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Yield, Quality and Composition of Cumin Essential Oil as Affected by Storage Period

Abdlazim Mohamed Ali*, Hana Jumma Ali Jumma

Faculty of Agriculture, Nile Valley University, Atbara, Sudan
Email: *azimali58@yahoo.com

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Abstract

Essential oils from newly produced (2017) and previous season crop (2016) of cumin *(Cuminum cyminum* L.) seeds collected from local farmers in Dongla area, north Sudan were extracted and analyzed to determine the effect of storage period on volatile oil yield and quality. The essential oil was extracted using hydro distillation method. Identification of the volatile compounds was performed using gas chromatography-mass spectrometry (GC/MS). Results revealed that oil obtained from cumin *(Cuminum cyminum* L.) was pale yellow. With respect to oil characteristics, the specific gravity, refractive index, acid value, ester value and other quality characters were not significantly different between the stored and newly harvested seeds. Thirty-eight compounds were separated and identified in cumin seeds essential oil in both seasons with some minor differences in some components between 2016 and 2017 samples. Major effective components of cumin seeds volatile oil were cumin alcohol (4-isopropylbenzyl alcohol), cumin aldehydes as 4-Isopropylbenzaldehyd and 7,7-dimethylbicyclo (4.1.0) hept-3-ene-4-carbaldehyde known as carenal, terpinenes as *γ*-terpinene and ***α***-terpinene (1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl) and 1,3-cyclohexadine-1-methanol, 4(1-methethyle)-), *β*-pinene, *o*-cymene and *p*-menthadienol in both stored and newly harvested samples. Differences between the samples season wise were very minor with respect to type of volatile components but percentage oil yield was largely decreased by storage period.

Keywords

Cumin, Essential Oil, Major Compounds

1. Introduction

Cumin (*Cuminum cyminum* L.), belonging to the family Umbelliferae, is believed to be a native from the East Mediterranean to East India. It is a herbaceous and medicinal crop and one of the oldest and popular seed spices worldwide after black pepper. It is known under various names in different countries [1] [2]. Cumin seed is generally used as a spice for foods in the form of powder for flavoring different food preparations [3]. It also has a variety of medicinal properties [4].

In Sudan, cumin spread along the Nile valley and some other parts where it continues to be sown by smallholders in the winter season to provide flavoring material. The cultivation of the crop is widely spreading especially in the Northern parts of Sudan, but very few or no attention was given by researchers to the plant [5].

Essential oils are the volatile fragrant components from various plants with significant trade value, nationally and internationally, for several uses [6]. All true essential oils are volatile aromatic secondary metabolites of plant origin. Their occurrences differ from one plant to other according to type of plant part containing oil [7]. Cumin seeds contain 2.5% - 4.0% volatile oil and aldehydes or cuminol, which attributes to the aroma, and are considered as the major oil constituents [1].

Very few works were devoted by Sudanese researchers to improve crop productivity and processing, therefore, the general and the main objective of this study is extraction and characterization of essential oil from cumin (*Cuminum cyminum* L.) seeds with specific objectives of:

– Extraction of the volatile oil of cumin (*Cuminum cyminum* L.) by hydro distillation.
– Identification of volatile components of oils using gas chromatography-mass spectrometry (GC/MS).
– Investigation of the effect of one season storage of cumin (*Cuminum cyminum* L.) schizocarp fruits (traditionally called seeds) on their volatile oil qualities.

2. Material and Methods

2.1. Plant Material and Essential Oil Extraction

Two samples of Cumin (*Cuminum cyminum* L.) seeds, newly harvested and other stored over season, were fetched from local farmers in Dongla area, north Sudan (production seasons 2016 and 2017). One hundred grams of old and newly harvested cumin seeds stored under shade at room temperature were hydro distilled and prepared for further detection of chemical and physical properties, including color, odor, specific gravity, refractive index, acid value, ester number, peroxide value and degeneration degree. SAS computer program was used to statistically analyze results as completely randomized design using duplicate readings.

For Gas Chromatography-Mass Spectrometry (GC-MS) five hundred grams of an old and newly harvested dried cumin seeds were distilled for further detection of chemical constituents. The extraction of the essential oil of cumin is achieved using hydro-distillation process, after seed crushing, with hydro distillation apparatus according to the European Pharmacopoeia [8]. Oil extraction continued for 3 h (until no more essential oil was obtained). The essential oil was collected, dried under anhydrous sodium sulphate and stored at 0˚C for further use.

2.2. Oil Analysis Procedure

The composition of the essential oil was analyzed using gas chromatography-mass spectroscopy technique. GC-MS instrument used was model QP2010-Ultra, Shimadzu Company, Japans, with serial number 020525101565SA equipped with capillary column (Rtx-5 ms-30 m × 0.25 mm × 0.25 μm) and flame ionization detector (FID). The sample was injected using split mode (Split ratio: 1:100), helium as carrier gas passed with flow rate 1.69 ml per minute, the temperature program was started from 50˚C with rate 7˚C per minute to 180˚C then the rate was changed to 10˚C per minute reaching 300˚C as final temperature degree, with 3 min hold time. The injection port temperature was 300˚C, the ion source temperature was 200˚C and the interface temperature was 250˚C. The sample was analyzed using scan mode in the range of m/z 40 - 550 charges to ratio and the total run time was 28 min. Identification of components for the sample was achieved comparing their retention times and mass fragmentation results automatically by instrument software with those available in the libraries of the National Institute of Standards and Technology (NIST), US National Library of Medicine (PUBCHEM), The National Center for Biotechnology Information (NCIB) and Royal Society of Chemistry (*Chem Spider*).

3. Results and Discussion

Oil obtained from cumin (*Cuminum cyminum* L.) was pale yellow. Hydro distillation of cumin seeds produced a percentage yield of cumin essential oil of 2.45% and 4% (v/w) of volatile oils, sample 2016 and sample 2017 respectively. This significant reduction in essential oil yield in stored sample was in line with that obtained by [9] in coriander stored oil. Flavor of essential oil of cumin seeds was slightly bitter, strongly aromatic with some strong dislikable odors. The essential oil characteristics detected according to standard procedures showed no significant differences in characteristics between samples of 2016 and 2017 seeds. The specific gravity ranged between 0.8772 in 2016 to 0.8767 in 2017 sample. Refractive index ranged between 1.505 in 2016 to 1.5035 in 2017 sample. Acid value ranged 1.1222 in 2016 to 2.435 in 2017 sample and ester value 56.39055 in 2016 to 74.76658 in 2017 sample (**Table 1**).

Identification of cumin seeds volatile oil components extracted by hydro distillation and analyzed using GC/MS are shown in **Table 2** and **Table 3**. From the results, 38 compounds were separated and identified in cumin seeds in both newly harvested and stored sample (**Figure 1** and **Figure 2**) compared with 49

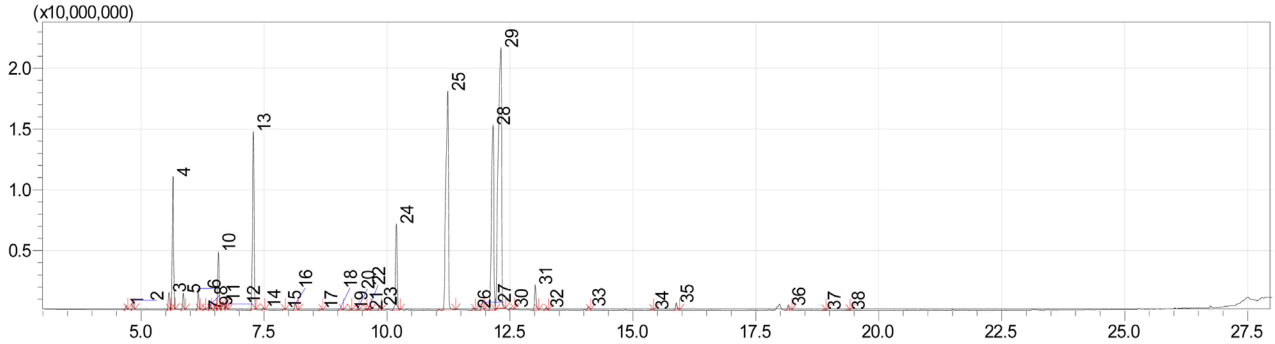


Figure 1. GC/MS chromatogram (2016 sample).

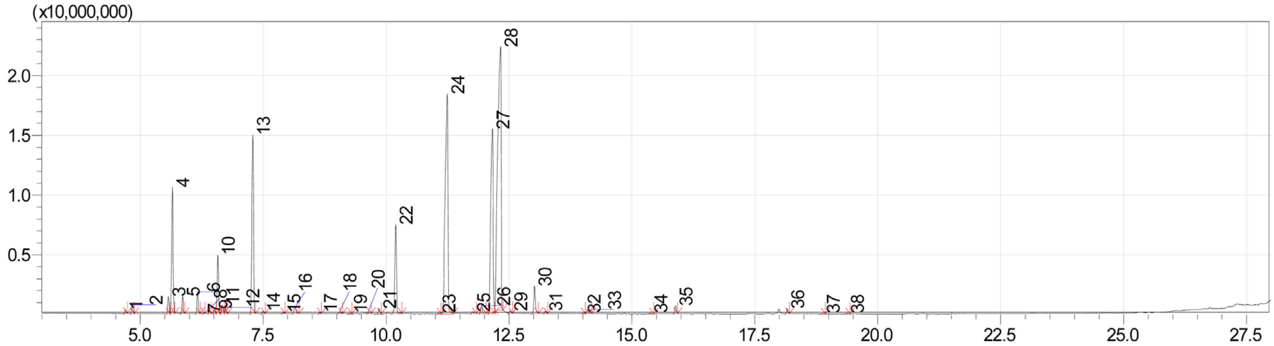


Figure 2. GC-MS chromatogram (2017 sample).

Table 1. Physical and chemical characteristics of essential oil of *Cuminum cyminum* L.

No.	Physical and chemical Properties	Sample 2016	Average 2016	Sample 2017	Average 2017
1	Color	Pale yellow		Pale yellow	
2	Odor	Strong and somewhat disagreeable		Strong and somewhat disagreeable	
3	Yield	2.45 - 2.55	2.5%	4 - 4	4%
4	Specific Gravity	0.8674 - 0.8870	0.8772	0.8614 - 0.892	0.8767
5	Refractive Index	1.505 - 1.505	1.505	1.505 - 1.502	1.5035
6	Acid Value (as mg KOH/g oil)	1.40275 - 0.84165	1.1222	1.222 - 3.64715	2.434575
7	Ester number (as mg KOH/g oil)	54.70725 - 58.07385	56.39055	77.4318 - 72.10135	74.76658
8	Peroxide value (as meq.O_2/Kg oil)	0.025 - 0.045	0.035	0.05 - 0.015	0.0325
9	Degeneration degree (as mg KOH/g oil)	56.11 - 58.92	57.515	78.554 - 75.7485	77.15125

Physicochemical properties	R-Square	P value	Sig. (0.05)
Specific gravity	0.950445	0.2226	Not Significant
Refractive index	0.000000		NS
Degree of acidity	0.517928	0.5635	NS
Peroxide value	0.076336	0.9423	NS
De generation degree	0.979964	0.0904	NS
Ester number	0.947114	0.1479	NS

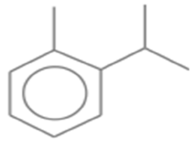
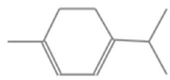
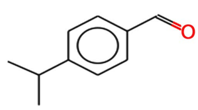
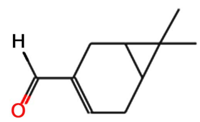
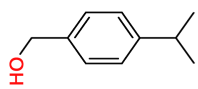
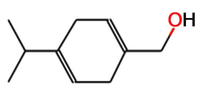
Table 2. The results of the analysis of the GC/MS spectrum of the different constituents of the essential oil extracted from cumin (2017) by hydrodistillation.

Peak	Retention time (min)	Area %	Compound Name
1	4.695	**0.19**	*α*-Phyllanderene
2	4.834	**0.42**	*α*-Pinene
3	5.565	**0.77**	Bicyclo(3.1.0)hexane,4-ethylene-1-(1-m
4	5.647	**6.80**	Bicyclo(3.1.1)heptane6-6 dimethyl 1-2 methylene (***β*-Pinene**)
5	5.855	**0.72**	*β*-myrecene
6	6.162	**0.97**	*β*-Phyllanderene
7	6.282	**0.03**	*β*-ocimene
8	6.404	**0.22**	(+)-2-careen
9	6.509	**0.21**	Cyclohexane-1-methyle-4(1-methleethyle)-
10	6.571	**2.59**	Benzene, 1-methyl-2-(1-ethylethyl)-) **(*O*-Cymene)**
11	6.658	**0.57**	D-limonene
12	6.728	**0.24**	Eucalyptal
13	7.282	**10.17**	1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl)-**(*γ*-terpinene)**
14	7.485	**0.11**	Bicyclo(3-1-0)hexane-2-ol,2methyl1-5 (1-me
15	7.903	**0.05**	Cyclohexane-1-methyle-4(1-methleethyle)-
16	8.131	**0.45**	1-6-octadien-3-ol,3-7dimethyl 1-
17	8.645	**0.06**	2 Cyclohexane-1-methyle-4(1-methleethyle)-
18	9.041	**0.15**	*P*-Menth-8-en-1-ol steroisomer
19	9.256	**0.03**	Cyclo hexane, 3 butyl
20	9.397	**0.03**	2-Decyn-1-ol
21	9.565	**0.04**	3-cyclohexane-1-carboxaldehyde,134-tri
22	9.621	**0.05**	2,6-dimethyle-357-octatriene-2-ol,E,E
23	9.854	**0.23**	Terpinene-4-ol
24	10.191	**4.73**	1,3-cyclohexadine-1-methanol,4(1-methethyle)-**(*α*-Terpinene)**
25	11.238	**20.60**	4-Isopropylbenzaldehyd **(cumin aldehyde)**
26	11.757	**0.13**	Santolinatriene
27	11.937	**0.40**	1-cyclohexane-1-carboxaldehyde, 4(1-meth)
28	12.155	**15.28**	7,7-dimethylbicyclo (4.1.0) hept-3-ene-4-carbaldehyde **(3-caren-10-al)**
29	12.318	**31.03**	4-isopropylbenzyl alcohol **(cuminol)**
30	12.519	**0.16**	2,3,5-trimythlanizol
31	13.015	**1.47**	1,4-cyclohexadine-1-methanol,4(1-methylethyle)-**(*p*-Mentha-1,4-dien-7-ol)**
32	13.237	**0.13**	Bicyclo(311)hepta-2-ene-2-carboxaldehyde
33	14.097	**0.21**	2-Propionic acid3-phenyl-,methyl ester
34	15.374	**0.05**	(E)-beta Famesene
35	15.884	**0.37**	Spiro(45)dec-7-ene,1,8-dimethyl1-4(1-methylethyle)-
36	18.159	**0.26**	Carotol
37	18.875	**0.04**	Gama-Muurolene
38	19.367	**0.04**	Cyclo hexane methanol,4-ethenyl-alpha

Table 3. The results of the analysis of the GC_MS spectrum of the different constituents of the essential oil extracted from cumin (2016) by hydrodistillation.

Peak	Retention time (min)	Area %	Compound Name
1	4.699	**0.18**	*α*-Phyllanderene
2	4.838	**0.38**	*α*-Pinene
3	5.570	**0.75**	Bicyclo(3.1.0)hexane,4-ethylene-1-(1-m
4	5.653	**6.56**	Bicyclo(3.1.1)heptane6-6 dimethyl 1-2 methylene (***β*-Pinene**)
5	5.861	**0.71**	*β*-myrecene
6	6.167	**0.95**	*β*-Phyllanderene
7	6.288	**0.03**	*β*-ocimene
8	6.410	**0.22**	(+)-2-careen
9	6.513	**0.20**	Cyclohexane-1-methyle-4(1-methleethyle)-
10	6.577	**2.64**	Benzene, 1-methyl-2-(1-ethylethyl)-) (***O*-Cymene)**
11	6.664	**0.54**	D-limonene
12	6.734	**0.26**	Eucalyptal
13	7.290	**9.92**	1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl)-(***γ*-terpinene)**
14	7.492	**0.12**	Bicyclo(3-1-0)hexane-2-ol,2methyl1-(1-methylethyl)-
15	7.908	**0.05**	Cyclohexane-1-methyle-4(1-methleethyle)-
16	8.140	**0.48**	1-6-octadien-3-ol,3-7 dimethyl 1-
17	8.650	**0.06**	2 Cyclohexane-1-methyle-4(1-methleethyle)-
18	9.046	**0.14**	*P*-Menth-8-en-1-ol steroisomer
19	9.261	**0.04**	Cyclo hexane, 3 butyl
20	9.626	**0.06**	2,6-dimethyle-357-octatriene-2-ol,E,E
21	9.857	**0.24**	Terpinene-4-ol
22	10.196	**4.88**	1,3-cyclohexadine-1-methanol,4(1-methethyle)-**(*α*-Terpinene)**
23	11.063	**0.06**	Phenol,3-(1-methyl ethyl)
24	11.244	**20.66**	4-Isopropylbenzaldehyd **(cumin aldehyde)**
25	11.763	**0.14**	Santolinatriene
26	11.941	**0.46**	1-cyclohexane-1-carboxaldehyde, 4(1-methyethyle)-
27	12.162	**15.41**	7,7-dimethylbicyclo (4.1.0) hept-3-ene-4-carbaldehyde**(3-caren-10-al)**
28	12.328	**30.99**	4-isopropylbenzyl alcohol **(cuminol)**
29	12.522	**0.13**	2,3,5-trimythlanizol
30	13.018	**1.56**	1,4-cyclohexadine-1-methanol,4(1-methylethyle)-**(*p*-Mentha-1,4-dien-7-ol)**
31	13.240	**0.14**	Bicyclo(311)hepta-2-ene-2-carboxaldehyde
32	14.017	**0.03**	Naphthalene, 124a, 568a-hexahydro-47
33	14.100	**0.24**	2-Propionic acid3-phenyl-,methyl ester
34	15.377	**0.05**	(E)-beta Famesene
35	15.886	**0.39**	Spiro(45)dec-7-ene,1,8-dimethyl1-4-(1-methylethyle)_
36	18.160	**0.25**	Carotol
37	18.877	**0.04**	Gama-Muurolene
38	19.370	**0.04**	Cyclo hexane methanol,4-ethenyl-alpha

Table 4. Major effective compounds of Sudanese cumin essential oil, their molecular weight and their chemical structure.

	Compound	Formula	Molecular weight	Chemical structure	Liberary
1	Bicyclo(3.1.1)heptane6-6 dimethyl 1-2 methylene (***β*-Pinene**)	$C_{10}H_{16}$	136		NIST PUBCHEM ChemSpider
2	Benzene, 1-methyl-2-(1-ethylethyl)-) (***O*-Cymene**)	$C_{10}H_{14}$	134		NIST
3	1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl)-) ***γ*-terpinene**	$C_{10}H_{16}$	136		NIST
4	1,3-cyclohexadine-1-methanol,4(1-methethyle)-(***α*-terpinene**)	$C_{10}H_{16}$	136		NIST PUBCHEM ChemSpider
5	4-Isopropylbenzaldehyd (**cumin aldehyde**)	$C_{10}H_{12}O$	148		PUBCHEM ChemSpider
6	7,7-dimethylbicyclo (4.1.0) hept-3-ene-4-carbaldehyde **(3-caren-10-al)**	$C_{10}H_{14}O$	150		PUBCHEM
7	4-isopropylbenzyl alcohol (**cuminol**)	$C_{10}H_{14}O$	150		PUBCHEM ChemSpider
8	1,4-cyclohexadine-1-methanol,4(1-methylethyle)- **(p-Mentha-1,4-dien-7-ol)**	$C_{10}H_{16}O$	152		PUBCHEM

compounds isolated by [10]. There were some differences in some components between 2016 and 2017 samples (compounds No.20 and 21 **2-Decyn-1-ol** and **3-cyclohexane-1-carboxaldehyde, 134-tri** in Table 2 and No. 23 and 32 **Phenol, 3-(1-methyl ethyl)-and Naphthalene, 124a, 568a-hexahydro-47** in Table 3 each of them were absent in the other table). Major effective components of cumin seeds volatile oil were; 4-isopropylbenzyl alcohol known as **cuminal** (31.03%), 4-Isopropylbenzaldehyd known as **cumin aldehyde** (20.60%), 7,7-dimethylbicyclo (4.1.0) hept-3-ene-4-carbaldehyde known as **3-caren-10-al** (15.28%), 1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl)-known as ***γ*-terpinene** (10.17%), Bicyclo (3.1.1) heptanes 6-6 dimethyl 1-2 methylene known as ***β*-pinene**, (6.80%) 1,3-cyclohexadine-1-methanol, 4(1-methethyle)-known as ***α*-terpinene** (4.73%), Benzene, 1-methyl-2-(1-methylethyl)-known as ***o*-cymene** (2.59%) and 1,4-cyclohexadine-1-methanol, 4(1-methylethyle)-known as ***p*-mentha-1,4-dien-7-ol** (1.47%) obtained from 2017 sample. However, in 2016 major effective components were, 4-isopropylbenzyl alcohol known as **cuminal** (30.99%) 4-Isopropylbenzaldehyd known as **cumin aldehyde** (20.66%), 7,7-dimethylbicyclo (4.1.0) hept-3-ene-4-carbaldehyde known as **3-caren-10-al**, (15.41%), 1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl)-known as ***γ*-terpinene** (9.92%), Bicyclo (3.1.1) heptane 6-6 dimethyl 1-2 methylene known as ***β*-pinene** (6.56%), 1,3-cyclohexadine-1-methanol, 4(1-methethyle)-known as ***α*-terpinene** (4.88%), Benzene, 1-methyl-2-(1-methylethyl)-known as ***o*-cymene** (2.64.), and 1,4-cyclohexadine-1-methanol, 4(1-methylethyle)-known as ***p*-mentha-1,4-dien-7-ol** (1.56%). Results obtained were comparable to that obtained by [11]. Major compounds and their molecular weight, formula and chemical structure were presented in **Table 4**. Most cited literature refer to the fact that cuminaldehyde considered the major compound followed by alpha or gamma terpinene [12] [13] [14]. Many researchers stated that the composition and characteristics of herb essential oil differ according to many factors among them cultivar, growing condition, harvesting method and time, storage condition and finally oil extraction technique [15] [16] [17] [18]. Differences between the sample season wise were very minor with respect to percentage and type of volatile components.

4. Conclusion

Storage period reduced oil yield but oil characteristics including chemical, physical and constituent properties remain with negligible change.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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