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ORIGINAL ARTICLE

Aromatic Plants from the Sudan: Part II. Chemical composition of the essential oil of *Xylopia aethiopica* (Dunal)A.Rich. – Existence of chemotype species

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ABSTRACT

The essential oil obtained by hydrodistillation from the fruits of Sudanese native *Xylopia aethiopica* (Annonaceae) was analysed by gas chromatography-mass spectrometry GC-MS). Forty five compounds which constitute 97.43 % of the total oil were identified. The oil was dominated by monoterpene fraction which accounted for 78.58 % of the oil. The most abundant components of monoterpene hydrocarbons are alphapinene (11.36%), alpha-phellandrene (10.50 %), Beta-phellandrene (8.94%) and gamma- terpinene (3.19%). 4-isopropylbenzyl alcohol (16.67 %), $C_{10}H_{16}O$ (8.12%), 1,8-cineole (5.28%) and $C_{10}H_{14}O$ (2.57) are the main constituents of oxygenated monterpens. Sesquiterpene hydrocarbons contains gamma-cadinene (11.11%), $C_{15}H_{24}$ (1.73%) and copaene (0.95%) as main constituents while alpha-eudesmol (1.08%) is the most abundant oxygenated sesquiterpene.

Key words: Xylopia aethiopica, Annonaceae, essential oil, chemical composition, 4-isopropylbenzyl alcohol.

Introduction

Xylopia aethiopica (Dunal) A. Rich, (Annonaceae) is a medicinal plant of great repute in Africa which produces a variety of complex chemical compounds. It is commonly known as "African pepper", "Ethiopian pepper" or "Guinea pepper" and locally known as "Komba". (urkill, 1985). In Sudan and Nigeria, the fruits are used in cough medicines as well as a carminative and as spice (EL-Kamali *et al.*, 2007; Oliver-ever, 1986). In Cameroon, *X. aethiopica* fruits are used in the treatment of cough, bronchitis, dysentery and female fertility (Tatsadjieu *et al.*, 2003).

The essential oil of spice tree "African pepper" dried fruits from Cameroon contains more than 100 identified volatiles, and the main components are Beta-pinene (18%), terpinen-4-ol (8.9%), sabinene (7.2%), alpha-terpineol (4.1%), 1,8-cineole (2.5%), mytenol (2.4%) and kaurane derivatives (4.2%) (Jirovetz *et al.*, 2005), while Tatsadjien *et al.*, 2003 fruits essential oil obtained by hydrodistillation (yield 2.5%) from plants growing in Cameroon contain Beta-pinene (18.3%), terpinen-4-ol (8.9%), sabinene (7.2%), alpha-phellandrene (7.1%), alpha-terpineol (4.1%) and trans-Beta-ocimene (3.1%). Jirovetz *et al.*, 1997 gave a semblance of the aroma from the essential oil in the fruit of *X. aethiopica* from Cameroon. The oil extracted from fruits of this plant comprised of mainly monoterpenoids 1,8-cineole (15.15%) and terpinen-4-ol (6.6%) are the most abundant compounds (Aseku ad Adeniyi, 2004).

Ekundayo ,1989 reported that *X. aethiopica* fruits consist mainly of mono- and sesquiterpenoids with typical constituents being alpha-and Beta-pinene, myrcene, p-cymene, limonene, linalool and 1,8-cineole. Sesquiterpenes, elemol and guaiol and some other terpenes like p-mentha3,8-diene and p-mentha-3,8-triene were found in the essential oil of the fruit from the Republic of Benin (Ayedoun *et al.*, 1996).

Tairu et al., 1999 showed that linalool, Beta-trans-ocimene, alpha- farnesene, alpha-pinene, Beta-pinene,

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myrtenol, Beta-phellandrene and 3-ethylphenol were the most important odorants present in the volatile oil of the fruit with linalool being the most intense giving the pepperish note, characteristic of the ground, dried, smoked fruits of *X. aethiopica*.

A number of diterpenes from *X. aethiopica* fruits have been reported (Faulkner *et al.*, 1985; Rabunmi and Pieera, 1992; Harrigan *et al.*, 1994).

The fruits essential oil of *X. aethiopica* showed activity against some fungi and cytotoxicity to carcinoma cells (Hep-2cell line) at 5 mg/ml concentration (Asekun and Adeniyi, 2004). The essential oil of spice tree fruits at dilution in methanol (1:5) showed moderate antibacterial activity against *Escherichia coli*, *Proteus vulgaris* and *Klebsiella pneumoniae* (EL-Kamali *et al.*, 2007). Antibacterial properties in the *X. aethiopica* ethanolic extract of fruits against *E. coli*, *P.vulgaris* and *K. pneumoniae* was reported by Okeke *et al.*, 2001.

The objectives of this study are to: (i) qualitatively and quantitatively assessing the oil components and (ii) comparing these findings with literature data on some African countries.

Materials and Methods

Plant material:

Fruits of X. aethiopica were collected in April 2007 from Nuba Mountains, Western Sudan. It was identified at the Department of Botany, Faculty of Science and Technology, Omdurman Islamic University.

Preparation of the essential oil:

The finely dry powdered fruits (200 grams) were subjected to Hydrodistillation using Clevenger-type apparatus (British Pharmacopaeia, 1988). The obtained oil (3 ml) was collected, and dried over anhydrous sodium sulphate and kept at 4 °C until analysis.

GC/MS analysis

GC/MS analysis was conducted using a Shimatzu QP 2010 GC/MS instrument equipped with reference libraries.. The flow rate of helium as carrying gas was (1 ml/min). The temperature program consisted of 60 - 270 °C, at rate of 4 °C /min. MS were taken at ionization voltage 70 eV. Library search was carried out using Wiley GC/MS library. The individual identifications were made by the comparison of fragmentation patterns with those found in the library of the Mass spectrometer and literature (Adam, 2001).

Results and Discussion

Xylopia aethiopica growing in Sudan has not previously been subjected to any chemical investigation. The essential oil obtained from the fruits of this plant by hydrodistillation, when subjected to GC-MS analysis showed 45 identified components representing 97.43% of the total components (60 compounds) (Table 1). The GC-MS results revealed the presence of 78.58% monoterpenes (42.31% monoterpene hydrocarbons and 36.27% oxygenated monoterpenes) and 18.85% sesquiterpenes (15.88% sesquiterpene hydrocarbons and 2.97% oxygenated sesquiterpenes).

Upon comparing the composition of Sudanese oil with that of some other African origins , some variation was noted. The chemical composition of Sudanese *X. aethiopica* oil was characterized by a high content of 4-isopropylbenzyl alcohol, alpha-pinene, alpha-phellandrene and gamma-cadinene. The essential oils of various *X. aethiopica* populations from the Cameroon, Benin and Nigeria were previously studied (Table 2) and three chemotypes can be defined based on their major oil components. These chemotypes are alcohol type, ester type and Beta-pinene type. This work shows, once more, the variability of compounds of the oils of species growing in different biotypes.

Conclusion

The GC-MS analysis of a Sudanese native *Xylopia aethiopica* essential oil revealed the presence of 34 monoterpenes and 16 sesquiterpenes. This comprehensive study has highlighted the posibility of using w/w % differences between 4-isopropylbenzyl alcohol and alpha-pinene, to distinguish between Sudanese and other African *X. aethiopica* oils. This however, will require more extensive investigation. We will continue our study of the Sudanese *X. aethiopica* oils in an attempt to gain a better understanding of the secondary metabolite profile of this economically valuable plant species.

| Table 1: Chemical composition of the essential oil from Xylopia aethiopica of Western Sudan | | | | | | |
|---|---------------|---|------------|---------------------|--|--|
| Compound | % | Formula | Class Type | Retention Time (RT) | | |
| delta-3-carene | 2.22 | $C_{10}H_{16}$ | MH | 11.653 | | |
| alpha-pinene | 11.36 | $C_{10}H_{16}$ | MH | 11.931 | | |
| camphene | 0.27 | $C_{10}H_{16}$ | MH | 12.619 | | |
| unidentified | 0.06 | $C_{10}H_{16}O$ | OM | 12.819 | | |
| Beta-phellandrene | 8.94 | $C_{10}H_{16}$ | MH | 13.589 | | |
| Beta-pinene | 0.27 | $C_{10}H_{16}$ | MH | 14.585 | | |
| unidentified | 0.09 | $C_{10}H_{16}$ | MH | 14.660 | | |
| ocimene | 0.02 | $C_{10}H_{16}$ | MH | 14.711 | | |
| alpha-terpinene | 1.80 | $C_{10}H_{16}$ | MH | 14.975 | | |
| cymene | 1.75 | $C_{10}H_{14}$ | MH | 15.194 | | |
| alpha-phellandrene | 10.50 | $C_{10}H_{16}$ | MH | 15.340 | | |
| 1,8-cineole | 5.28 | $C_{10}H_{18}O$ | OM | 15.392 | | |
| p-ocimene | 1.18 | $C_{10}H_{16}$ | MH | 15.556 | | |
| gamma-terpinene | 3.19 | $C_{10}H_{16}$ | MH | 16.062 | | |
| sabinene hydrate | 0.38 | $C_{10}H_{18}O$ | OM | 16.366 | | |
| 2-carene | 0.68 | $C_{10}H_{16}$ | MH | 16.685 | | |
| cis-p-2-menthen-1-ol | 0.37 | $C_{10}H_{18}O$ | OM | 17.049 | | |
| alpha-thujone | 0.05 | $C_{10}H_{16}O$ | OM | 17.408 | | |
| unidentified | 0.01 | $C_{10}H_{16}O$ | OM | 17.489 | | |
| unidentified | 0.40 | $C_{10}H_{18}O$ | OM | 17.543 | | |
| 2,4,6-octatriene | 0.04 | $C_{10}H_{16}$ | MH | 17.594 | | |
| pulegone | 0.03 | $C_{10}^{10}H_{16}^{10}O$ | OM | 17.632 | | |
| l-pinocarveol | 1.27 | $C_{10}H_{16}O$ | OM | 17.869 | | |
| sabinaketone | 0.22 | $C_9H_{14}O$ | | 18.197 | | |
| pinocarvone | 0.30 | $C_{10}H_{14}O$ | OM | 18.289 | | |
| unidentified | 8.12 | $C_{10}H_{16}O$ | OM | 18.640 | | |
| 4-isopropylbenzyl alcohol | 16.67 | $C_{10}H_{14}O$ | OM | 18.641 | | |
| unidentified | 2.57 | $C_{10}H_{14}O$ | OM | 18.907 | | |
| bornyl acetate | 0.08 | $C_{12}H_{20}O_2$ | | 19.319 | | |
| phellandral | 0.19 | $C_{10}H_{16}O$ | OM | 19.729 | | |
| unidentified | 0.24 | $C_{10}H_{12}O$ | OM | 20.325 | | |
| unidentified | 0.09 | $C_{10}H_{16}O$ | OM | 20.401 | | |
| terpineol | 0.05 | $C_{10}H_{18}O$ | OM | 20.466 | | |
| linalool | 0.09 | $C_{10}H_{18}O$ | OM | 20.528 | | |
| terpinen-4-ol | 0.02 | $C_{10}H_{18}O$ | OM | 21.004 | | |
| unidentified | 0.08 | $C_{10}H_{16}O$ | OM | 21.099 | | |
| delta-elemene | 0.69 | $C_{15}H_{24}$ | SH | 21.198 | | |
| alpha-cubebene | 0.27 | $C_{15}H_{24}$ | SH | 21.380 | | |
| ylangene | 0.11 | $C_{15}H_{24}$ | SH | 21.741 | | |
| copaene | 0.95 | $C_{15}H_{24}$ | SH | 21.835 | | |
| 1-chloroocta-decane | 0.03 | $C_{18}H_{37}Cl$ | CII | 22.095 | | |
| Beta-caryophyllene | 0.47 | $C_{15}H_{24}$ | SH | 22.501 | | |
| Beta-cubebene | 0.04 | $C_{15}H_{24}$ | SH | 22.580 | | |
| humulene | 0.07 | $C_{15}H_{24}$ | SH | 22.632 | | |
| unidentified | 0.22 | $C_{15}H_{24}$ | SH SH | 23.008 | | |
| 1(10),4(14),5-germacratriene | 0.02 11.11 | $C_{15}H_{24}$ | SH SH | 23.076 23.356 | | |
| gamma-cadinene | | C ₁₅ H ₂₄ | | | | |
| elemol unidentified | 0.73 1.73 | $C_{15}H_{26}O$ | OS SH | 23.765 24.215 | | |
| guaiol | 0.35 | $ C_{15}H_{24} C_{15}H_{26}O $ | OS | 24.213 | | |
| unidentified | 0.33 | $C_{15}H_{26}O$ $C_{15}H_{24}$ | SH | 24.413 | | |
| alpha-eudesmol | 1.08 | $C_{15}H_{26}O$ | OS | 25.427 | | |
| unidentified | 0.81 | $C_{15}H_{26}O$ $C_{15}H_{26}O$ | OS | 25.634 | | |
| phthalic acid | 0.81 | $C_{15}\Pi_{26}O$ $C_{16}H_{22}O_4$ | OB | 27.858 | | |
| kaur-16-ene | 0.23 | $C_{16}H_{22}O_4$ $C_{20}H_{32}$ | | 29.133 | | |
| unidentified | 0.21 | $C_{20}H_{32}$ $C_{27}H_{46}$ | | 30.067 | | |
| (+)-manoyl oxide | 0.04 | $C_{27}H_{46}$ $C_{20}H_{34}O$ | | 30.087 | | |
| pregnane-3-17-diol | 0.08 | $C_{20}H_{34}O$ $C_{21}H_{32}O_4$ | | 32.558 | | |
| unidentified | 0.12 | $C_{21}H_{32}O_4$ $C_{10}H_{18}N_2O_2$ | | 32.561 | | |
| methylene bis methyl butyl-phenol | 0.40 | $C_{10}H_{18}N_2O_2$ $C_{23}H_{32}O_2$ | | 34.007 | | |
| MH - Monoterpene hydrocarbons: OM | | | | 2007 | | |

MH = Monoterpene hydrocarbons; OM = Oxygenated Monoterpenes; SH = Sesquiterpene Hydrocarbons; OS = Oxygenated Sesquiterpenes.

Table 2: Comparison between the relative percentage of the major constituents of X. aethiopica fruit essential oils from different origin

| Origin | Major constituents | % | Reference |
|----------|---------------------------|-------|--------------------------|
| Sudan | 4-isopropylbenzyl alcohol | 16.67 | |
| | alpha-pinene | 11.36 | Present study |
| Cameroon | Beta-pinene | 18 | |
| | Terpinen-4-ol | 8.9 | Jirovetz et al., 2005 |
| | Beta-pinene | 18.3 | |
| | Terpinen-4-ol | 8.9 | Tatsadjien et al., 2003 |
| Benin | alpha- & Beta- pinene | | Ayedoun et al., 1996 |
| Nigeria | 1,8-cineole | 15.15 | |
| | Terpinen-4-ol | 6.6 | Asekun and Adeniyi, 2004 |

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