



GC-MS Analyses of the Volatile Oil Constituents of the Leaf of *Landolphia owariensis* P. Beauv (Apocynaceae)

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Authors' contributions

This work was carried out in collaboration between all authors. Authors SEO and OFK designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript.

Author HOE managed the analyses of the study and the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Aim: Evaluation of the volatile oil constituents of the leaves of *Landolphia owariensis* collected from Suleja, Niger State, North Central Nigeria in July 2015, where it is valued as an important medicinal plant used in folk medicines.

Methodology: Fresh leaves were hydrodistilled in an all-glass Clavenger apparatus and their chemical constituents were analyzed by GC-MS.

Results: The examined material contained 0.06% w/w of essential oil. A total of thirty-two compounds were identified in the essential oil, accounting for 86.33% of the oil composition. The main components of the essential oil were pentadecanal (13.63%), 1-dodecanol (6.32%),

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tetradecanol (5.83%), hexadecatrienal (5.62%), squalene (4.63%), β -ionone (3.25%), α -ionone (2.38%), supraene (3.01%), α -farnesene (3%), carophyllene oxide (2%) and (-)-spathulenol (1.78%).

Conclusion: *Landolphia owariensis* leaf essential oil could be used in pharmaceutical formulations or in perfumery and as a renewable source of pentadecanal and 1-dodecanol.

Keywords: *Landolphia owariensis*; Apocynaceae; leaves; essential oil; pentadecanal.

1. INTRODUCTION

The *Landolphia* genus (Apocynaceae), which consists of 63 species, is well distributed from Guinea to West Cameroon and extending across Central Africa to Sudan, Uganda and Southern Tanganyika [1]. *Landolphia owariensis*, with a characteristic odour, is the only one in West Africa out of the three varieties recognised, and its botanical characters have been described in literature [2]. In Congo, sap expressed from the leaves is dipped into the eyes and used to wash the patient's face as a treatment for giddiness and epilepsy. In Ghana the acid-pronounced pulp of the fruit is edible and is sometimes used to season food [3]. In some Nigeria localities, decoction of the leaf is used to cure malaria, fever and rheumatic pain (Personal communication, Muazzam Ibrahim 2015).

Nwaogu et al. [4] reported the phytochemical composition of *Landolphia owariensis* leaf extract namely alkaloids, tannins, saponins and flavonoids. *Landolphia owariensis* leaf extract had good antioxidant activity in DPPH assay, compared to ascorbic acid [5]. Large amount of fat in the form of oil with agreeable colour and odour which have potentials for use as domestic oil has been reported to be present in the seed extract of *Landolphia owariensis* [6]. It has also been reported that the gas chromatographic analysis on the methyl ester mix of *Landolphia owariensis* string seed pulp gave hexadecanoic acid (palmitic acid) as the principal fatty acid Palmitoleic acid and linoleic acid ranked second and third respectively [7]. However, to our knowledge no phytochemical investigations of the leaf essential oil of *Landolphia owariensis* have been reported to date. Thus the present study aims to evaluate the chemical constituents of *Landolphia owariensis* leaf essential oil.

2. MATERIALS AND METHODS

2.1 Chemicals

Hexane and anhydrous sodium sulphate are of analytical grade obtained from Sigma-Aldrich (Germany).

2.2 Plant Material

The leaves of *L. owariensis* were collected from Suleja, Niger State, North Central Nigeria in July 2015 by Mallam Muazzam Ibrahim. The identification and authentication of the plant was done by Dr. Grace Ugbabe at the Herbarium of the National Institute for Pharmaceutical Research and Development, Abuja, where the voucher specimen (NIPRD/H/6692) was deposited.

2.3 Essential Oil Isolation

The dried leaves of *L. owariensis* were chopped into small pieces. 400 g of the dried plant material was subjected to hydrodistillation using Clavenger type apparatus of 2 litre capacity. One litre of distilled water was added to the material. The mixture was heated on heating mantle at 100°C. The hydrodistillation was continued for four hours. The light yellow essential oil obtained was dried over anhydrous sodium sulphate, stored in sealed vials and used within 30 minutes for analysis.

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

GC-MS analysis was performed using a Shimadzu GCMS QP-2010 system, with QP-2010 mass selective detector MSD, operated in the EI mode (electron energy = 70 eV), scan range = 45-400 amu, and scan rate = 3.99 scans/sec (Shimadzu Corporation, Kyoto Japan) equipped with a fused silica capillary column (30 m x 0.25 mm, i.d.) coated with 5% phenylmethylpolysiloxane (film thickness 0.25 μ m) Optima 5 ms [Macherey-Nagel GmbH & Co, Germany] and Shimadzu GCMSsolution software. Helium (flow rate 1.61 ml/min) was used as a carrier gas. The program used for GC oven temperature was 60°C, then it was incremented to 180°C at a rate of 10°C/min, held at 180°C for 2 minutes, then it was increased to 280°C at a rate of 15°C/min, then again held at 280°C for 4 minutes. Injector temperature was 250°C, ion source temperature was 200°C and interface temperature was 250°C. Diluted sample

(1/100 in hexane, v/v) of 1.0 µl was injected using auto-sampler and in the split mode with split ratio of 1:100.

The analysis was performed in triplicate. Individual constituents were identified by referring to compounds known in the literature data [8] and also by comparing their mass spectra with known compounds and NIST Mass Spectral Library (NIST 11).

3. RESULTS AND DISCUSSION

The light yellow essential oil from *Landolphia owariensis* leaf was obtained in 0.32 ml (0.25 g; 0.06% w/w) yield. The oil was subjected to GC-MS analysis. A total of 32 compounds were identified in the essential oil, accounting for 86.33% of the oil composition. The components identified, their retention times and their percentage area are summarized in Table 1, and

Table 1. Chemical composition of leaf essential oil from *Landolphia owariensis*

Compounds	% Composition	Retention time
4α-Methyldecalin-1-yl-acetate	0.4±0.01	8.136
Tridecane	0.99±0.05	8.285
Citronellyl acetate	4.05±0.02	8.939
Tetradecane	2.02±0.01	9.608
α-Ionone	2.38±0.01	9.977
cis-Geranylacetone	3.3±0.08	10.231
1-Dodecanol	6.32±0.04	10.521
β-Ionone	3.25±0.06	10.715
Hexadecane	1.36±0.02	10.858
α-Farnesene	3±0.05	10.956
5,5,8a-Trimethyl-3,5,6,7,8,8a-hexahydro-2H-chromene	1.19±0.01	11.14
β-Nerolidol	1.73±0.01	11.629
n-Tridecan-1-ol	1.12±0.05	11.733
Supraene	3.01±0.04	11.813
(-)-Spathulenol	1.78±0.03	11.92
Caryophyllene oxide	1.99±0.1	12.001
Hexadecane	2.19±0.01	12.057
1-Tetradecanol	5.83±0.02	13.023
Pentadecanal	13.63±0.01	13.598
6-Phenyldodecane	1.2±0.02	13.867
Eicosane	1.34±0.03	14.941
2-Phenyldodecane	0.92±0.01	15.033
6-Phenyl-tridecane	1.06±0.03	15.309
5-Phenyl-tridecane	1.15±0.01	15.4
Hexahydrofarnesyl acetone	2.68±0.05	15.496
Benzyl salicylate	1.88±0.01	15.81
Hexadecatrienal <7,10,13-cis,cis,cis->	5.62±0.04	16.058
Farnesyl acetone	2.14±0.02	16.319
Methyl palmitate	1.68±0.03	16.386
Ethyl palmitate	1.49±0.04	17.053
Squalene	4.63±0.04	17.242
Isopropyl palmitate	1.00±0.02	17.328
Total	86.33%	
Oxygenated monoterpenes	4	
Oxygenated sesquiterpenes	10	
Triterpene hydrocarbons	4	
Fatty acids	6	
Ketones	4	
Ethers	2	
Aliphatic hydrocarbons	14	
Aliphatic alcohols	8	
Aldehydes	4	
Aromatic compounds	12	

are arranged in their order of elution on Optima 5 ms capillary column. The essential oil from *Landolphia owariensis* leaf showed diverse composition. The most representative class of compounds in the oil was aliphatic hydrocarbons (14%), followed by aromatic compounds (12%), oxygenated sesquiterpenes (10%), aliphatic alcohols (8%), fatty acids (6%), triterpene hydrocarbons (4%), oxygenated monoterpenes (4%), aldehydes (4%), ketones (4%), and small amount of ethers (2%). The major components of the essential oil identified were pentadecanal (13.63%) an aliphatic aldehyde, 1-dodecanol (6.32%), tetradecanol (5.83%), hexadecatrienal (5.62%), squalene (4.63%), 3-buten-2-one (3.25%), supraene (3.01%), α -farnesene (3%), α -ionone (2.38%), carophyllene oxide (2%) and (-)-spathulenol (1.78%). The oil contained important oxygenated monoterpenes such as citronellylacetate and geranylacetone <cis>, as well as the alcohols dodecan-1-ol, tetradecan-1-ol, spathulenol and tridecan-1-ol. Also the oil showed presence of two oxygenated sesquiterpenes namely hexahydrofarnesyl acetone and caryophyllene oxide.

Pentadecanal has been reported as the major constituent (38.5%) of *Mitracarpus scaber* leaf essential oil [9] and *Coriandrum sativum* L. herb had been reported to contain 4.2% of 1-dodecanol. 1-Dodecanol and α -ionone are used as flavor and fragrance agents [10]. α -ionone α -ionone has a floral type odour and flavour. β -ionone is a constituent of rose flower (*Rosa damascena*) and is also used in fragrances. It is used in all areas of perfumery. The ionones are derived from the degradation of carotenoids [11].

4. CONCLUSION

GC-MS analysis of the compositions of *Landolphia owariensis* leaf essential oil identified thirty-two volatile constituents. The main components of the essential oil were pentadecanal (13.63%), 1-dodecanol (6.32%), tetradecanol (5.83%), hexadecatrienal (5.62%), squalene (4.63%), 3-buten-2-one (3.25%), α -ionone (2.38%), supraene (3.01%), α -farnesene (3%), carophyllene oxide (2%) and (-)-spathulenol (1.78%). Based on the results, this plant is a possible natural source of pentadecanal and 1-dodecanol.

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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