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Volatile composition of the floral essential oil of *Hibiscus sabdariffa* L. from Nigeria

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Abstract
The chemical composition of essential oil obtained from the hydrodistillation of air-dried flowers of *Hibiscus sabdariffa* L (Malvaceae) are reported. The components of the essential oils were analysed by means of gas chromatography (GC) and gas chromatography coupled with mass spectrometry (GC-MS). The major compounds identified in the essential oil were hexadecanoic acid (64.3%) and linoleic acid (22.7%).

Keywords: *Hibiscus sabdariffa*, essential oil composition, fatty acids, hexadecanoic acid, linoleic acid

1. Introduction
Hibiscus sabdariffa L. (Malvaceae) is an erect, mostly branched, annual shrub. It is native to tropical Africa and can also be found in India and Southeast Asia including Thailand and Malaysia. The stems are reddish in colour and up to 3.5 m tall, with a deep penetrating taproot. The alternate leaves are variously colored, dark green to red with long-petiolate, palmately divided into 3-7 lobes and serrate margins. The flowers are large, short-peduncled, red to yellow with dark center. The large and fleshy sepals become enlarged and succulent, making excellent jelly [1]. The calyces of the plant are used as a refrigerant in the form of tea (Zobo), to make jellies and jams [2]. The red calyces of *H. sabdariffa* are used in the preparation of a flavorful and tart cold or hot beverage. These calyces have been shown to contain numerous bioactive compounds.
In Nigeria, the aqueous extract of the calyx is prepared and taken as beverage after adding spices and flavoring agents. This beverage popularly known as 'zobo' has received widespread acceptance and has replaced canned and bottled carbonated drinks in many communities in Nigeria.
H. sabdariffa was reported to contain proteins, fats, carbohydrates, flavonoids, acids, minerals and vitamins [2]. Several reports describing the antihypertensive, hepatoprotective, antihyperlipidemic, anticancer, antidiabetic, cytotoxicity, antibacterial, immunomodulatory, antinociceptive, anti-inflammatory, antidiarrheal and antioxidant activities of the plant among others have been published [2-15]. There are published reports on the phytochemical constituents of different plant parts of H. sabdariffa. Two anthocyanins present in the plant namely delphinidin-3-sambubiside and cyanidin-3-sambubioside are responsible for the deep red pigment of the calyces and were also found to be the major contributors to antioxidant activity [16- 18]. Other compounds found in the calyces include delphinidin-3-glucoxyloside, hibiscin, gossypetin, gossypetin, quercetin, myricetin, hibiscetin, hibiscetrin, sabdaritrin, galactose, galacturonic acid, rhamnose, gallic and protocatechuic acid which has also been shown to inhibit the carcinogenic effect of various chemicals in different tissues of rats, including the liver, oral cavity, colon, glandular stomach tissue, bladder, and skin [17]. The chemical compositions of some essential oils of the different parts of *H. sabdariffa* from other parts of the world have been reported. The oil of *H. sabdariffa* from China was characterised by the abundance of tributyl phosphate (18.63%), nonacosane (11.84%) and heneicosane (8.62%) [19]. The composition of the seed oil from Cuba was dominated by linalool and α-terpineol [20] while linoleic acid (43.2%), oleic acid (24.7%) and palmitic acid (17.3%) were the main constituents identified in the seed oil from Austria [21]. In addition, the major volatiles identified in different samples

of *H. sabdariffa* were hexanal, *cis* and *trans*-dehydroxylinalool oxide, octanal, 1-octanol, linalool, furfural, decanal, nonanal, 1-nonanol, acetic acid, 6-methyl-5-hepten-2-one, 1-octen-3-ol, (*E*)-2-nonenal, (*E,E*)-2,4-nonadienal and (*E,E*)-2,4-decadienal [22]. The calyx oil of *H. sabdariffa* was reported to be rich in geraniol, menthol and γ-undecalactone [18]. However, (*Z*)-3-hexenol (32.8%), 2-hexenol (27.7%), 1-hexanol (21.9%) and α-terpineol (12.7%) were present in the fresh calyces from Taiwan [23] while the frozen samples contained α-terpineol (18.0%), eugenol (9.0%) and linalool oxide (7.1%). The main volatile components of the plant from Mexico were known to be made up of limonene, ethyl hexadeanoate, furfural and *cis*-linalool oxide [24]. It could be seen that there is no homogeneity in the volatile constituents of *H. sabdariffa*.

In continuation of our studies on the chemical composition of essential oils from aromatic and medicinal plants growing in Nigeria [25], the present investigation reports the chemical compositions of the floral essential oil of *H. sabdariffa* growing in Nigeria.

2. Materials and methods

2.1 Plant collection

Mature and fresh flowers of *H. sabdarifa* were collected from a location in Lagos, Nigeria, in June 2013 and were authenticated by the Curators at the Herbarium, Forestry Research Institute of Nigeria (FRIN), Ibadan, Nigeria, where a voucher specimen (FHI 5723) was deposited.

2.2 Extraction of essential oil

The air-dried and pulverised flowers of *H. sabdarifa* (250 g) were subjected to hydrodistillation in an all glass Clevenger apparatus for 4 h according to the established procedure [26]. The yield of the essential oil 0.13% (v/w), calculated on a dry weight basis.

2.3 Analysis of the oil sample

Gas Chromatography (GC) analyses 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 °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 200 °C and 240 °C, 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. Gas chromatography-mass spectrometry (GC-MS) analysis of the essential oil was performed on a Hewlett Packard Gas Chromatograph HP 6890 interfaced with a Hewlett Packard 5973 mass spectrometer system equipped with a DB-5 capillary column (30 m x 0.25 mm id, film thickness 0.25 μm) under the same condition as the GC column. The oven temperature was programmed from 70-240 °C at the rate of 5 °C/min. The ion source was set at 240 °C and electron ionization at 70 eV. Helium was used as the carrier gas at a flow rate of 1 mL/min. The scanning range was 35 to 425 amu. Diluted oil in *n*-hexane (1.0 μL) was injected into the GC/MS.

2.4 Identification of compounds

The components of the oil were identified base on the comparison of their retention indices and mass spectra with those standards, Wiley library mass spectra database of the GC/MS system and published data [27, 28].

Table 1: Chemical constituents of essential oil of *H. sabdariffa*

Compounds [a]	RI[b]	RI[c]	Percent composition (%)
n-Nonanoic acid	1273	1272	0.6
Eugenol	1345	1351	0.1
β-Caryophyllene	1417	1419	0.1
10-*epi*-γ-Eudesmol	1613	1622	0.3
τ-Cadinol	1640	1640	0.5
α-Selina-6-en-4-ol	1648	1650	0.2
Bisabolol oxide	1652	1652	0.2
Cadalene	1677	1674	0.1
Tetradecanoic acid	1760	1750	2.1
Hexadecanoic acid methyl ester	1921	1915	2.3
Isophytol	1950	1949	1.6
Hexadecanoic acid	1970	1975	64.3
Heptadecanoic acid	2088	2080	1.2
Linoleic acid methyl ester	2092	2096	2.1
Oleic acid	2110	2097	0.9
Stearic acid methyl ester	2113	2117	0.5
Linoleic acid	2125	2123	22.7
Total			**99.8**
Sesquiterpene hydrocarbons			**0.2**
Oxygenated sesquiterpenes			**1.2**
Diterpenes			**1.6**
Fatty acids			**96.1**
Aliphatic acids			**0.6**
Phenylpropanoids			**0.1**

[a] Elution order on DB-5 column; [b] Retention indices on DB-5 column; [c] Literature retention indices

3. Results & Discussion

The essential oil of *H. sabdariffa* contained 17 compounds representing 99.8% of the total oil content. The percentage of the constituents identified in the oil samples as well as the experimental and literature retention indices are summarized in Table 1. The compounds were arranged in order of elution on DB-5 capillary column. The chemical classes of compounds present in the oil were sesquiterpene hydrocarbon (0.2%), oxygenated sesquiterpenes (1.2%), diterpenes (1.6%), aliphatic compounds (0.6%), phenylpropanoids (0.1%) and fatty acids (96.1%). Monoterpene compounds were conspicuously absent in the essential oil. The major compounds identified in the essential oil were the fatty acids represented by hexadecanoic acid (64.3%) and linoleic acid (22.7%). The minor constituents include hexadecanoic acid methyl ester (2.3%), tetradecanoic acid (2.1%), linoleic acid methyl ester (2.1%), heptadecanoic acid (1.2%) and isophytol (1.6%). The oil composition was low in ubiquitous terpenes. Recently we reported that the major constituents of the leaves of *Hibiscus surattensis* were β-caryophyllene (12.9%), menthol (10.6%), methyl salicylate (9.7%) and camphor (9.2%), and contained low content of hexadecanoic acid (4.3%) [29]. A comparison of the present oil composition and previous studies revealed some qualitative and quantitative variations. It could be seen that some notable compounds such as limonene, α-terpineol, furfural, geraniol. palmitic acid, tributyl phosphate, nonacosane, (*E*,*E*)-2,4-nonadienal, (*E*,*E*)-2,4-decadienal, linalool oxide, aliphatic alcohols such as hexanol and nonanol, as well as 6-methyl-5-hepten-2-one, that were characteristics of previous samples were not identified in the present study. Moreover, hexadecanoic acid was not described previously to be of significant quantity in the oils of *H. sabdariffa.*

This compositional variation between the Nigerian grown sample and the samples analyzed from other parts of the world may be due to the geographical, environmental and ecological variations between as well the age of the plant, handling and processing conditions, genotype etc.

4. Conclusions

The chemical constituents of essential oil obtained from the leaf of *H. sabdariffa* from Nigeria are reported. The result indicated that both qualitative and quantitative variation exists between the present results and previous analysis from other parts of the world. This may be attributable to factors such as environmental conditions and the nature of the plant samples.

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6. References

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