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The Effect of Two Methods of Nigella oil Extraction on Chemical Composition.

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ABSTRACT

Black Cumin is an annual herbaceous plant which is cultivated for its seeds and is classified as an edible plant. Two methods of Nigella oil extract(Folklore method and Mechanical cold press method) and the composition of Nigella fixed and volatile oils were studied. Some fatty acids and main compounds of Nigella oil were also determined.Using the mechanical cold press method gave higher percentages of oil and main compounds of Nigella volatile and fixed oils than the Folklore method.

Key words: *Nigella sativa L.*, Nigella Oil, Methods of extraction, Fatty Acid,Volatile Oil, Linoleic Acid,Thymoquinone.

INTRODUCTION

Among the most important medicinal crops in Egypt are *Nigella sativa* L.(Nigella) which belonging to family Ranunculaceae Nigella plants are widely distributed in countries which border the Mediterranean Sea, Central Europe and Western Asia (Hedrick, 1972).

There are many species of Nigella (About 20 spp. of annual herb) such as *sativa, aristata, arvensis, damascena, diversifolia, integrifolia,* ... etc. (Bailey, 1978). The growing conditions for all Nigella species are approximately the same.

Nigella sativa L commonly known as Nigella, sometimes known as Haba El-Barka or Black Cumin in some Middle East countries.*Nigella sativa L.* is only source of Nigella oil as compared with other Nigella species (Cevdet and Semih, 1993).

The economical importance of Nigella plants is attributed to the seeds, which contain the Nigella oil.

The whole seed contains 30-35% of oil (Ustun et at. 1990). Nigella oil consists of fixed oil and essential oil, the percentage of essential oil is around 0.091-0.105% (Peter et al. 1995). Fixed oil of Nigella is rich in Linoleic acid (above 60%) (Ustun et al. 1990). Moreover the principal compounds of essential oil of Nigella are mainly α-Pinene, Sabinene, Limonene, Camphore, Thymoquinone (Nigellon) and Dithymoquinone (Awad et al., 1993, and Peter et al., 1995). The pharmaceutical effects of Nigella oil are due to the essential oil (Peter et al., 1995).

Nigella sativa L. seeds are used almost entirely for edible and medical purposes, such as for seasoning many kinds of cookies (as a condiment). Nigella plants (seeds or oil) have a long history of folkloric medicine in Arabian and other countries for treatment of various diseases. The seeds, on account of their aromatic nature, are used as a spice in cooking, particularly in Italy and Southern France. They are also used as a carminative and diuretic (Hedrick, 1972).

The expressed oil from the seeds is sold in the markets of the Middle East and neighbouring countries specifically as a topical treatment for pain and stiffness of the joints. This indication together with reports of the use of the seeds as a treatment for bronchial asthma and for eczema. The oil may affect the formation of inflammatory agents associated with this condition (Sayed, 1980).

The seeds of *Nigella sativa L* have been subjected to arrange of pharmacological investigation in recent years and a recent report has demonstrated analgesic activity in mice (Khanna, et al., 1993).

In modern study to undertake the effect of some medicinal plant extracts on wound healing in farm animal, the result revealed

that healing was the best with the use of *Nigella sativa L.*lotion (in the form of oil preparation for both washing and dressing of wounds) and other extract (Ahmed et. at, 1995).Further, the fixed oil isolated from *Nigella sativa L.* seeds showed insecticidal activity against the stored grain insects (Deshpande et al., 1974) .

Generally, most of the changes in the chemical composition of fixed oils refer to the condition of extraction method (Owen, 1996)

The aim of the present work is to study the effect of two methods of oil extraction on chemical composition of Nigella oil (fixed and volatile).

Materials and Methods

Nigella oils used in this study were obtained from two sources of Nigella oils .The first source was purchased from local condiments markets in Alexandria.In this case , Nigella oil was extracted by a folklore method (different sequence steps: crush of seeds, roste of powdered seeds and the mechanical press was used). The other source of Nigella oil was extracted by a mechanical cold press machine. Seeds (samples of 100 gm) were extracted for 15 mins. using CARVER model 2759S/N 2759-595 FREDS CARVER INC. in Medicinal and Aromatic Plants Branch laboratory in Sabahia Horticulture Researches Station in Alexandria , Egypt. .

The layout of the experiment was complete randomized design, ten samples were used for every methods(Snedecor and Cochran , 1974).

The crude oil (fixed oil + Essential oil) was obtained by mechanical cold pressing of the seeds with a screw press machine . The oil percentage, some physical properties (Refractive index, Iodine value, Acid value and Saponification value) and chemical composition of the cold pressed oil were determined (Official Methods of the American oil Chemists Society (1989)).

Nigella essential oil was extracted by adding one ml of methanol to 1 ml of the crude oil (fixed oil + essential oil) in a glass centrifuge tube with cover. Vortex mix for 2 mines, the methanol top layer (essential oil) was transferred to a small glass vial (Abou-Basha et al. ,1995).

Gas chromatography - Mass Spectrum technique was used to determine the principal components of essential oil of Nigella oil.

Conditions of GC-Ms:

Information	Condition
Instrument	Hewlett Packard (HP)
Column	Stainless steel capillary column (12m.)
Stationary phases	SMG
Flow rate	1.5 ml. H/min.
Column temp.	30-180 ^{0}C
Rate temp.	5^{0}C/min.
Injection temp.	250 ^{0}C
Detector temp.	280 ^{0}C
Recorder	HP

Gas chromatography technique was used to determine the fatty acids of Nigella oil.

Conditions of GC:

Information	Condition
Instrument	Fisons 8000
Column	Stainless steel capillary
Stationary phases	MGE
Flow rate	1.5 ml. H/min.
Column temp.	30-180 ^{0}C
Rate temp.	5 ^{0}C / min.
Injection temp.	250 ^{0}C
Detector temp.	280 ^{0}C
Recorder	HP

The percentage of oil constituents (Fatty acids and main compounds of the essential oil) were estimated by measuring the peak area of the different compounds of the chromatogram according to Heftman (1967) and Gunther and Joseph (1978).

Authentic samples of the main compounds of Nigella volatile oil and fatty acids of Nigella fixed oil were obtained from Buch Boake Allen Limited, London, England for Camphene and α-Pinene; and Dithymoquinone and Thymoquinone from Haarmann and Reimner, Holzminden, Germany; and fatty acids (Palmitic, Stearic, Oleic and Linoleic.) from Ciba Gigi, NY, USA.

Results and Discussion

The effect of different methods of oil extraction on Nigella oil percentage , physiocomercial properties of the crude Nigella oil , the percentage of the main compounds of Nigella volatile oil , and fatty acids percentage of Nigella fixed oil are shown in Tables (1 and 2) and illustrated in Figs.(1,2 and 3).The results indicated that using two methods of oil extraction had significant effect on most of the studied physiocomercial and chemical properties of the crude oil of Nigella oil except the percentage of Unsaponifiables (Table 1 and Fig 1).

These results were probably due to that physiocomercial properties of Nigella oil were mainly linked with the conditions of oil extraction method. (Guenther, 1961)

Generally, using Mechanical Press gave the heights percentage of Nigella fixed oil (35.09%), and the main compounds of Nigella volatile oil (α - Pinene, Camphene, Thymoquinone and Dithymoquinone); and fatty acids percentages of Nigella fixed oil (Linoleic, Oleic, Palmitic and Stearic) compared with folkloric method. The chromatographic fractionation shows that the major fatty acid is Linoleic acid (39.34%).

The lowest percentage of the major constituents of the volatile oil may be probably due to the effect of heat used in folkloric method

on the chemical components. Some compounds were reduced and some were broken down (Owen, 1996).

On the other hand, using the heat in folkloric method led to the increase in saturated fatty acids (Palmitic and Stearic) percentage of Nigella fixed oil. This point is very important for human health as a relationship between them and the concentration of cholesterol in blood serum (Mathews and Holde, 1996).

References

Abou-Basha,L.; M. Rashed; and H. Aboul - Enein. 1995. TLC assay of thymoquinone in black seed oil (*Nigella sativa* L.) and identification of dithymoquinone and thymol. J.L. Chromatography, 18 (1), 105-115.

Ahmed,I; M.Awad ; M. El-Mahdy ;H. Gohar, and A. Ghanem. 1995. The effect of some medicinal plant extracts on wound healing in farm animals.Assiut Vet.Med. J. Vol. 32 No. 64.

Awad, N.; F. Hafez, and A. Ali. 1993. The production of *Nigella sativa L.* oil constituents through genetically engineered yeast strain. Assiut J. of Agr. Sci., 24:4, 225-240.

Bailey, H. 1978. A Concise Dictionary of Plants Cultivated in United States and Canada. Macmillan Publishing Co., Inc. New York.

Cevdet. N., and O. Semih. 1993. Chemical composition of *Nigella sativa L.* seeds. Food Chem. 48:259-261.

Deshpande, R.; P. Adhikary, and H.Tipnis. 1974. Stored grain pest control agents from *Nigella sativa L.* and *Pogostemon heyneanus.* Bull, Grain Technol. 12;3,232-234.

Guenther, E. 1961. The Essential Oils. Vol. I D. Van Nostrand Company Inc. New York.

Gunther, z., and S. Joseph. 1978: Hand Book Series in Chromatography. CRC press, Inc.

Hedrick, U. 1972. Sturtevant's Edible Plants of the World. Dover, New York, pp. 388 - 89.

Heftman, E. 1967. Chromatography. Reinhold Pub. Crop. New York.

Khanna, T.; F. Zaida, and P. Dandiya. 1993. Uses of Some Fixed Oils. Fitoterapia, 64: 407-410.

Mathews, C., and K. Holde. 1996. Biochemistry, 2nd. Ed., The Benjamin / Cummings Publishing Company, Inc. New York, U.S.A.

Peter, H; R.Zarka; B. Heras, and J. Hoult. 1995. Fixed oil of *Nigella sativa* and derived thymoquinone inhibit eicosanoid generation in leukocytes and membrane lipid peroxidation. Planta Med. 61, 33-36.

Sayed,M. 1980. Uses of Nigella oil. J. Ethnopharmacology 2,19-22.

Snedecor, G., and W. Cochran. 1974. Statistical Methods. Sixth Edition, Iowa State College Press, Ames , Iowa, USA.

Owen,F.1996.FoodChemistry.Third Edition,Marcel Dekker Inc.,N.Y.,USA.

Ustun, G.; L. Kent; N. Cekin; and H. Civelekoglu. 1990. Investigation of the technological properties of Nigella sativa (Black Cumin) seed oil. J.AOCS, Vol. 67, No. 12.

Table (1): Effect of different methods of oil extract on some physical Properties (Acid value , Saponification value , Iodine value, Unsaponifiables (%) Density g/mL. 20 ^{0}C and Refractive Index, 20 ^{0}C).s

Methods	Acid value	Saponification value	Iodine value	Unsaponif-iables (%)	Density g/mL.20 ^{0}C	Refractive index,20 ^{0}C
Folklore	**30.01**	**192.5**	**109.8**	**0.58**	**0.9432**	**1.4918**
M.Press	**29.29**	**189.2**	**111.9**	**0.59**	**0.9212**	**1.5432**
L.S.D $_{0.5}$	**0.23**	**6.55**	**1.05**	**n.s.**	**0.84**	**0.092**

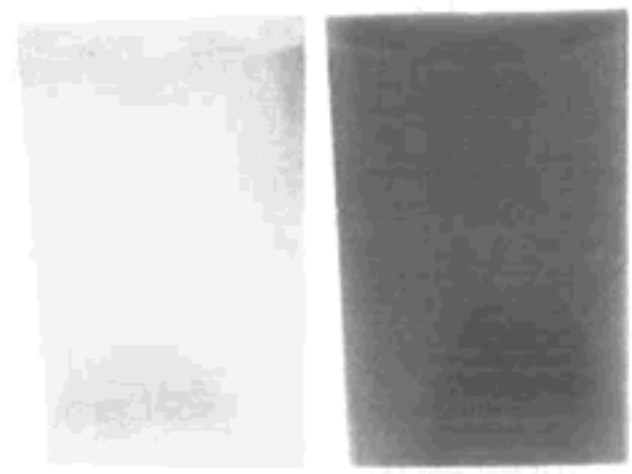

Fig (1):Nigella oil product , (left : mechanical press) and (right : folklore method).

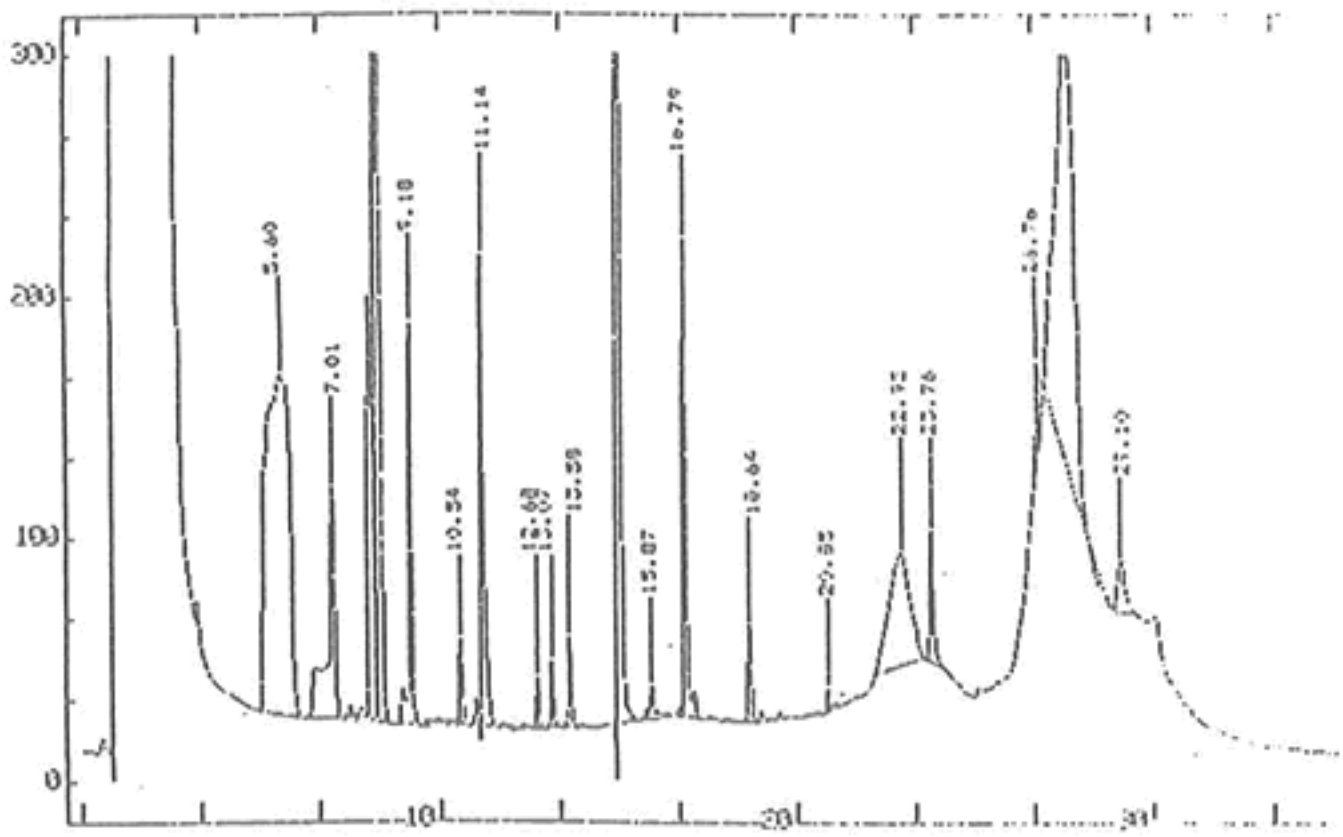

Fig (2): Typical chromatogram of chemical composition of Nigella volatile oil (α - Pinene, Camphene, Thymoquinone and Dithymoquinone) (Mechanical Press Method).

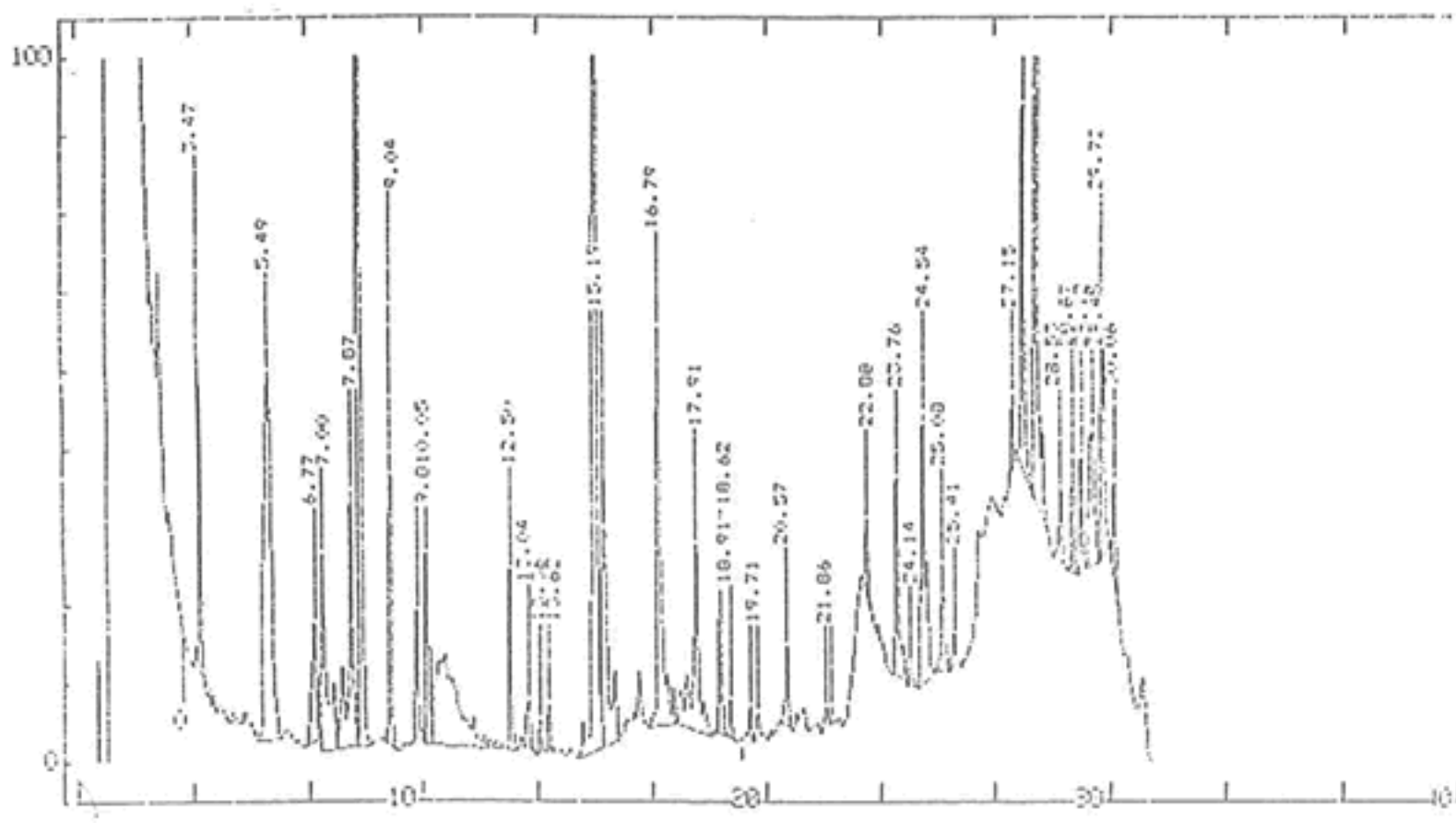

Fig (3): Typical chromatogram of chemical composition of Nigella volatile oil (α- Pinene , Camphene , Thymoquinone and Dithymoquinone) (Folklore Method).

Table (2):Means of the percentage of Nigella oil, some main compounds percentage of Nigella volatile oil, and Fatty acids percentage of Nigella fixed oil, as influenced by two methods of oil extract.

Methods	Nigella Oil %	PrincipalsCompounds % of NigellaVolatile oil				Fatty Acids % of Nigella fixed oil			
		α - Pinene	Camphene	Thymoquinone	Dithymoquinone	Linoleic	Oleic	Palmitic	Stearic
Folklore	33.05	13.53	5.23	0.50	0.11	34.23	23.02	14.91	3.89
M. Press	35.09	18.04	7.92	1.68	0.97	39.34	26.65	12.10	3.11
L.S.D $_{0.05}$	1.98	0.33	0.29	0.20	0.11	0.92	0.78	0.71	0.42

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تأثير طريقتي استخلاص لزيت حبة البركة على المكونات الكيميائية

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ملخص :

نبات حبة البركة من النباتات الحولية الصالحة للأكل التـــى تـــزرع بالبـــذور. درســـت طريقتين لاستخلاص زيت حبة البركة (طريقة شعبية و طريقة اسـتخلاص على البارد) ومكونات الزيت الثابت و الطيار .أيضا قدرت بعض الأحماض الدهنيـــة و المكونات الرئيسية لزيت حبة البركة .استخدام طريقة الاستخلاص علـــى البـــارد أعطت نسبة مئوية عالية من الزيت والمكونات الرئيسية للزيت العطـــري والثـــابت بالمقارنة بالطريقة الشعبية

كلمات مفتاحية: نبات حبة البركة , زيت حبة البركة , طرق الاستخلاص , حامض دهني , زيت عطري , لينوليك , ثيموكينون.