



Comparison of Chemical Constituents of Essential Oils of Black Cumin (*Nigella sativa* L.)

FAHAD AL JUHAIMI^{1*}, GILLES FIGUEROA², MEHMET MUSA ÖZCAN³ and PIERRE CHALARD⁴

¹Department of Food Science & Nutrition, College of Food and Agricultural Sciences, King Saud University, Riyadh, Saudi Arabia

²Laboratoire de Chimie des Huiles Essentielles Université Blaise Pascal de Clermont, 63177 Aubiere Cedex, France

³Department of Food Engineering, Faculty of Agriculture, University of Selçuk, 42031 Konya, Turkey

⁴Laboratoire de Chimie des Hétérocycles et des Glucides - EA987/UBP Ensemble Scientifique des Cézeaux, France

*Corresponding author: E-mail: faljuhaimi@ksu.edu.sa

(Received: 4 April 2013;

Accepted: 18 November 2013)

AJC-14413

Essential oils from dried aerial parts of *Nigella sativa* were analyzed using gas chromatography-mass spectrometry (GC-MS). The air dried plant materials were subjected to hydrodistillation for 4 h using a Clevenger type apparatus to give yellow oils. The major compounds in the essential oil of Turkey sample were α -thujene (15.78 %), *p*-cymene (48.25 %), *trans*-4-methoxythujane (6.56 %), β -pinene (4.60 %), α -pinene (3.52 %) and limonene (2.88 %) while *p*-cymene (51.42 %), α -thujene (16.36 %), *trans*-4-methoxythujene (6.11 %), β -pinene (3.98 %), α -pinene (3.60 %) and limonene (2.84 %) were major components of Syria essential oil. The main compound of Ethiopia essential oil was *p*-cymene (55.03 %) followed by α -thujene (18.19 %), *trans*-4-methoxythujene (6.33 %), β -pinene (4.50 %), α -pinene (3.96 %) and limonene (2.85 %).

Key Words: *N. sativa*, Essential oil, Constituents, *p*-Cymene, α -Thujene, β -Pinene.

INTRODUCTION

Nigella sativa (black cumin) belongs to the family Ranunculaceae had originated in Turkey and Italy is now commonly grown in Europe, Middle East and Western Asia¹. It is an annual herbaceous plant and commonly known as black cumin seed. The role of the essential oils is predominant in many industries, particularly in the pharmaceutical, clinical and food industries²⁻⁴. The volatile oils are complex mixtures of the compounds which mainly having monoterpenes, sesquiterpenes hydrocarbons with general formula (C₅H₈)_n^{5,6}. The black cumin seeds are trigonous black rugose tubercular and used as a flavouring agent and as medicinal purposes⁷. In the seed oil thymoquinone was identified as the main component besides *p*-cymene, α -pinene, dithymoquinone, thymohydroquinone and nigellone. Other terpene derivatives were found only in traces *e.g.*, carvacrol, carvone, limonene, terpineol-4, citronellal^{1,3,8,9}. For many centuries, *N. sativa* seeds have been used as a food additive as well as for medicinal purposes in many countries¹⁰. This plant is one of the most extensively studied, both phytochemically and pharmacologically¹¹⁻¹³. Another use of *N. sativa* seeds is as seasoning for food stuffs like bread and pickles, especially widespread among Turkish people¹⁴. In the framework of research program, the aim of current study was to establish the chemical composition of

essential oil *N. sativa* seeds purchased from local markets in Saudi Arabia and Turkey.

EXPERIMENTAL

The seeds of *Nigella sativa* were provided from local market in Turkey, Syria and Ethiopia. The seeds were dried in air conditions until a constant weight was reached and then the seeds were ground to a fine powder, packaged and stored at 4 °C until analyses. Seeds were transported to the laboratory in polypropylene bags and held at refrigerator. They are cleaned in an air screen cleaner to remove all foreign matter such as dust, dirt and immature and broken seeds were discarded as well. Seeds were preserved until ground using a hammer mill. After grinding, the seed flour was kept until using.

Recovery of the essential oils: Dried aerial parts of the plants (200 g) were ground and submitted to hydrodistillation for 4 h using a Clevenger-type apparatus and the oils obtained were dried over anhydrous sodium sulfate.

Identification of components: For identification of components, analytical HP 5890 gas chromatograph equipped with FID (GC) was performed a DELSI 121 C apparatus fitted with a flame ionization detector and a CP WAX 51 fused silica column (25 m × 0.3 mm; 0.25 μ m film thickness). Temperature

was programmed from 50 °C for 5 min and to reach 220 °C at the rate of 3 °C per min. ACP WAX 51 fused silica WCOT column (60 m × 0.3 mm) for GC/MS was used with helium as carrier gas (flow rate 1 mL/min) and coupled to a HP mass spectrometer: ionization energy 70 eV. Temperature programming was from 50-240 °C at the rate 3 °C/min. The samples

were injected at 240 °C. The components were identified by comparing linear Kovats indices (KI), their retention times (RT) and mass spectra with those obtained from the authentic samples and/or the MS library. Qualitative analysis was based on a comparison of retention times and mass spectra with corresponding data in the literature¹⁵.

TABLE-1
CHEMICAL COMPOSITION OF ESSENTIAL OIL OF *N. sativa* SEEDS PROVIDED FROM TURKEY, SYRIA AND ETHIOPIA (%)*

RT	Constituents	Turkey	Syria	Ethiopia
8.36	1-Hexanol	—**	0.09***	—
10.38	α-Thujene	15.78	16.36	18.19
10.62	α-Pinene	3.52	3.59	3.96
11.0	Butylbenzene	—	0.05	—
11.13	Thuja-2,4(10)-diene	—	—	0.06
11.21	Camphene	0.06	0.05	0.08
12.06	Sabinene	1.96	1.77	1.69
12.23	β-Pinene	4.59	3.98	4.50
12.66	Myrcene	—	—	0.01
12.69	2-Pentylfuran	0.12	0.09	—
13.24	α-Phellandrene	—	—	0.03
13.63	α-Terpinene	0.22	0.38	0.45
13.99	p-Cymene	48.25	51.42	55.03
14.08	Limonene	2.88	2.84	2.85
14.2	Eucalyptol	0.14	0.20	0.13
15.03	γ-Terpinene	1.64	1.40	1.46
15.47	cis-Hydrate de saminene	0.04	0.08	0.07
15.91	Terpinolene	0.04	0.05	0.10
16.08	p-Cymenene	—	0.11	—
16.26	cis-4-Methoxythujane	1.09	—	1.06
16.41	Linalool	0.05	0.06	0.09
17.01	trans-4-Methoxythujane	6.56	6.11	6.33
17.9	Camphre	0.02	—	0.26
18.35	Verbenol	0.51	0.33	0.27
18.88	Terpinene-4-ol	0.61	0.62	0.45
19.37	Methyl chavicol	—	0.07	0.06
20.66	Cuminaldehyde	1.67	—	—
20.67	Carvone	—	0.13	0.09
21.09	iso-3-Thujylacetate	—	0.07	—
21.11	Neo-3 Thujylacetate	0.08	—	—
21.79	Anethole + bornyl acetate	—	0.42	0.11
21.82	Anethole	0.63	—	—
21.87	Thymol	—	0.05	—
21.97	γ-Terpinene-7-al	0.38	—	—
22.04	2E,4Z-Decadienal	0.42	—	—
22.14	Carvacrol	0.55	0.31	0.20
22.69	2E,4E-Decadienal	2.52	—	—
23.34	α-Terpinyol acetate	—	0.05	—
23.5	α-Longipinene	0.23	0.14	0.12
24.10	α-Copaene	—	0.18	—
25.03	Longifolene	1.23	0.79	0.63
25.24	β-Caryophyllene	0.08	0.12	0.11
27.62	δ-Cadinene	—	—	0.02
28.95	Spathulenol	0.62	—	—
29.08	Oxyde de caryophyllene	1.52	—	—
34.76	Phthalate	0.05	—	—
36.55	13-epi-manool	0.15	0.10	—
36.56	Pimaradiene	—	—	0.05
36.74	Hexadecanoic acid	—	1.12	0.43
39.92	Linoleic acid	—	2.76	0.35
40.03	Oleic acid	—	1.81	0.25
Total (%)		31	35	33

*Compound listed in the order of elution from a HP-5MS column; **Unidentified; ***Each compound is mean of two values.

RESULTS AND DISCUSSION

The hydrodistillation of the extract from the seeds of *N. sativa* gave a yellowish essential oils. Generally the major constituents of oils were found close. About 31, 35 and 33 compounds were identified. The major compounds in the essential oil of Konya sample were α -thujene (15.78 %), *p*-cymene (48.25 %), *trans*-4-methoxythujane (6.56 %), β -pinene (4.60 %), α -pinene (3.52 %) and limonene (2.88 %) while *p*-cymene (51.42 %), α -thujene (16.36 %), *trans*-4-methoxythujene (6.11 %), β -pinene (3.98 %), α -pinene (3.60 %) and limonene (2.84 %) were major components of Syria essential oil. The main compound of Ethiopia essential oil was *p*-cymene (55.03 %) followed by α -thujene (18.19 %), *trans*-4-methoxythujane (6.33 %), β -pinene (4.50 %), α -pinene (3.96 %) and limonene (2.85. %). These results are partly different to the qualitative results obtained from other investigations^{3,6,16}. Singh *et al.*³ reported that the major component of *N. sativa* seeds oil was *p*-cymene (36.2 %) followed by thymoquinone (11.27 %), α -thujene (10.03 %), longifolene (6.32 %) and carvacrol (2.12 %). They reported that this essential oil contains thymoquinone as the major component, whereas our essential oil contains *p*-cymene as the major component. Rizvi *et al.*¹ determined *p*-cymene (10.6 %), menthol (2.9 %) and thymoquinone (15.9 %), palmitic acid (6.8 %), linoleic acid (41.1 %) and oleic acid (3.5 %) in crushed *N. sativa* seeds. Gerige *et al.*⁶ reported that *p*-cymene (9.0 %), limonene (4.3 %), thymoquinone (11.8 %), *trans*-anethole (27.1 %), carvacrol (3.7 %), longifolene (5.7 %) were identified as the major constituents of *N. sativa* volatile oil. According to our survey of the available literature on the composition of *Salvia* species, our data partially agrees with previous studies. As a result, it can be said that plant derivatives are good phenol resources. Also, several derivatives such as essential and extracts of medicinal and aromatic plants can be used as preservatives for food. Further studies should be carried out on biofunctional properties of these plant derivatives *in vitro*.

Conclusion

These results show that these plants are remarkably variable species. The high quantities of *p*-cymene, α -pinene, β -pinene and α -thujene. Especially, *N. sativa* contained monoterpene hydrocarbons constituents.

ACKNOWLEDGEMENTS

This project was financially supported by King Saud University, Deanship of Scientific Research, College of Food and Agricultural Sciences, Research Center.

REFERENCES

1. A.H. Rizvi, M.M. Abid Ali Khan, G. Saxena and A.A. Naqvi, *J. Biol. Chem. Res.*, **29**, 44 (2012).
2. K. Knobloch, A. Pauli, B. Iberl, H. Weigand and N. Weis, *J. Essen. Oil Res.*, **1**, 119 (1989).
3. G. Singh, P. Marimuthu, C.S. de Heluani and C. Catalan, *J. Sci. Food Agric.*, **85**, 2297 (2005).
4. S. Pepeljnjak, I. Kosalec, Z. Kolodera and D. Kustrak, in eds.: M. Rai and D. Mares, *Natural Antimycotics from Croatian Plants*, in *Plant-derived Antimycotics. Current Trends and Future Prospects*, Harworth Press, New York, pp. 41-84 (2003).
5. K.P. Svoboda and J.B. Hampson, *Bioactivity of Essential Oils of Selected Temperature Aromatic Plants: Antibacterial, Antioxidant, Anti-inflammatory and other related pharmacological activities*, Proceedings NAHA, 25-28 september, St. Louis, Missouri, USA 105-127 (1999).
6. S.J. Gerige, M.K.Y. Gerige and M.R. Muralidhara Rao, *Braz. Arch. Biol. Technol.*, **52**, 1189 (2009).
7. S. Vardharajan, *The Wealth of India-A Dictionary of Indian Raw Materials and Industrial Products*, Publication and Information Directorate, CSIR, Vol. 1A, New Delhi (1985).
8. M. El-Dakhkhany, *Planta Med.*, **11**, 465 (1963).
9. A.M. Gad, M. El-Dakhkhany and M.M. Hassan, *Planta Med.*, **11**, 134 (1963).
10. P.C.M. Jansen, *Spices, Condiments and Medicinal Plants in Ethiopia, Their Taxonomy and Agricultural Significance*, Center for Agricultural Publishing and Documentation, Addis Ababa, pp. 76-85 (1981).
11. M. Riaz, M. Syed and F.M. Chaudhary, *Hamdard Medicus*, **39**, 40 (1996).
12. N. El-Sayed and S. El-Din, M.Sc. Thesis, *Some Pharmacological Studies of Nigella sativa*. Faculty of Pharmacy, Cairo University (1998).
13. D.R. Worthen, O.A. Ghosheh and P.A. Crooks, *Anticancer Res.*, **18**, 1527 (1998).
14. L.F. D'Antuono, A. Moretti and A.F.S. Lovato, *Ind. Crops Prod.*, **15**, 59 (2002).
15. R. Adams, *Essential oil components by Quadrupole GC/MS*, Allured Publishing Corp., Carol Stream, IL, USA (2001).
16. H.M. Adamu, E.O. Ekanem and S. Bulama, *Pak. J. Nutr.*, **9**, 966 (2010).