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Microwave heating and conventional roasting of cumin seeds (*Cuminum cyminum* L.) and effect on chemical composition of volatiles

Sushmita Behera, S. Nagarajan, L. Jagan Mohan Rao *

Plantation Products, Spices and Flavour Technology Department, Central Food Technological Research Institute, Mysore 570 013, India

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Abstract

Microwave processing and cooking of foods is a recent development, which is gaining momentum in household as well as large-scale food applications. Processing of spices using microwaves is a newer dimension. This alternative methodology is preferred, due to the convenience and ease of handling. In Indian tradition, most of the spices are subjected to roasting before addition to food preparations. In the present study, cumin seeds are subjected to heating by microwaves, using various power levels, and conventional roasting at different temperatures. The conditions were standardized in both methods. The volatile oils distilled from these samples were analysed by GC and GC–MS. The results indicated that the microwave-heated samples showed better retention of characteristic flavour compounds, such as aldehydes, than did the conventionally roasted samples. Comparative data on yield, chemical composition, flavour quality and physicochemical parameters of the volatile oils are presented.

Keywords: *Cuminum cyminum*; Umbelliferae; Cumin seeds; Microwave heating and conventional roasting; Volatile compounds; GC–MS; Chemical composition; Flavour quality

1. Introduction

Processing and preservation of spices are important for assuming the quality of the end-product (Charalambous, 1994). Spices are extensively used and their different uses for flavouring and also for bacteriocidal, fungistatic, antifertility, anthelmintic and medicinal applications have been reported (Pruthi, 1980). *Cuminum cyminum* L. is one widely used spice. Crushed cumin seeds are used as a condiment in a variety of dishes. Cumin seeds contain volatile oil (2%–5%) that imparts the characteristic aroma to the seeds. The proximate composition of the seeds indicates that it contains fixed oil (~10%), protein, cellulose, sugar and other mineral elements (Winton & Winton, 1939), and the physicochemical properties of the volatile oil have been reported (Guenther, 1950).

Volatile oil of cumin is employed advantageously, instead of the seeds, in many types of flavouring compounds. The essential oil present in cumin seeds prevents butter from deterioration and improves its acid value. It has an antihydrolytic effect and is better than conventional synthetic antioxidants (The Wealth of India, 2001). The oil is used in perfumery and for flavouring liquors and cordials. It is also used as a carminative. Cumin seeds possess an aromatic odour and have a spicy and bitter taste. They are used as an essential ingredient in mixed soups, sausages, pickles, cheese and meat dishes, and for seasoning breads, cakes and candies.

In indigenous medicine, cumin seeds have long been considered as a stimulant, carminative and are used for therapeutic purposes. They are also used in veterinary medicines (Varo & Heinz, 1970). The chief constituent of the volatile oil is cuminaldehyde (*p*-isopropyl benzaldehyde, *p*-cuminaldehyde). The comparative chemical composition of cumin seed oil produced in different ways, has been reported, and steam distillation results in

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* Corresponding author.
E-mail address: ljnatpro@yahoo.com (L. Jagan Mohan Rao).

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good retention of character impact flavour constituents. Also, the cumin oil, shows fungi-toxic activity, which could be linked to the cuminaldehyde content (Lawrence, 1992). The ovicidal activity of cumin essential oil, against insects, has been reported (Tunc, Berger, Erler, & Dagli, 2000). The composition of Indian cumin oil has been reported. It shows significant flavour components, such as cuminaldehyde 18.3% and perillaldehyde 8.17%, in addition to the terpenic hydrocarbons, whereas Egyptian cumin oil contains 39.2% cuminaldehyde (Srinivas, 1986).

Generally, some spices are processed, for their microbial stability and removal of extraneous matter. Roasting is one of the important phases in the cooking process to release characteristic flavour volatiles (Sushеela, 2000) and undesirable **constituents**. Hence, roasting of spices affects flavour quality. This study aimed to standardize the roasting conditions of cumin seeds. The effect of microwave heating and the retention of volatile oil constituents, as compared to conventional roasting, were studied. Comparative data on yield, composition, flavour quality and physicochemical parameters of the volatile oils are presented.

2. Materials and methods

2.1. Processing of raw material

Seeds of cumin (*Cuminum cyminum*) were procured from commercial sources locally, and were identified and a voucher specimen was deposited at the Manasgangotri herbarium, Botany Department, Mysore University, Mysore, India. Fresh seed samples (100 g) were cleaned and coarse ground (mesh size-5) in a spice mix grinder (Amrut, Mumbai, India). Six samples (60 g each) were roasted in a conventional roaster (Bharat electric roaster, Bangalore, Horizontal drum type, Capacity: 0.5 kg) at different temperature of 100(±2), 125(±2) and 150(±2) °C and different time intervals (5 and 10 min).

Similarly, ten samples (60 g each) were subjected to microwave heating at different wattages of 175, 385, 595, 730 and 800 W in a microwave oven (BPL-Sanyo, India) and different time intervals (10 and 20 min). These seed samples were coarse-ground (mesh-size 5) in a spice mix grinder. The powdered samples were packed in low-density polyethylene (LDPE) air locked pouches and stored at room temperature in the dark.

2.2. Distillation of essential oils

The fresh cumin seed powdered sample (50 g), and distilled water (350 ml) were placed in a round bottom flask and connected to the Clevenger distillation unit and distilled for 4 h. At the end of the specified time, heating was discontinued and, after at least five minutes, the volatile oil was collected in a graduated test tube and the amount of volatile oil was noted. Essential oils were distilled from conventional roasted and microwave heated and powdered samples. Moisture from the oil samples was removed by storing over anhydrous sodium sulphate and preserved in a refrigerator at 4 °C for further analysis.

2.3. Analysis of physicochemical parameters

2.3.1. Optical rotation

Optical rotations were recorded in a 0.5 ml cell of 1cm length using a polarimeter (Perkin Elmer-243, Germany) at 589 nm.

2.3.2. Refractive index

The refractive index was determined using an Abbe refractometer (Atago-3T, Japan).

2.4. Analysis of volatile compounds

2.4.1. GC–MS analyses

The GC–MS analysis of hydro-steam distilled volatile oil, was carried out on a Shimadzu – 17A (Japan) gas chromatograph fitted with a QP-5000 quadrupole mass spectrometer, using a SPB-1 column (30 m × 0.32 mm, 0.25 μm film thickness) for three samples: fresh cumin oil, conventionally roasted (125 °C, for 10 min), and microwave heated (730 W, 10 min). The analytical conditions employed were: carrier gas helium, with flow rate 1 ml/min, injection port temperature 250 °C, detector temperature 250 °C, oven temperature was kept at 50 °C for 2 min initially, and then increased to 250 °C at the rate of 2 °C per minute and maintained at 250 °C for 5 min. Retention indices of all the constituents were determined by the Kovats method using *n*-alkanes as standards (Jennings & Shibamoto, 1980; Davies, 1990). The constituents (Table 3 see below), of the volatile oil from fresh cumin were identified by comparison of their Kovat's indices with those reported in the literature, and their identities were confirmed by computer matching of their mass spectral fragmentation patterns with those of compounds in the NIST-MS Library and with MS Data reported in literature (Stenhagen, Abrahamsson, & Mc Lafferty, 1974; Ten Noever de Bravw, Bouwman, Gramberg, & LaVos, 1988; Adams, 1989).

2.4.2. GC analysis

Samples were prepared by the AOAC (2000, Chap. 43) method. The analytical conditions employed were: SS column [10′ × 0.125″ (o.d.)] packed with 10% SE-30 coated on Chromosorb-W, carrier gas-nitrogen, flow rate 30 ml/min, detector (FID) temperature 250 °C, injection port temperature 250 °C; temperature programme: column starting temperature 50 °C for 2 min, rate of temperature increases 4 °C/min; final temperature 250 °C for 5 min.

3. Results and discussion

Cumin seeds subjected to conventional roasting and microwave heating were analysed, and a comparison was made between these two methods. In the case of fresh sample, volatile oil recovered after 4 h of distillation was 5.6%. But when the samples were roasted conventionally in an electric roaster at different temperature and time intervals, volatile oil yield decreased from 4.4% to 1.8% with increase in temperatures and time, as shown in Table 1. The samples subjected to microwave heating at different watts from 175 to 800W, and time intervals showed that the yield decreased from 4.4% to 2.0% (Table 2). When comparison was made with the fresh sample, loss of essential oils observed in both cases (microwave- heated and conventional-roasted) were found to be same.

The volatile oil yields and physicochemical parameters of conventionally and microwave heated samples are shown in Tables 1 and 2. Optical rotation of fresh sample was found to be +2.86° and refractive index was found to be 1.5007. Optical rotations at 20 °C, were found to be in the range of +0.545° to +2.860° for conventional and +1.538° to +3.615° for microwave-treated samples, respectively. The difference between these values indicates change in chemical composition. Refractive indices for all these were found to be in the range of 1.493–1.500. The range of the refractive index values indicates, that the components were neither degraded nor polymerized and remained as mono- or sesquiterpenoids and their derivatives, as identified.

The optimum condition, in the case of conventionally roasted sample, was found to be 125 °C for 10 min and, in the case of microwave-heated sample, the best condition was found to be 730 W for 10 min. The samples processed under these conditions had the better aroma, which is shown by the aldehyde/hydrocarbon ratio (Table 5). The samples gave comparable yields of volatile oils. Samples roasted at lower temperatures, wattages and for lesser times, were found to possess raw aroma. On the other hand, the samples roasted at higher temperatures, and higher wattages, using the microwave oven for longer periods, were charred and the aroma deteriorated considerably.

The GC–MS analysis of volatile oil showed the presence of 24 constituents (Table 3). All these components were identified by Kovats index values and MS Spectra (Jennings & Shibamoto, 1980; Davies, 1990; Stenhagen et al., 1974; Ten Noever de Bravw et al., 1988; Adams, 1989). Seven are major, and constitute

Table 1
The volatile oil yields obtained by conventional roasting and their physicochemical parameters

Roasting temperature (°C)	Time (min)	Yield[a] (%)	Optical rotation $[\alpha]_D^{20}$	Refractive index (30 °C)
Fresh	–	5.6(±0.05)	+2.860	1.4958
100	5	4.4(±0.10)	+1.830	1.5052
100	10	3.8(±0.05)	+1.260	1.4953
125	5	3.6(±0.03)	+0.545	1.4956
125	10	3.0(±0.05)	+2.090	1.5000
150	5	2.6(±0.08)	+2.270	1.4950
150	10	1.8(±0.03)	+2.519	1.4930

The values in the brackets are standard deviations.

[a] All analyses done in triplicate and the mean values are given.

Table 2
The volatile oil yields obtained from the microwave heated samples and physicochemical parameters of the volatile oil

Watt	Time (min)	Yield[a] (%)	Optical rotation $[\alpha]_D^{20}$	Refractive index (30 °C)
Fresh	–	5.6(±0.05)	+2.860	1.4958
175	10	4.4(±0.03)	+1.538	1.4958
175	20	4.0(±0.05)	+1.565	1.4903
385	10	4.0(±0.08)	+1.994	1.5002
385	20	3.6(±0.05)	+3.615	1.4804
595	10	3.6(±0.03)	+2.355	1.4504
595	20	2.8(±0.10)	+2.395	1.4950
730	10	2.8(±0.08)	+2.284	1.4908
730	20	2.4(±0.10)	+2.552	1.4966
800	10	2.4(±0.08)	+1.775	1.4900
800	20	2.0(±0.10)	+2.355	1.4906

The values in the brackets are standard deviations.

[a] All analyses done in triplicate and the mean values are given.

Table 3
Chemical constituents of essential oil of cumin using GC–MS

Sl. No.	RT	KI	COMPOUND	Identification by
1	5.67	927	α-pinene	RI, MS, CoI
2	6.88	960	sabinene	RI, MS
3	7.10	966	β-pinene	RI, MS, CoI
4	7.92	985	β-myrcene	RI, MS
5	9.13	1012	*p*-cymene	RI, MS, CoI
6	11.03	1051	γ-terpinene	RI, MS, CoI
7	14.82	1117	fenchol	RI, MS
8	16.30	1143	β-pinene oxide	RI, MS
9	16.98	1154	terpinene-4-ol	RI, MS, CoI
10	17.55	1163	*trans*-verbenol	RI, MS
11	18.20	1173	myrtenal	RI, MS
12	21.10	1218	cuminaldehyde	RI, MS, CoI
13	22.88	1248	*p*-mentha-1,3-dien-7-al	RI, MS
14	24.12	1267	*p*-mentha-1,4-dien-7-al	RI, MS
15	24.67	1275	cumin alcohol	RI, MS
16	24.93	1279	perilla alcohol	RI, MS
17	30.87	1373	α-cadinene	RI, MS
18	32.23	1393	*cis*-β-farnesene	RI, MS
19	35.13	1441	germacrene	RI, MS
20	35.35	1445	*trans*-β-farnescene	RI, MS
21	35.55	1448	α-zingiberene	RI, MS
22	38.20	1490	β-bisabolene	RI, MS
23	41.11	1540	sesquiphellandrene	RI, MS
24	41.30	1543	*cis*-nerolidol	RI, MS

RI – Retention Indices as determined on SPB-1 using the homologous series of *n*-alkanes.

MS – Mass spectrum.

CoI – Co-injection.

KI – Kovats indices.

Table 4
Comparison of chemical compositions of essential oils of fresh and optimally processed cumin seeds (μl/100 g)

Sl. No.	Compound	Fresh	Conventional 125 °C (10 min)	Microwave 730 W (10 min)
1	α-pinene	18.7	3.94	2.58
2	β-pinene	544	286	8.05
3	β-myrcene	44.6	18.9	174
4	*p*-cymene	943	992	845
5	γ-terpinene	1565	425	115
6	terpinene-4-ol	12.4	21.7	83.2
7	*trans*-verbenol	4.28	–	45.1
8	Myrtenal	6.88	6.81	–
9	Cuminaldehyde	1038	819	920
10	*p*-mentha-1,3-dien-7-al	316	100	349
11	*p*-mentha-1,4-dien-7-al	1034	297	2.24
12	*cis*-farnesene	5.99	–	2.18

upto 95% of the total volatile oil and the rest are minor constituents. Comparisons of chemical compositions and the quantitative data of the character impact compounds from the essential oils of fresh and optimally processed cumin seeds by conventional roasting and microwave heating are given in Table 4.

Table 5
Comparison of terpene hydrocarbons and aldehydes from the essential oils of fresh and optimally processed (conventional and microwave) cumin seeds using GC analysis

Compounds	Fresh sample (%)	Conventional roasting (125 °C, 10 min) (%)	Microwave heating (730 W, 10 min) (%)
Monoterpenes	56.4	58.1	45.0
Sesquiterpenes	0.108	–	0.085
Aldehydes	43.2	41.0	50.0
Alcohols	0.3	0.73	7.68
Ratio of aldehyde/hydrocarbons	0.765	0.705	1.11

Comparisons of terpene hydrocarbons and aldehydes from the essential oils of fresh and optimally processed (conventional and microwave) cumin seeds, using GC analysis, are presented in Table 5.

GC analysis has confirmed the presence of major constituents; terpene hydrocarbons (α-pinene, β-pinene, sabinene, *p*-cymene, terpinene), aldehydes (cuminaldehyde, *p*-mentha-1,3-dien-7-al, *p*-mentha-1,4-dien-7-al) and alcohols (cumin alcohol, perilla alcohol, *trans*-verbenol, fenchol). Aldehydes contribute to the characteristic flavour of cumin seed (Boelens, 1991).

In the microwave-heated sample, the terpene hydrocarbons (mono- and sesquiterpenes) accounted for 45% of the constituents. Aldehydes that contribute to the characteristic flavour of seed spice accounted for 50% of the oil whereas, in the conventional-roasted sample, the aldehydes were about 41% and hydrocarbons 58%. This is well illustrated in the aldehyde to hydrocarbon ratio in which the microwave-heated sample was 1.11, whereas the fresh and conventional-roasted samples were 0.765 and 0.705, respectively, which indicated more retention of the flavour impact compounds in microwave heating than conventional roasting.

4. Conclusions

Conventionally roasted and microwave heated samples were compared; the optimum condition in the former method was found to be 125 °C for 10 min and in the latter method, the best condition was found to be 730 W for 10 min. Under these conditions, the yields of the volatile oils were similar in both cases. Physicochemical properties, such as refractive index, for both sample oils, showed no significant difference from the fresh sample. Changes were observed in the optical rotation values, which indicated differences in the chemical compositions. GC and GC–MS analysis of optimized condition samples showed that microwave heated samples showed better retention of characteristic flavour compounds of cumin (i.e., total aldehydes), than did conventionally-roasted samples. Earlier GC reports showed the presence of only cuminaldehyde as the major aldehyde present in Indian cumin oil. But the present studies resulted in identification of two more aldehydes (*p*-mentha-1,3-dien-7-al, *p*-mentha-1,4-dien-7-al) in Indian cumin oil. Thus, the microwave treatment, in spite of losing terpene hydrocarbons, retained aldehydes in the volatile oil, making microwaves the best choice as an alternative-heating medium for processing.

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