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Chemical composition of *Gardenia ternifolia* Schumach & Thonn (Rubiaceae) leaf essential oil

Moses S Owolabi, Lanre Akintayo Ogundajo, Balogun Olaoye Solomon, Ambika Poudel and William N Setzer

Abstract
Gardenia, a genus of flowering plants in the Rubiaceae family, is used in traditional medicine to treat various maladies and illnesses. In this work, the essential of *Gardenia ternifolia* leaves has been obtained by hydrodistillation and analyzed by gas chromatography-mass spectrometry. The major components were found to be sesquiterpene hydrocarbons (56.2%) with *trans*-α-bergamotene (16.4%), trans-calamenene (8.8%), and δ-cadinene (8.0%), and the oxygenated sesquiterpenoids (39.5%), spathulenol (10.9%), τ-muurolol (6.1%), α-cadinol (5.9%), and τ-cadinol (5.0%).

Keywords Rubiaceae, essential oil composition, sesquiterpenoids, hydrodistillation

1. Introduction
Gardenia is a genus of evergreen shrubs and small flowering plants in the madder or Rubiaceae family, growing up to 15 m tall. The leaves are arranged in whorls of three or four about 5-50 cm long and up to 25 cm broad. The Rubiaceae has more than 563 genera and around 10, 900 species [1], commonly found in China and Japan and distributed over tropical and subtropical Africa, Asia, and Pacific islands [2]. *Gardenia ternifolia* Schumach. & Thonn. (Rubiaceae) is an evergreen flowering plant that grows up to 10 m tall with intertwined short twigs branches that are hard and thorny. The leaves are generally in whorls of three, 10-18 cm in length and 7-11 cm in diameter withobovate shape of grayish white [3, 4]. Locally in Nigeria, it is called gáudȅnkúúráá (Hausa) and langbà (Nupe). *G. ternifolia* is used traditionally for the treatment of various ailments and diseases [5-8]. Ethno-medically, a decoction extract from the fruit has been used as a remedy for the treatment of malaria and eye complaints [9, 10]. Locally, *G. ternifolia* branches are chewed as toothbrushes [11]. Research has shown that both fresh leaves and fruit extracts of *G. ternifolia* could be used in managing hemorrhoids, cancer, and diabetes [12]. Phytochemical evaluation on the leaves, fruits, and roots of *G. ternifolia* revealed the presence of saponins, reducing compounds, sterols, triterpenes, and polyphenolic substances such as tannins, flavonoids, coumarins, and anthocyanins [13]. However, to the best of our knowledge, there have been no reports on the essential oil compositions of *G. ternifolia*. We report herein the chemical composition of *G. ternifolia* leaf essential oil aimed at characterizing the constituents of the essential oil for potential future use in pharmaceutical applications.

2. Materials and Methods
2.1 Plant Materials and Identification
The fresh leaves of *G. ternifolia* were collected in February 2021 from Kufena Hills (11° 06′ 40.61″ N, 7° 43′ 21.72″ E), located in Zaria Local Government in the Northern part of Kaduna State. The plant sample was authenticated by Mr. Namadi Sanusu of the Botany Department, Ahmadu Bello University, Zaria, where voucher specimen 070717 was deposited. The leaves of *G. ternifolia* were air-dried in the shade for 5-7 days and then pulverized using an electric blender before extraction.

2.2 Isolation of Essential Oil
The oil samples from the leaves of *G. ternifolia* were isolated using hydrodistillation. The plant material (500 g) was introduced into a 5-L flask, and distilled water was added until it covered the sample.

Hydrodistillation was carried out for four h in an all-glass modified Clevenger apparatus according to the British pharmacopeia. The distillate was extracted with *n*-hexane, transferred to a pre-weighed amber sample bottle, and dried with anhydrous sodium sulfate to eliminate traces of water. The oils were kept under refrigeration (4 °C) until ready for analysis. The yield of the oil was 0.62% on dry weight basis for leaves of *G. ternifolia*.

2.3 Gas Chromatographic-Mass Spectral Analysis

The essential oil was analyzed by Gas Chromatography-Mass spectrometry (GC-MS) using a Shimadzu GCMS-QP2010 Ultra operated in the electron impact (EI) mode (electron energy = 70 eV), scan range = 40-400 atomic mass units, scan rate = 3.0 scan/s, and GC-MS solution software. The GC column was a ZB-5 fused silica capillary column (30 m length × 0.25 mm inner diameter) with a (5% phenyl)-polydimethylsiloxane stationary phase and a film thickness of 0.25 µm. The carrier gas was helium with a column head pressure of 553 kPa and a flow rate of 1.37 mL/min. The injector temperature was 250 °C, and the ion source temperature was 200 °C. The GC oven temperature was programmed for 50 °C initial temperature, the temperature increased at a rate of 2 °C/min to 260 °C. A 5% *w/v* solution of the sample in CH_2Cl_2 was prepared, and 0.1 µL was injected with a splitting mode (30:1). Identification of the constituents of the volatile oil was achieved based on their retention indices and by comparison of their mass spectral fragmentation patterns with those reported in the databases [14-17]. The quantification of the constituents of each essential oil was done by the external standard method using the calibration curves generated by running the GC analyses of representative standard compounds for each class [18].

3. Results and Discussion

The essential oil was light yellow in color, with a yield of 0.62% (v/w). The GC-MS analysis revealed a total of 66 components identified, representing 98.6% of the total composition (Table 1). The essential oil was dominated by sesquiterpene hydrocarbons (56.2%), oxygenated sesquiterpenoids (39.5%), with minor amounts of oxygenated mono terpenoids and trace quantities of mono terpene hydrocarbons. The main sesquiterpene hydrocarbon components were *trans*-α-bergamotene (16.4%), *trans*-calamenene (8.8%), and δ-cadinene (8.0%). The main oxygenated sesquiterpenoids were spathulenol (10.9%), τ-muurolol (6.1%), α-cadinol (5.9%), and τ-cadinol (5.0%), all other constituents identified occurred in minute quantity.

Table 1: Chemical composition of the leaf essential oil of *Gardenia ternifolia*

S/N	RT	RI_{calc}	RI_{db}	Compound	% Composition
	12.5	934	933	α-Pinene	t
	15.0	979	978	β-Pinene	t
	15.4	987	986	6-Methylhept-5-en-2-one	t
	17.9	1026	1025	*p*-Cymene	t
	18.1	1030	1030	Limonene	t
	18.5	1036	1034	(*Z*)-β-Ocimene	t
	19.2	1047	1045	(*E*)-β-Ocimene	t
	21.8	1087	1087	Terpinolene	t
	22.8	1102	1101	Linalool	0.3
	23.2	1107	1107	Nonanal	0.1
	29.4	1197	1195	α-Terpineol	0.2
	31.0	1220	1219	β-Cyclocitral	0.1
	35.7	1290	1287	Dihydroedulan IA	t
	35.8	1292	1293	Thymol	0.1
	39.6	1348	1349	α-Cubebene	0.3
	41.0	1371	1371	α-Ylangene	0.1
	41.5	1377	1377	α-Copaene	2.3
	41.6	1380	1380	(*E*)-β-Damascenone	0.2
	42.2	1389	1387	β-Cubebene	0.1
	43.5	1408	1406	α-Gurjunene	0.1
	43.9	1414	1416	*cis*-α-Bergamotene	1.1
	44.2	1420	1420	α-Santalene	2.4
	44.4	1423	1421	(*E*)-α-Ionone	0.1
	44.9	1431	1430	β-Copaene	0.1
	45.1	1435	1432	*trans*-α-Bergamotene	16.4
	45.4	1440	1438	Aromadendrene	0.1
	46.0	1449	1450	Geranyl acetone	1.6
	46.1	1450	1451	*trans*-Muurola-3,5-diene	0.3
	46.3	1454	1452	(*E*)-β-Farnesene	0.2
	46.5	1457	1454	α-Humulene	0.4
	46.8	1461	1458	*allo*-Aromadendrene	0.3
	46.9	1463	1461	*cis*-Cadina-1(6),4-diene	0.1
	47.5	1573	1475	*trans*-Cadina-1(6),4-diene	0.7
	47.7	1476	1478	γ-Muurolene	1.2
	47.9	1479	1481	(*E*)-β-Ionone	0.3
	48.1	1482	1482	*ar*-Curcumene	0.2
	48.3	1485	1483	*trans*-β-Bergamotene	3.6
	48.6	1490	1489	β-Selinene	0.6
	48.8	1493	1493	*trans*-Muurola-4(14),5-diene	0.7
	49.0	1497	1497	α-Selinene	1.0

	49.2	1500	1500	α-Muurolene	2.2
	49.5	1506	1505	(*E*,*E*)-α-Farnesene	0.1
	49.7	1509	1508	β-Bisabolene	0.3
	49.9	1512	1511	(*Z*)-γ-Bisabolene	0.2
	50.1	1514	1514	γ-Cadinene	2.7
	50.4	1520	1520	δ-Cadinene	8.0
	50.6	1523	1521	*trans*-Calamenene	8.8
	51.2	1534	1533	*trans*-Cadina-1,4-diene	0.7
	51.5	1538	1538	α-Cadinene	0.6
	51.7	1542	1541	α-Calacorene	0.6
	52.9	1563	1563	(*E*)-Nerolidol	0.9
	53.6	1575	1568	Dendrolasin	0.2
	53.9	1579	1576	Spathulenol	10.9
	54.1	1583	1582	Caryophyllene oxide	0.5
	54.4	1588	1590	Globulol	0.5
	55.5	1606	1605	Ledol	1.5
	55.7	1611	1613	Humulene epoxide II	0.3
	56.1	1617	1616	1,10-di-*epi*-Cubenol	0.6
	56.8	1630	1631	1-*epi*-Cubenol	2.1
	57.1	1636	1629	*iso*-Spathulenol	1.4
	57.6	1645	1643	τ-Cadinol	5.0
	57.7	1647	1645	τ-Muurolol	6.1
	57.9	1649	1651	α-Muurolol (= δ-Cadinol)	3.1
	58.4	1658	1655	α-Cadinol	5.9
	59.3	1674	1677	Cadalene	0.4
				Mono terpene hydrocarbons	traces
				Oxygenated mono terpenoids	0.6
				Sesquiterpene hydrocarbons	56.2
				Oxygenated sesquiterpenoids	39.4
				Others	2.4
				Total identified	98.6

RT = Retention time (min). RI_{calc}= Calculated retention index values. RI_{db}= Retention index values from the databases. t = trace (< 0.05%).

While the floral essential oils of *Gardenia species* have been the subject of several studies [19-22], leaf essential oil compositions of the genus are relatively sparse. The leaf essential oil of *Gardenia jasminoides* J. Ellis from Nigeria was found to contain pentadecanal (49.2%), geranial (12.3%), *ar*-turmerone (8.1%), and 10-*epi*-γ-eudesmol (6.2%) as major components [23]. Thus, the chemical compositions of *G. ternifolia* and *G. jasminoides* leaf essential oils are vastly different and there are no obvious phytochemical trends in the genus.

4. Conclusions

Since no information has been reported on the chemical composition of *G. ternifolia*, this represents the first report of the chemical composition of *G. ternifolia* leaf essential oil. Additional analyses of *Gardenia* essential oils are needed to provide a more complete picture of the volatile phytochemistry of the genus.

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7. Conflicts of Interest

The authors declare no conflict of interest.

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