GC-MS ANALYSIS OF THE VOLATILE CONSTITUENTS FROM THE AIR-DRIED LEAVES OF *Terminalia catappa* (LINNAEUS)

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Abstract: The volatile constituents of air-dried leaves (200 g) of *Terminalia catappa* growing in Nigeria was obtained by hydrodistillation using an all glass Clevenger-type glass apparatus. The extracted oil was analyzed using a gas chromatography coupled with mass spectrometry (GC-MS). A total of fourteen constituents' representing 74.32% of *T. catappa* oil with a yield of 0.25% (v/w) was obtained. Hexahydrofarnesyl acetone (12.34%) was the main constituent of the oil followed by 1,3,8-p-Menthatriene (9.38%), 1,2-dimethyl-Cyclooctene (7.30%) Undecane (6.73%), Trans-geranylacetone (6.02%) and 4-ethyl-m-xylene (5.83%). The other constituents' were; Cis-13-Octadecenal (4.83%), Trans-2-Decenol (4.28%), 3-Eicosene, (E)- (4.17%), 1,7-Hexadecadiene (3.47%), m-Propyl-toluene (3.01%), Neophytadiene (2.56%) trans-β-Ionone (2.53%) and 2-ethyl-p-xylene (1.87%). The common classes of compounds in the leave oil are; Hydrocarbons (21.67%), Oxygenated Sesquiterpenes (14.87%), Benzene derivatives (10.71%), Monoterpenes (9.38%), Oxygenated hydrocarbons (9.11%), Oxygenated monoterpene (6.02%) and Sesquiterpenes (2.56%).

Keywords: Combretaceae, essential oil, hexafarnesyl acetone and *Terminalia catappa*

Introduction

The genus Terminalia includes 200 species, some of them with economic importance as ornamentals and timber yielding plants (Mabberley, 2008). *Terminalia catappa* L., a member of the Combretaceae family is a perennial tree species found in almost all the regions of the Country as it thrives well in the tropics. It is commonly called tropical almond, wild almond, Indian almond, sea almond, beach almond and Malabar almond (Untwal and Kondawar, 2006; Orwa *et al.*, 2009). It is mainly found in the southern part of Nigeria, especially in the south-east where they are usually planted for provision of shade and ornamental purposes (Ezeokonkwo and Dodson, 2004; Agu and Menkiti, 2017). It is widely planted throughout the tropics, especially along sandy seashores, for shade, ornamental purposes, sand-dune stabilizer and edible nuts (Brown and Cooprider, 2013). Furnitures and interior building materials are derived from the hardword of the Plant (Lex and Barry, 2006).

Tropical almond has a characteristic 'pagoda' shape because it sends out a single stem from the top centre (Chen *et al.*, 2000). The fruit has an endocarp, which contains an edible oily seed that tastes like almond. The dried raw seeds of tropical almond are highly relished by children in India, Malaysia and Nigeria (Sosulski *et al.*, 1998; Henn *et al.*, 2014). Fruits are produced from about 3 years of age, and the nutritious, tasty seed kernels may be eaten immediately after extraction (Mohale *et al.*, 2009).

The leaves of *Terminalia catappa* are used as a maturant and emollient; the juice is used in the preparation of ointment for scabies, leprosy and other cutaneous diseases (Nair and Chanda, 2008). It has also been investigated for its medicinal activities. These include the *in vitro* and *in vivo* antimetastic effects (Chen *et al.*, 2007), antidiabetic activities (Rao and Nammi, 2006), actinociceptive activity, antiparasitic, antibacterial, antifungal, antimicrobial (Elizabeth, 2005; Nair and Chanda, 2008; Rajarajan *et al.*, 2010), antioxidant (Ko *et al.*, 2002; Mety and Mathad, 2011), anticancer properties (Chu *et al.*, 2007), hepatitis and liver-related diseases in Taiwan (Lin and Kan, 1990).

In addition, some of the phytochemicals identified from *T. catappa* leaves extracts includes; triterpenic acids responsible for the antiinflammatory activity (Fan *et al.*, 2004), hydrolysable tannins, such as punicalagin, punicalin, terflavins A and B, tergallagin, tercatain, chebulagic acid, geraniin, granatin B, and corilagin, but no caffeine (Tanaka *et al.*, 1986). Also identified are Six phenolic compounds; phydroxybenzoic acid, 4-hydroxyphenylpropionic acid, m-coumaric acid, 3,4-dihydroxybenzoic acid, p-coumaric acid and gallic acid (Chyau *et al.*, 2006). Furthermore, apigenin 6-*C*-(2-*O*-galloyl)-â-*D*glucopyranoside, apigenin 8-*C*-(2-*O*-galloyl)- â -*D*-glucopyranoside, glycosides, sovitexin, vitexin, isoorientin, and rutin, were isolated from the dried fallen leaves of *Terminalia catappa* (Yun-Lian *et al.*, 2000). The Leaf also contains 1-degalloyl-eugeniin, 2,3-(4,4',5,5',6,6'-hexahydroxy-diphenoyl)-glucose, chebulagic-acid, corilagin, gentisic-acid, geraniin, granatin-b, kaempferol, punicalagin, punicalin, quercetin, tercatain, terflavin-a, terflavin-b, tergallagin (Duke, 2008).

The chemical compositions of some essential oils from some other Terminalia species have been reported. For example the water-distilled essential oil of *Terminalia bentzoë* from the Island of Rodrigues analyzed by GC and GC/MS gave Twenty-eight constituents which was dominated by citronellyl acetate (64.87%) (Gurib-Fakim and Demarne, 1994).

Furthermore, the composition of essential oil from the fruit oil of *T. chebula* obtained from India was found to consist mainly of palmitic acid (35.7%), furfural (26.8%) and phenylacetaldehyde (13.1%) (Naik *et al.*, 2010). In addition, the main compounds of the oil from *Terminalia ivorensis* flower from Northern Nigeria were δ-3-carene (29.4%) and α-pinene (20.9%) (Ogunwande *et al.*, 2019).

Although, the essential oil of the different parts (fruits, leaves, nuts) of *T. catappa* has been analyzed with some of the following major phytochemicals identified; α-farnesene (21.3%), octadedecane (11.7%), hexadecanoic acid (9.5%), dibutyl phthalate (9.1%), 1,2,3-trimethoxy-5-(2-propenyl)-benzene (6.6%), neoisothujol (5.8%), 1,2,4-trimethoxy-5-(1-propenyl)-benzene (4.5%) 6,10,14-trimethyl-2-pentadecanoic, 1-(2,3,6-trimethyl phenyl)-(E)-3-buten-2-one, geranyl acetone, hexadecanoic acid (21.0%) and 2-ethyl-3,6-dimtheylpyrazine (19.2%), (*Z*)-phytol (41.2%), fatty acid palmitic acid (11.0%), and the (*E*)-nerolidol (4.7%) (Moronkola and Ekundayo, 2000; Wang *et al.*, 2000; Lasekan *et al.*, 2012; Owolabi *et al.*, 2013).

In continuation of our research into the volatile oil components of some poorly studied plants grown in Nigeria, we report in this paper the essential oil constituents of the air-dried leaves of *T. catappa* as our contribution towards increasing the available literature on the plant due to limited work on the essential oil of the leave.

Materials and Methods

Sample collection and identification

The leaves of *Terminalia catappa* were collected from a tree sample in Olabisi Onabanjo University (permanent site), Ago-Iwoye, Ogun State, Nigeria. Identification and authentication was done at the Herbarium of Forestry Research Institute of Nigeria (FRIN), Ibadan, Nigeria where a voucher specimen with herbarium no. FHI 110462 was deposited.

Extraction of essential oil

The pulverized air-dried leaves of *T. catappa* (200 g) were subjected to hydrodistillation in an all glass Clevenger-type all glass apparatus for 3 h in accordance with established procedure (British Pharmacopoeia, 1980). The oil collected was preserved in a sample tube and stored in a Refrigerator until analysis.

Gas chromatography-mass spectrometry (GC/MS) analysis of the leaf oil

GC-MS analysis of the oil was done on an Agilent model 7890A gas chromatograph equipped with a FID and fitted with a fused silica capillary HP-5 MS column (30 m x 0.32 mm id, film thickness 0.25 µm). The oven temperature was programmed from 80 – 240°C at the rate of 8°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 2 mL/min. Scanning range was 35-425 amu. Diluted oil in n-hexane (1.0 µL) was injected into the GC/MS spectrometer.

Identification of the constituents of the leaf oil

The individual components of the oil were identified on the basis of their retention indices (RI) determined by co-injection with reference to a homologous series of n-alkanes, under identical experimental conditions. Further identification was performed by comparison of their mass spectra with those from National Institute of Standards and Technology NIST (Data base 69) and the home-made MS library built up from pure substances and components of known essential oils, as well as by comparison of their retention indices with literature values (Adams, 2007).

Results and Discussion

The essential oil (0.25% v/w), obtained was cloudy light yellow with a strong odour. The result of the chromatographic analysis done on the essential oil is shown in Fig. 1 while the identities of the constituents as well as their percentage composition are shown in Table 1. Fourteen constituents were identified representing 74.32% of the oil from *T. catappa* with the following components; Hexahydrofarnesyl acetone (12.34%) as the major constituent, followed by 1,3,8-p-Menthatriene (9.38%), 1,2-dimethyl-Cyclooctene (7.30%), Undecane (6.73%) Trans-geranylacetone (6.02%) and 4-ethyl-m-xylene (5.83%). The other constituents of the oil were Cis-13-Octadecenal (4.83%), Trans-2-Decenol (4.28%), 3-Eicosene, (E)- (4.17%), 1,7-Hexadecadiene (3.47%), m-Propyl-toluene (3.01%), Neophytadiene (2.56%) trans-β-Ionone (2.53%) and 2-ethyl-p-xylene (1.87%). Hydrocarbons (21.61%) were the most abundant class of compounds in the oil composition while the others are Oxygenated Sesquiterpenes (14.87%), Benzene derivatives (10.71%), Monoterpenes (9.38%), Oxygenated hydrocarbons (9.11%), Oxygenated monoterpene (6.02%) and Sesquiterpenes (2.56%).

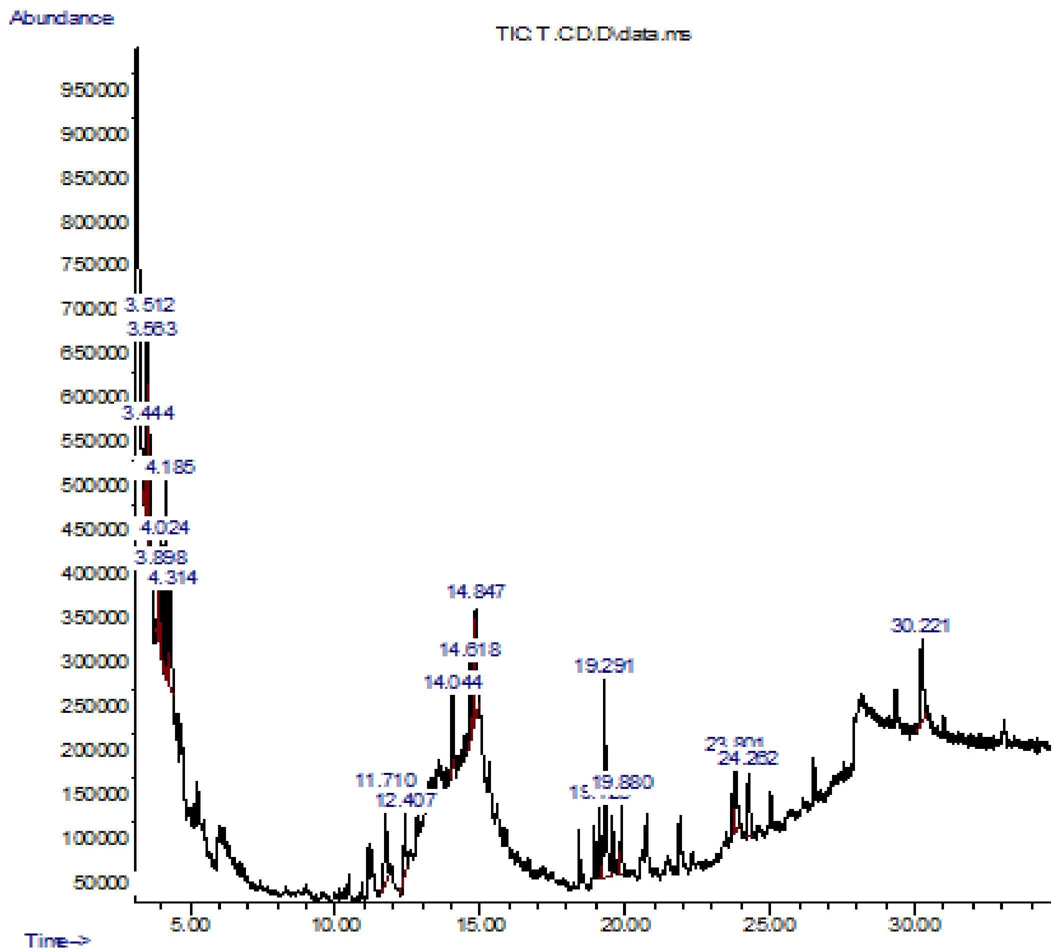

Fig. 1: Chromatogram of the air-dried leaves of *Terminalia catappa* essential oil components

Table 1: Chemical composition of essential oil from the air-dried leaves of *Terminalia catappa*

S/N	Name	KOVAT index	Retention time	% Composition	Molecular formula
1	m-Propyl-toluene	981	3.442	3.01	$C_{10}H_{14}$
2	1,2-dimethyl- Cyclooctene	1000	3.510	7.30	$C_{10}H_{18}$
3	1,3,8-p-Menthatriene	1000	3.562	9.38	$C_{10}H_{14}$
4	2-ethyl-p-xylene	1000	3.899	1.87	$C_{10}H_{14}$
5	4-ethyl-m-xylene	1044	4.025	5.83	$C_{10}H_{14}$
6	Undecane	1000	4.185	6.73	$C_{11}H_{24}$
7	Trans-2-Decenol	1103	4.311	4.28	$C_{10}H_{20}O$
8	Trans-geranylacetone	1274	11.710	6.02	$C_{13}H_{22}O$
9	Trans-β-Ionone	1366	12.408	2.53	$C_{13}H_{20}O$
10	Neophytadiene	1788	19.125	2.56	$C_{20}H_{38}$
11	Hexahydrofarnesyl acetone	1912	19.291	12.34	$C_{18}H_{36}O$
12	1,7-Hexadecadiene	1800	19.881	3.47	$C_{16}H_{30}$
13	Cis-13-Octadecenal	1958	23.800	4.83	$C_{18}H_{34}O$
14	3-Eicosene, (E)-	1835	24.264	4.17	$C_{20}H_{40}$
			Toluene/Benzene derivatives	10.71	
			Hydrocarbons	21.67	
			Oxygenated hydrocarbons	9.11	
			Monoterpene	9.38	
			Oxygenated monoterpene	6.02	
			Sesquiterpene	2.56	
			Oxygenated sesquiterpenes	14.87	

Hexahydrofarnesyl acetone, which was the major constituent present in the oil extracted in this study belong to the sesquiterpenoids class of compound and its quantitative amount of (12.34%) in the oil is noteworthy since it has not been previously reported to be a major compound of *T. catappa* (Owolabi *et al.,* 2013).
A comparison of the present result with previously analysed samples of *T. catappa* revealed some qualitative and quantitative variations. For example, Sixty-six constituents accounting for 100% of the composition included (*Z*)-phytol (41.2%) an acyclic diterpenoid, palmitic acid (11.0%) a fatty acid being the dominant compounds and lesser quantities of the sesquiterpenoid- (*E*)-nerolidol (4.7%), alkane hydrocarbons; heptadecane (3.0%), hexadecane (2.3%), pristane (2.2%), and phytane (2.0%) (Owolabi *et al.,* 2013).
Similarly, (*Z*)-phytol (41.2%), the main compound of *T. catappa* (Owolabi *et al.,* 2013), was not identified in this sample while the content of trans β-ionone (0.8%) was also insignificant compared to the 2.53% of this present work. It was also observed that common constituents like linalool, camphor, menthol, α- Humulene and (E)-Caryophyllene present in the leaf oil of previous study from Nigeria, were not detected in the current sample.
However, hexahydrofarnesyl acetone (12.34%) which was the major constituent, followed by 1,3,8-p-Menthatriene (9.38%), 1,2-dimethyl-Cyclooctene (7.30%) Undecane (6.73%) and 4-ethyl-m-xylene (5.83%) were not identified in previous studies (Owolabi *et al.,* 2013). Also, Hexahydrofarnesyl acetone was previously reported to exhibit a potent antimicrobial activity against gram-positive and gram-negative bacteria (Filipowicz *et al.,* 2003), antibacterial, antifungal, (Radulovic *et al.,* 2006), antimicrobial (Radulovic*et al.,* 2011), had allopathic (Razavi and Nejad-Ebrahimi, 2010) and pest control potential (Mohamed *et al.,* 1992).
Nevertheless, the presence of (5E,9E)-fernesyl acetone, geranylacetone, trans β-ionone and other saturated isomeric compounds in previously reported data are in accordance with the current study though non-quantitatively (Mau *et al.,* 2003; Owolabi *et al.,* 2013). The chemical compositions of some essential oils of the different parts of *T. catappa* from other parts of the world have been reported. The oil of *T. catappa* leaves from Taiwan was characterised by the abundance of 6,10,14-trimethyl-2-pentadecanoic, 1-(2,3,6-trimethyl phenyl)-(E)-3-buten-2-one and geranyl acetone (Wang *et al.,* 2000). The composition of the roasted *T. catappa* nut from Malaysia was dominated by Hexadecanoic acid (21.0%) and 2-ethyl-3,6-dimtheylpyrazine (19.2%) (Lasekan *et al.,* 2012).
From the fore-going, there are obvious qualitative and/or quantitative compositional variations in the volatile compounds present in the different parts of *T. catappa* as well as other species from the same genus and this may be due to ecological factors, age of the plant, period of collection, handling procedure and climatic condition (Inikpi *et al.,* 2014; Rehman *et al.,* 2016; Ibanez and Blazquez, 2019).

Conclusion

The essential oil of the leaves of *T.catappa* investigated revealed the presence of fourteen constituents as determined by GC-MS analysis constituting 74.32% of the total oil composition. The presence of hexahydrofarnesyl acetone as the major constituent in the oils justifies the traditional use of the plant in treating pains, headaches and as an antimicrobial agent. Also, some of the identified chemical constituents from the plant are new additions to the chemical data base for the plant.

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Conflict of Interest

Authors declare that there is no conflict of interest related to this study.

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