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PRODUCTION OF ESSENTIAL OIL FROM CUMIN SEEDS

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Steam distillation of cumin (Cuminum cyminum *L.*) *seeds was studied to show the effects of particle size, batch size, and distillation rate on their essential oil recovery. Experiments were carried out both on bench and pilot scale. The composition of the oil was analyzed by GC/MS.*

Key Words: *Cuminum cyminum L.*, cumin seed, essential oil, steam distillation, GC/MS.

Cumin (*Cuminum cyminum* L.) is an annual plant of the *Apiaceae* family. Although not a native plant of Turkey, it is cultivated in Cyprus, Lebanon, Morocco, Malta, Turkey, Spain, Russia,and China, and, on a small scale, in Central America [1]. In Turkey it is widely cultivated in the Central Anatolian region where it is used as condiment and as an ingredient of sucuk (a kind of Turkish sausage) [2].

Cumin oil is a pale yellow to brownish yellow liquid; it occasionally displays a greenish tint. The oil is quite sensitive to daylight, air, moisture, and metals as well as to alkali. It is used in perfumes in trace amounts to introduce green-spicy and green-woody top notes, particularly in the woody-floral perfume types. It is also used for "special effects" in modern aldehydic fragrances [2].

Cumin oil is obtained by steam distillation. Distillation is performed right after grinding the seeds in order to prevent oil loss and decomposition. In steam distillation there are certain parameters that greatly influence the recovery of the oil. The main parameters such as particle size, batch size, and distillation rate should be considered before commercial scale production. These parameters can be determined only by experimentation [3].

For bench scale experiments, seeds were ground and sieved through a set of screens of 1.00, 0.710, 0.500, and 0.177 mm openings. The oil yields of water distillation are given in Fig. 1.

Particle size, mm	Oil yield, %
0.177	2.8
0.297	2.6
0.500	1.9
0.710	1.4

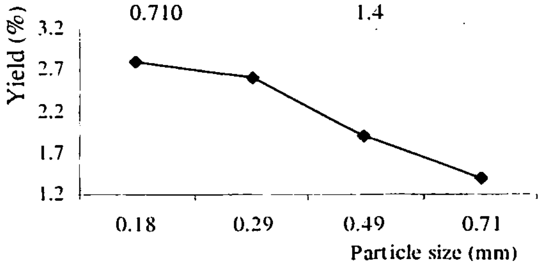

Fig. 1. The effect of particle size on oil yield (bench scale), distillation time: 3 hours.

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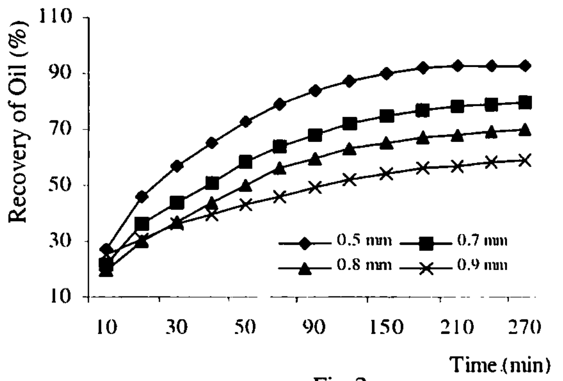

Fig.2

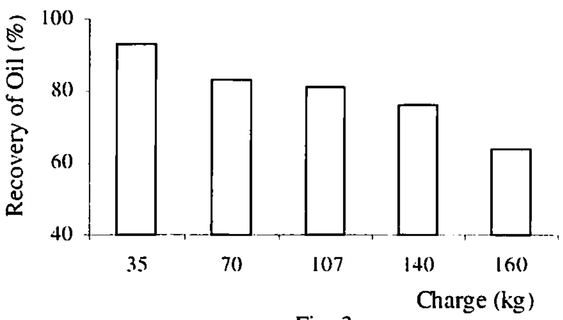

Fig. 3

Fig. 2. The effect of particle size on recovery of oil (pilot scale), distillation time: 3 hours, distillation rate: 1 kg $kg^{-1}h^{-1}$, batch size: 35 kg.

Fig. 3. The effect of batch size on recovery of oil (pilot scale), distillation time: 3 hours, distillation rate: 1 kg $kg^{-1}h^{-1}$, particle size: 0.5 mm (average).

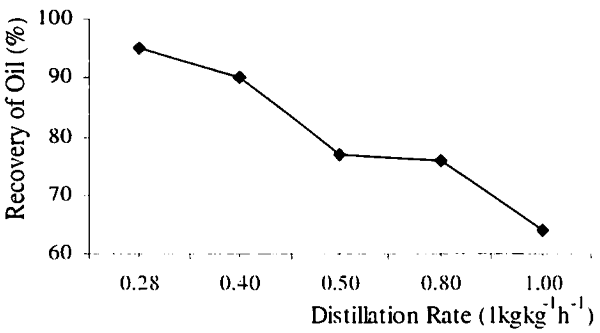

Fig. 4. The effect of steam rate on recovery of oil (pilot scale), distillation time: 3 hours, particle size: 0.5 mm (average), batch size: 160 kg.

Seeds were ground into four different average particle size lots (0.5, 0.7, 0.8, and 0.9 mm) for pilot scale experiments.

Cumin seed oil recovery results of pilot scale experiments performed using different average particle size lots by the steam distillation method are given in Fig. 2.

The effect of batch size on oil recovery was also established in pilot scale experiments by steam distillation (Fig. 3). The effect of distillation rate on oil recovery is shown in Fig. 4.

The essential oil composition of cumin seed obtained by steam distillation was determined by GC/MS analysis (Table 1). As far as the composition of the oil produced is concerned, the major components were cumin aldehyde (27.60%), γ-terpinene (17.25%), *p*-mentha-1,3-dien-7-al (15.18%), β-pinene (10.22%), and *p*-mentha-1,4-dien-7-al (9.48%).

It is well known that diffusion plays a very important part in plant distillation. It is always a slow process, and if the plants or parts of plants were left intact, the rate of recovery of oil would be determined entirely by the rate of diffusion. Consequently, before distillation, the seeds must be thoroughly crushed in order to rupture as many of the cell walls as possible, to render the oil easily accessible to the passing steam. This is clearly seen in Fig. 1 for water distillation on the bench scale and in Fig. 2 for steam distillation on the pilot scale. Reducing the particle size of the cumin seeds from 0.710 to 0.177 mm increased the oil yield from 1.4 to 2.8% for bench scale water distillation. The same particle size effect is observed on pilot scale steam distillation experiments (Fig. 2). Average particle size reduction from 0.9 mm to 0.5 mm increased the oil recovery from 60 to 93%.

TABLE 1. Essential Oil Composition of Cumin Seed Oil

Components	%	Components	%
α-Pinene	0.63	*trans*-Sabinenehydrate	0.09
Camphene	0.01	*cis*-Sabinenehydrate	0.19
β-Pinene	10.22	Linalool	0.04
Sabinene	0.58	*p*-Mentha-3-en-7-al	2.91
Δ_3-Carene	0.03	β-Caryophyllene	0.45
Myrcene	0.83	Terpinen-4-ol	0.13
α-Phellandrene	1.60	(*Z*)-β-Farnesene	0.60
α-Terpinene	0.11	α-Terpineol	0.05
Limonene	0.39	**Cumin aldehyde**	**27.60**
β-Phellandrene+1.8-cineole	0.49	*p*-Mentha-1,3-dien-7-al	15.18
γ-Terpinene	17.25	*p*-Mentha-1,4-dien-7-al	9.48
p-Cymene	5.51	*p*-Mentha-1,3-dien-7-ol	0.31
Terpinolene	0.06	Cumin alcohol	0.36
Total		95.10	

The effect of batch size on oil yield is important especially for comminuted seeds. The height of the packing increases with the still charge and this results in channeling steam through the mass of plant material, thus reducing oil yield because of poor contact between steam and charge. This effect is indicated in Fig. 3, which shows that when the batch size was increased from 35 to 160 kg the oil recovery decreased from 93 to 64%, a 30% decrease.

A partial solution of the steam channeling problem may be possible by reducing the distillation rate. A 40% increase in oil recovery was achieved by reducing the distillation rate from 1 kg distillate kg charge^{-1} hour^{-1} to 0.28 kg distillate kg charge^{-1} hour^{-1} (Fig. 4).

EXPERIMENTAL

In this study, the influence of particle size, batch size, and distillation rate on cumin seed oil recovery were investigated. The seeds were purchased locally, in Eskishehir, Turkey.

For bench scale experiments, ground seeds were subjected to water distillation using a Clevenger type apparatus. The seeds were ground and sieved through a set of screens of 1.000, 0.710, 0.500, 0.297, and 0.177 mm openings. Samples weighing 100 g from each screen were water distilled for 3 hours. Oil yields on a moisture free basis were calculated and plotted against particle size in Fig. 1.

Pilot scale steam distillation experiments were conducted in a 500 L capacity stainless steel distillation unit. Seeds ground into four different average particle size lots (0.5, 0.7, 0.8, and 0.9 mm) were subjected to steam distillation. In parallel with these runs, small samples taken from each size lot were water distilled on the bench scale. Oil recovery calculations were then based on the ratio of the oil volume obtained on the pilot scale over that obtained on the bench scale. Figure 2 shows the oil recovery against time for the indicated average particle sizes. The charge for each case was 35 kg and the distillation rate was taken to be 1 kg distillate kg charge^{-1} hour^{-1}. The batch size effect was investigated with a 0.5 mm average particle size lot. Five different charges of seeds (35, 70, 107, 140, and 160 kg) were distilled with a 1 kg distillate kg charge^{-1}hour^{-1} distillation rate for three hours. The oil recovery are plotted in Fig. 3.

Similarly, the effect of distillation rate was studied with a 160 kg charge of average particle size 0.5 mm. The rate of distillation was changed from 0.28 kg distillate kg charge^{-1} hour^{-1} and for each run the time of distillation was 3 hours. The data thus obtained are shown in Fig. 4.

The composition of the oil obtained from pilot scale experiments was determined by GC-MS techniques (Table 1). GC-MS analysis was carried out with an HP-GCD system. The column was HP-Innowax (60m × 0.25 mm ⌀ with 0.25 mm film thickness). The oven temperature was held at 60°C for 10 min and programmed to 220°C at a rate of 4°C/min, then held at

220°C for 10 min, then programmed to 240°C. Helium was used as a carrier gas with a 1 ml/min constant flow rate. The split ratio was 50:1 and the mass range was 35–425 *m/z*. MS were taken at 70 eV. Identification of individual components was achieved using "The TBAM Library of Essential Oil Constituents" and library search software from "The Wiley/NBS Registry Mass Spectral Data".

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