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SHORT COMMUNICATION

CHEMICAL INVESTIGATION OF THE VOLATILE CONSTITUENTS OF *CLEOME VISCOSA* FROM NIGERIA

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ABSTRACT. The major volatile constituents of the oils from the integral parts of *Cleome viscosa* L. from Nigeria have been identified by GC, GC/MS and ^1H NMR. The main constituents of the non-polar fraction of the oils were monoterpene hydrocarbons (21% in stem/leaves, 15% in seed/fruits, 12% in roots) and some oxygenated derivatives (3% in leaves/stem; 1% in seeds/fruits and 1.5% in roots). The monoterpenes occurred frequently in the oils. Fatty acid esters especially ethyl palmitate which constituted a major constituent in the oil from the seeds/fruits was not detected in the oils from the roots.

KEY WORDS: *Cleome viscosa* L, Capparaceae, Essential oil, Volatile constituents

INTRODUCTION

Cleome viscosa L is a common weed found in the tropical regions of the world where it finds application in traditional medicine. Some significant phytochemical investigations of the crude extract of *C. viscosa* have been reported [1-4]. Ray *et al.* [5-7] reported extensively on the coumarino-lignoids of seeds of *C. viscosa.* A cembrane diterpenoid (macrocyclic unsaturated diterpenoid) has been isolated from the stem/leaves [8]. In addition, we have established that a petroleum ether extract of the whole aerial part of *C. viscosa* exhibited significant insecticidal activity (in laboratory tests and field trials) against the bean pest *Epilachna varivestis*.

However, as far as we are aware, no report has appeared on a comprehensive study of the volatile constituents of all integral parts of the plant at the same time of collection. We report this study to bridge the gap.

RESULTS AND DISCUSSION

Purification of the crude ethyl acetate extracts of all integral parts of *C. viscosa* was achieved by column and Kugel-rohr (KR) fractionation and flash chromatography (FC). The results of this study are discussed in the priority order leaves/stem (CV/L), roots (CV/R) and seeds/fruits (CV/S).

CV/L/A/KR1. (Hexane, 100 mL). This non-polar fraction (oil) had a very strong pinene smell and the GC showed a total of 73 peaks comprising mainly of monoterpenoid hydrocarbons and a few sesquiterpenoids. Altogether, 63 peaks (86.3%) could be identified by a combination of the RRI and GC/MS data. Table 1 shows list of compounds whose GC concentration is not less than 1% of total peak concentration. Six compounds were represented at greater than 5% namely:

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heptan-4-one (CV/I, 5.63%); dehydrosabinene (CV/II, 13.03%); 6-methylhept-5-ene-2-one (CV/III, 5.78%); myrcene (CV/IV, 18.9%); *p*-cymene (CV/V, 5.41%); α-and β-pinene together (CV/VI, 5.65%).

CV/L/A/KR2. (Hexane/diethyl ether, 10:1, 150 mL). GC analysis of this non-polar fraction (oil) after three .FC afforded 36 peaks out of which 24 were assigned. Enrichment FC and co-injection techniques enabled the identification of the two major constituents. These are α-amorphene (CV/VII, 6.5% enriched to 54.5%) and ethyl palmitate (CV/VIII, 5.7% enriched to 45.1%).

Table 1. List of compounds identified by GC, GC/MS, ^{1}H NMR in leaves, roots and seeds of *C viscosa*.

RRI	Identification	Leaves (CV/L)		Roots (CV/R)		Seeds (CV/S)	
		A/KR1	A/KR2	A/KR1	A/KR2	B/KR1	B/KR2
787	Oct-1-ene			++		++	
861	Heptan-4- one	++				+	
872	Heptan-2-one					+	
878	Non-1-ene					++	
936	α-Pinene	+		+		+	
945	Camphene	+					
955	Dehydrosabinene	+++				+	+
963	6-Methylhept-5-ene-2-one	++					+
974	β-Pinene	+		+			
989	Myrcene	+++		+++			+
1016	*p*-Cymene	++	+		++		+
1024	Limonene	+				+	+
1044	E-Ocimene	+			++	+	+
1051	γ-Terpenene	+		++			
1075	Dehydrolinalool			++			+
1102	Undecane				+	+	+
1118	Allo-ocimene		++		++		
1127	Limonene oxide			+++		+	+
1166	Benzoic acid					+	+
1177	α-Terpineol	+			++	+	+
1197	Decan-2-ol			+			+
1238	Geraniol					+	+
1272	Undec-10-en-1-al					+	++
1291	Deca-2, 4-dien-1-al				+		++
1300	Citronellic acid		+	++			
1399	Coumarin		+			+	+
1419	Cedrene		+				
1469	α-Amorphene		++				
1983	Ethyl palmitate		++				

Note: GC concentration symbol: (+) = 1–5%; (++) = 5–10%; (+++) = 10%$^+$. RRI: relative retention indices.

CV/R/A. KR distillation of this non-polar fraction (oil) afforded CV/R/A/KR1 (0.33%, 100 °C, 0.02 mmHg of the crude) and CV/R/A/KR2 (1.25%, 120 °C, 0.02 mmHg of the crude). GC analysis of CV/R/A/KR1 (EFC, pentane, 100 mL) resulted in 39 peaks. Compounds which represented not below 1% of total peak concentration are listed in Table 1. The frequently occurring myrcene (CV/IV) and limonene oxide (CV/XIII) peaks were enriched, being the two most prominent peaks in the oil.

CV/R/A/KR2. (Pentane/diethyl ether, 10:1, 150 mL). Total of 36 peaks resulted from the GC run, from which 32 (88.9%) were identified. The three main constituents in this fraction (oil) are *p*-cymene (CV/V, 7%), allo-ocimene (CV/XIV, 13%), and α-terpineol (CV/XV, 20%).

CV/S/A. The GC investigation gave results identical to that of CV/L/A/KR1. The oil was therefore not further investigated.

CV/S/B. KR distillation afforded two fractions (oils): CV/S/B/KR1 (130 °C, 0.02 mmHg) and CV/S/B/KR2 (150 °C, 0.02 mmHg). The GS analysis of the former gave a total of 19 mainly hydrocarbon peaks. The two major peaks obtained after EFC (pentane/diethyl ether, 10:1, 100 mL)/GC were assigned to oct-1-ene (CV/XVI) and non-1-ene (CV/XVII). Of a total of 54 peaks obtained for CV/S/B/KR2, 29 were identified and after EFC (pentane/diethyl ether, 10:3, 150 mL)/GC, the two major peaks obtained were assigned to undec-10-en-l-al (CV/XVIII, 13.7%) and deca-2, 4-dien-1-al (CV/XIX, 7.39%. The GC investigations showed that the polar fractions and non-polar fractions both contained similar monoterpeniod hydrocarbons. *p*-Cymene and other alkyl benzenes are well represented in all the plant parts (Table 1).

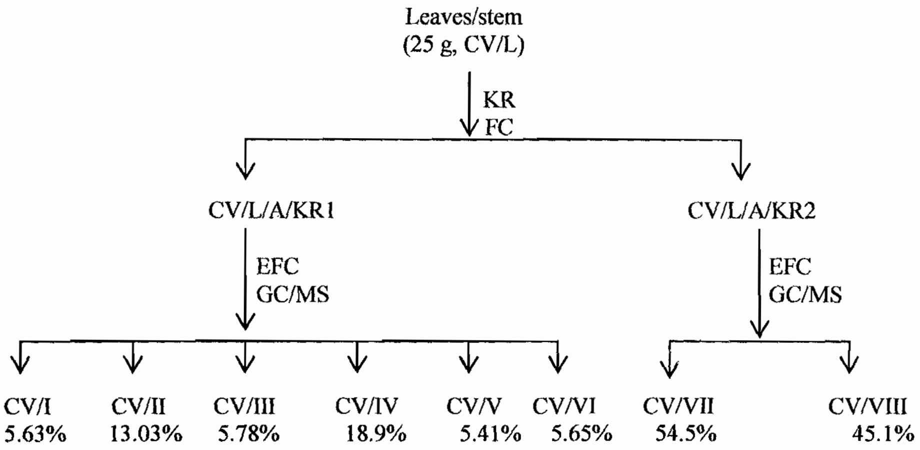
EXPERIMENTAL

General. ^{1}H NMR ($CDCl_3$): Bruker AM400; ^{13}C NMR ($CDCl_3$): Bruker AH 270 with DEPT programmed. GC/MS: Varian MAT 44S (70 eV) combined with Varian 3700, 25 m CP Sil. 5 CB columns, carriers gas He. GC: Packard 439 with a 25 m CP Sil. 5 CB columns, carrier gas N_2. Flash chromatography (FC): ICN Biochemical's silica (mesh 32-63) eluted with hexane or pentane and increasing amounts of diethyl ether. TLC precoated silica gel F_{254} (Merck No. 5554). Guided partly by the relative retention indices (RRI), the identification of compounds was based on comparison of their GC, GC/MS and/or ^{1}H NMR spectra with our spectra collection of authentic samples.

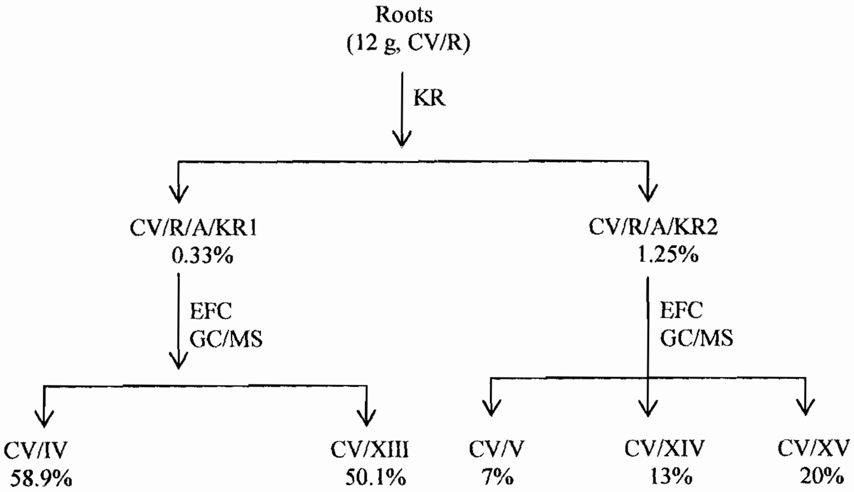
Plant materials. Mature, fresh whole plants (10 kg) of *C. viscosa* were collected during the peak of the raining season in the environs of the University of Ilorin, Nigeria. A voucher (No CV/BL/107) of the specimen was deposited at the Herbarium of the Plant Biology Department, University of Ilorin.

Extraction, isolation and identification of compounds. The whole fresh plants were separated into the integral parts. (1.7 kg seeds/fruits; 6 kg leaves/stem; 2.3 kg roots), chopped into small pieces and defatted first with petroleum spirit and then extracted with cold ethyl acetate. The ethyl acetate extracts were concentrated under reduced pressure and the residue dried to give dark brown, 12 g root crude (CV/R); 25 g of leaves/stem crude (CV/L); and 7.5 g seed/fruit crude (CV/S).

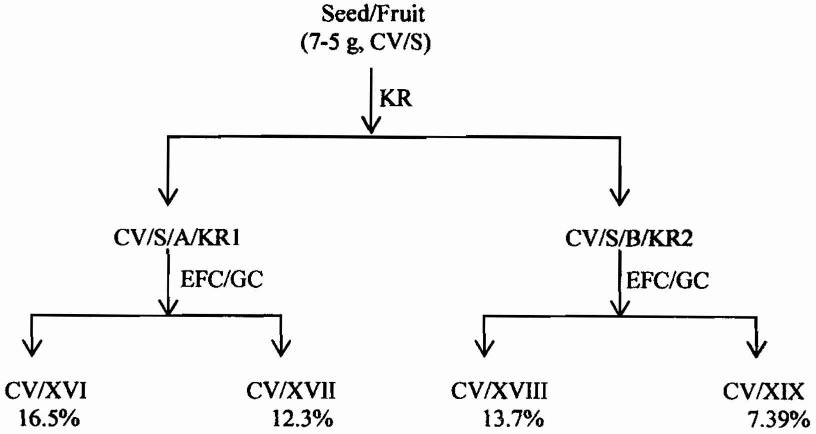
The initial filtration over silica gel using hexane/diethyl ether (10:1) afforded the polar and non-polar fractions (oils), which were then separately subjected to Kugel-rohr (KR) distillation (110-160 °C, 0.01 mmHg). This was further separated and purified by repeated flash chromatography, which then afforded different components. These were subjected to GC and appropriate spectroscopic analysis (Scheme 1).



Scheme 1a. Identification of compounds isolated from the leaves/stem crude extract of *Cleome viscosa* L.



Scheme 1b. Identification of compounds isolated from the roots crude extract of *Cleome viscosa* L.



Scheme 1c. Identification of compounds isolated from the seed/fruit crude extract of *Cleome viscosa* L.

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