

Full Length Research Paper

## Physical and chemical analysis of some imported essential oils in the Sudanese market

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Accepted 8 May, 2012

A market survey in Sudan indicated the growing trend of import of essential oils as crude raw materials or in a form of semi-finished or finished products and commodities. Quality control and assessment of the imported essential oils need to be promoted bearing in mind the research and development in this field. The main objectives of the present paper were to evaluate some of these imported essential oils namely: mint oil, clove oil and olibanum oil and to compare their physical and chemical properties with essential oils prepared from plants available in the local market. The essential oils were investigated according to international standard methods: hydrodistillation, thin layer chromatography, gas chromatography - mass spectroscopy and British Pharmacopoeia procedures. According to the results obtained, it was obvious that the physico-chemical properties and chemical composition of traded (imported) essential oils were different when compared with essential oils prepared from local plant samples and were not compatible with the international standards. Quality control and assessment of imported and locally produced essential oils should be promoted with regard to their uses in cosmetics, pharmaceuticals, aromatherapy and food industries.

**Key words:** Essential oils, Sudanese market, quality control, gas chromatography - mass spectroscopy (GC-MS) analysis, adulteration.

### INTRODUCTION

Essential oil bearing plants are important source of secondary metabolites that are used extensively in our daily life and play an important role in the economy of man. The drugs and oils are used for their valuable medicinal properties and as spices and flavors, as well as in perfumes and cosmetics (Guenther, 1962; Evans, 2002). A survey of the market in Sudan indicated the growing trend of import of essential oils as crude raw materials or in a form of semi-finished products or end-products and commodities. The essential oils of mint (*Mentha spicata* L., family: Lamiaceae), clove (*Syzygium aromaticum* L., family: Myrtaceae) and olibanum

(*Boswellia papyrifera* Del., family: Burseraceae) are used as drugs, flavors and in perfumes and cosmetics (Abd Elwahab, 1987; Kokkini and Vakou, 1989; Kokkini et al., 1995; Hayashi et al., 1998; Evans, 2002). Quality control and assessment of the imported and locally produced essential oils need to be promoted bearing in mind the research and development in this field (Lawrence, 1993; Samgwam et al., 2001).

The objectives of the present work were to evaluate the physical and chemical properties of the imported mint, clove and olibanum essential oils based on a comparative study with the same oils produced locally, and to investigate their chemical composition by thin layer chromatography (TLC) and gas chromatography-mass spectroscopic (GC-MS) analysis. The results of investigation are reported in the present paper with reference to possible adulteration of samples or their

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**Table 1.** Physico-chemical properties of mint, clove, and olibanum local and imported essential oils.

Sample of essential oil	Physico-chemical property						
	Colour	Specific gravity	Refractive index	Specific rotation	Acid value	Sapon. value	Ester value
I	Colorless	0.910	1.465	-49°	1.50	17.50	16.00
II	Yellow	1.050	1.433	-2.50°	3.00	35.00	32.0
III	Yellow	0.860	1.443	-26°	2.00	11.50	9.5
IV	Yellow	0.897	0.464	-20°	6.1	28.30	22.20
V	Yellow	0.990	1.479	-12°	5.3	33.23	27.90
VI	Yellow	0.970	1.475	18.0°	6.7	26.53	19.23
M	Colorless	0.917 to 0.934	1.460 to 1.440	-48 to -61	1.10 to 2.10	17.0 to 18.0	15.9 to 16.9
C	Yellow	1.040 to 1.050	1.400 to 1.448	-2 to zero	3.8	42.07	39.9
O	Yellow	0.872 to 0.892	1.471 to 1.482	-16 to -35	Up to 3	4.0 to 13	5.0 to 16

Local samples: I, Mint essential Oil; II, clove essential Oil; III, olibanum essential oil; Imported samples: IV, Mint essential oil; V, Clove essential oil; VI, Olibanum essential oil; Published data (Guenther, 1962; Green and Hone, 1992; Hayashi et al., 1998; Sangwan et al., 2001): M, mint essential oil; C; clove essential oil; O, olibanum essential oil.

or their aromatherapeutic applications.

## MATERIALS AND METHODS

The leaves of *M. spicata* L. (Lamiaceae), buds of *S. aromaticum* (L) Merr & Perry (Myrtaceae) and gum of *B. papyrifera* (Burseraceae) were purchased from the local market (Souk Alarabi, Khartoum), during march, 2009. Imported samples of mint, clove and olibanum essential oils were purchased from the same market during the same period. The samples were identified by Dr. WE Abdullah, Medicinal and Aromatic Plants Research Institute, Khartoum, Sudan.

### Preparation of essential oils

The essential oil content of the three local samples was determined by hydrodistillation. In brief, 500 g of each sample were introduced in a Clavenger apparatus according to the British Pharmacopeia (2003). The prepared oils were dried over anhydrous sodium sulphate and measured in V/W and stored in brown vials under refrigeration. The imported samples of essential oils were subjected to steam distillation and the usual work- up. The volatile and non-volatile portions were stored in brown vials under refrigeration. Physical and chemical properties of the prepared and imported essential oils samples were also determined (BP, 2003; Guenther, 1962).

### Chromatographic procedures

The prepared and imported essential oils samples and the non-volatile portions of imported samples were examined by TLC on pre-coated silica gelF<sub>254</sub> plates, thickness 0.20 mm, using the following solvent systems: Toluene/ethyl acetate (7:3,v/v); petroleum ether (40-60°C)/diethyl ether/ gl; acetic acid (80:20:01, v/v/v) for non-volatile portions of imported samples. The chromatograms were visualized by spraying with vanillin-concentrated sulphuric acid (1%) and heated at 105°C for 5 min.

The gas chromatographic analysis and detection (GC- MS analysis) was done in a gas chromatograph mass spectrometer (QP-2010- Shimadzu/ Japan) in a fused silica capillary column (30

m × 0.25 mm diameter, film thickness 0.25 µM DB-5). The carrier gas was helium at 1.00 ml/min. The column temperature was programmed from 50 to 280°C at 8°C/min. The injection port and detector were set at 250°C. Identification of components was achieved by comparison with data in the instrument library dataset (NIST 1998 MS).

## RESULTS AND DISCUSSION

The essential oil content in the local and imported samples was determined by hydrodistillation and results were compared with those in literature. Mint leaves gave 1.5% essential oil compared to the literature (not less than 1.5%), clove buds gave 8.8% compared to the literature (6 to 18%) and olibanum gum gave 4.5% compared to the literature (5 to 9%). The yields of imported oils were not compatible to the standards (Guenther, 1962; Evans, 2002).

The physico-chemical properties of local and imported essential oils were determined according to standard methods and results were summarized in Table (1) compared to available literature (Guenther, 1962; Yates and Wenninger, 1970; Kokkini et al., 1995; Lawless, 1996; Ozel and Ozguren, 2002). The results of analyses of local samples prepared from drugs available in the market (clove buds) or grown in Sudan (mint leaves and olibanum gum), were within the limits published in the current literature (Guenther, 1962; Green and Hone, 1992; Sangwan et al., 2001). The volatile portions of imported samples of mint, clove and olibanum essential oils gave yields and physico-chemical parameters different from what is known in the literature. The low content of volatile portion in the three imported samples of mint, clove and olibanum claimed to be essential oils and the different physico-chemical properties compared with published data in the literature (table 1), prompted us to investigate the chemical composition of the volatile and

**Table 2.** Results of GC-MS analysis of essential oils prepared from mint, clove and olibanum local samples.

Peak no.	Retention time (t <sub>R</sub> )	Composition (%) from total peak area	Name of component
<b>1. Mint essential oil</b>			
1	5.71	22.53	D-limonene
2	5.85	2.68	Eucalyptol
3	13.22	2.31	Terpineol
4	19.87	0.64	Piperitone
5	14.36	0.41	Pulegone
6	14.52	<u>70.20</u>	Carvone
<b>2. Clove essential oil</b>			
1	14.27	<u>81.85</u>	Eugenol
2	15.52	2.74	β-caryophyllene
3	16.14	0.44	α-caryophyllene
4	16.91	1.80	Anethole
5	17.01	13.17	Acetyeugenol
<b>3. Olibanum essential oil</b>			
1	6.49	1.14	α-pinene
2	6.82	0.61	Camphene
3	7.25	0.59	β-phellandrene
4	7.43	0.18	β-pinene
5	7.89	0.11	Carvacrol
6	8.23	0.77	p-cymene
7	8.35	1.50	D-Limonene
8	10.30	1.11	Eucalyptol
9	11.31	<u>94.00</u>	Octyl acetate

non-volatile portions of the imported samples along with the local samples by TLC and GC-MS procedures (Adams, 1995; Wagner and Bladt, 1996).

TLC profiles in different solvent systems of the volatile portions of imported samples and local samples of essential oils were similar to the composition of mint, clove and olibanum oils described in the literature, while the TLC profiles of non-volatile portions were close to the composition of fixed oils (Wagner and Bladt, 1996; Evans, 2002). An important conclusion reached could be that imported essential oils as randomly purchased samples from the local market could be adulterated by cheaper fixed oils. The qualitative and quantitative composition of essential oils and volatile portions was determined by GC-MS technique and identification of components was achieved by comparison with data in the library dataset (NIST 1998 MS). The results were reported in Tables 2 to 5). Carvone (70.20%), eugenol (81.85%) and octyl acetate (94.00%) were the main components of local mint, clove and olibanum essential oils respectively, and were compatible with the published data in the literature (Wagner and Bladt, 1996; Evans,

2002). Menthol (24.82) and dibutyl phthalate (30.60%) were the main components of imported mint oil; eugenol (24.03%) and an unidentified component (21.91%) represented almost half of the imported clove oil, while p-xylene (23.18%) was the main component of imported olibanum oil. The detection of saturated and unsaturated hydrocarbons and other adulterants of aromatic nature in the imported samples modified their physico-chemical properties, hence their incompatible results compared to international standards.

In conclusion, according to the results obtained it was obvious that imported essential oils of mint, clove and olibanum were not pure samples of essential oils and contained other components as adulterants. Comparison with local samples of mint, clove and olibanum essential oils and published data in the literature indicated their adulteration if they were under use as pure essential oils. Another possibility would be their use in aromatherapy practices, where carriers represent considerable part of the essential oils. Quality control and assessment of the imported essential oils need to be promoted with regard to the ongoing research and development in this field.

**Table 3.** Results of GC-MS analysis of imported mint essential oil.

Peak no.	Retention time (t <sub>R</sub> )	Composition (%) from total peak area	Name of component
1	5.84	8.71	Eucalyptol
2	11.69	7.22	Menthone
3	12.57	24.82	Menthol
4	14.51	4.71	Piperitone
5	15.53	2.98	Isobornyl acetate
6	20.62	2.72	Heptadec-1-ene
7	23.10	3.02	Nonadec-1-ene
8	23.18	2.04	Heptadecane
9	23.42	2.39	2,6,10,14-Tetramethyl/hexadecane
10	24.30	3.32	Heptadecane
11	25.10	30.60	Dibutyl phthalate
12	25.49	1.97	Triacos-1-ene
13	26.67	2.62	Pentatriacontane

**Table 4.** Results of Gc-MS analysis of imported clove essential oil.

Peak no.	Retention time (t <sub>R</sub> )	Composition (%) from total peak area	Name of component
1	12.01	3.20	Menthol
2	13.82	6.75	Heneicosane
3	15.03	21.91	Unidentified
4	16.95	<u>24.03</u>	Eugenol
5	21.44	5.40	Diphenylamine
6	21.63	6.03	Octadecane
7	23.19	14.07	Unidentified
8	25.08	2.40	Pentadecane
9	26.84	1.77	Unidentified
10	27.34	4.99	Unidentified
11	33.93	0.96	Nonadecan-1-ol
12	41.77	1.32	1,2-Benzene dicarboxylic acid

**Table 5.** Results of GC-MS analysis of imported olibanum essential oil.

Peak no.	Retention time (t <sub>R</sub> )	Composition (%) from total peak area	Name of component
1	2.88	12.90	Unidentified
2	3.91	5.65	1-Ethoxypentane
3	5.29	23.18	p-xylene
4	5.64	11.37	1-Ethyl-4-methylcyclohexane
5	6.01	9.93	1-Ethyl-4-methylcyclohex-1-ene
6	6.44	10.38	Propylcyclohexane
7	17.12	12.19	Octadec-1-ene
8	20.37	14.40	Nonadec-1-ene

## ACKNOWLEDGEMENTS

We thank the administration of UMST and Faculty of Pharmacy, for their technical assistance. Part of this work was carried out in the Central Laboratory, Ministry of Science and Technology, Sudan.

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