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Zierone: A Sesquiterpene Ketone from the Essential Oil of *Cyperus distans* L. (Cyperaceae)

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Authors' contributions

This work was carried out in collaboration between all authors. Author OAL isolated the oils and wrote part of the manuscript. Author IAO managed the literature searches and wrote the final draft of the manuscript. Authors ARO and AOO supervised the work. All authors read and approved the final manuscript.

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ABSTRACT

Aims: The study investigates the essential oil obtained from the rhizomes of *Cyperus distans* collected from different location, and compared the result with previous sample and other reports on the chemical composition of essential oils from several species of the genus *Cyperus*.
Study Design: Isolation of essential oil from the rhizomes of *Cyperus distans* and identification of its chemical constituents.
Place and Duration of Study: Fresh plant materials of *Cyperus distans* were collected at full flowering stage from Main road, Vulindlela, along KwaDlangezwa road, opposite University of Zululand, KwaDlangezwa, KwaZulu-Natal, South Africa, in October, 2006.
Methodology: Fresh rhizomes of *Cyperus distans* were hydrodistilled in a Clevenger-type

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apparatus and the isolated oil analyzed by GC and GC/MS.
Results: Twenty-two constituents were identified, representing over 80.0% of the oil composition. The chemical profile of the oil was very different from the essential oils of several species of the genus *Cyperus*, with little amount of cyperene (3.1%). The most interesting feature of the oil was the presence of a sesquiterpene ketone, zierone (33.9%). Other significant constituents are caryophyllene oxide (14.2%) and α-cyperone (9.0%).
Conclusion: The chemical pattern of this oil and the previously investigated sample differs greatly, suggesting a chemotype.

Keywords: Cyperus distans; Cyperaceae; essential oil composition; zierone.

1. INTRODUCTION

Cyperus distans L.f. (Cyperaceae) is an annual herb of about 0.5-1.4 m high, commonly found in damp locations along rivers, roadside ditches and as weeds of cultivation in coastal and midlands of KwaZulu-Natal to Maputaland and Madagascar [1]. The plant has many flowers and stolons with a solitary trigonous culm invested by 3-ranked sheaths bearing blades that are green adaxially and glaucous abaxially. Inflorescences are anthelate, delicate and diffuse with long spikelets, obtuse apex and wide membranous margins [1,2]. Previous phytochemical study on *C. distans* revealed the isolation of scabequinone, which possessed antifeedant activity [3].

The present report continues our studies on the essential oils from the species of the genus *Cyperus* growing wild in different locations in KwaZulu-Natal Province, South Africa. We have investigated the essential oil of *C. distans* collected from the location described in the previous work and found it to contain cyperene (47.6%), α-pinene (18.8%), 1,8-cineole (14.5%) and caryophyllene oxide (7.3%) [4]. In addition, the volatile constituents from the rhizomes oils of *Cyperus rotundus* [5] and *Cyperus papyrus* [6] collected from two different locations each have been reported. Likewise, we have reported the antioxidant activity, total phenolic and flavonoids contents of essential oils of *Cyperus distans*, *C. papyrus* and *C. rotundus* collected from two different locations each [7]. In the present study, we report the presence of zierone, a sesquiterpene ketone as a constituent of essential oil of *Cyperus distans* collected from another location in the city of uMhlathuze district, KwaZulu-Natal Province, South Africa.

2. MATERIALS AND METHODS

2.1 Plant Materials

Fresh plant materials of *Cyperus distans* were collected at full flowering stage from Main road, Vulindlela, KwaDlangezwa, KwaZulu-Natal, South Africa. The material was authenticated by Dr S. J. Siebert of the Department of Botany, University of Zululand, KwaDlangezwa. A voucher specimen [Lawal, OA 03 (ZULU)] was deposited at the University of Zululand, Herbarium.

2.2 Isolation of Essential Oil

Fresh rhizomes (500 g) of *Cyperus distans* were subjected to hydrodistillation in a Clevenger-type apparatus for 4 h in accordance with the British Pharmacopoeia specification [8]. The isolated oil was preserved in a sealed sample tube and stored under refrigeration until analysis.

2.3 Gas Chromatography Analyses

GC analysis of the oil was carried out on a Hewlett Packard HP 6820 Gas Chromatograph equipped with a FID detector and DB-5 column (60 m x 0.25 mm id), film thickness was 0.25 µm and the split ratio was 1:25. The oven temperature was programmed from 50℃ (after 2 min) to 240℃ at 5℃/min and the final temperature was held for 10 min. Injection and detector temperatures were 200℃ and 240℃, respectively. Hydrogen was the carrier gas. An aliquot (0.5 µL of the diluted oil) was injected into the GC. Peaks were measured by electronic integration. A homologous series of *n*-alkanes were run under the same conditions for determination of retention indices.

2.4 Gas Chromatography - Mass Spectrometry

GC/MS analyses of the oil was performed on a Hewlett Packard Gas Chromatography HP 6890 interfaced with Hewlett Packard 5973 mass spectrometer system equipped with a HP 5-MS capillary column (30 m x 0.25 mm id, film thickness 0.25 µm). The oven temperature was programmed from 70-240℃ at the rate of 5℃/min. The ion source was set at 240℃ and electron ionization at 70eV. Helium was used as the carrier gas at a flow rate of 1 mL/min. Scanning range was from 35 to 425 amu. Diluted oil in *n*-hexane (1.0 µL) was injected into the GC/MS.

2.5 Identification of Components

Identification of the oil components was carried out by comparison of their relative retention times with those of authentic samples, by comparison of their relative retention indices and mass spectra with Wiley library mass spectra database of the GC/MS system, and published data [9-11].

3. RESULTS AND DISCUSSION

Table 1 shows the identity, retention index and the percent composition of the oil of *C. distans*, where compounds are listed in order of their elution on the DB-5 column. Twenty-two compounds were identified, accounting for over 80.0% of the oil composition. Among the identified compounds were 4 oxygenated monoterpenes (2.8%), 8 sesquiterpene hydrocarbons (12.7%), 10 oxygenated sesquiterpenes (69.1%) and 9 unidentified compounds accounting for 12.4% of the total oil. The major component of the oil was zierone (33.9%). Other significant constituents were caryophyllene oxide (14.2%), α-cyperone (9.0%), humulene epoxide II (3.9%), cyperene (3.1%), endesma-2,4,11-triene (2.8%), nor-copernone (2.8%) and germacrene D (2.7%). Comparing the present study with previous reports on essential oil of *C. distans* [4,7], the oil samples showed an array of varied chemical composition.

Table 1. Chemical composition of essential oil of *Cyperus distans*

Compound	RI[a]	% Composition
Artemesia ketone	1058	0.6
Pinocarveol	1139	0.5
Myrtenol	1194	0.9
(*Z*)-Ocimenone	1226	0.8
α-Copaene	1374	0.6
Cyperene	1396	3.1
β-Caryophyllene	1419	0.6
Unknown	1439	2.3
Endesma-2,4,11-triene	1451	2.8
Germacrene D	1476	2.7
ar-Curcumene	1480	0.7
α-Selinene	1489	1.0
cis-Calamenene	1514	1.2
Caryophyllene oxide	1582	14.2
Humulene epoxide II	1603	3.9
Unknown	1638	1.8
Caryophylla-3,8 (13)-dien-5-β-ol	1649	1.7
Carypohyllenol II	1657	1.4
nor-Cypernone	1668	2.8
Unknown	1721	0.7
Unknown	1729	3.0
Aristolone	1747	1.6
Zierone	1751	33.9
Unknown	1754	0.6
Unknown	1759	1.4
Unknown	1762	0.8
Unknown	1766	0.4
Unknown	1768	1.4
α-Cyperone	1771	9.0
Oxygenated monoterpenes		2.8
Sesquiterpene hydrocarbons		12.7
Oxygenated sesquiterpenes		69.1
Unidentified		12.4
Total identified		84.6

[a]RRI = Retention indices relative to C_9-C_{24} n-alkanes on the DB-5 column

In the oil of previous report, 7 constituents were identified, representing 99.6% of the total oil with 3 monoterpene hydrocarbons (24.9%), 1 oxygenated monoterpene (14.5%), 2 sesquiterpene hydrocarbons (51.6%) and 2 oxygenated sesquiterpenes (8.6%). In addition, only three compounds (cyperene, caryophyllene oxide and humulene epoxide II) were present in both oils. Furthermore, α-pinene, β-pinene, 1,8-cineole and β-cadinene found in the oil of previous report were not detected in the oil of present sample (Please rewrite this statement). Finally, zierone is the major compound of our present study, whereas, cyperene was the most abundant constituent in the oil of previous report. It is obvious, that the chemical pattern of both oils differs greatly. This suggests chemotypic forms of *C. distans*.

A comparison of the previous reports on the chemical composition of essential oils from several species of the genus *Cyperus* with the present data, it is evident that the essential oils of *Cyperus* species from different countries contained sesquiterpenes as the dominant compounds. However, there are qualitative and quantitative variations among these sesquiterpenes [7,12-33]. Cyperene, a major component of essential oil of several species [7,12,18,20,21,27-29,31,32] was detected in small quantity in our present study. Other major constituents of *Cyperus* oils such as β-selinene, hexahydrofarnesyl acetone, 1,4-hydroxy-9-epi-β-caryophyllene, α-humulene, piperitone, δ-cadinene and cedrol were not present in this study [13,15,16,19,26]. Also, monoterpenes compounds including α-pinene, β-pinene, *p*-cymene, limonene, 1,8-cineole and *trans*-pinocarveol that were previously reported as major constituents of volatile oils of some *Cyperus* species were conspicuously absent in our sample [4,22]. Previously, sesquiterpene ketones have been identified as constituents of essential oils of some *Cyperus* plants [13,15,16,23,25,27,34]. However, the presence of zierone in large amount, as seen in the present oil sample, could not been found in the literature.

4. CONCLUSION

Although, the chemical pattern of this oil and the previous sample differs greatly, suggesting a chemotype. On the other hand, the predominantly sesquiterpenoids nature of the oil, makes it similar to others species in genus *Cyperus*. In addition, the observed differences between the essential oils of *Cyperus distans* and other species of *Cyperus* growing around the world could be due to environmental, climatic and other factors which can influence essential oil composition.

CONSENT

It is not applicable.

ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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