Investigation of the Technological Properties of *Nigella sativa* (Black Cumin) Seed Oil

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Three commercially cultivated *Nigella sativa* seed varieties of Turkish origin were analyzed, and the characteristics and constituents of the seed oils were reported. Presence of lipase enzyme in seed results in enzymatic hydrolysis at ordinary temperature; the free acid content of oil may increase up to 40% or higher. Black cumin seed oil might serve as a source of semi-drying oil and fatty acids of technical grade, and the removal of free fatty acids from oil and the recovery of fatty acids were investigated.

KEY WORDS: Black cumin seed oil, lipase enzymatic hydrolysis, *Nigella sativa*, vegetable oil.

Nigella sativa seeds are used almost entirely for edible and medical purposes, such as for seasoning many kinds of cookies and for treatment of some diseases. However, the whole seed contains 30–35% of oil that is rich in linoleic acid (content above 60%) (1–6). Presence of lipase enzyme in seed (7) results in enzymatic hydrolysis at ordinary temperature, therefore the free acid content of oil may be as high as 40% or higher. This fact may render the oil more easily hydrolyzed, with less chemicals required than used in ordinary hydrolysis procedures in the manufacture of fatty acids for surface coatings and other industrial applications.

In consideration of potential utilization, detailed knowledge on the composition of *Nigella sativa* seed oil is of major importance. Little factual information is available concerning the exact composition of *Nigella sativa* seed oils originating from Turkish sources (8). In one study (8), it was mentioned that *N. sativa* seed contained 26.6% oil, of which the major fatty acids were linoleic (64.6%) and palmitic (20.4%) acids.

In this study, the general characteristics and composition of three *Nigella sativa* seed oil samples of Turkish origin were investigated. The deacidification of the black cumin seed oil with a low refining loss and the recovery of fatty acids as a by-product were also considered.

MATERIALS AND METHODS

Three samples of fully matured *Nigella sativa* seeds were collected from the Kütahya, Denizli and Konya districts of Turkey.

For determination of oil characteristics and composition, pressing and solvent extraction methods were used for oil production. Both oils were analyzed separately.

Each air-dried seed sample was extracted for 4 hr with petroleum-ether (b.p.: 40–60°C) in a Soxhlet apparatus. The solvent was removed with a Büchi vacuum distillation apparatus and the remaining oil was weighed. The oil content of seed was calculated on a moisture-free basis.

Seed oils were also obtained with a laboratory-type of Carver hydraulic press under 10,000 lb/in^2 (psi) pressure for 1 hr at 80°C.

The oils either extracted or pressed were analyzed for acid, saponification and iodine (Hanus) values; unsaponifiable matter content; density and refractive index according to the AOCS (9) and AOAC (10) procedures.

The fatty acid compositions of seed oils were determined by saponification, acidification, conversion of the liberated acids to methyl esters and analysis by gas chromatography of the methyl esters. The analyses were carried out with a Varian 3700 unit equipped with a hydrogen flame ionization detector, a data processor and a stainless-steel column (2.0 m × 2.0 mm) packed with 10% DEGS/Chromosorb G (80–100 mesh). The column, injection port and detector were maintained at 160–185°C, 240°C and 260°C, respectively. Nitrogen was used as a carrier gas at a flow rate of 30 mL/min.

A black cumin seed oil (with 21.22% acidity) was subjected to a laboratory-scale cross-current multistage extraction with 90% aqueous methanol (11). In preliminary experiments, during extraction of samples with high free fatty acid content, the occurrence of a strong emulsion was observed. However, the addition of common salt to the solvent prevented formation of such emulsions. In the series of extractions, 4 parts of 90% aqueous methanol (containing 0.2 g NaCl/L) was used for 1 part of oil in 4 stages.

RESULTS AND DISCUSSION

The oil contents of *Nigella sativa* seeds collected from the Kütahya, Denizli and Konya regions are quite similar, 29.4, 29.5 and 29.7%, respectively.

Due to close similarity between extracted and pressed seed oil samples, both in their composition and characteristics, average values are given in the following Tables. Table 1 presents a comparison of the average oil characteristics of Turkish seed oils with those reported in the literature (1,2,4,6).

The identification of individual fatty acids of *Nigella sativa* seed oil samples was carried out by gas chromatography. Fatty acid compositions of *Nigella sativa* seed oils reported in the literature are summarized in Table 2. Our quantitative gas chromatography (GC) evaluations of fatty acid contents are also included.

Table 2 reveals that the major components of fatty acids are linoleic and oleic acids. These oils also contain appreciably larger amounts of saturated normal chain fatty acids, especially palmitic acid. In addition

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TABLE 1

Oil Characteristics of *Nigella sativa* Seed Oils from Some Countries (1,2,4,6)

Country	Acid value	Saponification value	Iodine value	Unsaponifiables (%)	Density g/mL, 20°C	Refractive index, 20°C
Czechoslovakia (1,2)	14.68	210.6	107.4	—	0.8930	—
	29.42	201.98	110.9	—	0.8960	—
India (4)	40.64	169.9	116.9	4.0	0.9164	1.466 (35°)
Egypt (6)	30.3	196.3	114.5	0.66	0.9207	1.4718
Turkey (this study)						
Kütahya	39.61	212.75	116.63	3.47	0.9221	1.4731
Denizli	36.74	230.50	112.32	5.38	0.9188	1.4711
Konya	42.60	196.10	122.13	4.00	0.9044	1.4690

TABLE 2

Fatty Acid Compositions of *Nigella sativa* Seed Oils from Some Countries (4–6)

	Fatty Acid (%)								
Country	Myristic	Palmitic	Stearic	Oleic	Linoleic	Linolenic	Arachidic	Behenic	Eicosadienoic
India (4)	0.29	7.08	2.74	49.67	40.22	—	—	—	—
U.S.A. (5)	0.16	12.08	3.11	24.64	56.12	0.7	—	—	2.5
Egypt (6)	11.8	11.8	11.8	48.76	37.56	1.88	—	—	—
Turkey (this study)									
Kütahya	0.45	12.97	2.08	21.84	61.84	0.33	0.22	0.19	—
Denizli	0.34	12.70	1.99	22.03	62.53	—	0.24	0.17	—
Konya	0.49	15.12	1.62	23.76	58.38	—	0.37	0.26	—

to the common fatty acids, measurable amounts of 14:0, 20:0 and 22:0 saturated fatty acids were detected in the investigated samples. A 0.33% of linolenic acid was only identified in the Kütahya seed oil sample. Contrary to Babayan *et al.* (5), eicosadienoic acid was absent from all samples that were investigated.

Since *Nigella sativa* seeds are subject to strong enzymatic hydrolysis during harvesting, handling and processing of the oil (7), seed oils were obtained containing about 20% of free acids, calculated as oleic acid. During storage of the Konya seed oil samples at room temperature, the free fatty acid contents of both the extracted and cold-pressed oils increased significantly compared with the pressed oil, which was only slightly affected (Table 3). These facts may be the result of catalytic lipase activity in water-in-oil microemulsions present in oils (12) and of the loss of enzyme activity during pressing of seeds at 80°C, respectively.

It was also noticed that the cold-pressed oil sample had relatively low initial FFA content. During the extraction and hot-pressing procedures, the FFA contents of oils increased. Preheating moist seeds prior to extraction or pressing may eliminate the enzyme activities to a great extent.

In order to prepare glyceride oils with low FFA values, the Konya black cumin seed oil (with 21.22% acidity) was subjected to deacidification treatment. A laboratory-scale cross-current multistage extraction procedure was applied to this sample, and at the end of this extraction, 70.7 parts of a raffinate with 3.6% free fatty acid content and 29.3 parts of an extract with 68.2% free fatty acid content were obtained, as seen in Table 4.

TABLE 3

Effect of Storage Time on the Free Fatty Acid Contents of the Konya Seed Oils

Storage time (day)	FFA (%)		
	Extracted oil	Pressed oil	Cold-pressed oil
0	21.22	21.64	9.28
5	25.73	22.33	11.46
10	27.99	23.12	13.60
15	29.19	23.23	14.35
20	30.04	24.16	16.77
30	31.71	24.16	17.50
40	33.48	25.26	18.91
53	32.85	25.65	18.64
68	33.90	24.99	19.82

Removal of free fatty acids from the high acidity black cumin seed oil can be performed with a low refining loss, and a prerefined oil, and fatty acids of technical grade can be obtained.

It may be concluded that *Nigella sativa* seed oils are oleic-linoleic type oils (semi-drying oil). Fruitful

TABLE 4

Deacidification of the Konya Seed Oil (with 21.22% acidity) with Aqueous 90% Methanol by Cross-Current Multistage Extraction

Extraction procedure	Amount of raffinate[a] (%)	Amount of total extracts[a] (solvent-free) (%)	FFA content of raffinate (%)	FFA content of total extracts (solvent-free) (%)	Recovery of FFA[b] (%)	Loss of neutral oil[c] (%)
1 stage	88.40	11.60	14.91	69.38	37.93	4.51
2 stage	81.40	18.61	9.41	72.90	63.90	6.40
3 stage	76.03	23.97	5.87	69.92	78.97	9.15
4 stage	70.74	29.26	3.59	68.18	94.03	13.44

[a]Crude oil basis.
[b]Free fatty acid content of crude oil basis.
[c]Neutral oil content of crude oil basis.

utilization of indigenously produced seed oil is expected to be realized.

ACKNOWLEDGMENTS

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