

Research Article

Comparative GC Analysis of Seed Essential Oils from Tunisian and German Caraway (*Carum carvi* L.) Ecotypes

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ABSTRACT: **Background:** Caraway (*Carum carvi* L.) seeds have been used numerously throughout history as a condiment and for its medicinal properties. **Objective:** The present study aims to compare the Tunisian and German caraway ecotypes regarding their seed essential oil compositions. **Materials and Methods:** Seed essential oil composition of two caraway ecotypes from Tunisia and Germany, cultivated under the same pedoclimatic and cultural conditions has been analysed by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). **Results:** The essential oil yields differed significantly between the two caraway ecotypes: 1.41% and 0.48% for Tunisian and German seeds, respectively. Forty one (41) volatile compounds were identified in the two oil samples where carvone and limonene constituted the main components but with significantly different proportions. Consequently, the two caraway ecotypes displayed the same chemotype, namely carvone. Additionally, the proportions of some minor essential oil compounds such as ketones, oxygenated monoterpenes and sesquiterpenes were found to be significantly different between the German caraway seed essential oil and the Tunisian one. **Conclusion:** Since the influence of different environmental factors has been eliminated, the observed differences in seed essential oil yield and composition between the two studied ecotypes seem likely to result from the genetic variability.

KEYWORDS: *Carum carvi* L., seeds, essential oil, carvone, limonene

INTRODUCTION

Nowadays, essential oils are gaining increasing interest and are thus widely used in pharmaceutical, cosmetic, agricultural and food industries. Investigations on the evaluation of essential oils biological activities of some aromatic and medicinal plant species have revealed that some of them exhibited interesting properties such as antibacterial,^[1] antifungal,^[2] antiparasitic^[3] and insecticidal.^[4] Essential oils are volatile mixtures of organic compounds, usually extracted from various aromatic

and medicinal plants generally localized in temperate to warm countries like Mediterranean and tropical regions where they represent an important part of the traditional pharmacopoeia.^[3]

An example of an aromatic plant is caraway (*Carum carvi* L.), a member of the Apiaceae family. It is an annual or biennial herb with white flowers and small green to yellow seeds, which it's mainly cultivated in the Netherlands, Finland, Hungary, Morocco, Iran, India and Russia.^[5] Caraway seeds have been used numerously throughout history as a condiment and in traditional medicine for its diuretic,^[6] hypoglycaemic^[7] and hypocholesterol^[8] properties. Moreover, caraway was proved to be a class of potential chemopreventive agents^[9] since its anticarcinogenic, antiinflammatory, and antiproliferative ability is correlated with enhanced repair or remodeling of precursor lesions.^[10]

Previously, we showed that Tunisian caraway essential oil is characterized by the prevalence of carvone as the main

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volatile component^[11] In the present study, we compared for the first time the Tunisian and German caraway ecotypes regarding their seed essential oil compositions. The results will be important to indicate the effect of geographic origin on the essential oils profiles of caraway seeds

MATERIALS AND METHODS

Plant material and growth conditions

Two caraway (*C. carvi* L.) seed ecotypes of cultivated origin were used in this study. The first (Tunisian caraway) was collected from a field in the region of Souassi (South of Tunisia; latitude 35° 34' 12" N; longitude 10° 16' 6" E; altitude 41 m) whilst the other (German caraway) was imported from Germany (Munich, latitude 48° 08' N; longitude 11° 34' E; altitude 519 m). The two ecotypes were cultivated under the same pedoclimatic and cultural conditions at the National Agronomic Institute of Tunisia (36° 55' N, 10° 11' E, 10 m above sea level). The experiment was carried out in a greenhouse at day light (photoperiod varying from 13 to 16h) and at a temperature varying from 18 to 20°C during the day and from 10 to 12°C during the night. The experimental design was the complete random blocks with three replications. Each ecotype sown area was of 15 m² (10 m × 1.5 m). Seeds were sown on November 28, 2005 with row spacing of 0.40 m and a density of 125 plants m⁻². Fertilization consisted of 250, 200 and 100 kg ha⁻¹ of P₂O₅, K₂O and N, respectively, incorporated uniformly to the soil before sowing, and supplemented by 100 kg ha⁻¹ of N brought twice during the crop cycle. Pre-irrigation was done immediately after sowing for uniform emergence and establishment of seedlings. Irrigation was done by submersion one to twice frequencies per week. In addition, weeds were controlled by hand when needed. Harvest was on May 27, 2006. Harvested seeds were air-dried and stored at 4 °C until further analysis.

Essential oil extraction

Whole air-dried seeds (50 g) were subjected to hydrodistillation for 90 min (time fixed after a kinetic survey for 30, 60, 90 and 120 min). The hydrodistillation was performed by a simple laboratory Quik-fit apparatus which consisted of a 1,000 ml steam generator flask, a distillation flask, a condenser and a receiving vessel. Essential oils were extracted from the distillate using diethyl-ether as solvent (v/v) dried over anhydrous sodium sulphate then concentrated at +35°C using a Vigreux column and stored at -20°C prior to analysis. All experiments were done in triplicates and results were expressed on the basis of dry matter weight (DMW).

Chromatographic analysis

Gas chromatography (GC-FID)

Gas chromatography analyses were carried out on a Hewlett-Packard 6890 gas chromatograph (Agilent Palo Alto, CA, USA) equipped with a flame ionization detector (FID) and an electronic pressure control (EPC) injector. A polar HP Innowax (PEG) column and an apolar HP-5 one (30 m × 0.25 mm, 0.25 µm film thickness) were used. The oven temperature was held at 35°C for 10 min then programmed at 3°C min⁻¹ to 205 °C and finally isotherm during 10 min. The injector and detector temperatures were programmed, respectively, at 250 and 300°C. The flow of the carrier gas (N₂) was 1.6 ml min⁻¹ and the split ratio was 60:1. Injection volume for all samples was 1 µL.

Gas Chromatography-Mass Spectrometry (GC-MS)

The GC-MS analyses were performed on a gas chromatograph HP 6890 (II) interfaced with a HP 5973 mass spectrometer (Agilent Technologies, Palo Alto, California, USA) with electron impact ionization (70 eV). A HP-5MS capillary column (60 m × 0.25 mm, 0.25 µm film thickness) was used. The column temperature was programmed to rise from 40 to 280°C at a rate of 5°C min⁻¹. The carrier gas was helium with a flow rate of 1.2 ml min⁻¹. The scan time and mass range were 1 s and 50–550 m/z, respectively.

Compounds identification

The volatile components were identified by comparison of their retention index (RI) relative to (C₇–C₂₀) *n*-alkanes with those of literature and/or with those of authentic compounds available in our laboratory, and by matching their recorded mass spectra with corresponding data (Wiley 275. L library) and other published mass spectra.^[12] Relative percentage amounts of the identified compounds were obtained from the electronic integration of the FID peak areas without the use of correction factor.

Statistical analysis

Data were subjected to statistical analysis using the program package STATISTICA^[13] and expressed as mean ± standard deviation (SD). One-way analysis of variance (ANOVA) followed by Duncan's multiple range test at the significance level of 5 % was used to compare means.

RESULTS AND DISCUSSION

Essential oil yield

The essential oil yields (based on dry matter weight) obtained from Tunisian and German caraway seeds are given in Table 1. Interestingly, the Tunisian ecotype displayed higher

Table 1: Essential oil yield and composition (%) of Tunisian and German caraway (*Carum carvi* L.) seed ecotypes.

Essential oil yield (% on the basis of dry matter weight)				Tunisian caraway	German caraway
				1.41 ± 0.05	0.48 ± 0.12
Volatile compound*	RI ^a	RI ^b	Identification ^c	% Composition ^d	
α-Pinene	1032	934	RI, MS	0.09 ± 0.06 ^b	0.16 ± 0.01 ^a
Camphene	1086	951	RI, Co-GC	0.03 ± 0.00 ^b	0.28 ± 0.08 ^a
β-Pinene	1123	980	RI, Co-GC	0.03 ± 0.00 ^b	0.25 ± 0.08 ^a
β-Myrcene	1166	991	RI, MS	0.16 ± 0.03 ^a	0.11 ± 0.01 ^a
Limonene	1206	1030	RI, MS	19.52 ± 3.31 ^a	16.15 ± 0.95 ^b
γ-Terpinene	1255	1062	RI, MS, Co-GC	0.04 ± 0.01 ^b	0.16 ± 0.05 ^a
(<i>E</i>)-β-Ocimene	1266	1050	RI, MS, Co-GC	0.08 ± 0.06 ^b	0.18 ± 0.05 ^a
<i>p</i> -Cymene	1280	1026	RI, MS, Co-GC	0.03 ± 0.00 ^a	0.07 ± 0.01 ^a
Terpinolene	1290	1092	RI, MS, Co-GC	0.01 ± 0.00 ^b	0.12 ± 0.03 ^a
Z-3-Hexenol	1370	855	RI, MS, Co-GC	0.01 ± 0.00 ^b	0.13 ± 0.03 ^a
<i>trans</i> -Limonene oxide	1463	1136	RI, MS	0.01 ± 0.00 ^b	0.11 ± 0.01 ^a
Cuminaldehyde	1785	1238	RI, MS, Co-GC	0.03 ± 0.00 ^a	0.01 ± 0.01 ^b
Perilla-aldehyde	1789	1272	RI, MS, Co-GC	0.09 ± 0.00 ^b	0.19 ± 0.00 ^a
<i>Trans</i> -Dihydrocarvone	1627	1204	RI, MS	0.15 ± 0.02 ^b	0.29 ± 0.07 ^a
<i>cis</i> -Dihydrocarvone	1645	1197	RI, MS	0.08 ± 0.02 ^b	0.25 ± 0.02 ^a
Carvone	1740	1241	RI, MS	76.37 ± 4.73 ^a	77.35 ± 0.17 ^a
Camphor	1532	1143	GC/MS, Co-GC	0.01 ± 0.00 ^b	0.06 ± 0.01 ^a
Linalool	1545	1100	GC/MS, Co-GC	0.02 ± 0.00 ^b	0.07 ± 0.01 ^a
Terpinene-4-ol	1611	1178	RI, MS, Co-GC	0.06 ± 0.01 ^b	0.10 ± 0.06 ^a
α-Terpineol	1700	1189	RI, MS, Co-GC	0.06 ± 0.01 ^a	0.07 ± 0.04 ^a
Dihydrocarveol	1720	1253	RI, MS	0.14 ± 0.02 ^a	0.08 ± 0.01 ^b
Citronellol	1766	1229	RI, MS, Co-GC	0.09 ± 0.06 ^a	0.08 ± 0.01 ^a
Nerol	1797	1228	GC/MS, Co-GC	0.02 ± 0.00 ^a	0.05 ± 0.01 ^a
<i>trans</i> -carveol	1841	1218	RI, MS	0.05 ± 0.01 ^b	0.51 ± 0.08 ^a
<i>cis</i> -carveol	1869	1230	RI, MS	0.05 ± 0.01 ^b	0.08 ± 0.01 ^a
Perilla-alcohol	2001	1296	RI, MS, Co-GC	0.03 ± 0.01 ^a	tr
β-Elemene	1600	1594	RI, MS, Co-GC	0.03 ± 0.00 ^b	0.27 ± 0.08 ^a
β-Caryophyllene	1612	1419	RI, MS, Co-GC	0.06 ± 0.01 ^b	0.15 ± 0.05 ^a
Allo-aromadendrene	1661	1474	RI, MS, Co-GC	0.04 ± 0.01 ^b	0.17 ± 0.02 ^a
Germacrene-D	1719	1480	RI, MS, Co-GC	tr	0.01 ± 0.01 ^a
β-Selinene	1742	1481	RI, MS, Co-GC	0.41 ± 0.41 ^a	0.10 ± 0.10 ^b
α-Selinene	1745	1485	RI, MS, Co-GC	0.82 ± 0.82 ^a	0.14 ± 0.04 ^b
α-Farnesene	1755	1508	RI, MS, Co-GC	0.69 ± 0.02 ^a	0.26 ± 0.03 ^b
δ-Cadinene	1772	1517	RI, MS, Co-GC	0.37 ± 0.06 ^b	0.77 ± 0.00 ^a
γ-Cadinene	1776	1511	RI, MS, Co-GC	0.16 ± 0.00 ^b	0.37 ± 0.02 ^a
Spathulenol	2125	1575	RI, MS, Co-GC	0.04 ± 0.01 ^b	0.65 ± 0.60 ^a
Eugenol	2192	1356	RI, MS, Co-GC	0.02 ± 0.01 ^a	0.01 ± 0.01 ^a
Thymol	2198	1290	RI, MS, Co-GC	0.04 ± 0.01 ^a	0.03 ± 0.03 ^a
Carvacrol	2215	1296	RI, MS, Co-GC	0.02 ± 0.00 ^b	0.06 ± 0.02 ^a
Linalyl acetate	1556	1257	RI, MS, Co-GC	0.04 ± 0.01 ^b	0.08 ± 0.01 ^a
Nonadecane	1900	1900	RI, MS, Co-GC	0.02 ± 0.00 ^a	0.02 ± 0.02 ^a

*Components are listed in order of elution in apolar column (HP-5); RI^a, RI^b: Retention indices calculated using respectively an apolar column (HP-5) and polar column (HP-Innowax); ^c: RI: retention indices relative to (C₇-C₂₀) *n*-alkanes on the HP-Innowax, MS = mass spectrum, Co-GC = co-injection with authentic compound; ^d: The percentage composition was calculated from the chromatograms obtained on the HP-Innowax column; tr: trace; values within raw followed by the same letter are not significantly different at *P* < 5% (Duncan test).

essential oil yield (1.41%) than the German one (0.48%). Generally, variation in essential oil yield can be attributed to genetic factors, maturity stage and environmental factors as well as ontogenic factors and analytical methods.^[14] In the present work, the two seed essential oil yields were obtained from caraway ecotypes cultivated under the same pedoclimatic and cultural conditions. In addition, they were isolated and analysed under the same operating

conditions. So, the influence of the environmental and technical parameters was considered negligible and the main source of variance could be related to genotype.^[15, 16] Thus, the difference observed between these two seed essential oil yields was closely related to the geographical origin of the ecotype. Indeed, the Tunisian ecotype originated from the Mediterranean region whereas the German one is native temperate seasonal climate.

Essential oil composition

The GC-MS analysis of the essential oils obtained from the two caraway ecotypes is presented in Table 1. A total of 41 volatile compounds were identified. Carvone was the major component of caraway seed essential oil (76.37 and 77.35% in Tunisian and German caraway ecotypes, respectively) followed by limonene (19.52 and 16.15% in Tunisian and German caraway ecotypes, respectively). Comparison of our results with those of international literature confirms the prevalence of carvone and limonene.^[17-19] In addition, carvone and limonene proportions significantly differed from those reported in previous papers and ranged from 50 to 60% and from 25 to 35%, respectively.^[20, 21]

Furthermore, it is important to note that the lower proportion of carvone in German ecotype was accompanied by a lower limonene proportion. This is predictable, since limonene is both an intermediate in the biosynthesis of carvone as well as an end product.^[22] In contrast to our findings, it is worth mentioning that other different major compounds were reported by Razzaghi-Abyneh et al.^[23] and Jalali-Heravi et al.^[24] who found that the major constituent of caraway seed essential oil were simultaneously cuminaldehyde (22.08%) and γ -terpinene (24.40%), respectively.

Other minor monoterpene compounds (i.e. α -pinene, camphene, β -pinene, γ -terpinene and terpinolene) were identified in the two caraway essential oils. However, their proportions were significantly higher in the German caraway seeds in comparison to the Tunisian ones. The same tendency was also observed for aldehydes with Z-3-hexenol, *trans*-limonene oxide and perilla-aldehyde being present in lower percentages that did not pass 0.1% in Tunisian caraway seeds.

Additionally, the proportions of some other ketones and oxygenated monoterpene compounds (i.e. *trans*-dihydrocarvone, *cis*-dihydrocarvone and *trans*-carveol) were found to be significantly higher in the German caraway seeds in comparison to the Tunisian ones. On the other hand, Tunisian caraway seeds contained more sesquiterpene hydrocarbon components mainly represented by β -selinene (0.41%), α -selinene (0.82%) and α -farnesene (0.69%), than the German ones. However, the German seeds presented significantly higher amounts of β -elemene (0.27%), β -caryophyllene (0.15%), allo-aromadendrene (0.17%), δ -cadinene (0.77%) and γ -cadinene (0.37%) in comparison to the Tunisian caraway seeds. Besides, spathulenol (0.65%) was the most abundant oxygenated sesquiterpene component in the German caraway seeds.

In addition, germacrene-D and perilla alcohol were present in trace amounts in both Tunisian and German caraway seed ecotypes. Consequently, it seems that caraway seed essential oil of the two ecotypes preserves the same qualitative composition with the prevalence of carvone. Although the same main compounds were present in the two ecotypes, but there were some differences in their percentages. So, discrimination is possible by comparison of their proportions in the two ecotypes. This variation in the main component proportions was closely related to genetic background since the two ecotypes were cultivated, harvested and processed under the same conditions. Accordingly, this finding agrees with literature data which attributed the variation in essential oil composition to genetic factors.^[16, 25, 26]

Overall, these results indicate that the essential oils from the two caraway ecotypes displayed the same chemotype, namely carvone which appears origin independent. Hence, our findings agree with literature data.^[17, 20, 27] So, caraway seed essential oil constitutes a valuable source of carvone which has various applications, as fragrance and flavour, potato sprouting inhibitor, antimicrobial agent and also in the medical field.^[21]

Chemical class characterization of seed essential oil

As shown in Table 2, the chemical class characterization of caraway seed essential oils from both ecotypes showed the prevalence of ketones (76.60 and 77.89% in Tunisian and German caraway ecotypes, respectively) exclusively represented by carvone. The monoterpene hydrocarbons formed the second main class with respectively 19.99 and 17.48% in Tunisian and German caraway seed ecotypes. The remaining fractions, such as oxygenated monoterpenes, aldehydes, sesquiterpene hydrocarbons, oxygenated sesquiterpenes and esters, formed the minor essential oil chemical classes of caraway seeds in both ecotypes. Our results are similar to those found in previous study dealing with the essential oil composition of three Tunisian caraway seed ecotypes.^[11] However, German caraway seed essential oil presented more aldehydes (0.44%), oxygenated monoterpenes (1.09%) and oxygenated sesquiterpenes (0.76%) than the Tunisian one. On the other hand, the two ecotypes were not significantly different for the proportion of sesquiterpene hydrocarbons (2.57 and 2.24% in Tunisian and German caraway ecotypes, respectively).

It is worth mentioning the therapeutic properties of the two main chemical classes of caraway seed essential

Table 2: Proportions (%) of essential oil chemical classes of Tunisian and German caraway (*Carum carvi* L.) seed ecotypes

Chemical class	Tunisian caraway	German caraway
Monoterpene hydrocarbons	19.99 ± 2.49 ^a	17.48 ± 1.33 ^b
Aldehydes	0.14 ± 0.04 ^b	0.44 ± 0.07 ^a
Ketones	76.60 ± 4.02 ^a	77.89 ± 4.50 ^a
Oxygenated monoterpenes	0.51 ± 0.04 ^b	1.09 ± 0.14 ^a
Sesquiterpene hydrocarbons	2.57 ± 0.31 ^a	2.24 ± 0.22 ^a
Oxygenated sesquiterpenes	0.13 ± 0.05 ^b	0.76 ± 0.31 ^a
Esters	0.04 ± 0.02 ^b	0.08 ± 0.01 ^a
Others	0.02 ± 0.01 ^a	0.02 ± 0.03 ^a

Values within row followed by the same letter are not significantly different at $P < 5\%$ (Duncan test).

oil which are ketones and monoterpene hydrocarbons. However, the presence of synergistic functions of the various molecules contained in the essential oil, in comparison to the action of one or two main components of the oil, seems questionable. In fact, it is possible that the activity of the main components is modulated by other minor molecules. In that sense, for biological purposes, it is more informative to study entire oil rather than some of its components because the concept of synergism appears to be more meaningful.^[3]

CONCLUSION

The essential oil yields of two caraway ecotypes from Tunisia and Germany and their composition were investigated. The Tunisian ecotype presented the highest yield with 1.41% compared with the German one. The two ecotypes were characterized by the same predominant compounds: carvone and limonene but with significant different proportions. The remaining chemical classes were weakly represented. Moreover, the two ecotypes displayed the same chemotype, namely carvone. The caraway seeds were also rich in essential oil, especially the Tunisian ecotype. Since the influence of different environmental factors has been eliminated, the observed differences in seed essential oil yield and chemical composition between the two studied ecotypes seem likely to result from the genetic variability.

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