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Chemical Composition of Essential Oils of *Plumeria rubra* L. Grown in Nigeria

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Authors' contributions

This work was carried out in collaboration between all authors. Author OAL designed the study, isolation of the oils and wrote part of the manuscript. Author IAO managed the literature searches and wrote the final draft of the manuscript. Author ARO managed the analyses of the GC and GC/MS. All authors read and approved the final manuscript.

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ABSTRACT

The chemical compositions of essential oils obtained by hydrodistillation of the leaves and flowers of pink-flower *Plumeria rubra* L., grown in Nigeria were being reported. The chemical analysis was performed by means of gas chromatography-flame ionization detector (GC-FID) and gas chromatography-mass spectrometry (GC-MS) techniques. The major leaves oil constituents were (*Z*)-β-farnesene (16.0%), α-patchoulene (13.0%), limonene (12.1%), (*E*)-β-farnesene (10.8%), α-copaene (7.2%) and phytol (6.3%). However, the quantitative significant compounds of the flowers oil were (*E*)-non-2-en-1-ol (15.7%), limonene (10.8%), phenyl acetaldehyde (9.0%), *n*-tetradecanal (8.8%), γ-elemene (6.5%) and (*E,E*)-α-farnesene (6.1%). This is the first report on the volatile constituents from the leaves of *Plumeria rubra.*
Aims: The aim of the of the present study was to examine the constituents of the leaves and flowers oils of *P. rubra* grown in Southwest Nigeria in details, and to compare the results obtained

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with those reported earlier.
Study Design: Isolation of essential oils from the leaves and flowers of *Plumeria rubra* and determination of their chemical constituents.
Place and Duration of Study: Fresh plant materials of *P. rubra* (flowers and leaves) were collected from a location within the Campus of Lagos State University, Ojo, Lagos State, Nigeria, in October 2013.
Methodology: Fresh leaves and flowers were hydodistilled in an all glass Clevenger apparatus and their chemical constituents were analyzed by GC and GC/MS.
Results: A total of twenty six compounds were identified in the leaves and the major ones were (*Z*)-β-farnesene (16.0%), α-patchoulene (13.0%), limonene (12.1%), (*E*)-β-farnesene (10.8%), α-copaene (7.2%) and phytol (6.3%) while the flowers had twenty seven compounds with (*E*)-non-2-en-1-ol (15.7%), limonene (10.8%), phenyl acetaldehyde (9.0%) and *n*-tetradecanal (8.8%) occurring in higher percentages.
Conclusion: The chemical composition of the volatile compounds differed from each other and from data reported previously from other parts of the world.

Keywords: Plumeria rubra; Apocynaceae; essential oil composition; fatty acids; terpenes.

1. INTRODUCTION

Plumeria rubra L. (Apocynaceae) grows as a spreading shrub or small tree to a height of 2-8 m and similar width. It has a thick succulent trunk and sausage-like blunt branches covered with a thin grey bark. The large green leaves can reach 30 to 50 cm long and are arranged alternately and clustered at the end of the branches. The flowers are terminal, appearing at the ends of branches over the summer. Often profuse and very prominent, they are strongly fragrant, and have five petals. The colours range from the common pink to white with shades of yellow in the centre of the flower. They produce seed of about 20-60 winged seeds [1]. The decoction of *P. rubra* has traditionally been used to treat asthma, constipation, promote menstruation and reduce fever. The fruit was reported to be been used as an abortifacient [2]. The flowers are aromatic and are used for the control of diabetes mellitus while the leaves are used to ameliorate ulcers, leprosy, inflammation and rubifacient. The milky sap of the stem and leaf has been applied to skin diseases such as herpes and scabies [2]. Extracts of the plants are known to possess some biological activities of importance such as antimicrobial, anti-inflammatory, analgesic, anthelmintic, antioxidant, antipyretic, abortifacient, antiulcer, antifertility, antitumor, anticancer and hypolipidemic [2-7].

Phytochemical screening showed that *P. rubra* contained several biologically active compounds such as plumericin and isoplumericin that displayed molluscicidal, cytotoxic and antibacterial activities as well as cyanidin 3-*O*-β-(2″-glucopyranosyl-*O*-β-galactopyranoside) and cyanidin-3-*O*-β-galactopyranoside [8] which were responsible for the attractive colours of the flowers of red *P. rubra*. Antimicrobial iridoids plumeridoids A-C, epiplumeridoid C [9] and cytotoxic iridoids, fulvoplumierin, allamcin, plumericin and allamandin were the other constituents of *P. rubra* [2].

Literature reports on the essential oil contents of *P. rubra* have focussed mostly on the flowers. The essential oils of *P. rubra* and its cultivars exhibited high chemical variations depending on the origin. Alkanes, terpene hydrocarbons, oxygenated terpenes, aromatic compounds, alcohols and fatty acids were the main classes of compounds present in the analysed oils from different parts of the world [10-22].

Although the composition of the flower oils of *P. rubra* from Nigeria [10] and other parts of the world have been substantially investigated (Table 1), much less is known about the composition of the leaf oil. The objective of the present study was to examine the chemical constituents of the leaves and flowers oils of *P. rubra* grown in Southwest Nigeria in details, and to compare the results obtained with those reported earlier. The chemical analysis of essential oil of some plant species of Nigeria flora has been reported [23].

2. MATERIALS AND METHODS

2.1 Plant Materials

Fresh plant materials of *P. rubra* (flowers and leaves) were collected from a location within the

Table 1. Major compounds identified in the essential oils of *P. rubra* from literature

Parts	Origin	Major constituents	References
Flower (ro)[hy]	Nigeria	heneicosane (19.15%) nonadecane (15.63%), citronellol (14.63%), geraniol (9.17%)	10
Flower (ro)[hy]	Malaysia	phenylethyl benzoate (12.3%), dodecanoic acid (11.8%), hexadecanoic acid (9.3%)	11
Flower (red) [hy]	Malaysia	hexadecanoic acid (27.2%), linoleic acid (20.7%), tetradecanoic acid (18.9%), dodecanoic acid (10.6%)	11
Flower (pink) [hy]	Malaysia	dodecanoic acid (30.8%), tetradecanoic acid (17.4%), hexadecanoic acid (9.8%), nonadecane (8.2%)	12
Flower (or) [hy]	Malaysia	benzyl salicylate (20.9%), (*E*)-nerolidol (14.1%), benzyl benzoate (8.6%)	12
Flower [mw]	China	9-hexacosene (14.6%), *n*-octadecanal (11.5%), *n*-octadecanol(8.4%), lupeol acetate (8.3%), *n*-hexadecanoic acid (7.9%)	13
Flower [a, b, std]	China	d-nerolidol, farnesol, benzyl benzoate, geranyl benzoate, neryl linalool isomer, terpinyl isovalerate	14
Flower [a, std]	China	d-nerolidol, farnesol, benzyl benzoate, geranyl benzoate	15
		palmitic acid, tetradecanoic acid	16
Flower [a, b, scde]		1,2-benzenedicarboxylic acid (66.11%)	17
Flower [b, sde]	Hawaii	β-phenylethyl alcohol, phenyl acetaldehyde, methyl cinnamate	18
Flower [b, sde]	Hawaii	linalool, phenylacetaldehyde, *trans,trans*-farnesol, phenylethyl alcholo, geraniol, α-terpineol, neral,geranial	19
Flower [b, std]	Egypt	α-pinene, 2-carene, β-pinene, α-phellandrene, p-cymene, linalool, phenylalcohol, citral	20
Leaf [a, b, hy]	Egypt	farnesol, geraniol, phenylethyl benzoate, methyl pentadecane, terpinolene	21
Flower [b, std]	Cuba	butyl oleate (13.8%), butyl palmitate (11.5%), methyl palmitate (9.7%), methyl oleate (9.3%), linalool (8.2%)	22

[a]Plumeria rubra var. acutifolia; [b] quantitative data not available to authors; hy, hydrodistillation; mw, microwave extraction; sdce, supercritical carbon dioxide fluid extraction ; sde, steam distillation and extraction; std, steam distillation; ro, red-orange; or, orange

Campus of Lagos State University, Ojo, Lagos State, Nigeria, in October 2013. Identification of the plant materials was carried out at the Department of Botany, University of Lagos. A voucher specimen (LUH 5805) was deposited at the University Herbarium.

2.2 Oil Isolation

Fresh flowers (50 g) and leaves (300 g) were separately subjected to hydrodistillation in a Clevenger-type glass apparatus for 3 h in accordance with the established specification [24]. The distilled oils were preserved in sealed sample tubes and stored under refrigeration until analysis.

2.3 Gas Chromatography (GC) Analysis

GC analysis of the oil was carried out on a Hewlett Packard HP 6820 Gas Chromatograph equipped with a FID detector and HP-5MS column (30 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°C (after 2 min) to 240°C at 5°C/min and the final temperature was held for 10 min. Injection and detector temperatures were maintained at 200°C and 240°C respectively. Hydrogen was the carrier gas at a flow rate of 1 mL/min. 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. Each analysis was performed thrice.

2.4 Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

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

2.5 Identification of Compounds

The constituents of the essential oils were identified by comparing their retention indices with an analysis done under the same temperature-programmed conditions for n-alkanes and the oil on a HP-5 MS column under the same chromatographic conditions. Individual compounds were identified by comparing their mass spectra with the internal reference mass spectra library or with authentic compounds. Confirmation of identity was done by comparing their retention indices with the GC-MS library data [25] and with the mass spectra from literature data [26,27].

3. RESULTS AND DISCUSSION

The identities and the percentage composition of compounds present in *P. rubra* are presented in Table 2. The yields of the volatile oils were 0.12% and 0.23% (v/w) respectively for the leaves and flowers. Sesquiterpene hydrocarbons (54.1%) and monoterpene hydrocarbons (13.9%) were the main classes of compounds identified in the leaves oil. The major constituents of the leaves oil were (*Z*)-β-farnesene (16.0%), α-patchoulene (13.0%), limonene (12.1%) and

Table 2. Chemical composition of essential oils of *Plumeria rubra*

Compounds[a]	RI (Cal.)	RI (Lit.)	% composition		MI
			Flower	Leaf	
α-Pinene	938	932	0.4	-	RI, MS
β-Pinene	979	974	2.9	-	RI, MS
n-Ocotanal	1007	998	2.0	-	RI, MS
(*E,E*)-2,4-Heptadienal	1016	1005	2.0	-	RI, MS, CI
Limonene	1032	1024	10.8	12.1	RI, MS, CI
3,5,5-Trimethylhexanol	1037	1041	-	1.2	RI, MS
Phenyl acetaldehyde	1046	1049	9.0	3.1	RI, MS, CI
γ-Terpinene	1063	1054	0.8	-	RI, MS
1-Octanol	1076	1063	1.1	1.8	RI, MS
Terpinolene	1089	1086	-	1.8	RI, MS
(*E*)-Non-2-en-1-ol	1097	1097	15.7	-	RI, MS, CI
Linalool	1101	1099	-	1.7	RI, MS
n-Nonanal	1105	1100	4.3	3.3	RI, MS
n-Nonanol	1175	1165	-	0.3	RI, MS
β-Cyclocitral	1217	1217	-	1.1	RI, MS
Citronellol	1231	1223	-	0.3	RI, MS
Geraniol	1254	1249	-	0.5	RI, MS, CI
Geranial	1260	1264	-	0.3	RI, MS, CI
n-Tridecane	1300	1300	-	1.1	RI, MS
Eugenol	1364	1356	1.2	1.1	RI, MS
α-Copaene	1378	1374	30	7.2	RI, MS, CI
β-Elemene	1389	1389	0.7		RI, MS
α-Cedrene	1416	1410	1.2		RI, MS

Compounds[a]	RI (Cal.)	RI (Lit.)	% composition		MI
			Flower	Leaf	
β-Caryophyllene	1420	1417	1.5		RI, MS
γ-Elemene	1432	1434	6.5	0.2	RI, MS
(*Z*)-β-Farnesene	1440	1440	2.0	16.0	RI, MS, CI
α-Patchoulene	1457	1454	-	13.0	RI, MS, CI
(*E*)-β-Farnesene	1465	1454	-	10.8	RI, MS, CI
γ-Muurolene	1476	1478	-	1.4	RI, MS
(*E,E*)-α-Farnesene	1500	1505	6.1	-	RI, MS, CI
B-Bisabolene	1510	1505	-	3.5	RI, MS
δ-Cadinene	1523	1522	1.9	-	RI, MS
β-Sesquiphellandrene	1526	1525	-	2.0	RI, MS
Spathulenol	1575	1577	0.7	-	RI, MS
Caryophyllene oxide	1586	1582	1.6	0.3	RI, MS
Viridiflorol	1593	1592	-	0.6	RI, MS
Ledol	1608	1602	1.5	3.4	RI, MS
n-Tetradecanal	1614	1612	8.8	-	RI, MS, CI
Hexadecanal	1812	1817	2.2	-	RI, MS
n-Hexadecanol	1879	1874	1.6	-	RI, MS
n-Octadecanal	2017	2012	5.0	-	RI, MS
Phytol	2125	2122	-	6.3	RI, MS, CI
cis-9-Tricosene	2298	2299	4.4	-	RI, MS, CI
Total			**98.9**	**94.6**	
Monoterpene hydrocarbons			**14.9**	**13.9**	
Oxygenated monoterpenes			**1.2**	**5.0**	
Sesquiterpene hydrocarbons			**22.9**	**54.1**	
Oxygenated sesquiterpenes			**3.8**	**4.3**	
Fatty acids			**22.0**	**-**	
Diterpenes			**-**	**6.3**	
Aliphatic compounds			**25.1**	**7.7**	
Aromatic compounds			**9.0**	**3.1**	

[a]Elution order on HP-5MS column; RI (Cal.) Retention indices relative to n-alkanes on HP-5MS column; RI (Lit.) Literature retention indices; - Not identified; MI, Mode of identification; MS, Mass spectrum; CI, Co-injection with an authentic sample

(*E*)-β-farnesene (10.8%). Also present in significant amounts were α-copaene (7.2%) and phytol (6.3%). The minor compounds include β-bisabolene (3.5%), ledol (3.4%), *n*-nonanal (3.3%) and phenyl acetaldehyde (3.1%).

The chemical classes of compounds present in the flowers oil were aliphatic compounds (25.1%), sesquiterpene hydrocarbons (22.9%), fatty acids (22.0%) and monoterpene hydrocarbons (14.9%). The quantitatively significant compounds of the flower oil were (*E*)-non-2-en-1-ol (15.7%), limonene (10.8%), phenyl acetaldehyde (9.0%) and *n*-tetradecanal (8.8%), with significant quantities of γ-elemene (6.5%), (*E*,*E*)-α-farnesene (6.1%), octadecanal (5.0%), *cis*-9-tricosene (4.4%), *n*-nonanal (4.3%) and α-copaene (3.0%).

Few compositional variations were observed between the studied oil samples. (*E*)-Non-2-en-1-ol, the main compound of the flower oil was not detected in the leaf while α-patchoulene and (*E*)-β-farnesene, present in leaves oil were not identified in the flowers oil. In addition, while fatty acids were conspicuously absent in the leaves oil, none of the diterpene compound could be detected in the flower. However, compounds such as limonene, phenyl acetaldehyde and (*Z*)-β-farnesene were identified in both samples though with varying proportion. These observations may be based on the fact that different parts of the same plant may contain different chemical substances [23].

Although heneicosane and nonadecane were previously identified as the major compounds of *P. rubra* flowers oil from Nigeria [10], these compounds were not present in this study which also has low contents of citronellol and geraniol. The main compounds of essential oil of pink-flower *P. rubra* from Malaysia namely dodecanoic acid, tetradecanoic acid, hexadecanoic acid and nonadecane, were not identified in this oil sample from Nigeria. Moreover, the main constituents of the present

oil samples namely non-2-en-1-ol, limonene, phenyl acetaldehyde, γ-elemene and (*E*,*E*)-α-farnesene, were conspicuously absent in the Malaysian oil sample [12]. It was noted that non-2-en-1-ol and limonene were not previously reported to be main compounds of previously investigated *P. rubra* oils (Table 1). Although, phenyl acetaldehyde was present in the Nigeria, Egypt [20,21] and Hawaii [18,19] samples, other variations were observed in the compositions of these samples. Some compounds such as 1,2-benzenedicarboxylic acid 9-hexacosene, *n*-octadecanal, *n*-octadecanol, lupeol acetate, palmitic acid, tetradecanoic acid and *n*-hexadecanoic acid commonly observed from China oil samples [13-17] were not detected in the Nigeria grown *P. rubra* oils. The chemical compounds present in the essential oil of *P. rubra* from Cuba [22] were not identified in the Nigerian samples. The origin, environmental conditions, handling procedure, extraction methods, age and nature of the plant etc are some of the factors that may be responsible for the variations in the chemical composition of essential oils of *P. rubra* from different parts of the world.

The components present in the essential oils may be of economic importance. For example, limonene among others posses antimicrobial activity [28] while (*E*)-β-farnesene was used as an insecticide for the control of aphids [29]. On the other hand, (*E*)-non-2-en-1-ol is a scent material and also useful as an acaricide [30].

4. CONCLUSION

For the first time the compositions of essential oil from the leaf of *P. rubra* grown in Nigeria are being reported. In addition qualitative and quantitative differences were observed between the oil compositions from Nigeria and other parts of the world. These differences may be probably due to ecological and geographical conditions between Nigeria and other parts of the world as well as the age and nature of the plant, handling procedure etc.

CONSENT

Not applicable.

ETHICAL APPROVAL

Not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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