Effect of Geographical location on essential oil Content and Composition of *Xylopia aethiopica*

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Abstract

The essential oil from the dried fruit of *Xylopia aethiopica* (Dunal) A. Richard from Sudan was analyzed using combined GC-MS and ^{1}HNMR. The main constituents identified in the oil were 4-terpineol (11.30%), β-pinene (6.12%), α-terpineol (6.02%), 1,8-cineole (5.42%), *cis*-α-copaene-8-ol (4.68%),13-epimanoyl oxide (4.62%), (+)-spathulenol (4.26%), L-pinocarveol (3.26%), myrtenol (2.94%), o-cymene (2.82%), eudesma-1,3-dien-11-ol (2.35%), eudesma,4-11(13)-dien-2-ol (2.32%), cumic alcohol (2.29%), Kaur-16-ene (2.21%) and α-pinene (1.88%). Fifteen compounds were identified for the first time in *X. aethiopica* essential oil, among which thujol, 1,5-epoxysalvial,4(14)-ene, salvial-4(14)-en-1-one and 13-epimanool. The oil yield and composition were compared, respectively, to those of the dried fruits of the plant from other African countries.

Key words: *Xylopia aethiopica*, essential oil, oil yield, oil composition, GC-MS

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Introduction

Xylopia aethiopica (Family: Anononaceae), commonly known as Negro pepper or Ethiopian pepper, is an ever green aromatic tree, growing up to 20 m high. It is a native of low land rain forest and moist fringe forests in the Savanna zones and coastal regions of Africa (Dalziel, 1955; Irvine, 1961), located in West and Central Africa (Hutchinson, 1954; Kieta, 2003). It is distributed in high rain fall Savanna and swampy forests of Sudan (Elamin, 1990; Elgazali *et al*., 2004).

The composition of the oil of *X. aethiopica* is very diverse. Ogan (1971), identified β-phellandrene, (E)-β-ocimene, α-terpineol, bisabolene, and cuminal in the volatile oil of the fruit of *X. aethiopica*. Karawya *et al.* (1979) studied the composition of the essential oil in *X. aethiopica* collected from a local market in Egypt and identified more than twenty one compounds. The major constituents were terpinen-4-ol (23.4%), 1,8 cineole (16.3%), β-pinene (14.6%), α-terpineol (11.1%), cumin aldehyde (6.3%) and cuminyl alcohol (3.3%). Fourty-one compounds were detected in essential oil of *X. aethiopica* dried fruit from Benin with sabinene (36.0%) as the main component. The other major constituents were 1,8-cineole (12.8%), terpinen-4-ol (7.0%), linalool (3.9%), β-eudesmol (1.9%) and β-elemene (0.81%) (Poitou *et al*., 1996; Ayedoun *et al.*,1996). The constituents of the essential oil of *X. aethiopica* from Guinea were identified by Tomi and Casanova (1996). They found twenty one compounds with dominant constituents being β-pinene (37– 40.5%), α-pinene (13.6–18.4%), sabinene (7.1–7.6%) and 1,8-cineole (6.5–8.4%). Keita *et al*. (2003) detected thirty compounds in essential oil of *X. aethiopica* fruit from Mali. The principal constituents identified in the oil were β-pinene (19.1%), γ-terpinene (14.7%), trans-pinocarveol (8.6%), p-cymene (7.3%) and myrtenal (4.3%).

Similar oil from Ghana showed thirty nine compounds among which germacrene D was the highest (25.1%) in addition to β- pinene.(21.6%), α-pinene.(8.0%) and 1,8 cineole (7.4%). A diterpene (kaur-16-ene) was also detected in a trace amount (0.1%) (Karioti *et al*., 2004). Noudjou *et al*. (2007) identified sixty three compounds in essential oil of *X. aethiopica* from Cameroon. The main constituents were β-phellandrene+1,8-cineole (31.0%), β-pinene (8.0%) and α- pinene (3.4%). A minor diterpene, 13-ent-epimanoyl oxide (0.4–0.6 %), was also identified. Onayade-Sontan (1991) studied essential oil of *X. aethiopica* fruit from Nigeria followed by Olonisakin *et al.* (2007) and identified twenty three compounds. The predominant compounds were β-pinene (13.78%), β-phellandrene (12.36%), γ-terpinene (7.66%), eucalyptol (6.9%) and α-pinene (5.56%).

The objectives of this study are to investigate constituents of the essential oil of the dried fruits of *X. aethiopica* grown in Sudan and to compare the essential oil yield and composition to those of the dried fruits of the plant from other African countries. .

Materials and Methods

Plant material

The dried fruits of *X. aethiopica* were brought from a local market in Khartoum and authenticated (herbarium no. H.A. 11/2004) by Medicinal and Aromatic Plants Research Institute (MAPRI), Sudan.

Essential oil isolation

One hundred grams of the dried fruits of *X. aethiopica* were crushed and subjected to hydrodistillation for 4 h using Clevenger apparatus. The distilled essential oil was dried over anhydrous sodium sulphate, filtered and stored in a sealed vial at 4°C until analysis. The yield of the oil (v/w %) was calculated based on the plant dry matter.

GC-MS analysis

The essential oil was analyzed by gas chromatography coupled with mass spectrometry (GC-MS) using HP 6890 (GC) and HP 5973 (MSD). The sample was dissolved in dichloromethane and injected at 250 °C (Injector temperature) into a capillary column type HP-1, 30 × 0.25 mm × 0.25 µm, stationary phase (95 % diethyl-5% diphenylsiloxane), using helium as a carrier gas at a flow rate of 1 ml/min. The injected volume was 1 µl and the injection mode used was splitless. The oven temperature was programmed from 45–280 °C at the rate of 4 °C/min. Detector temperature was 250 ^{o}C. The MS was operated in the El mode at 70 eV. The mass and scan range was set at *m/z* 30-500.

On the other hand, some of the oil constituents were isolated in a pure form using column chromatography and semi-preparative high performance liquid chromatography. The pure isolated constituents were analyzed using ^{1}HNMR technique, in addition to MS. The mass spectra of the oil constituents were compared with those of NIST-98 and Wiley libraries spectral database as well as the published data.

Results and Discussion

The oil yield was found to be about 4.00% (v/w), which was considered high compared to the essential oil content of dried fruits of the plant from other African countries (Fig. 1). The oil was pale yellow, with spicy characteristic odor.

GC-MS analysis of the essential oil showed that it was a mixture of seventy one compounds (Fig. 2).

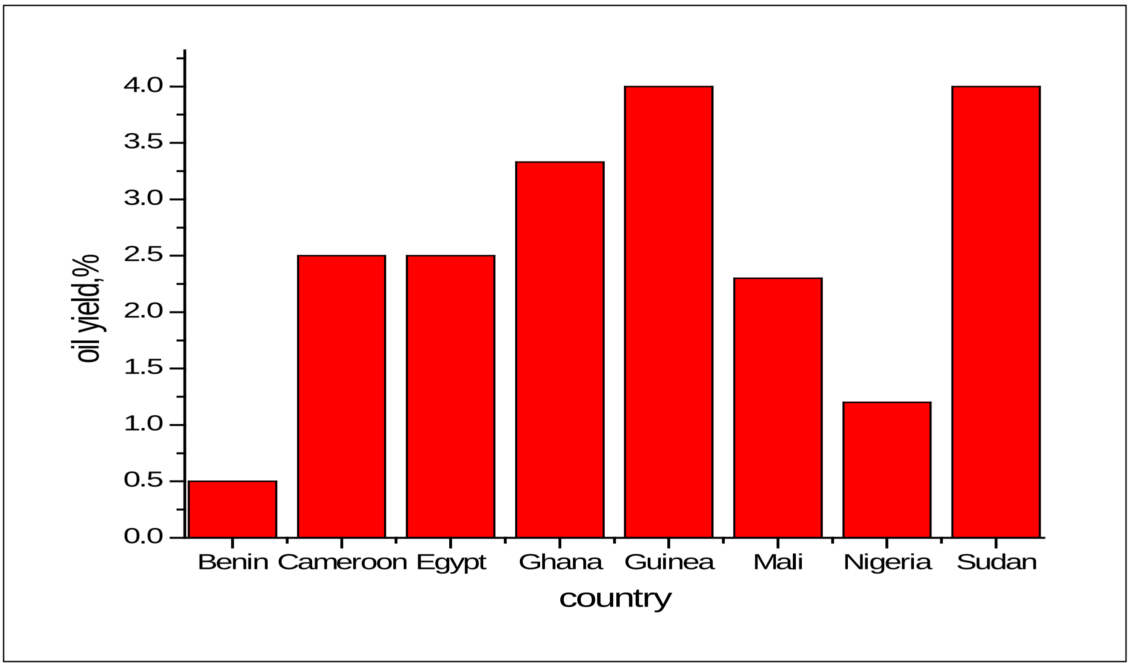

Figure 1. Essential oil yield of *X. aethiopica* dried fruit from African countries.

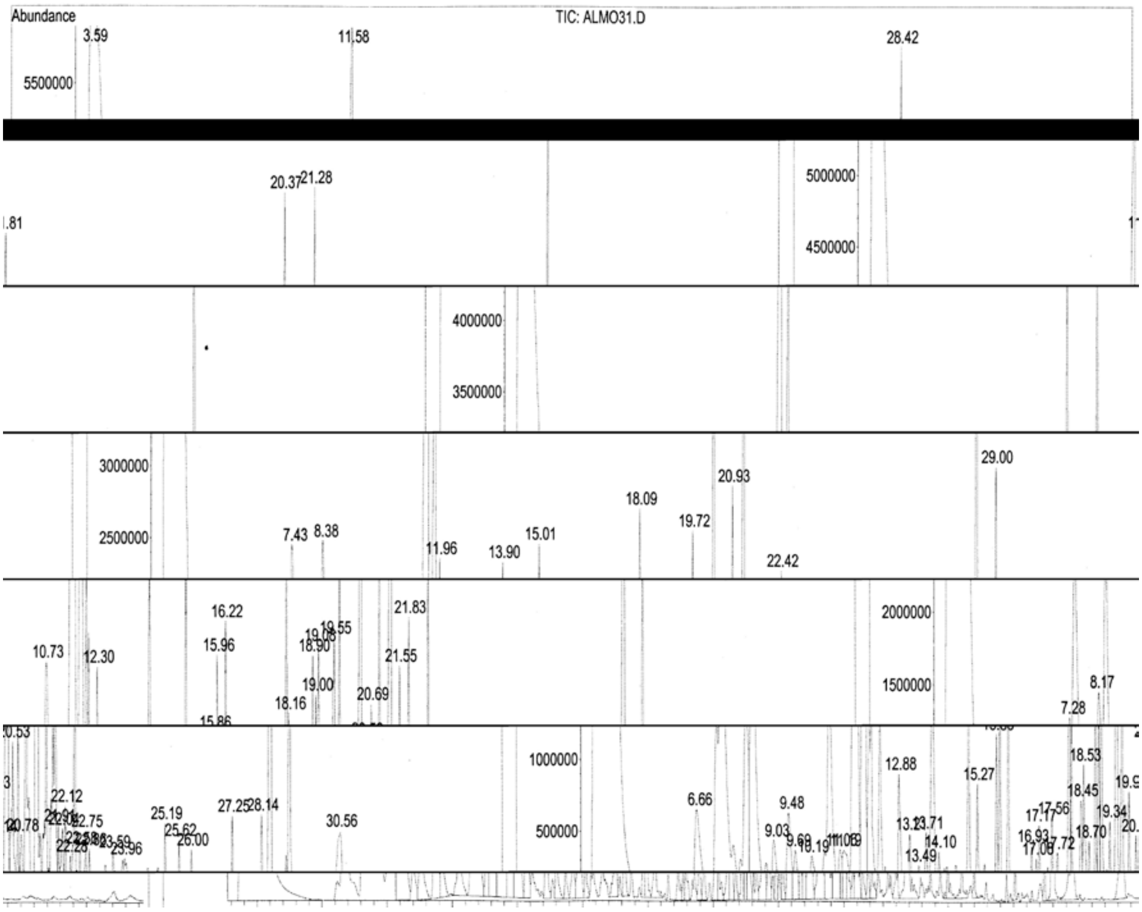

Figure 2. Gas chromatogram of essential oil from dried fruit of *X. aethiopica* from Sudan

From the preliminary investigation of the essential oil of *X. aethiopica* from Sudan, it was observed that, there was a clear difference, quantitatively and qualitatively, between the composition of the essential oil under investigation and the essential oils of *X. aethiopica* (Dunal) A. Rich. from other producing African countries (Figures 3, 4, 5 and 6). These variations may be attributed to the fact that, the amount and quality of secondary metabolites (essential oils, alkaloids, …etc) affected by genetic factors, climatic conditions, soil and cultivation techniques (Pitarevic *et al*. 1985; Telic and Sahbaz, 2005; Orav *et al*. 2006; Bhatti *et a*l., 2007). On the other hand, factors that specifically determine the composition and yield of the essential oil obtained include seasonal and maturity variation, geographical origin, genetic variation, growth stages, part of plant utilized and postharvest drying and storage (Marotti *et al.* 1994; Hussain *et al.,* 2008; Anwar *et al.,* 2009b).

There are many reports in the literature showing the variation in the yield and chemical composition of the essential oil with respect to geographical regions (Uribe-Hernandez *et al.,* 1992; Souto-Bachiller *et al.,* 1997; Celiktas *et al.,* 2006; Van Vuuren *et al*., 2007; Viljoen *et al*., 2006 and Chalchat *et al*., 1995).

It was noticed that the oxygenated fraction was greater than the hydrocarbon fraction only in the essential oil of the plant grown in Sudan and that analyzed in Egypt, which supposed to be brought from Sudan. Among the constituents detected for the first time in the essential oil *X. aethiopica*, sesquiterpene alcohols of eudesmane skeleton. Also sesquiterpenes of isodaucane skeleton has been detected for the first time in the oil.

Hexadecanoic acid methyl ester was detected as a trace compound (0.19%) among the constituents of the oil under investigation for the first time. It is suggested to be resulted from methylation of the Hexdecanoic acid, which has been previously detected by Tariu *et al.* (1999) in the essential oil *X. aethiopica* from Nigeria, with the dichloromethane used to dissolve the sample for analysis. Among the oxygenated fractions, alcohols were the predominant constituents in the oil from Sudan, in contrast to other producing countries where hydrocarbons were the dominant constituents.

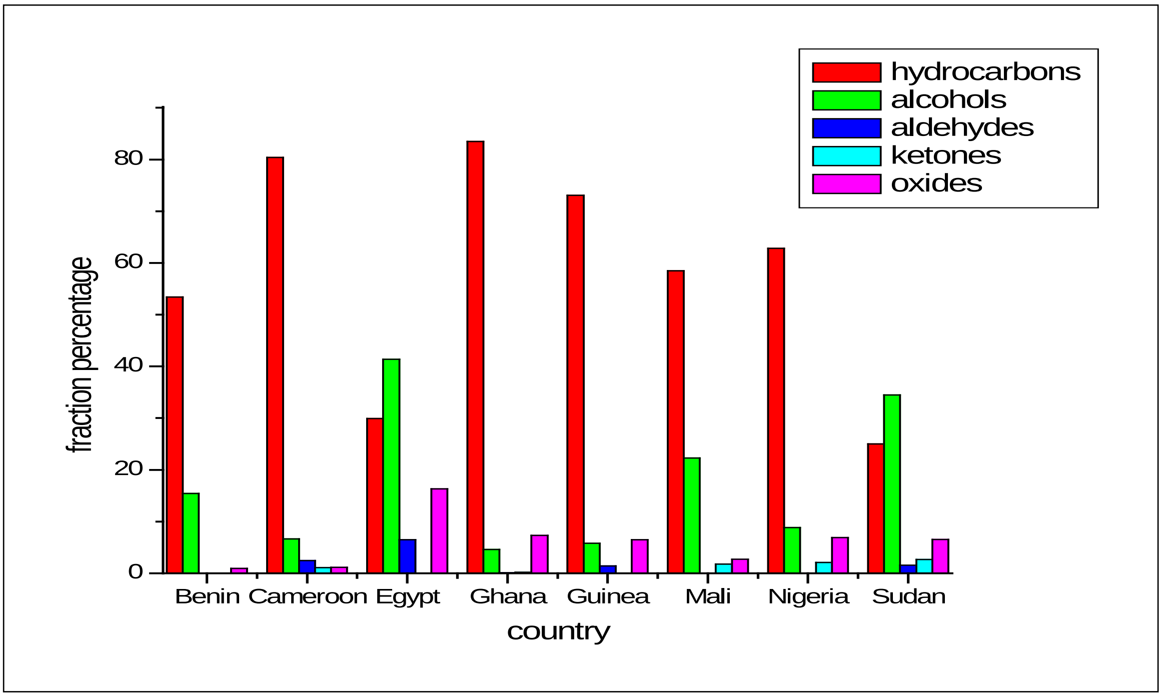

Figure 3. Constituents of *X. aethiopica* essential oils from different countries

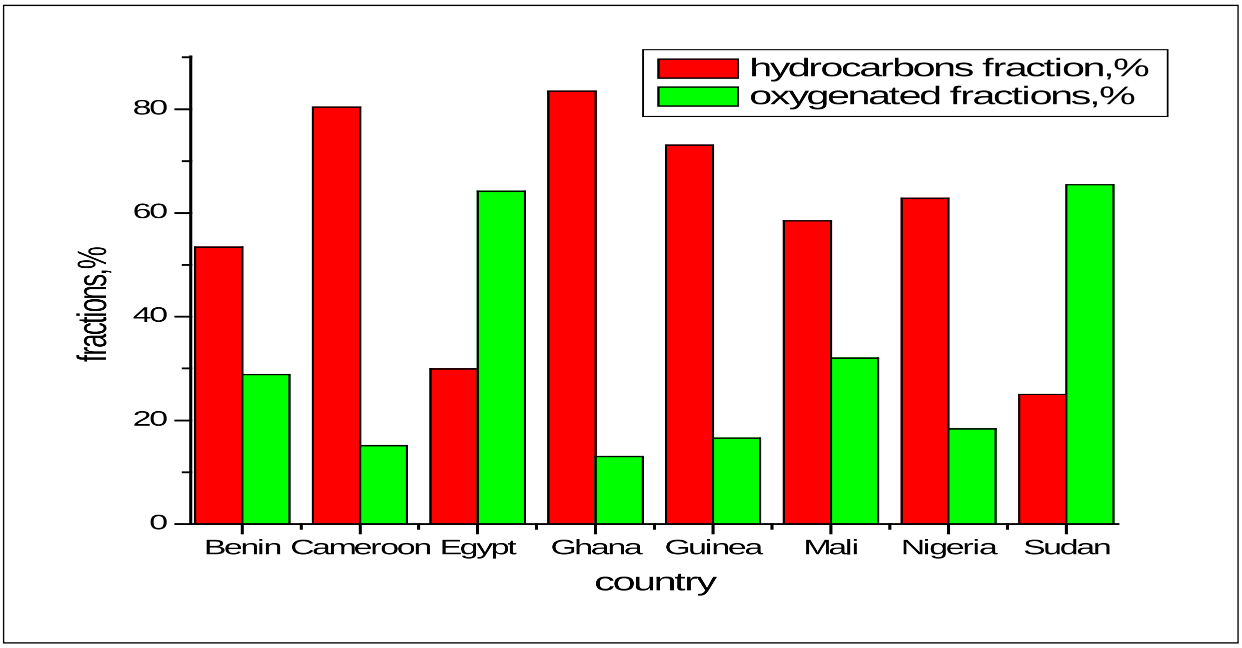

Figure 4. Hydrocarbon and oxygenated fractions of *X. aethiopica* essential oil from different countries

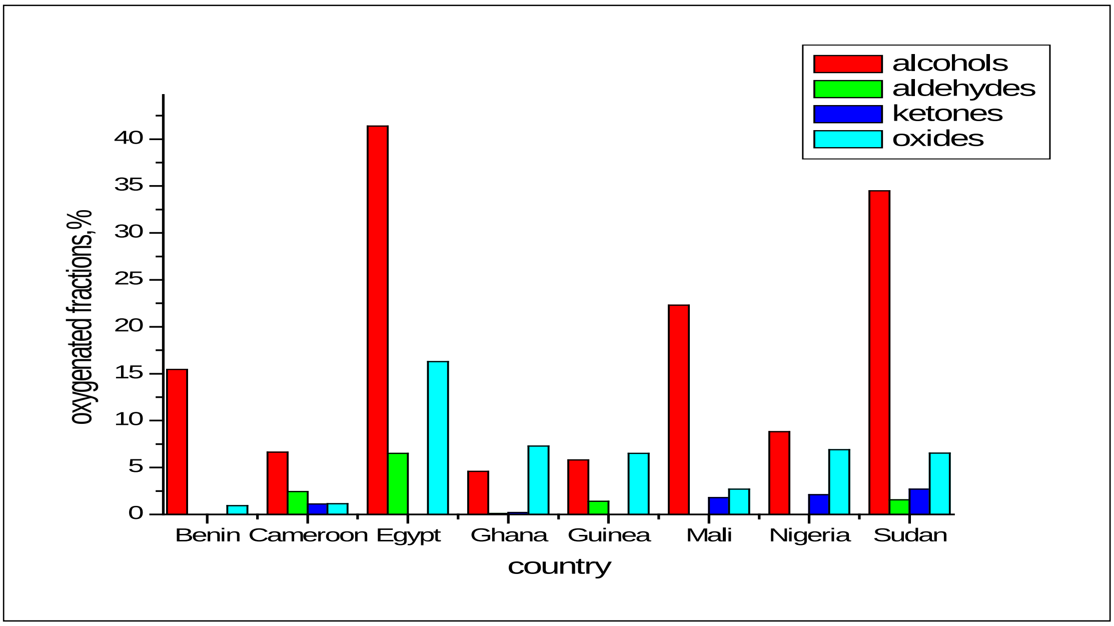

Figure 5. Oxygenated constituents of *X. aethiopica* essential oil from different countries

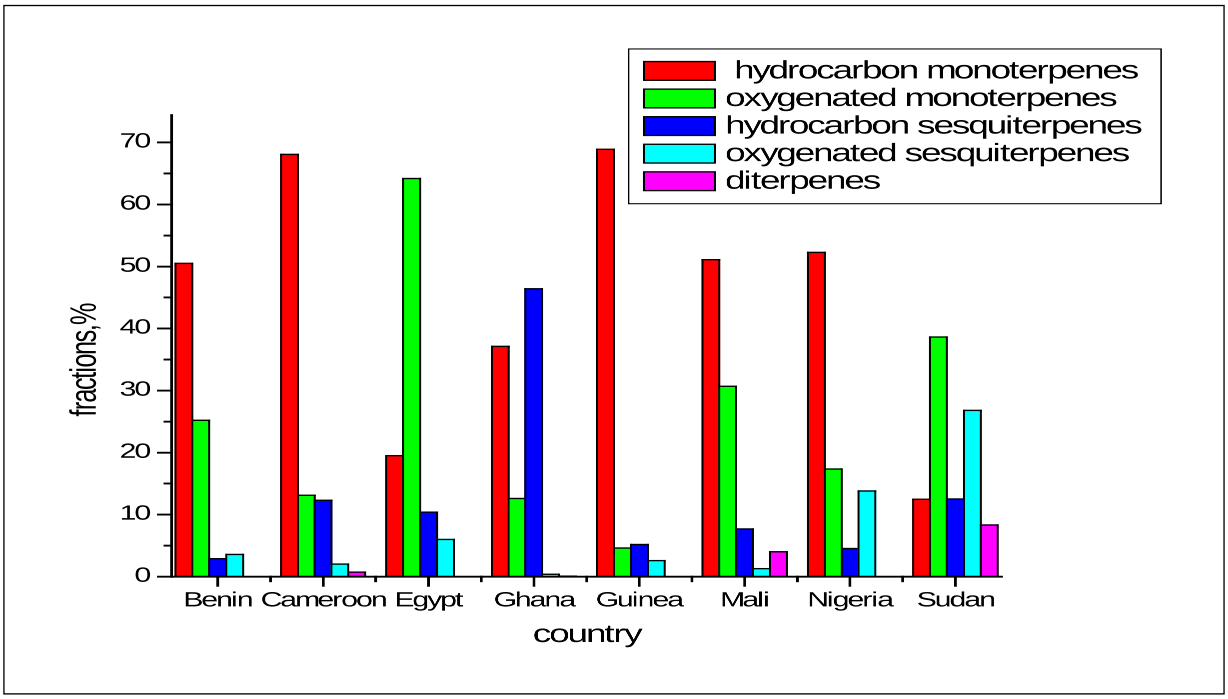

Figure 6. Terpenoid classes in *X. aethiopica* essential oils from different countries

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