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Variability in the Content of Essential Oils and Selected Mycotoxins in Spices of the Apiaceae Family

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ABSTRACT

Essential oils (EOs) obtained from spices, herbs and medicinal plants are well known in traditional medicine and are an area of interest due to their various biological activities. On the other hand, spices are consumed on a daily basis and have the potential to be contaminated with mycotoxins. Therefore, it is crucial to consider them as a factor in health safety. The present study investigated the yield, EOs composition and mycotoxins (T-2 toxin and HT-2 toxin) content in five spices (caraway, anise, coriander, fennel, dill) of the *Apiaceae* family. EOs obtained by hydrodistillation were analysed and identified by gas chromatography coupled to mass spectrometry (GC–MS). Mycotoxins were determined using liquid chromatography coupled to a mass spectrometer (LC–MS). The yields of EOs ranged between 0.66% and 5.26% depending on the type of spice. The results showed that the major components present in caraway samples were carvone (53.02%–59.65%) and limonene (39.34%–46.08%); in anise trans-anethole (93.79%–95.70%); in coriander linalool (62.02%–67.91%); in fennel trans-anethole (0.82%–92.87%) or estragole (2.53%–89.51%); and in dill samples limonene and carvone (about 47.00% each), or dill apiole (42.08%) and limonene (29.80%). T-2 and HT-2 toxins were detected in small amounts (0.37–2.69 μg/kg) in three of 24 analysed samples.

1 | Introduction

The *Apiaceae* (also known as *Umbelliferae*) family is one of the largest plant families in the world and includes over 420 genera and more than 3500 plant species. *Apiaceae* are widespread nearly worldwide, with most genera occurring in Asia (almost 300), followed by Europe and Africa (around 120 both). Members of this family have been known for thousands of years as vegetable, culinary and medicinal plants used as a dietary supplement but also in cosmetology [1–4].

Most of the herbs and spices of this family are aromatic plants rich in essential oils (EOs) [5]. EOs are volatile, low molecular weight mixtures of different bioactive components, mostly obtained by hydrodistillation [6]. They have been used for their biological properties such as antibacterial, antifungal, antimutagenic,

Abbreviation: EOs essential oils.

antiviral, antioxidant, antidiabetic, anti-inflammatory and insecticidal in different applications: pharmaceutical, cosmetic, agronomic and food industries [3, 4, 7–9]. EOs are natural mixtures containing approximately 20–60 different constituents in varying concentrations. The properties of the EOs are often determined by two or three main compounds that are present in relatively high concentrations (20%–70%) compared to other components that are present in trace levels. The chemical composition of EOs is influenced by many factors, for example, plant age, part and phenological stages; extraction techniques; soil character; geographical origin, harvesting season and the environment [4].

Currently, numerous worldwide public health authorities have turned their attention to the safety of botanical substances, particularly phytochemicals, as a result of the recent rapid expansion of the global market for herb-based products. Consumers typically believe that plant-derived substances are safe and have no side effects [10].

A common food contaminant that causes serious health problems (due to its hepato-toxic, mutagenic, immunosuppressive, nephrotoxic and carcinogenic effects) in humans and animals is mycotoxins—secondary metabolites produced by different fungi genera, mainly the *Aspergillus, Fusarium* and *Penicillium* genera. Spices are prone to mycotoxin contamination; the rate of production and the levels of its contamination heavily depend on several environmental factors (mainly temperature and humidity) and can arise in the different stages (pre- and post-harvest, during storage and processing). Mycotoxins are generally stable and resistant compounds. Fungi belonging to the *Fusarium* genus produce a group of mycotoxins called trichothecenes, which represent frequent contaminants of numerous agricultural commodities (such as wheat, barley, maize, rice and cereal grains). According to their functional groups, trichothecenes are categorised into four groups (type A–D), with types A and B being the most prevalent [11, 12].

One of the most frequent trichothecenes is the T-2 toxin, which is a member of the type A trichothecenes [13]. T-2 and HT-2 toxins are very similar in structure, differing only at the C-4 functional group. T-2 toxin is very easily metabolised to HT-2 toxin, often resulting in the co-occurrence of the two toxins. Particularly in cold temperature regions or under moist storage conditions, several *Fusarium* species that can grow on a range of seeds frequently produce T-2 and HT-2 toxins [13–15]. Environmental factors like temperature, humidity and substrate type can have a big impact on T-2 toxin production in addition to fungal genetics [16].

From 13 mycotoxins regulated in foods and beverages in Europe, only aflatoxins and ochratoxin A in selected spices (*Capsicum* spp., *Piper* spp., *Myristica fragrans*, *Zingiber officinale*, *Curcuma longa* and mixtures of spices containing one or more of these spices) are regulated (EC 915/2023). Furthermore, the European Food Safety Authority (EFSA) established a tolerable daily intake (TDI) value of 0.02 μg/kg body weight per day for the sum of T-2 and HT-2 toxins [17].

The aim of the present study was to analyse the EOs composition and T-2 and HT-2 mycotoxins content of five spices (caraway, anise, coriander, fennel and dill) of the *Apiaceae* family available on the local market. EOs were obtained by hydrodistillation of selected spices, and their yields were calculated. The chemical composition of EOs was determined by gas chromatography coupled to mass spectrometry (GC–MS), and the content of mycotoxins T-2 and HT-2 toxins was determined by liquid chromatography with mass detection (LC–MS). The obtained data were also statistically evaluated.

2 | Materials and Methods

2.1 | Chemicals

Solvents' purity LC–MS grade such as methanol, acetonitrile, 2-propanol, acetone, dimethylsulfoxide and GC–MS grade n-hexane, as well as standards of mycotoxins T-2 toxin, HT-2 toxin, n-alkanes and terpenes: Terpene Mix A, sabinene, myrcene, α-terpineol, β-caryophyllene, *p*-cymene, linalool, terpinolene, myrtenyl acetate, geranyl acetate, terpinene-4-ol, phellandrene, borneol, estragole, anethole were purchased from Merck (Merck KGaA, Darmstadt, Germany). Additives for LC–MS (purity LC–MS) such as ammonium acetate, formic acid, acetic acid and citric acid were also purchased from Merck (Merck KGaA, Darmstadt, Germany). Sodium chloride was obtained from Lach-ner (Neratovice, Czech Republic). Ultrapure water was produced by Aqua Osmotic 06 (Tišnov, Czech Republic), and the quality of deionised water was characterised by a conductivity of 0.06 μS/cm.

2.2 | Plant Material

Five different samples of plants from the *Apiaceae* family, *Carum carvi* (caraway), *Pimpinella anisum* (anise), *Coriandrum sativum* (coriander), *Foeniculum vulgare* (fennel) and *Anethum graveolens* (dill) were obtained in 2022 from four local companies focused on the production of spices (the letters G, S, B and V in the labelling of samples belong to each company). The origin of individual samples was verified by Helena Pluháčková. Prior to analysis, whole samples of achenes were ground on a laboratory grinder AR 1105 (Moulinex, France). Determination of dry matter in the monitored samples was performed according to the method of Horáčková et al. [18]. The photos of the achenes used for analysis are shown in Figure S1.

2.3 | Extraction of EOs

The ground achenes (10 g per sample; particle size approximately 0.5–1.0 mm) were subjected to hydrodistillation using a standard Clevenger-type apparatus in accordance with the method described in the European Pharmacopoeia (10th edition, 2019). Each sample was placed in a 500 mL round-bottom flask with 200 mL of distilled water. Distillation was performed at a constant boiling rate for 3 h using a heating mantle as the heat source. Upon completion, the volume of the separated essential oil was measured directly in the graduated collection tube. The oils were subsequently collected into amber glass vials, tightly sealed and stored at 4°C in the dark until further analysis.

2.4 | Analysis of EOs by GC–MS

The GC–MS analyses of EOs were carried out using a Thermo Trace GC Ultra equipped with a TriPlus autosampler, coupled with an ion-trap Polaris Q mass spectrometer (EI mode at 70 eV; Thermo Fisher Scientific Inc., Waltham, USA). The chromatographic separation was performed on the DB-5 capillary column (30 m × 0.25 mm i.d., film thickness 0.25 μm; Agilent J&W Scientific, Folsom, USA); the following temperature programme was used: 45°C held for 2 min, then increased to 250°C at a rate of 10°C/min, then increased to 300°C at a rate of 30°C/min and finally held at 300°C for 2 min. The carrier gas was helium, with a flow rate of 0.8 mL/min. The sample (1 μL) dissolved in n-hexane was injected according to a splitless mode. The mass scan range (*m/z*) was 50–650 amu, and data were acquired at full scan mode with solvent delay for 4.7 min. Both the injector and the transfer line temperatures were set at 225°C and the ion source at 220°C. The data were analysed using Xcalibur 2.2 (Thermo Fisher Scientific Inc., Waltham, MA, USA), with identification of the individual components performed by comparison with co-injected pure standards and by matching the MS fragmentation patterns and retention indices with the built-in libraries or literature data [19] or commercial mass spectral libraries (NIST MS Search 2.0 library; National Institute of Standards and Technology, Gaithersburg, USA). The relative amounts (%) of compounds were calculated based on the GC peak area.

2.5 | Analysis of T-2 Toxin and HT-2 Toxin by LC–MS

The LC–MS analysis of selected mycotoxins was performed according to the method set by Martiník et al. [15] after immunoaffinity columns EASI-EXTRACT T-2 and HT-2 (R-Biopharm AG, Darmstadt, Germany) clean up adapted from the work of Boško et al. [20]. Briefly, 25 g of sample was weighed with 5 g of NaCl and added to 125 mL 90% methanol/water (v/v) and shook for 50 min on an orbital shaker. After centrifugation at 4800 rpm for 10 min, 20 mL of extract was diluted with 80 mL 2% sodium chloride and left on the bench for 5 min with occasional gentle shaking of the solution to allow precipitation to occur. The supernatant was filtered through Whatman No. 4 filter paper. The 25 mL aliquot of diluted filtrate was passed through the immunoaffinity column EASI-EXTRACT T-2 and HT-2, at a gravitational flow rate. The column was washed by passing 20 mL of water through at a flow rate of approximately 5 mL/min, then air was passed air through the column to remove residual liquid. The toxins were eluted using 2 mL 100% methanol. This eluate was evaporated to dryness under a vacuum in a rotary evaporator and the residues were dissolved in 1 mL of 10% methanol/water (v/v) for UPLC–MS/MS analysis according to Martiník et al. [15].

In the present study, matrix effects for T-2 and HT-2 toxin analysis in spice samples were not evaluated in detail. This decision was based on the use of immunoaffinity columns Easy-Extract T2 and HT-2 (R-Biopharm, Germany), which provide a highly selective sample cleanup specifically designed for trichothecene toxins. The application of this immunoaffinity-based extraction method significantly reduces co-extracted matrix components and potential ion suppression or enhancement effects during LC–MS/MS analysis. The limit of detection (LOD) and limit of quantification (LOQ) were determined by successive dilution of both analytes until the signal disappeared and calculated as the concentration at which the signal-to-noise ratio equals to 3 and 10, respectively.

2.6 | Statistical Analysis

Statistical analysis was carried out using Microsoft Office Excel 2019 (Redmond, WA, USA) and software STATISTICA.CZ version 14 (StatSoft, Prague, Czech Republic).

First, normality tests (Kolmogorov–Smirnov and Shapiro–Wilk) were performed, which showed that the data did not have a normal distribution. Therefore, the non-parametric Kendall rank correlation coefficient was used to determine the correlation, which is more appropriate for non-normally distributed data. It was calculated for all pairs of variables, and its statistical significance was evaluated by the appropriate test of the independence hypothesis.

3 | Results and Discussion

Investigating the chemical composition of commonly used therapeutic herbs is important for the elimination of health-threatening substances in the herb material.

3.1 | The Yield of EOs

The yield of EOs was calculated as a volume of oil collected after hydrodistillation by the weight of the dry plant sample expressed in percentages (%, *v/w*). An average EOs yield of three separate hydrodistillations of individual samples is shown in Figure 1. The EOs of all samples yielded from 0.66% to 5.26% (*v/w*). These results are in good agreement with previously reported data [21–24]. Statistically significant differences were found among *Foeniculum vulgare* samples (0.93%–5.26%, *v/w*) as well as between the two samples of *Anethum graveolens* (1.90% and 3.23%, *v/w*).

3.2 | Chemical Composition of EOs Evaluated by GC–MS

3.2.1 | *Carum carvi* (Caraway)

Eighteen compounds were identified in caraway EOs. As shown in Table 1, carvone (53.02%–59.65%) and limonene (39.34%–46.08%) were the most represented in all samples. The remaining components were found in negligible amounts (< 0.20% each). A correlation analysis was performed between the observed components of essential oils (Figure 2). The results obtained show a strong positive statistical dependence between the essential oil component carvone and α-pinene at the 99% confidence level. Similarly, a strong positive statistical correlation was found between the components *trans*-carveol and *cis*-limonene oxide, *cis*-limonene oxide and α-pinene, perilla aldehyde and *trans*-dihydrocarvone. A strong negative statistical correlation was

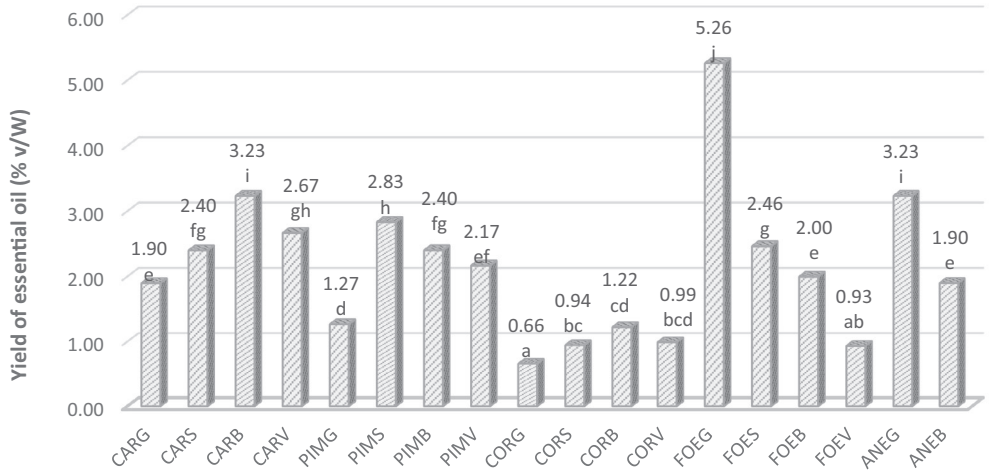

FIGURE 1 | The EOs yield (%, *v*/*w*) obtained by hydrodistillation of the selected *Apiaceae* spices. ANE, dill; CAR, caraway; COR, coriander; FOE, fennel; PIM, anise. The letters G, S, B and V belong to individual local producers of spices. Different letters in a row are statistically different.

TABLE 1 | Chemical composition of essential oils (% ± SD) in achenes of *Carum carvi* (caraway) determined by GC–MS.

No.	RT	Compound	RI calc	RI lit	CARG, % ± SD	CARS, % ± SD	CARB, % ± SD	CARV, % ± SD
1	7.49	α-Pinene	937	932	0.01 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.02 ± 0.00
2	8.18	Sabinene	976	969	0.08 ± 0.01	0.13 ± 0.02	0.13 ± 0.01	0.09 ± 0.01
3	8.40	Myrcene	989	988	0.13 ± 0.01	0.15 ± 0.01	0.15 ± 0.01	0.14 ± 0.01
4	9.15	**Limonene**	1034	1024	**43.16 ± 0.25**	**39.34 ± 0.23**	**44.16 ± 0.26**	**46.08 ± 0.27**
5	9.38	*trans*-Ocimene	1048	1044	0.04 ± 0.01	0.07 ± 0.02	0.04 ± 0.01	0.02 ± 0.00
6	9.62	γ-Terpinene	1062	1054	0.01 ± 0.00	0.01 ± 0.00	0.02 ± 0.01	0.02 ± 0.00
7	10.67	*trans*-p-Mentha-2,8-dien-1-ol	1127	1119	0.10 ± 0.01	0.07 ± 0.01	0.06 ± 0.01	0.05 ± 0.01
8	10.84	*cis*-Limonene oxide	1138	1132	0.05 ± 0.01	0.02 ± 0.01	0.02 ± 0.00	0.02 ± 0.00
9	10.91	*trans*-Limonene oxide	1142	1137	0.13 ± 0.02	0.05 ± 0.00	0.06 ± 0.01	0.05 ± 0.00
10	11.82	α-Terpineol	1201	1186	0.04 ± 0.00	0.07 ± 0.01	0.03 ± 0.00	0.04 ± 0.00
11	11.88	*cis*-Dihydrocarvone	1205	1191	0.04 ± 0.00	0.01 ± 0.00	0.04 ± 0.00	0.05 ± 0.01
12	11.98	*trans*-Dihydrocarvone	1212	1200	0.05 ± 0.01	0.03 ± 0.00	0.04 ± 0.01	0.03 ± 0.00
13	12.16	*trans*-Carveol	1225	1215	0.18 ± 0.01	0.10 ± 0.01	0.09 ± 0.01	0.08 ± 0.01
14	12.35	*cis*-Carveol	1238	1226	0.10 ± 0.01	0.07 ± 0.01	0.07 ± 0.00	0.05 ± 0.01
15	12.55	**Carvone**	1251	1239	**55.41 ± 0.24**	**59.65 ± 0.24**	**54.76 ± 0.24**	**53.02 ± 0.29**
16	13.04	Perilla aldehyde	1284	1269	0.13 ± 0.01	0.07 ± 0.01	0.10 ± 0.00	0.10 ± 0.01
17	13.14	*trans*-Anethole	1291	1282	0.18 ± 0.01	0.04 ± 0.00	0.13 ± 0.01	0.06 ± 0.01
18	15.09	β-Caryophyllene	1435	1417	0.16 ± 0.01	0.11 ± 0.01	0.08 ± 0.00	0.07 ± 0.01

Note: The letters G, S, B and V belong to individual local producers of spices. Results are reported in % ± SD (standard deviation) as the mean of three experiments. Significance of bold value indicates. The dominant components of the essential oil are highlighted.
Abbreviations: CAR, caraway; No., peak number; RI calc, retention index calculated with respect to the homologous series of n-alkanes (C9-C23) on a DB-5 column; RI lit, retention index from literature—entered from Robert P. Adam's library; RT, retention time.

found between the essential oil component α-terpineol and *trans*-anethole. The GC–MS chromatogram of the representative sample is shown in Figure S2.

The obtained data are in good accordance with previously reported studies. Khalil et al. [22] reported carvone and limonene as the main identified compounds in caraway

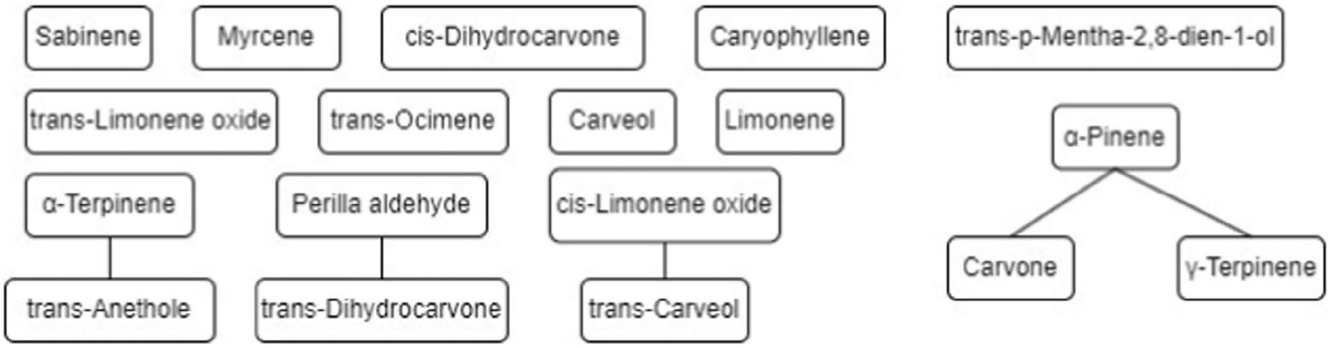

FIGURE 2 | Correlations between the chemical composition of essential oils in fruits of *Carum carvi* (caraway), ($r = 0.7$).

oil (50.60% and 46.48%, respectively). Similar results were also reported by Šťastník et al. [25], 54.50% for carvone and 45.50% for limonene. Mahboubi [26] compared 20 commercial caraway samples from different countries. The content of carvone and limonene varied from 44.50% to 95.90% and from 1.50% to 51.30%, respectively. Kim et al. [27] identified in caraway seeds from Egypt carvone (41.00%), limonene (27.00%), *cis*-carveol (0.50%) and *trans*-carveol (0.40%). Raal et al. [24] identified 18 compounds in 20 commercial caraway samples. The main components of analysed essential oils were carvone (44.50%–95.90%) and limonene (1.50%–51.30%), while the minor compounds were identified as β-myrcene, *trans*-dihydrocarvone, *trans*-carveol, α-pinene, sabinene, *n*-octanal, *trans*-β-ocimene, δ-terpinene, linalool, *cis*- and *trans*-limonene oxide, *cis*-dihydrocarvone, *cis*-carveol, perillaldehyde, *trans*-anethole and *trans*-β-caryophyllene.

3.2.2 | *Pimpinella anisum* (Anise)

A total of 32 compounds were identified in anise EOs of all analysed samples. *Trans*-anethole (93.79%–95.70%) was the predominant component followed by γ-himachalene (2.22%–2.59%) and estragole (0.37%–1.09%) in all samples (Table 2). Statistical evaluation revealed a strong positive statistical correlation at the 99% confidence level between the essential oil components *p*-cymene, linalool, estragole and α-pinene, as well as between linalool and *p*-cymene, germacrene D and limonene, estragole and linalool, *trans*-anethole and sabinene, germacrene D, and *trans*-methyl isoeugenol. The component γ-terpinene showed a statistically strong positive correlation with the component β-pinene. The component sabinene had a statistically strong positive correlation with germacrene D and *trans*-methyl isoeugenol, which also showed a strong positive correlation with germacrene D. The component α-zingiberene had a statistically strong positive correlation with the components *cis*-verbenol and δ-elemene. A strong positive statistical correlation was also observed between α-longipinene and ethyl *trans*-cinnamate, *trans*-methyl isoeugenol; ethyl *trans*-cinnamate and spathulenol; α-curcumene and pseudoisoeugenol-2-methylbutanoate. Furthermore, a statistically strong negative correlation at the 99% confidence level was found between germacrene D and limonene (Figure 3). The GC–MS chromatogram of the representative sample is shown in Figure S3.

The results were in good agreement with those published by Salem et al. [28] and El-Kersh et al. [29], who reported about 90% of *trans*-anethole and 5% of γ-himachalene in anise oils. Özcan and Chalchat [30] reported the presence of *trans*-anethole (93.90%), estragole (2.40%) and γ-himachalene (1.10%) in EOs from Turkey *Pimpinella anisum* L. dried seeds.

3.2.3 | *Coriandrum sativum* (Coriander)

In coriander EOs of all samples, linalool (62.02%–67.91%) was the main constituent determined, followed by γ-terpinene (12.11%–13.91%) and α-pinene (5.24%–8.36%) from a total of 21 identified compounds. The camphor (2.99%–3.86%), limonene (2.21%–2.82%), geranyl acetate (1.07%–2.20%) and geraniol (0.95%–1.69%) were detected as minor components (Table 3). Statistical evaluation revealed a strong positive statistical correlation between the essential oil components limonene and myrcene, *p*-cymene; γ-terpinene and α-pinene, myrcene, linalool; α-terpineol and myrcene, *p*-cymene, limonene; sabinene and γ-terpinene; β-pinene and α-pinene, myrcene, linalool, γ-terpinene, sabinene; *cis*-sabinene hydrate and myrcene, limonene; terpinolene and myrcene, *p*-cymene, limonene, γ-terpinene, α-terpineol, *cis*-sabinene hydrate; camphor and γ-terpinene, β-pinene; terpinen-4-ol and α-pinene, linalool; geranyl acetate and myrcene, limonene, terpinolene; β-caryophyllene and *cis*-sabinene hydrate, terpinen-4-ol, geranyl acetate (Figure 4). The GC–MS chromatogram of the representative sample is shown in Figure S4.

Similar results were previously reported by Khalil et al. [22] in a coriander sample cultivated in Egypt, where the dominant linalool accounted for 70.93%, γ-terpinene for 4.17% and α-pinene for 2.58%. A higher amount of γ-terpinene (12.11%–13.91%) correlated with the study of Matsushita et al. [31], who found 12.39% of γ-terpinene in the coriander sample from Morocco. Ebrahimi et al. [32] determined linalool (40.90%–79.90%), γ-terpinene (0.10%–13.60%), neryl acetate (2.30%–14.20%), α-pinene (1.20%–7.10%) and *p*-cymene (0.80%–3.60%) as the main components of several coriander EOs from Iran.

3.2.4 | *Foeniculum vulgare* (Fennel)

The most abundant compounds in fennel EOs, from 21 identified, were *trans*-anethole (0.82%–92.87%), estragole (2.53%–89.51%), limonene (1.05%–6.61%) and fenchone (0.50%–7.62%) (Table 4). A significant difference in the representation of the components

TABLE 2 | Chemical composition of essential oils (% ± SD) in achenes of *Pimpinella anisum* (anise) determined by GC–MS.

No.	RT	Compound	RI calc	RI lit	PIMG, % ± SD	PIMS, % ± SD	PIMB, % ± SD	PIMV, % ± SD
1	7.49	α-Pinene	937	932	0.01 ± 0.00	—	—	0.04 ± 0.01
2	8.19	Sabinene	976	969	0.02 ± 0.00	0.03 ± 0.00	0.03 ± 0.00	0.02 ± 0.00
3	8.28	β-Pinene	982	974	—	—	—	0.01 ± 0.00
4	8.80	3-Carene	1013	1008	—	—	—	0.08 ± 0.00
5	9.05	p-Cymene	1028	1020	0.02 ± 0.00	—	0.01 ± 0.00	0.02 ± 0.00
6	9.14	Limonene	1033	1024	0.05 ± 0.00	0.01 ± 0.00	0.01 ± 0.00	0.03 ± 0.00
7	9.62	γ-Terpinene	1062	1054	—	—	—	0.03 ± 0.00
8	10.26	Linalool	1101	1095	0.01 ± 0.00	—	—	0.20 ± 0.01
9	11.02	*cis*-Verbenol	1150	1138	0.01 ± 0.00	0.01 ± 0.00	0.02 ± 0.00	0.02 ± 0.00
10	11.84	Estragole	1203	1195	1.09 ± 0.01	0.45 ± 0.01	0.37 ± 0.02	0.81 ± 0.06
11	12.54	Carvone	1251	1239	0.16 ± 0.01	0.10 ± 0.01	0.04 ± 0.00	0.07 ± 0.00
12	12.65	*cis*-Anethole	1258	1249	0.29 ± 0.01	0.15 ± 0.01	0.16 ± 0.03	0.22 ± 0.02
13	12.72	p-Anis aldehyde	1263	1247	0.37 ± 0.04	0.13 ± 0.01	0.06 ± 0.01	0.09 ± 0.01
14	13.16	***trans*-Anethole**	1293	1282	**93.84 ± 0.04**	**95.30 ± 0.15**	**95.70 ± 0.13**	**93.79 ± 0.08**
15	13.85	δ-Elemene	1344	1335	0.03 ± 0.00	0.03 ± 0.00	0.06 ± 0.00	0.07 ± 0.01
16	14.14	α-Longipinene	1364	1350	0.03 ± 0.00	0.03 ± 0.00	0.02 ± 0.00	0.02 ± 0.00
17	14.37	α-Copaene	1381	1374	0.02 ± 0.00	0.02 ± 0.01	0.02 ± 0.00	0.03 ± 0.00
18	14.51	Anisketone	1391	1380	0.02 ± 0.00	0.01 ± 0.00	0.02 ± 0.00	0.02 ± 0.00
19	14.60	β-Elemene	1398	1389	0.04 ± 0.00	0.04 ± 0.01	0.03 ± 0.00	0.03 ± 0.01
20	15.09	β-Caryophyllene	1435	1417	0.01 ± 0.00	0.01 ± 0.00	0.01 ± 0.00	0.04 ± 0.00
21	15.51	α-Himachalene	1468	1449	0.16 ± 0.00	0.14 ± 0.02	0.17 ± 0.01	0.15 ± 0.02
22	15.64	Ethyl trans-cinnamate	1478	1465	0.06 ± 0.00	0.05 ± 0.00	0.04 ± 0.00	0.04 ± 0.00
23	15.76	α-Curcumene	1487	1479	0.15 ± 0.00	0.12 ± 0.00	0.06 ± 0.00	0.20 ± 0.01
24	15.83	*trans*-Methyl isoeugenol	1492	1491	0.06 ± 0.00	0.07 ± 0.00	0.05 ± 0.00	0.05 ± 0.00
25	15.86	γ-Himachalene	1495	1480	2.33 ± 0.06	2.59 ± 0.08	2.41 ± 0.08	2.22 ± 0.08
26	15.93	α-Zingiberene	1500	1494	0.06 ± 0.00	0.04 ± 0.01	0.06 ± 0.01	0.14 ± 0.03
27	16.11	β-Himachalene	1515	1500	0.22 ± 0.01	0.16 ± 0.04	0.24 ± 0.03	0.33 ± 0.02
28	16.32	2-Methoxycinnamaldehyde	1532	1527	0.09 ± 0.01	0.09 ± 0.02	0.06 ± 0.00	0.31 ± 0.05
29	17.09	Spathulenol	1595	1577	0.04 ± 0.00	0.04 ± 0.01	0.02 ± 0.00	0.02 ± 0.00
30	17.62	Alloaromadendrene epoxide	1641	1639	0.05 ± 0.00	0.05 ± 0.02	0.03 ± 0.01	0.04 ± 0.01
31	19.77	Pseudoisoeugenol-2-methylbutanoate	1835	1822	0.75 ± 0.05	0.41 ± 0.00	0.35 ± 0.01	0.74 ± 0.01
32	20.37	Epoxypseudoisoeugenol-2-methylbutyrate (Thellungianin G)	1892	1882	0.08 ± 0.00	0.04 ± 0.01	0.05 ± 0.01	0.07 ± 0.00

Note: The letters G, S, B and V belong to individual local producers of spices. Results are reported in % ± SD (standard deviation) as the mean of three experiments. Significance of bold value indicates. The dominant components of the essential oil are highlighted.
Abbreviations: PIM, anise; No., peak number; RI calc, retention index calculated with respect to homologous series of n-alkanes (C9-C23) on a DB-5 column; RI lit, retention index from literature—entered from Robert P. Adam's library; RT, retention time.

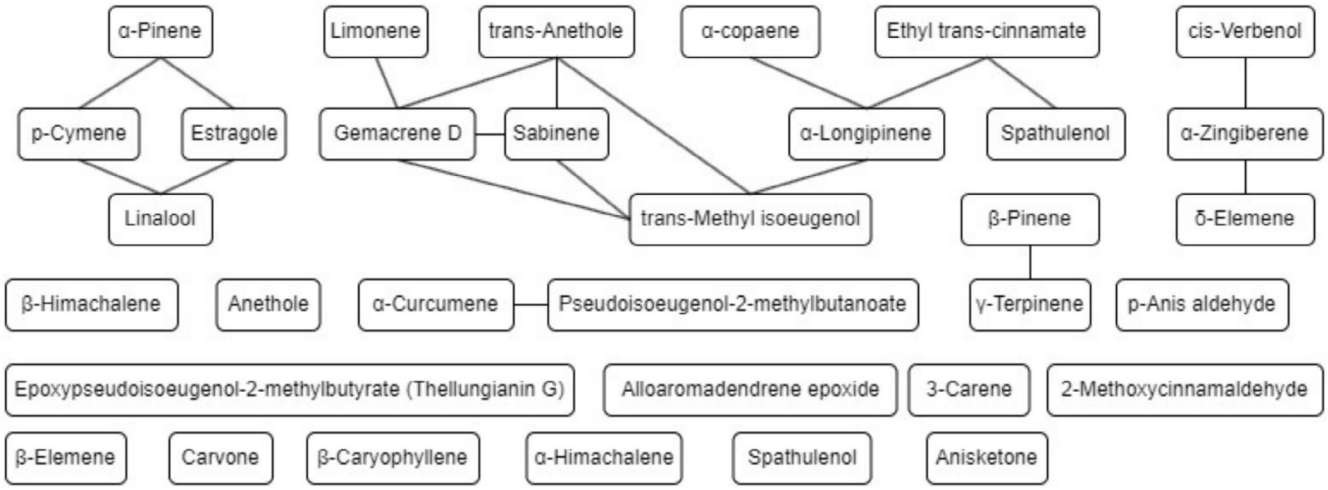

FIGURE 3 | Correlations between the chemical composition of essential oils in fruits of *Pimpinella anisum* (anise), ($r = 0.7$).

TABLE 3 | Chemical composition of essential oils (% ± SD) in achenes of *Coriandrum sativum* (coriander) determined by GC–MS.

No.	RT	Compound	RI calc	RI lit	CORG, % ± SD	CORS, % ± SD	CORB, % ± SD	CORV, % ± SD
1	7.50	α-Pinene	937	932	5.93 ± 0.07	5.24 ± 0.06	8.36 ± 0.10	5.53 ± 0.05
2	8.18	Sabinene	976	969	0.60 ± 0.02	0.47 ± 0.02	0.54 ± 0.02	0.48 ± 0.02
3	8.29	β-Pinene	983	974	0.56 ± 0.02	0.52 ± 0.03	0.80 ± 0.02	0.58 ± 0.02
4	8.40	Myrcene	990	988	0.80 ± 0.03	0.88 ± 0.04	0.90 ± 0.02	0.81 ± 0.03
5	8.94	α-Terpinene	1020	1014	0.11 ± 0.01	0.06 ± 0.00	0.10 ± 0.00	0.12 ± 0.00
6	9.05	p-Cymene	1028	1020	1.33 ± 0.03	2.68 ± 0.03	2.10 ± 0.04	2.00 ± 0.04
7	9.14	Limonene	1033	1024	2.21 ± 0.03	2.82 ± 0.03	2.54 ± 0.04	2.25 ± 0.04
8	9.38	*trans*-Ocimene	1048	1044	0.13 ± 0.02	0.11 ± 0.01	0.11 ± 0.00	0.16 ± 0.01
9	9.62	γ-Terpinene	1062	1054	12.85 ± 0.09	12.73 ± 0.08	13.91 ± 0.10	12.1 ± 0.10
10	9.83	*cis*-Sabinene hydrate	1074	1065	0.09 ± 0.01	0.10 ± 0.01	0.09 ± 0.00	0.07 ± 0.00
11	10.07	Terpinolene	1089	1086	0.62 ± 0.03	0.73 ± 0.03	0.66 ± 0.02	0.58 ± 0.02
12	10.25	**Linalool**	1099	1095	**66.66 ± 0.22**	**64.50 ± 0.20**	**62.02 ± 0.28**	**67.91 ± 0.30**
13	11.13	Camphor	1156	1141	3.60 ± 0.08	2.99 ± 0.06	3.45 ± 0.05	3.86 ± 0.04
14	11.50	Borneol	1181	1165	0.22 ± 0.02	2.11 ± 0.04	0.07 ± 0.00	0.06 ± 0.00
15	11.62	Terpinen-4-ol	1188	1174	0.12 ± 0.01	0.13 ± 0.01	0.14 ± 0.01	0.16 ± 0.01
16	11.82	α-Terpineol	1201	1186	0.17 ± 0.01	0.28 ± 0.02	0.21 ± 0.02	0.16 ± 0.01
17	12.55	Geraniol	1251	1249	1.69 ± 0.03	1.22 ± 0.05	1.26 ± 0.04	0.95 ± 0.04
18	13.15	*trans*-Anethole	1291	1282	0.86 ± 0.03	0.11 ± 0.01	0.37 ± 0.02	1.00 ± 0.04
19	13.66	Myrtenyl acetate	1329	1324	0.05 ± 0.00	0.15 ± 0.01	0.04 ± 0.00	0.04 ± 0.00
20	14.31	Geranyl acetate	1377	1379	1.34 ± 0.03	2.06 ± 0.05	2.20 ± 0.04	1.07 ± 0.06
21	15.09	β-Caryophyllene	1435	1417	0.08 ± 0.01	0.09 ± 0.01	0.13 ± 0.01	0.08 ± 0.01

Note: The letters G, S, B and V belong to individual local producers of spices. Results are reported in % ± SD (standard deviation) as the mean of three experiments. Significance of bold value indicates. The dominant components of the essential oil are highlighted.
Abbreviations: COR, coriander; No., peak number; RI calc, retention index calculated with respect to homologous series of n-alkanes (C9-C23) on a DB-5 column; RI lit, retention index from literature—entered from Robert P. Adam's library; RT, retention time.

in the FENV sample was observed. In this sample, the highest amount of estragole and limonene was found, 89.51% and 6.61% respectively, while the lowest amount of *trans*-anethole was found to be 0.82%, which is in good correlation with the results previously reported by Salem et al. [28]. In this study, a fennel sample obtained from the Agricultural Research Centre, Cairo,

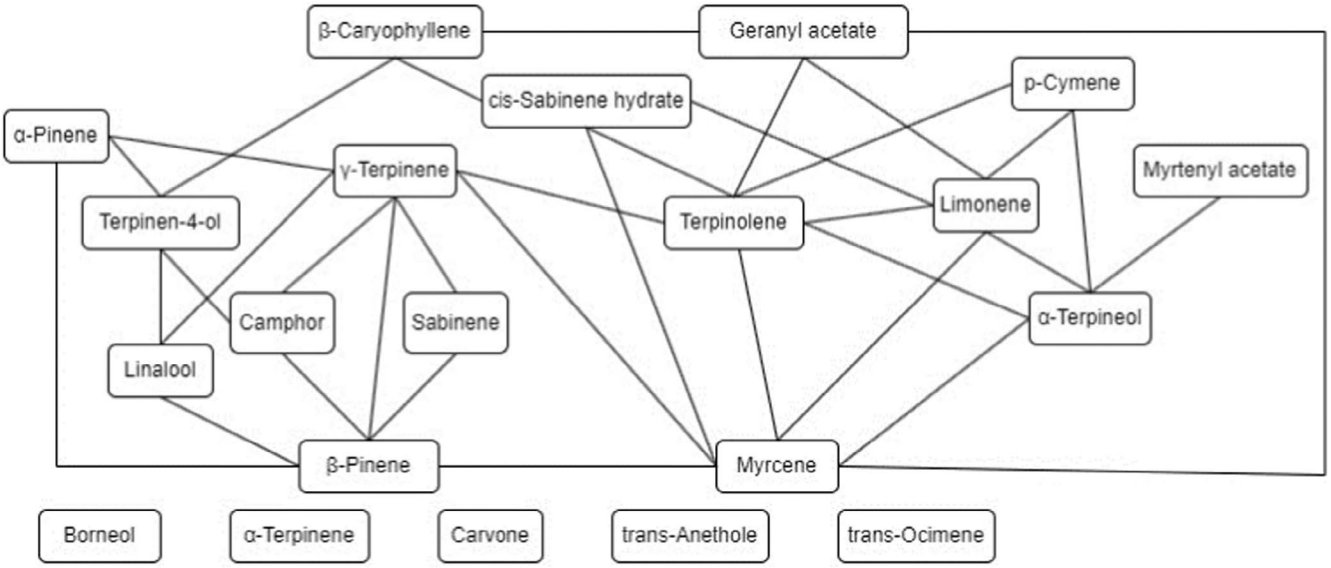

FIGURE 4 | Correlations between the chemical composition of essential oils in fruits of *Coriandrum sativum* (coriander), ($r = 0.7$).

TABLE 4 | Chemical composition of essential oils (% ± SD) in achenes of *Foeniculum vulgare* (fennel) determined by GC–MS.

No.	RT	Compound	RI calc	RI lit	FOEG, % ± SD	FOES, % ± SD	FOEB, % ± SD	FOEV, % ± SD
1	7.49	α-Pinene	937	932	0.27 ± 0.01	1.19 ± 0.01	0.25 ± 0.01	0.77 ± 0.01
2	7.80	Camphene	955	946	0.01 ± 0.00	0.14 ± 0.01	0.06 ± 0.01	0.10 ± 0.02
3	8.17	Sabinene	976	969	0.12 ± 0.02	0.08 ± 0.00	0.09 ± 0.01	0.21 ± 0.01
4	8.28	β-Pinene	983	974	0.02 ± 0.00	0.06 ± 0.01	0.01 ± 0.00	0.04 ± 0.00
5	8.41	Myrcene	990	988	0.05 ± 0.01	0.24 ± 0.03	0.04 ± 0.00	0.10 ± 0.00
6	8.75	α-Phellandrene	1010	1002	0.04 ± 0.00	0.17 ± 0.01	0.03 ± 0.00	0.07 ± 0.00
7	9.06	p-Cymene	1028	1020	0.06 ± 0.01	0.09 ± 0.00	0.07 ± 0.00	0.10 ± 0.00
8	9.15	Limonene	1033	1024	2.61 ± 0.12	1.05 ± 0.06	3.23 ± 0.04	6.61 ± 0.03
9	9.62	γ-Terpinene	1062	1054	0.10 ± 0.01	0.32 ± 0.01	0.08 ± 0.01	0.21 ± 0.02
10	9.83	*cis*-Sabinene hydrate	1074	1065	—	0.03 ± 0.00	0.01 ± 0.00	—
11	10.08	Terpinolene	1089	1086	0.01 ± 0.00	0.05 ± 0.00	0.01 ± 0.00	0.01 ± 0.00
12	10.17	L-Fenchone	1095	1083	1.02 ± 0.01	7.62 ± 0.00	0.50 ± 0.01	0.81 ± 0.01
13	11.14	Camphor	1156	1141	0.02 ± 0.00	0.18 ± 0.01	0.01 ± 0.00	0.02 ± 0.00
14	11.85	**Estragole**	1203	1195	2.53 ± 0.01	2.59 ± 0.02	3.46 ± 0.01	**89.51 ± 0.05**
15	12.55	Carvone	1251	1239	0.04 ± 0.00	—	0.11 ± 0.01	0.66 ± 0.06
16	12.66	*cis*-Anethole	1259	1249	0.06 ± 0.01	0.05 ± 0.00	0.06 ± 0.00	—
17	12.74	p-Anisaldehyde	1264	1247	0.04 ± 0.01	—	0.14 ± 0.01	—
18	13.15	***trans*-Anethole**	1292	1282	**92.87 ± 0.16**	**86.29 ± 0.18**	**92.01 ± 0.19**	0.82 ± 0.01
19	14.46	α-Copaene	1387	1374	0.01 ± 0.00	—	0.02 ± 0.00	0.02 ± 0.00
20	15.88	Gemacrene D	1496	1480	0.02 ± 0.00	0.02 ± 0.00	0.03 ± 0.00	0.04 ± 0.00
21	17.45	Dill apiole	1626	1620	0.01 ± 0.00	—	0.04 ± 0.00	0.02 ± 0.00

Note: The letters G, S, B and V belong to individual local producers of spices. Results are reported in % ± SD (standard deviation) as the mean of three experiments. Significance of bold value indicates. The dominant components of the essential oil are highlighted.
Abbreviations: FOE, fennel; No., peak number; RI calc, retention index calculated with respect to homologous series of n-alkanes (C9-C23) on a DB-5 column; RI lit, retention index from literature—entered from Robert P. Adam's library; RT, retention time.

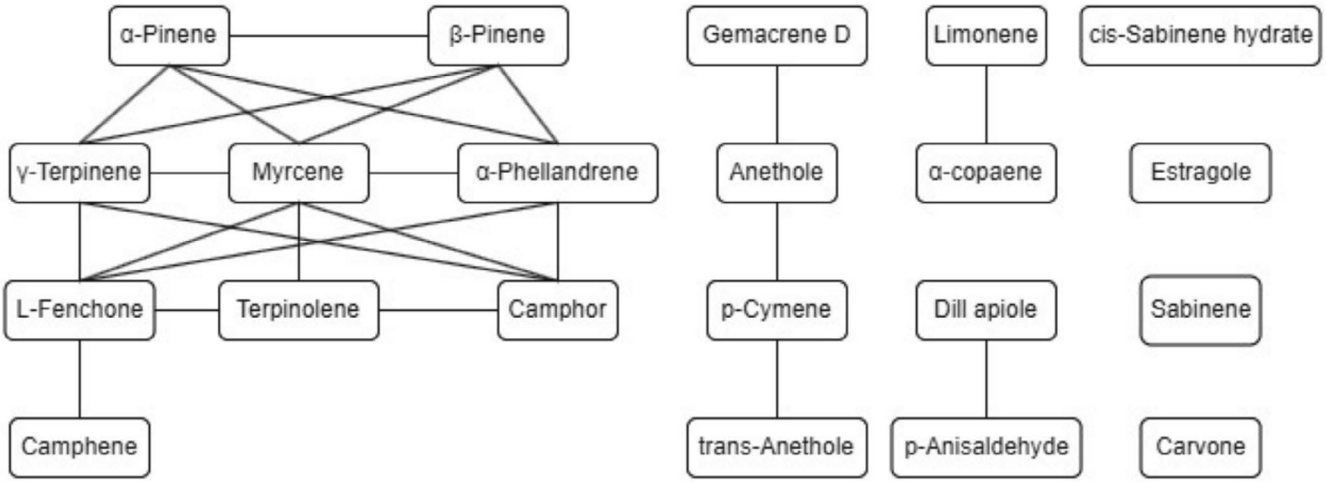

FIGURE 5 | Correlations between the chemical composition of essential oils in fruits of *Foeniculum vulgare* (fennel), ($r = 0.7$).

Egypt, consisted of 79.88% estragole, 12.50% limonene and no *trans*-anethole.

Statistical evaluation showed a strong positive statistical correlation at a 99% confidence level between the essential oil components myrcene and α-pinene; α-phellandrene and α-pinene, myrcene; *trans*-anethole and *p*-cymene; γ-terpinene and α-pinene, myrcene; β-pinene and α-pinene, myrcene, α-phellandrene, γ-terpinene; terpinolene and myrcene; camphor, L-fenchone and myrcene, α-phellandrene, γ-terpinene, terpinolene; estragole and carvone; anethole and *p*-cymene; *p*-anisaldehyde and dill apiole; α-copaene and limonene; germacrene D and anethole (Figure 5). The GC–MS chromatograms of the representative samples are shown in Figures S5 and S6.

Saharkhiz and Tarakeme [33] reported *trans*-anethol (84.10%–86.10%), fenchone (7.13%–8.86%), limonene (3.00%–3.30%) and methyl-chavicol (2.50%–2.70%) as the main components of fennel EOs from Iran. Wodnicka et al. [34] observed a significant difference in the composition of fennel EOs cultivated in Poland (70.00% of *trans*-anethole, 3.00% of estragole and 2.00% of limonene) and in Egypt (no *trans*-anethole, 87.00% of estragole and 9.00% of limonene). This study shows that the chemical composition of EOs depends on the geographical origin. However, there are numerous factors influencing the constitution of EOs from plants, such as plant variety, genotype and also environmental and agronomic conditions [4]. Estragole is a minor or major component of many herbs such as fennel, anise, tarragon and basil, which are used in various foodstuffs, as flavouring substances in seasonings, and in teas or tea-like beverages to prevent flatulence and spasms. Since estragole, a 4-allylalkoxybenzene derivative, has been described as a carcinogenic and genotoxic compound, a reduction in consumption has been recommended [35, 36]. All herbal species, especially fennel and anise, deserve attention since these products are designed for infants, young children, pregnant and breastfeeding women. Nowadays, in the food industry, regulatory agencies such as EFSA (European Food Safety Authority), FDA (US Food and Drug Administration) and Codex Alimentarius (International Food Standards Commission) have not yet set a maximum intake level for estragole. On the other hand, several research groups have reported estragole as non-toxic [37] and with anti-cancer effect [38]. So, these herbs need to be verified by toxicological tests with respect to their use. The differences between the samples are probably due to the processing of the different varieties of fennel. There are *Foeniculum vulgare* var. vulgare Mill. and *Foeniculum vulgare* var. dulce Mill. *Foeniculum vulgare* var. dulce is grown mainly in Asian countries and differs from each other both in morphology and in essential oil content. Its colour is yellow, sometimes tinged with green, with a sweet taste. This type is not bitter [39]. Recent research has concentrated on increasing the yield of fennel fruit and the quality of its essential oil by decreasing the undesirable component, estragole, under various plant treatments (using different fertilisers or biostimulants). The production and oil quality of fennel fruits were found to be improved by the use of organic fertilisation [40] or biostimulants such as a low concentration of yeast (2 g/L; [41]) or nitrogen-fixing bacteria and moringa leaf extract [42].

3.2.5 | *Anethum graveolens* (Dill)

Only two companies provided samples of dill (G and B). As shown in Table 5, the difference in the distribution of 20 identified compounds is evident. Limonene (47.59%) and carvone (47.29%) dominated in the ANEG sample. In contrast, dill apiole (42.08%), followed by limonene (29.80%), carvone (11.82%), *cis*- and *trans*-dihydrocarvone (7.30% and 7.20%, respectively), were the most abundant components in the ANEB sample. Compared to the plant matrices examined above, there were no very significant correlations between the constituents of dill EO. The GC–MS chromatograms of both samples are shown in Figures S7 and S8.

The chemical composition of dill oils varied in many ways. It depends on the part of the plant used for extraction, variety, chemotype and others such as climatic and agronomic conditions. There are two types of dill oil—dill weed oil extracted from the herb (leaves, stems, flowers) and dill seed oil—extracted from the seeds. The main constituents of them are either α-phellandrene (weed oil) or carvone with limonene (seed oil)

TABLE 5 | Chemical composition of essential oils (% ± SD) in achenes of *Anethum graveolens* (dill) determined by GC–MS.

No.	RT	Compound	RI calc	RI lit	ANEG, % ± SD	ANEB, % ± SD
1	7.50	α-Pinene	937	932	0.02 ± 0.00	0.03 ± 0.00
2	8.18	Sabinene	976	969	0.08 ± 0.01	0.18 ± 0.04
3	8.40	Myrcene	990	988	0.07 ± 0.01	0.07 ± 0.01
4	8.75	α-Phellandrene	1010	1002	0.83 ± 0.02	0.33 ± 0.05
5	9.05	p-Cymene	1028	1020	0.11 ± 0.01	0.09 ± 0.01
6	9.14	**Limonene**	1033	1024	**47.59 ± 0.55**	**29.80 ± 0.36**
7	10.15	p-Cymenene	1094	1089	0.16 ± 0.01	0.15 ± 0.03
8	10.24	Linalool	1099	1095	0.06 ± 0.00	—
9	10.84	*cis*-Limonene oxide	1138	1132	0.08 ± 0.01	0.07 ± 0.01
10	10.92	*trans*-Limonene oxide	1142	1137	0.02 ± 0.00	0.07 ± 0.01
11	11.70	Dill ether	1194	1184	0.07 ± 0.00	0.05 ± 0.01
12	11.87	*cis*-Dihydrocarvone	1205	1191	1.25 ± 0.04	7.30 ± 0.16
13	11.97	*trans*-Dihydrocarvone	1212	1200	1.28 ± 0.07	7.20 ± 0.16
14	12.14	*trans*-Carveol	1225	1215	0.33 ± 0.01	0.24 ± 0.03
15	12.35	*cis*-Carveol	1238	1226	0.42 ± 0.01	0.24 ± 0.02
16	12.54	**Carvone**	1251	1239	**47.29 ± 0.60**	11.82 ± 0.05
17	13.15	*trans*-Anethole	1291	1282	0.23 ± 0.01	0.20 ± 0.01
18	16.28	Myristicin	1529	1517	—	0.04 ± 0.00
19	16.51	Elemicin	1548	1545	—	0.05 ± 0.00
20	17.45	**Dill apiole**	1626	1620	0.12 ± 0.02	**42.08 ± 0.55**

Note: The letters G, S, B and V belong to individual local producers of spices. Results are reported in % ± SD (standard deviation) as the mean of three experiments. Significance of bold value indicates. The dominant components of the essential oil are highlighted.
Abbreviations: No., peak number; PIM, anise; RI calc, retention index calculated with respect to homologous series of n-alkanes (C9-C23) on a DB-5 column; RI lit, retention index from literature—entered from Robert P. Adam's library; RT, retention time.

[43]. Moreover, four different chemotypes of dill were described [44, 45]. ANEG sample is obviously the chemotype 3 (limonene 39.50%–50.70% and carvone 43.70%–57.70%) which is typical for European dill seed oil. ANEB sample is probably chemotype 1 (limonene 36.90%–46.70%, carvone 17.80%–45.60%, myristicin 0.20%–20.30% and dill apiole 8.00%–22.30%) with a higher amount of dill apiole. A higher amount of dill apiole (19.98%–48.90%) was found in dill cultivated in Thailand [46].

Ozliman et al. [47] analysed EOs of dill seeds after organic or inorganic fertilisation. As a function of treatment, the most abundant components were found to be dill apiole (13.55%–18.65%), carvantonacetone (5.04%–21.76%), limonene (10.23%–20.05%) and α-phellandrene (9.20%–10.79%).

3.3 | Content of T-2, HT-2 Toxins Evaluated by LC–MS

The main field mycotoxins T-2 and HT-2 toxins were monitored in all samples. The levels of selected mycotoxins T-2 toxin and HT-2 toxin in almost all samples were below the LOD (limit of detection), 0.04 and 0.25 μg/kg, respectively. Three spice samples exhibited detectable levels of trichothecene mycotoxins T-2 and HT-2. In the caraway sample (CARS), the average concentrations of T-2 and HT-2 toxins were 0.37 and 1.77 μg/kg, respectively. In the fennel sample (FOES), 0.49 μg/kg of T-2 toxin and 0.59 μg/kg of HT-2 toxin were detected. Finally, in the coriander sample (CORS), T-2 and HT-2 toxins were present at levels of 1.78 and 2.69 μg/kg, respectively. In Figure S9, the MRM transition of quantification ion for selected mycotoxins T-2 toxin and HT-2 toxin is shown. According to Commission Regulation (EU) 2023/915, as amended by Regulation (EU) 2024/1038, maximum limits for the sum of T-2 and HT-2 toxins have been established for cereals and cereal-based products (ranging from 50 to 200 μg/kg), but no specific limits currently exist for spice commodities. Therefore, the concentrations detected in the analysed samples remain well below any thresholds established for cereals. From the perspective of dietary risk assessment, the European Food Safety Authority (EFSA) has set a tolerable daily intake (TDI) of 0.02 μg/kg body weight per day and an acute reference dose (ARfD) of 0.3 μg/kg body weight for the sum of T-2 and HT-2 toxins. Given the very low concentrations found and the typical low daily consumption of spices (approximately 1–2 g per day), the resulting dietary exposure is negligible, representing less than 1% of the TDI for an average adult. These findings suggest that, although T-2 and HT-2 toxins were occasionally detected in spice samples, they are present at trace levels, posing no significant risk to consumer health.

There are not many studies monitoring T-2 and HT-2 toxins in spices. Potorti et al. [48] reported that the average T-2 toxin content in coriander samples from Tunisia was about two times lower (0.73 ng/g) than in our sample, and no mycotoxins were found in caraway and fennel samples from Tunisia and Sicily. The highest concentrations of T-2 and HT-2 toxins in different spices were found in dawadawa in Nigeria (32 and 76 μg/kg, respectively) and paprika in Italy (27 and 58 μg/kg, respectively) [11].

4 | Conclusions

The aim of this study was to compare the differences in the yield and essential oil composition of selected spices of the *Apiaceae* family and to screen for the T-2 and HT-2 toxins.

Approximately 20–30 components of EOs were found in samples. Carvone (60%) and limonene (40%) were observed as major constituents in caraway. Trans-anethole (94%–96%) was the predominant component in EOs from anise. Coriander EOs were characterised by linalool (62%–68%). Trans-anethole prevailed in FOEG, FOES and FOEB (86%–93%) samples, whereas estragole was the main component in the FOEV (90%) sample. The ANEG sample contained mainly carvone and limonene (47% each), and the ANEB sample consisted mostly of dill apiole (42%).

The differences in EOs components, particularly for fennel (FOEV), are alarming due to the potential carcinogenic effects of estragole. It is important that spice producers correctly label and identify the species they process into spices.

Further research is needed to assess the potential risk to human health and establish a limit of dangerous compounds, such as alkoxy-substituted allylbenzenes, in all raw materials, not only those used as pharmaceuticals.

The mycotoxin determination was focused on T-2 and HT-2 toxins. The levels of mycotoxins found in the samples were below the limit of detection, but in three samples (CARS, FOES, CORS) small amounts of T-2 and HT-2 toxins were detected. Therefore, to ensure health safety, special attention must be paid to the right conditions during harvesting and especially during transport and storage to prevent mycotoxin contamination of spices.

Author Contributions

Helena Pluháčková: Conceptualisation idea, manuscript editing and preparation for submission, methodology, samples plant material, sample preparation, extraction of EOs, extraction of T-2, HT-2 toxins, manuscript – original draft. Barbora Kudláčková: Methodology, manuscript – original draft, supervision. Lenka Svojanovská: Analysis of EOs by GC–MS, methodology, data processing, formal analysis, manuscript editing. Milan Křápek: Statistical analysis, methodology. Jan Martiník: Extraction of T-2, HT-2 toxins, analysis of T-2 toxin and HT-2 toxin by LC–MS. Rastislav Boško: Extraction of T-2, HT-2 toxins, manuscript editing. Marek Pernica: Analysis of T-2 toxin and HT-2 toxin by LC–MS, data processing, manuscript editing. All authors read and approved the final manuscript.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section. **Figure S1:** Photo of the dry achenes of analysed spices. A: caraway; B: anise; C: coriander; D: fennel; E: dill. **Figure S2:** GC–MS chromatogram (TIC) of the essential oil of *Carum carvi* (caraway) V sample. CAR = caraway; the letter V belongs to an individual local producer of spices. Peak identification: 1—α-Pinene, 2—Sabinene, 3—Myrcene, 4—Limonene, 5—*trans*-Ocimene, 6—γ-Terpinene, 7—*trans*-p-Mentha-2,8-dien-1-ol, 8—*cis*-Limonene oxide, 9—*trans*-Limonene oxide, 10—α-Terpineol, 11—*cis*-Dihydrocarvone, 12—*trans*-Dihydrocarvone, 13—*trans*-Carveol, 14—*cis*-Carveol, 15—Carvone, 16—Perilla aldehyde, 17—*trans*-Anethole, 18—β-Caryophyllene. **Figure S3:** GC–MS chromatogram (TIC) of the essential oil of *Pimpinella anisum* (anise) V sample. PIM = anise; the letter V belongs to an individual local producer of spices. Peak identification: 1—α-Pinene, 2—Sabinene, 3—β-Pinene, 4–3-Carene, 5—p-Cymene, 6—Limonene, 7—γ-Terpinene, 8—Linalool, 9—*cis*-Verbenol, 10—Estragole, 11—Carvone, 12—*cis*-Anethole, 13—p-Anis aldehyde, 14—*trans*-Anethole, 15—δ-Elemene, 16—α-Longipinene, 17—α-Copaene, 18—Anisketone, 19—β-Elemene, 20—β-Caryophyllene, 21—α-Himachalene, 22—Ethyl trans-cinnamate, 23—α-Curcumene, 24—*trans*-Methyl isoeugenol, 25—γ-Himachalene, 26—α-Zingiberene, 27—β-Himachalene, 28–2-Methoxycinnamaldehyde, 29—Spathulenol, 30—Alloaromadendrene epoxide, 31—Pseudoisoeugenol-2-methylbutanoate, 32—Epoxypseudoisoeugenol-2-methylbutyrate (Thellungianin G). **Figure S4:** GC–MS chromatogram (TIC) of the essential oil of *Coriandrum sativum* (coriander) V sample. COR = coriander; the letter V belongs to an individual local producer of spices. Peak identification: 1—α-Pinene, 2—Sabinene, 3—β-Pinene, 4—Myrcene, 5—α-Terpinene, 6—p-Cymene, 7—Limonene, 8—*trans*-Ocimene, 9—γ-Terpinene, 10—*cis*-Sabinene hydrate, 11—Terpinolene, 12—Linalool, 13—Camphor, 14—Borneol, 15—Terpinen-4-ol, 16—α-Terpineol, 17—Geraniol, 18—*trans*-Anethole, 19—Myrtenyl acetate, 20—Geranyl acetate, 21—β-Caryophyllene. **Figure S5:** GC–MS chromatogram (TIC) of the essential oil of *Foeniculum vulgare* (fennel) V sample. FOE = fennel; the letter V belongs to an individual local producer of spices. Peak identification: 1—α-Pinene, 2—Camphene, 3—Sabinene, 4—β-Pinene, 5—Myrcene, 6—α-Phellandrene, 7—p-Cymene, 8—Limonene, 9—γ-Terpinene, 11—Terpinolene, 12—L-Fenchone, 13—Camphor, 14—Estragole, 15—Carvone, 18—*trans*-Anethole, 19—α-Copaene, 20—Gemacrene D, 21—Dill apiole. **Figure S6:** GC–MS chromatogram (TIC) of the essential oil of *Foeniculum vulgare* (fennel) B sample. FOE = fennel; the letter B belongs to an individual local producer of spices. Peak identification: 1—α-Pinene, 2—Camphene, 3—Sabinene, 4—β-Pinene, 5—Myrcene, 6—α-Phellandrene, 7—p-Cymene, 8—Limonene, 9—γ-Terpinene, 10—*cis*-Sabinene hydrate, 11—Terpinolene, 12—L-Fenchone, 13—Camphor, 14—Estragole, 15—Carvone, 16—*cis*-Anethole, 17—p-Anisaldehyde, 18—*trans*-Anethole, 19—α-Copaene, 20—Gemacrene D, 21—Dill apiole. **Figure S7:** GC–MS chromatogram (TIC) of the essential oil of *Anethum graveolens* (dill) G sample. ANE = dill; the letter G belongs to an individual local producer of spices. Peak identification: 1—α-Pinene, 2—Sabinene, 3—Myrcene, 4—α-Phellandrene, 5—p-Cymene, 6—Limonene, 7—p-Cymenene, 8—Linalool, 9—*cis*-Limonene oxide, 10—*trans*-Limonene oxide, 11—Dill ether, 12—*cis*-Dihydrocarvone, 13—*trans*-Dihydrocarvone, 14—*trans*-Carveol, 15—*cis*-Carveol, 16—Carvone, 17—*trans*-Anethole, 20—Dill apiole. **Figure S8:** GC–MS chromatogram (TIC) of the essential oil of *Anethum graveolens* (dill) B sample. ANE = dill; the letter B belongs to an individual local producer of spices. Peak identification: 1—α-Pinene, 2—Sabinene, 3—Myrcene, 4—α-Phellandrene, 5—p-Cymene, 6—Limonene, 7—p-Cymenene, 9—*cis*-Limonene oxide, 10—*trans*-Limonene oxide, 11—Dill ether, 12—*cis*-Dihydrocarvone, 13—*trans*-Dihydrocarvone, 14—*trans*-Carveol, 15—*cis*-Carveol, 16—Carvone, 17—*trans*-Anethole, 18—Myristicin, 19—Elemicin, 20—Dill apiole. **Figure S9:** MRM transition of quantification ion for selected mycotoxins T-2 toxin (on left) and HT-2 toxin (on right).