical applications [9, 10, 11]. The phenylpropanoids (flavonoids, mono- and decaffeoylquinic acids) and sesquiterpene lactones present in them are responsible for these physiological characteristics [7, 12, 13].

C. Cardunculus is a crop that is used in many different applications. It contains sugars, beta-carotene, mineral salts, folic acid, vitamins C, B1, B3, B5, B6, and phosphorus. The plant's tender blooming heads, or capitula, are the edible portions. Inulin, which has nutritional and non-nutritional uses, can be found in *C. Cardunculus*.In addition, biofuels can be produced from *C. Cardunculus* seeds [14, 15]. Traditionally, the leaves of *C. Cardunculus* are used in soups, diuretics, cardiotonic, dyspepsia, antidiabetics' stews, and salads. Due to the abundance of 40 proteases in *C. Cardunculus* flowers, specifically cardosins A and B, aqueous floral extracts have been utilized for generations to make cheeses made from caprine or ovine milk [9, 16].

One popular method of preservation is herb drying. Nonetheless, it could have an impact on the quality of the herb due to potential changes in the concentration of essential oils in the herb as well as the formation of new volatile chemicals [5, 17, 18].

This study is a comparative analysis of essential oils of fresh and air-dried *Cynara Cardunculus*L. in the flowering stage in Jordan by using gas chromatography mass spectrometry analysis (GC-MS). Additionally, the antioxidant activity of the essential oils that were isolated from both fresh and air-dried *Cynara Cardunculus*L was analyzed by using DPPH free radical scavenging and $ABTS^{\cdot+}$ cation radical scavenging.

2. MATERIAL AND METHODS

2.1. Sample Preparation and Drying Techniques

The *C. cardunculus* plantmaterial was gathered in April 2023 from the western area of Irbid City, Jordan.

To ensure the consistency of plant materials in the treatments, plant samples were separated into two groups, taking into account the fresh and shad-drying procedures. The fresh flowering aerial portions of C. Cardunculus were spread out in the shade with natural airflow and a constant temperature of 22°C for approximately two weeks to carry out the shade-drying process.

2.2. Extraction and Analysis of Oils

Using a Clevenger apparatus, one hundred grams of fresh *C. Cardunculus* leaves were hydrodistilled. For three hours, 100 g of dried material was hydrodistilled in a Clevenger.

The plant oils that were extracted were refrigerated at 4 ± 1°C.

2.3. Gas Chromatography-Mass Spectrometry

Gas chromatography-mass spectrometry (Chromatic Crystal 9000 GC-MS, Yoshkar Ola, Russia) equipped with a CR-5 MS column (5% diphenyl, 95% dimethyl polysiloxane, 30 m × 0.25 mm × 0.25 μm film thicknesses) was used to analyze the essential oils. The electron ionization mode in the MS detector was run at 70 eV. The temperature column was set to rise steadily at a rate of 3°C per minute from 40°C for one minute (isothermal) to 280°C. Helium (1.0 mL/min) was used as the carrier gas. The relative percent concentrations of the chemicals found were computed using the relative peak regions. By comparing the calculated Kovats retention index (KI) of the essential oils to the n-alkanes C8–C30, matching their recorded mass spectra with the built-in library spectra (NIST, Gaithersburg, MD, USA, and Wiley Co., Hoboken, NJ, USA), and mass spectrum matching to genuine standards, the chemical constituents of the oils were identified [18].

3. Antioxidant Activity Evaluation

3.1. DPPH Free Radical-Scavenging Capacity

One of the most often used techniques for assessing antioxidant activity is the DPPH free radical scavenging activity [19, 20]. The procedure described in reference [20, 21] was followed in this work, and it covered a concentration range of (10–1000) μg/mL. A positive control was ascorbic acid. A Cary Bio 100 double-beam UV-VIS spectrophotometer was used to evaluate the mixture's absorbance at 517 nm in wavelength. The proportion of free radical-scavenging ability was calculated by using the following formula:

$$= \left(\frac{Ac - A_s}{A_c}\right) x100\%$$

where Ac is the absorbance of the blank and Asis the absorbance in the presence of extract. When exposed to radical scavengers, the concentration of DPPH radicals dramatically drops. IC50, or μg/mL, was used to express the antiradical activity. By performing a linear regression analysis on plots of the percent of radical inhibition against the logarithm of concentration, the IC50 values were determined.

3.2. $ABTS^{\cdot+}$ Radical-Scavenging Capacity

Using 2,2'-azinobis-3-ethylbenzothiazoline-6-sulfonic acid ($ABTS^{\cdot+}$) and a modified technique from reference [24], the essential oils from fresh and air-dried C. Cardunculus were tested for their ability to scavenge radicals over a concentration range of (10–1000) μg/mL. Ascorbic acid was used as a positive control. With the aid of a Cary Bio 100 UVVIS spectrophotometer, the mixture's absorbance was determined at a wavelength of 734 nanometers. The $ABTS^{\cdot+}$ inhibition ability was calculated by using the following formula:

$$\left(\frac{Ac - A_s}{A_C}\right) x100\%$$

Where As is the absorbance in the presence of extract And Ac is the blank's absorbance. By performing a linear regression analysis on plots of the percent of radical inhibition against the logarithm of concentration, the IC_{50} values were determined.

3.3. Statistical Analysis

The essential oils' ability to scavenge radicals such as DPPH and $ABTS^{\bullet+}$ was examined three times, and the results are presented as mean ± standard deviation. The one-way analysis of variance (ANOVA) test and Dunnett's test were used to conduct the statistical analysis. $P < 0.05$ values were regarded as statistically significant.

4. RESULTS AND DISCUSSIONS

4.1. Essential Oil Composition

The results of the GC/MS analysis of the essential oils extracted by hydro-distillation of both fresh and dried *C. Cardunculus*are shown in Table 1. The analysis resulted in the identification of 55 components in the hydro-distilled oils. In total, 53, and 41 components were identified in the essential oils in fresh and dried samples, respectively. The essential oil of the fresh plant was dominated by oxygenated sesquiterpenes (37.21%). In comparison to the results obtained from the oil of the dried samples, quantitative variations were noticed. Oxygenated sesquiterpenes were the second main group in the essential oil of the dried sample with a lower percentage (23.44%). The main group in the essential oil of the dried sample was diterpenes (28.27%). While a lower percentage of diterpenesin the essential oil of the fresh plant was found (18.68%). The oxygenated monoterpenes group has a significant percentage in both fresh and dried *C. Cardunculus* with percentages (19.56 %) and (17.02%) respectively. According to the compounds in both samples. Carissone (22.01%) was the main component detected in the oil of fresh C. Cardunculus followed by cubeban-11-ol (15.20%),(3z)-cembrene A (10.11%),limonen-10-ol (9.69%) and dimethyl-ionone (9.17%). While the percentage of carissonein the dried sample decreased by (9.79%) as compared to the fresh plant. According to the components obtained from the oil of the dried plant, (3z)-cembrene A (18.22%) was the main component detected, followed by carissone (12.22%), cubeban11-ol (11.22%), dimethyl-ionone (10.05%) and methyl tetradecanoate (9.10%).

Table 1 shows the 55 chemical composition, retention indices, and the relative percentages of the identified compounds of the essential oil obtained from fresh and air-dried *C.Cardunculus* growing wild in Jordan.

Table 1: Volatile organic compounds in fresh and air-dried dried of *CynaraCardunculus*L.

No.	KI	Compound	% Composition	
			Fresh	Air-dried
1	900	Heptanal	0.21	0.11
2	911	Butyl propanoate	0.31	-
3	914	Tiglic acid	1.91	-
4	925	α-Thujene	0.16	0.05
5	956	Camphene	0.22	0.55
6	965	Cyclohexylformate	0.12	-
7	970	6-methyl-Heptan-2-ol	0.02	-
8	972	n-Heptanol	0.03	0.04
9	980	Isopropyl tiglate	0.81	1.05
10	1044	Benzenelacetaldehyde	0.05	1.00
11	1089	Terpinolene	0.06	1.02
12	1092	Linalool	0.22	-
13	1095	α-Pinene oxide	0.18	1.09
14	1099	6-Camphenone	0.60	1.01
15	1105	Linalool	0.02	-
16	1108	Cis- Thujone	0.21	1.05
17	1111	1,3,8-ρ-Menthatriene	0.06	-
18	1114	6-Camphenol	0.22	-
19	1118	trans-Thujone	0.52	-
20	1197	α-Terpineol	0.17	1.22
21	1199	Verbenone	5.50	6.30
22	1210	p- Cymen-9-ol	-	0.04
23	1220	trans-Carveol	1.01	2.02
24	1226	Citronellol	0.02	1.03
25	1240	Z- Ocimenone	0.35	1.06

26	1244	Cumin aldehyde	0.05	-
27	1255	Isoamylhexanoate	0.14	1.52
28	1258	Butyrophenone	0.55	1.22
29	1272	Decanol	0.87	0.02
30	1282	Allyl octanoate	0.46	1.02
31	1288	p- Ethyl acetophenone	0.44	1.00
32	1291	Limonen-10-ol	9.69	-
33	1294	4-Undecanol	0.87	1.09
34	1300	5Z-Octenol propanoate	-	1.22
35	1338	cis-Piperitol acetate	0.85	1.65
36	1344	4-hydroxybenzenemethanol	0.66	1.21
37	1365	Neryl acetate	0.58	0.03
38	1372	n-Undecanol	0.98	-
39	1420	trans-α- Ambrinol	0.44	1.02
40	1450	Cis-Prenyl limonene	0.85	1.69
41	1567	Dimethyl-Ionone	9.17	10.05
42	1592	Cubeban-11-ol	15.20	11.22
43	1602	hydro-Cinnamaldehyde	0.99	0.24
44	1610	Z-Sesquilavandulol	0.14	-
45	1614	(2E)-Dodecenyl acetate	0.65	-
46	1725	Methyl tetradecanoate	8.08	9.10
47	1792	1-Octadecene	0.25	1.22
48	1802	n-Octadecane	0.21	-
49	1835	Isopropyl myristate	0.74	0.92
50	1852	Isoamyldodecanoate	0.58	0.62
51	1870	n-Hexadecanol	0.56	0.94
52	1923	Carissone	22.01	12.22
53	1945	Phytol	0.21	1.12
54	1962	(3Z)-Cembrene A	10.11	18.22
55	1990	1-Eicosene	0.35	0.95
56	1995	Ethyltetradecanoate	0.33	0.85

Classification	**% Composition**	
	Fresh	Dry
Oxygenated monoterpenes (OM)	2.13	-
Monoterpenes hydrocarbons (MH)	0.79	1.66
Oxygenated monoterpenes (OM)	19.56	17.02
Aliphatic compounds (AC)	10.84	15.75
Oxygenated sesquiterpenes (OS)	37.21	23.44
Sesquiterpene hydrocarbons (SH)	10.02	11.74
Diterpeneshydrocarbons (DH)	18.68	28.27
Oxygenated diterpenes (OD)	0.21	1.12

4.2. Antioxidant Activity

The concentrations between 1×10^4 and 1×10^6µg/mL for essential oils, using methanol as solvent, and the antioxidant activity was shown using the $ABTS^{\cdot+}$and DPPH techniques. Table 2 displays the findings as a percentage of inhibition (scavenging activity). Compared to ascorbic acid, it is evident that the essential oils are from both fresh and dried plants. C. Cardunculus has potent antioxidant and radical scavenging properties. Table 3 displays the equivalent IC_{50} values; a lower IC_{50} value denotes greater antioxidant activity. The essential oil from dried C. Cardunculus has a lower DPPH IC_{50} value (1.02 x 10^2µg/mL) than the essential oil from the fresh plant (3.91 x 10^2µg/mL). Similarly, the fresh plant's essential oil has a higher $ABTS^{\cdot+}$ IC_{50} value (2.93 x 10^2µg/mL) than the dried plant's (0.69 x 10^2µg/mL). These findings demonstrate that the essential oil extracted from air-dried C. Cardunculus possesses a little more antioxidant activity than the oil extracted from the fresh plant.

Table 2. Antioxidant activity of essential oils from air-dried and fresh C. cardunculus

DPPH scavenging activity%				**$ABTS^{\cdot+}$ scavenging activity%**		
Concentration (µg/mL)	**Dry**	**Fresh**	**Ascorbic acid**	**Dry**	**Fresh**	**Ascorbic acid**
1.00 x 10^1	20.62 ±0.98	26.25±0.94	22.14±0.54	24.56±0.75	12.69±0.21	32.67±0.56
2.00 x 10^1	27.62±0.96	36.36±0.22	36.10±0.42	38.86±0.41	22.56±0.14	41.82±0.69
1.00 x 10^2	39.81±0.16	45.67±0.21	45.67±0.11	49.25±0.69	27.96±0.11	48.02±0.15
3.50x 10^2	62.40±0.13	65.01±0.31	61.41±0.89	65.98±0.54	47.66±0.85	71.65±0.25
5.00 x 10^2	69.25±0.11	75.55±0.12	75.67±0.88	76.45±0.12	56.78±0.63	84.60±0.33
1.00 x 10^3	82.11±0.13	91.01±0.11	88.17±0.69	91.15±0.17	76.88±0.21	95.71±0.66

Table 3. IC50 values of essential oils from air-dried and fresh C. Cardunculus.

	DPPH IC50 value (µg/mL)	**$ABTS^{\cdot+}$ IC50 value(µg/mL)**
Dry	$(1.02 \pm 0.11) \times 10^2$	$(0.69 \pm 0.46) \times 10^2$
Fresh	$(3.91 \pm 0.35) \times 10^2$	$(2.93 \pm 0.05) \times 10^2$
Ascorbic acid	$(0.91 \pm 0.11) \times 10^2$	$(0.60 \pm 0.56) \times 10^2$

Conclusion

This is the first study to account for the chemical composition of the essential oil extracted from *C. Cardunculus* using hydrodistillation, as well as its antioxidant capacity. From fresh and air-dried *C. Cardunculus*oil, seven primary classes were identified: aliphatic compounds, oxygenated hemiterpenes hydrocarbons, oxygenated monoterpenes, sesquiterpene hydrocarbons, oxygenated sesquiterpenes, oxygenated diterpenes, and diterpene hydrocarbons. The current study's findings support the recommendation to use the air-drying method, which has a temperature mean value of 22 °C, as it did not result in appreciable changes to the primary classes of chemicals that make up the essential oils. New compounds that were not found in the essential oil from fresh material were also formed as a result of drying. The capacity of the essential oil constituents to scavenge DPPH and $ABTS^{\cdot+}$ radicals was used to evaluate their antioxidant activity. The results of this study indicate that C. Cardunculus both fresh and dried are note worthy providers of natural antioxidants, with the essential oil from the dried plant showing greater antioxidant activity than the sample from the fresh plant.C. Cardunculus essential oil has the potential to be a natural antioxidant and is used to stop oxidative stress, which is a factor in many degenerative disorders.

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