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Optimization of harvest date according to the volatile composition of Mediterranean aromatic herbs at different vegetative stages

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ABSTRACT

Most of the studies concerning the optimal harvest date of aromatic herbs have dealt with different parts of plant such as flowers, leaves, stems, roots and seeds, but none have evaluated the effect of different harvest date at different vegetative stages on shoots (leaves and stems). Therefore, the main objective was to investigate the effects of harvest date of two consecutive seasons on the volatile composition of shoots of 4 aromatic herbs (dill, parsley, coriander and mint) widely cultivated worldwide. The impact of harvest date during the evolution of vegetative part of four aromatic herbs (parsley, dill, coriander and mint) was investigated. Volatile compounds of shoots of the four herbs were identified by GC–MS. The main compounds were 1,3,8-*p*-menthatriene, beta-phellandrene, myristicin and myrcene for parsley, alpha-phellandrene, dillether and beta-phellandrene for dill, decanal, E-2-dodecenal, 1-decanol and dodecanal for coriander, and carvone and limonene in case of mint. There was a significant effect of harvest date on the content of volatile compounds of the four species. The results showed that highest total concentration of volatiles and therefore the optimal harvest date of parsley was found 9 weeks after planting date with a total concentration of 2543 mg kg^{-1}, for dill was 2619 mg kg^{-1} 14 weeks after planting date, for coriander was 2191 mg kg^{-1} 3 weeks after planting date, and 23329 mg kg^{-1} for mint 6 weeks after planting date.

1. Introduction

Aromatic herbs are worldwide used to improve the flavor of different types of food (Kivilompolo et al., 2007; Park, 2011; Vallverdú-Queralt et al., 2015). Essential oils of aromatic herbs can be used for bactericidal, virucidal, fungicidal, insecticidal, and cosmetic applications as well as in pharmaceutical, agricultural and food industries (Bakkali et al., 2008). For instance, leaves and stems of European dill (*Anethum graveolens* L.) from *Apiaceae* family contain essential oil and it has widely usage for flavoring foods and beverages (Callan et al., 2007). Fresh parsley (*Petroselinum crispum*) from *Apiaceae* family is used in salads and as a flavoring ingredient and its essential oil is used as flavoring agent or scent in creams, perfumes and soaps (Atta-Aly, 1999). Leaves of coriander (*Coriandrum sativum* L.), *Apiaceae* family, are a common ingredient in products such as salsa and seafood dishes (Potter, 1996). *Mentha* species belong to *Lamiaceae* family and are useful in traditional medicine, according to their antispasmodic and antiseptic; moreover, their essential oils are applied in several medical purposes such as: cosmetics, perfumes, toothpastes and mouthwashes. The herbage of mint is used as salad, spice, for tea and wool dyeing (Baytop, 1984; Edris et al., 2003; Kizil and Tonçer, 2006).

The essential oil is a mixture of many volatile compounds. Volatile compounds are affected by environmental factors such as the interaction between the genotype and environment, irrigation method, time of harvest, season, and others. The extraction method of the volatile compounds is also another important factor of worth consideration (Costa et al., 2014; de Faria and Costa, 1998; Verma et al., 2010). Yield and quality of sweet marjoram (*Origanum majorana* L.) herb was influenced by harvest time. The herb was harvested at the beginning of blossoming, i.e. in mid-July (1st harvest) and in early September (2nd harvest). More essential oil (2.39 %) and greater yield of fresh herb (0.78 kg m^{-2}) were obtained in the second harvest time (Zawiślak and

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Table 1
Different harvest dates at different vegetative stages of dill, parsley, coriander and mint shoots of two consecutive seasons.

Parsley		Dill		Coriander		Mint	
Code	Harvest date	Code	Harvest date	Code	Harvest date	Code	Harvest date
P1	22/10/2014-15	D1	22/10/2014-15	C1	22/10/2014-15	M1	22/10/2014-15
P2	29/10/2014-15	D2	29/10/2014-15	C2	29/10/2014-15	M2	29/10/2014-15
P3	05/11/2014-15	D3	05/11/2014-15	C3	05/11/2014-15	M3	05/11/2014-15
P4	12/11/2014-15	D4	12/11/2014-15	C4	12/11/2014-15	M4	12/11/2014-15
P5	19/11/2014-15	D5	19/11/2014-15	C5	19/11/2014-15	M5	19/11/2014-15
P6	26/11/2014-15	D6	26/11/2014-15	C6	26/11/2014-15	M6	26/11/2014-15
P7	04/12/2014-15	D7	04/12/2014-15	C7	04/12/2014-15	M7	04/12/2014-15
P8	11/12/2014-15	D8	11/12/2014-15	C8	11/12/2014-15	M8	11/12/2014-15
P9	18/12/2014-15	D9	18/12/2014-15	C9	18/12/2014-15	M9	18/12/2014-15
P10	24/12/2014-15	D10	24/12/2014-15	C10	24/12/2014-15	M10	24/12/2014-15
P11	31/12/2014-15	D11	31/12/2014-15	C11	31/12/2014-15	M11	31/12/2014-15
P12	05/01/2015-16	D12	21/01/2015-16	C12	05/01/2015-16	M12	05/01/2015-16
P13	21/01/2015-16	D13	28/01/2015-16	C13	21/01/2015-16	M13	21/01/2015-16
P14	28/01/2015-16	D14	11/02/2015-16	C14	28/01/2015-16	M14	28/01/2015-16
P15	11/02/2015-16	D15	25/02/2015-16	C15	11/02/2015-16	M15	11/02/2015-16
P16	25/02/2015-16						

Dzida, 2010). In addition, spearmint plant also showed a clear effect by harvest time, where results showed that the highest yield of fresh herbage was obtained at pre-flowering stage (36.0 t ha^{-1}). However, the highest essential oil yield (96.6 L ha^{-1}) was obtained at full flowering stage, while the highest plant height (62.8 cm), dry herbage yield (12.6 t ha^{-1}) and dry leaf yield (7.47 t ha^{-1}) were obtained after full flowering stage (Kizil and Tonçer, 2006). In fact, most of the studies concerning harvest time have dealt with different parts of plant (e.g. flowers, leaves, stems, roots and seeds), but none have evaluated the effect of different harvest date during the crop development (vegetative stages) on shoots (leaves and stems) of aromatic herbs. This aspect is very important since the farmers can decide which is the best harvest time according to the content of volatile compounds. Therefore, the main objective was to investigate the volatile content at different vegetative stages in order to establish the best harvest date of two consecutive seasons (years 2014 and 2015) of shoots of four worldwide used aromatic herbs (parsley, dill, coriander and mint).

2. Materials and methods

2.1. Plant material

The farm where the trials of **parsley** (*Petroselinum crispum*) and **dill** (*Anethum graveolens*) were carried out is located in a semi-arid region called Sucina (Murcia, Spain). The coordinates of the farm are 37.897433, −0.954283. The soil of culture has a salinity average with a value of 3.35 dS m^{-1}, a low content in organic matter (1.22 %) and a high level of sulfates (37.83 meq L^{-1}). In this area, the water used for irrigation has a conductivity of 1.26 dS m^{-1} and a basic pH (7.9). The farm where the **coriander** (*Coriandrum sativum*) and **mint** (*Mentha piperita*) trials were conducted is located in a semi-arid region called Librilla (Murcia, Spain). The coordinates of the farm are 37.912676, −1.299688. The soil of culture has a low salinity with a value of 0.62 dS m^{-1}, a low content in organic matter (1.12 %) and a high level of sulfates (2.60 meq L^{-1}). In this orchard, the water used for irrigation has a conductivity of 1.00 dS m^{-1} and a basic pH (8.5). All the crops were assayed during two consecutive seasons (2014 and 2015) in order to have more representative data.

The cultivations of **parsley** plants began with the transplant on September 24, 2014 and 2015, when the plant had a length of 5–6 cm. The seeds had been planted in seedbed 27 days before. In each root ball, between 12 and 16 seeds were deposited. The variety selected for the trial was the *Giant Italian Darkness* variety marketed by the seed company Diamond seeds. The frame of plantation used was in paired lines in quincunx at a distance of 90 cm between lines and 20 cm between plants.

The **dill** crop started with the transplant on October 4, 2014 and 2015, when the plant had a length of 5–6 cm. The seeds had been planted in seedbed 32 days before. In each root ball, 12–16 seeds were deposited. The variety selected for the trial was the *Aneto* variety marketed by the seed company Diamond seeds. The frame of plantation used was similar to parsley.

The **coriander** trials were developed in three different cycles, when making only one harvest per crop. The first cultivation of coriander started with direct sowing on September 30, 2014 and 2015. The variety selected for the trial was the *Salsa* variety marketed by the Sandrock seed company. The planting frame used was in line pairs in a 1.80 m culture table, planting 8 lines in the cultivation table, the sowing was done in steady flow.

The **mint** test was carried out on an implanted crop, starting after harvesting, on October 7, 2014 and 2015. The variety selected for the trial was a variety of local population, multiplied vegetatively. The frame of plantation used was in paired lines in quincunx at a distance of 90 cm between lines and 30 cm between plants.

Parsley, dill, coriander and mint were grown with high frequency localized irrigation (RLAF) using 16 mm diameter polyethylene pipes with drippers at a distance of 32 cm and a unit flow of 1.6 L/h, the discharge per hour of irrigation was 55.55 m^3 for parsley, dill and mint plants, and it was 111 m^3 in case of coriander. The fertilization was carried out with simple liquid fertilizers and the applied fertilization were 194, 179, 125 and 140 kg of N/ha, 105, 97, 70 and 85 kg/ha of P_2O_5 and 240, 216, 160 and 190 kg/ha of K_2O for parsley, dill, coriander and mint respectively. Most of the samples of the four herbs were collected weekly.

Table 1 showed the harvest dates of parsley, dill, coriander and mint, during the evolution of vegetative stage for two consecutive seasons (2014 and 2015).

It is necessary to mention that parsley, dill and coriander belong to the *Apiaceae* family, while mint plants belong to the *Lamiaceae* family. Although these two families have different types of gland formations that produce essential oils, comparisons have been made throughout the manuscript between these aromatic herbs, as well as with others belonging to different families, to obtain a global view of the effect of the harvest date.

2.2. Extraction of volatile compounds

Hydrodistillation (HD), using a Deryng system, was used for isolating the essential oil in fresh herbs (parsley, dill, coriander and mint). As described previously by El-Zaeddi et al. (2016) only 15 g of fresh

Table 2
Identification of volatile compound of parsley, dill coriander and mint.

Number	Compound	Rt[a] (min)	RI[a] (exp.)	RI[a] (lit.)	Parsley	Dill	Coriander	Mint
1	*trans*-2-Hexenal[b]	11.09	806	800		DV1		
2	Octane	12.12	808	800			CV1	
3	*α*-Thujene	13.19	899	905		DV2		
4	Santene	13.29	879	880				MV1
5	*α*-Pinene	13.58	906	909	PV1	DV3		
6	Camphene	14.38	944	945				MV2
7	Sabinene	14.89	975	975	PV2	DV4		
8	Myrcene	15.13	990	990	PV3	DV5		MV3
9	*β*-Pinene	15.23	995	990	PV4	DV6		MV4
10	*cis*-3-Hexenyl acetate	15.70	1008	1009	PV5		CV2	MV5
11	*α*-Phellandrene	16.13	1019	1013	PV6	DV7		
12	*α*-Terpinene	16.68	1031	1018				MV6
13	*p*-Cymene	16.88	1036	1034	PV7	DV8		MV7
14	Limonene	17.06	1040	1039	PV8	DV9	CV3	MV8
15	*β*-Phellandrene	17.23	1044	1034	PV9	DV10	CV4	
16	*trans-β*-Ocimene	17.38	1047	1047	PV10	DV11		MV9
17	γ-Terpinene	18.20	1066	1066	PV11			MV10
18	*trans*-Sabinene hydrate	19.00	1084	1087				MV11
19	Terpinolene	19.44	1095	1097	PV12	DV12		
20	Undecane	19.63	1100	1100		DV13	CV5	
21	Linalool	19.82	1103	1103			CV6	MV12
22	Nonanal	20.03	1107	1107			CV7	MV13
23	1,3,8-*p*-Menthatriene	20.74	1125	1115	PV13			
24	*cis*-Limonene oxide	21.87	1149	1140				MV14
25	*trans*-Limonene oxide	22.04	1153	1147				MV15
26	*cis-p*-Mentha-2,8-dien-1-ol	23.74	1192	1193				MV16
27	*trans-p*-Mentha-2,8-dien-1-ol	24.07	1199	1196				MV17
28	Dill ether	24.40	1196	1187		DV14		
29	*α*-Terpineol	24.74	1213	1200	PV14			
30	Decanal	24.79	1214	1207			CV8	
31	*cis*-Carveol	25.05	1220	1221				MV18
32	*trans*-Carveol	25.43	1228	1217				MV19
33	Carvone	27.13	1264	1262		DV15	CV9	MV20
34	E-2-Decenal	27.54	1273	1278			CV10	
35	1-Decanol	28.45	1292	1287			CV11	
36	Tridecane	29.06	1300	1300		DV16		
37	Bornyl acetate	29.10	1305	1291				MV21
38	Undecanal	29.63	1317	1310			CV12	
39	Carvomenthyl acetate	30.70	1339	1344				MV22
40	E-2-Undecenal	32.40	1375	1371			CV13	
41	1-Dodecanol (tent.)	33.88	1407	1386			CV14	
42	*β*-Bourbonene	34.08	1412	1407				MV23
43	Decyl acetate	34.13	1412	1410			CV15	
44	*β*-Caryophyllene	34.26	1416	1418				MV24
45	Dodecanal	34.39	1419	1420			CV16	
46	*trans*-β-Caryophyllene	35.68	1448	1455	PV15			MV25
47	Z-2-Dodecenal	36.37	1463	1467			CV17	
48	Alloaromadendrene	36.86	1475	1480				MV26
49	E-2-Dodecenal	37.12	1480	1468			CV18	
50	*α*-Humulene	37.48	1489	1489				MV27
51	E-2-Dodecen-1-ol	37.78	1495	1483			CV19	
52	1-Dodecanol	38.08	1502	1485			CV20	
53	Germacrene-D	38.38	1510	1516	PV16	DV17		
54	Tridecanal	38.96	1522	1518			CV21	
55	Nerolidol	38.97	1525	1528	PV17			
56	Myristicin	39.82	1535	1532	PV18	DV18		
57	E-2-Tridecenal	41.58	1582	1571			CV22	
58	1-Tetradecanol	42.81	1615	1618			CV23	
59	Tetradecanal	43.31	1632	1623			CV24	

[a] RT = retention time; RI = retention index; Exp. = experimental and Lit. = Literature.
[b] All compounds were identified using retention indexes, mass spectra and retention time of standards; SAFC (2015); www.pherobase.com; www.thegoodscentscompany.com.

material were required for a proper isolation of the volatile compounds. Briefly, 15.0 g of freshly chopped herbs shoots (aerial part of the plant, including stems and leaves) were put in a 500 mL round bottom flask, together with 1.0 g sodium chloride (NaCl), 150 mL of distilled water, and 50 μL of benzyl acetate as an internal standard (1470 mg L^{-1}) which were added before the hydrodistillation The total time of the hydrodistillation was of 60 min. All the extractions were conducted in triplicate.

2.3. Chromatographic analysis

Shimadzu GC-17A gas chromatograph coupled with a Shimadzu QP-5050A mass spectrometer detector (Shimadzu Corporation, Kyoto, Japan) was applied for the analysis of volatile compounds as described previously by El-Zaeddi et al. (2016). Briefly, the GC–MS system was equipped with a TRACSIL Meta. × 5 (95% dimethylpolysiloxane and 5% diphenylpolysiloxane) column (60 mx0.25 mm, 0.25 μm film thickness; Teknokroma S. Coop. C. Ltd, Barcelona, Spain). Analyses were carried out using helium as carrier gas at a column flow rate of 0.3 mL min^{-1} and a total flow of 3.9 mL min^{-1} in a split ratio of 1:11 and the following program: (a) 80 °C for 0 min; (b) increase of 3 °C min^{-1} from 80 °C to 210 °C and hold for 1 min; (c) increase of 25 °C min^{-1} from 210 °C to 300 °C and hold for 3 min. The temperatures of the injector and detector were 230 °C and 300 °C, respectively. The analysis was carried out from 45 to 400 m/z, with an electronic impact (EI) of 70 eV, in 0.5 scan/s mode. All compounds were identified using three different analytical methods: (i) comparison of experimental retention indexes (Equation (1)) (RI) with those of the literature; (ii) GC–MS retention times of authentic standards; and, (iii) mass spectra (authentic chemicals and NIST05 spectral library collection).

$$\boldsymbol{I} = 100\left[\boldsymbol{n} + (\boldsymbol{N}-\boldsymbol{n})\frac{\mathbf{log}\boldsymbol{t}_r^{'}(\boldsymbol{unknown}) - \mathbf{log}\boldsymbol{t}_r^{'}(\boldsymbol{n})}{\boldsymbol{log}\ \boldsymbol{t}_r^{'}(\boldsymbol{N}) - \mathbf{log}\boldsymbol{t}_r^{'}(\boldsymbol{n})}\right] \quad (1)$$

where,

n = number of carbon atoms in smaller alkane.

N = number of carbon atoms in larger alkane.

$t'_r(n)$ = adjust retention time of smaller alkane.

$t'_r(N)$ = adjusted retention time of larger alkane.

The semi-quantification of the volatile compounds was performed on a gas chromatograph, Shimadzu 2010, with a flame ionization detector (FID) as described previously by El-Zaeddi et al. (2016). Briefly, the column and chromatographic conditions were those previously reported for the GC–MS analysis. The injector temperature was 200 °C and nitrogen was used as carrier gas (1 mL min^{-1}). The quantification was obtained from electronic integration measurements using flame ionization detection (FID). Benzyl acetate (1000 mg L^{-1}) was added as internal standard at the beginning of the distillation procedure to simulate the behavior of volatile compounds; this chemical was used as an internal standard after checking that it was absent in herbs, it separates well from other volatiles, it possesses similar FID and MS response factors to most of the volatiles in the aromatic herb essential oil, it is stable at high temperatures, and does not react with water. Calibration curves were performed with the following compounds (Sigma-Aldrich, Madrid, Spain) as representative of each chemical family: α-phellandrene (monoterpenes), α-terpineol (terpenoids), trans-β-caryophyllene (sesquiterpenes), dill ether (terpene ethers), nonanal (aldehydes), myristicin (phenylpropanoids), bornyl acetate (esters), 1-decanol (alcohols), undecane (alkanes); the correlation coefficients (R^2) for all compounds were > 0.995, and results were expressed as mg kg^{-1} fresh weight, fw.

2.4. Statistical analysis

To compare the experimental data two consecutive tests were performed: (i) one-way analysis of variance (ANOVA) and (ii) Tukey's multiple range. Homogenous groups and the least significant difference (LSD) were determined at significance level of $p \leq 0.05$. Statgraphics Plus 5.0 software (Manugistics, Inc., Rockville, MD, USA) was the program used for the statistical analyses.

3. Results and discussion

3.1. Volatile composition of parsley oil as affected by harvest date

Eighteen compounds were identified by GC–MS (Table 2) in parsley shoots with an average volatile compounds yield of ≈992 mg kg^{-1} of fresh weight. 1,3,8-*p*-menthatriene, *β*-phellandrene, myristicin, terpinolene, myrcene, limonene, *α*-pinene, and *α*-phellandrene formed most of the composition of parsley aroma. Table 3 shows that 4 compounds (1,3,8-*p*-menthatriene, *β*-phellandrene, myristicin and myrcene) clearly dominated parsley aroma in all samples (P1-P16), representing 82–87% of the total concentration of volatile compounds. These results are largely consistent with those of Petropoulos et al. (2004) who reported that *β*-phellandrene, 1,3,8-*p*-menthatriene and *β*-myrcene were formed the major constituents of parsley oils obtained from leaves and stems of three types of parsley (turnip-rooted, plain leaf and curly leaf type) followed by myristicin and *α*-*p*-dimethylstyrene. Also, López et al. (1999) found that the main constitutes of parsley essential oil were *p*-1,3,8 menthatriene, *β*-phellandrene and apiole.

Results in Table 3 show that 1,3,8-*p*-menthatriene (85.8–1026 mg kg^{-1}, for P15 and P6 vegetative stages, respectively), *β*-phellandrene (75.5–637 mg kg^{-1}, for P15 and P6 vegetative stages, respectively), myristicin (13.1–410 mg kg^{-1} for P15 and P1 vegetative stages, respectively) and myrcene (14.4–165 mg kg^{-1} for P15 and P1 vegetative stages, respectively) were the most abundant constituents in all harvest stages (P1-P16). The highest concentrations of 1,3,8-*p*-menthatriene and *β*-phellandrene were found in P6 stage (9 weeks after planting date) while, myristicin and myrcene were in the maximum concentrations in P1 stage (4 weeks after planting date). Besides, the highest total concentrations of parsley shoots volatile compounds were 2543 and 2416 mg kg^{-1} in P6 and P1 respectively. The production of volatiles in parsley plant was found to be time-dependent. López et al. (1999) studied the effect of harvest time (5, 7, 9, 11 and 13 weeks) on the volatiles content of oil of parsley and their results indicated that *p*-1,3,8-menthatriene (0.62–398.8 ppm), *β*-phellandrene (44.9–255.1 ppm), and apiole (13.7–96.7 ppm) were the most abundant constituents in all stages, except for *p*-1,3,8-menthatriene, which was the lowest at 5 weeks (0.62 ppm). The essential oil of aerial parts of thyme (*Thymus vulgaris* L.) was analyzed at the beginning of the vegetative cycle, during the vegetative cycle, before the end of the cycle and after the cycle, determining that thyme collected before the end of the vegetative cycle, provided the best oil yield (1.2%) with also the highest % content of thymol 51.2% and carvacrol 4% (Hudaib et al., 2002). This last statement showed that the essential oils yield is strongly dependent on both the species and cultural practices.

3.2. Volatile composition of dill oil as affected by harvest date

Eighteen compounds were detected by GC–MS (Table 2) in dill shoots with an average volatile compounds yield of ≈1455 mg kg^{-1} of fresh weight. The eight main compounds in all samples were: *α*-phellandrene, dillether, *β*-phellandrene, limonene, *p*-cymene, *α*-pinene, trans-*β*-ocimene, and myristicin. In fact, *α*-phellandrene, dillether, *β*-phellandrene were represented 62–92% of the total concentrations of volatiles compounds of dill shoots in all samples (D1-D15). Rădulescu et al. (2010) analyzed the oil of dill leaves and they recorded that the main components in leaves oil were *α*-phellandrene (62.71 %), limonene (13.28 %) and dillether (16.42 %). The principal components of dill leaf oil were *α*-phellandrene (47.7–62.5 %), myristicin (1.7–28.2 %), dillether (0.9–14.8 %), *β*-phellandrene (7.4–7.5 %), and limonene (3.7–3.8 %) (Vokk et al., 2011).

Results tabulated in Table 4 indicate that the aroma of dill shoots was composed mainly from: *α*-phellandrene (357–1750 mg kg^{-1} for D5 and D15 vegetative stages, respectively), dillether (23.8–466 mg kg^{-1} for D5 and D10 vegetative stages, respectively) and *β*-phellandrene (46.1–230 mg kg^{-1} for D5 and D11 vegetative stages, respectively) in all harvest times (D1-D15). The highest concentration of *α*-phellandrene, dillether and *β*-phellandrene were obtained from D15, D10 and D11, respectively. In addition, the highest total concentrations of dill volatile compounds were found in D1 (2996 mg kg^{-1}), D11 (2620 mg kg^{-1}), D10 (2599 mg kg^{-1}) and D15 (2525 mg kg^{-1}). At three different growth stages (initial growth stage, before bud formation, and at flowering stages) aroma compounds of dill (*Anethum graveolens* L.) were detected. The content of limonene, dillether, and carvone increased, while the *α*-phellandrene, *β*-pellandrene, myristicin, and apiol contents decreased. These results indicated that the total amount of aroma compounds can change during the growth development (Huopalahti and Linko, 1983). In another study, the essential oil of Indian basil (*Ocimum basilicum* L.) was analyzed for four stages of harvest (40, 60,

Table 3
Volatile composition of parsley essential oil changing with harvest date at different vegetative stages of two consecutive seasons.

Compound	ANOVA†	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12	P13	P14	P15	P16
		Concentration (mg kg^{-1})															
PV1	***	41.9^{a‡}	9.22^{bc}	22.2^{b}	11.0^{bc}	7.49^{bc}	17.6^{bc}	2.70^{c}	12.2^{bc}	13.3^{bc}	11.4^{bc}	24.4^{b}	8.53^{bc}	4.14^{c}	3.16^{c}	2.29^{c}	9.86^{bc}
PV2	***	4.45^{a}	1.05^{bc}	1.71^{b}	0.81^{bc}	0.30^{bc}	1.70^{b}	0.146^{c}	1.08^{bc}	1.00^{bc}	0.69^{bc}	0.84^{bc}	0.38^{bc}	0.34^{bc}	0.08^{c}	0.03^{c}	0.54^{bc}
PV3	***	165^{a}	79.6^{abc}	100^{abc}	51.6^{c}	27.7^{c}	152^{ab}	21.5^{c}	66.1^{bc}	76.1^{bc}	47.1^{c}	73.4^{bc}	27.1^{c}	28.7^{c}	17.3^{c}	14.4^{c}	24.0^{c}
PV4	***	22.1^{a}	3.48^{cd}	9.25^{bc}	4.32^{bcd}	2.62^{cd}	6.64^{bcd}	0.380^{d}	3.76^{bcd}	6.07^{bcd}	4.38^{bcd}	10.6^{b}	4.14^{bcd}	1.07^{d}	1.05^{d}	0.99^{d}	3.68^{cd}
PV5	***	7.19^{ab}	8.10^{a}	3.04^{bc}	3.52^{abc}	1.71^{c}	2.96^{bc}	0.556^{c}	0.97^{c}	0.83^{c}	1.18^{c}	0.41^{c}	0.35^{c}	0.34^{c}	0.02^{c}	0.17^{c}	0.31^{c}
PV6	***	37.8^{ab}	16.8^{bc}	25.3^{abc}	12.2^{bc}	6.66^{c}	49.8^{a}	6.19^{c}	16.7^{bc}	15.9^{bc}	15.8^{bc}	17.7^{bc}	11.0^{bc}	9.63^{c}	5.59^{c}	3.73^{c}	8.48^{c}
PV7	**	4.54^{ab}	3.09^{ab}	4.62^{ab}	2.28^{ab}	1.48^{b}	6.19^{a}	1.06^{b}	3.95^{ab}	3.25^{ab}	2.74^{ab}	2.89^{ab}	1.41^{b}	1.17^{b}	0.74^{b}	0.79^{b}	1.75^{b}
PV8	***	59.9^{ab}	30.5^{abcd}	53.7^{abc}	27.9^{abcd}	13.1^{cd}	70.8^{a}	12.0^{cd}	30.0^{abcd}	27.7^{abcd}	26.5^{abcd}	32.0^{abcd}	11.3^{cd}	17.7^{bcd}	10.5^{cd}	5.59^{d}	13.7^{cd}
PV9	***	549^{ab}	234^{bc}	313^{abc}	169^{c}	103^{c}	637^{a}	95.3^{c}	240^{bc}	296^{abc}	289^{abc}	290^{abc}	122^{c}	140^{c}	88.4^{c}	75.5^{c}	110^{c}
PV10	***	9.94^{ab}	8.59^{ab}	10.0^{ab}	5.34^{ab}	3.03^{b}	12.8^{a}	2.43^{b}	6.72^{ab}	6.62^{ab}	3.42^{b}	4.80^{ab}	2.33^{b}	1.81^{b}	1.77^{b}	2.26^{b}	3.52^{b}
PV11	**	4.46^{a}	2.34^{ab}	3.28^{ab}	1.19^{ab}	0.47^{b}	3.66^{ab}	0.44^{b}	1.35^{ab}	0.99^{ab}	0.82^{b}	0.98^{ab}	0.29^{b}	0.61^{b}	0.34^{b}	0.10^{b}	0.30^{b}
PV12	***	198^{ab}	82.8^{bc}	101^{bc}	55.0^{c}	23.1^{c}	253^{a}	32.5^{c}	50.8^{c}	43.7^{c}	44.3^{c}	51.0^{c}	17.2^{c}	53.3^{c}	26.2^{c}	17.2^{c}	18.9^{c}
PV13	***	870^{ab}	560^{abc}	894^{ab}	476^{abc}	232^{bc}	1026^{a}	244^{bc}	391^{abc}	265^{bc}	386^{abc}	472^{abc}	159^{c}	317^{bc}	206^{bc}	85.8^{c}	195^{bc}
PV14	***	2.88^{a}	0.78^{bc}	1.68^{abc}	0.86^{bc}	0.26^{c}	2.39^{ab}	0.54^{bc}	2.52^{ab}	0.61^{bc}	0.34^{c}	0.12^{c}	0.40^{c}	0.37^{c}	0.02^{c}	0.03^{c}	0.83^{bc}
PV15	***	3.53^{b}	1.62^{b}	2.75^{b}	1.32^{b}	0.64^{b}	12.9^{a}	1.35^{b}	4.81^{b}	2.53^{b}	3.08^{b}	4.58^{b}	1.64^{b}	4.06^{b}	1.95^{b}	0.67^{b}	2.20^{b}
PV16	**	18.4^{ab}	11.4^{abc}	7.89^{abc}	3.67^{bc}	0.95^{c}	20.8^{a}	1.76^{bc}	7.32^{abc}	4.85^{abc}	1.68^{bc}	4.13^{abc}	1.64^{bc}	3.46^{bc}	0.98^{c}	0.50^{c}	1.37^{c}
PV17	***	7.94^{a}	1.20^{bc}	1.14^{bc}	0.77^{bc}	0.14^{bc}	3.24^{b}	0.15^{bc}	1.07^{bc}	0.41^{bc}	0.10^{bc}	0.14^{bc}	0.07^{c}	0.12^{bc}	0.00^{c}	0.00^{c}	0.21^{bc}
PV18	*	410^{a}	346^{ab}	100^{ab}	90.6^{ab}	45.0^{ab}	263^{ab}	55.8^{ab}	109^{ab}	99.1^{ab}	59.1^{ab}	147^{ab}	45.9^{ab}	68.2^{ab}	32.2^{ab}	13.1^{b}	24.4^{ab}
Total	***	2416^{ab}	1401^{abc}	1654^{abc}	918^{abc}	470^{c}	2543^{a}	479^{c}	949^{abc}	864^{bc}	898^{bc}	1137^{abc}	414^{c}	653^{c}	396^{c}	223^{c}	420^{c}

† *, ** and ***, significant at $p < 0.05$, 0.01 and 0.001, respectively.
‡ Values followed by the same letter, within the same column and factor (harvest date), were not significant different ($p < 0.05$), Tukey's multiple-range test.

80 and 100 days after transplanting) and the results showed that the highest oil content (0.73 % v/w) in fresh herb was obtained 60 days after transplanting and highest oil yield (132 kg ha^{-1}) was obtained in 80 days after transplanting harvest. It means that there are clearly effects of harvest date on the quality of oil of basil (Singh et al., 2010). Moreover, influence of harvesting stage on oil composition of rosemary (*Rosmarinus officinalis* L.) during vegetative, flower initiation, flowering and seed setting stages were investigated and maximum oil yield (110.94 kg ha^{-1}) was obtained in seed setting stage while, maximum oil content (1.17 %) was got in vegetative stage M. Singh and Guleria (2013). These last statements showed that the essential oils yield is strongly dependent on both the species and harvest time.

3.3. Volatile composition of coriander oil as affected by harvest date

Twenty-four compounds were detected (Table 2) in coriander shoots with an average volatile compounds yield of ≈323 mg kg^{-1} of fresh weight. The main seven volatile compounds of coriander oil were: decanal, *E*-2-dodecenal, 1-decanol, dodecanal, *E*-2-tridecenal, octane and undecanal. Decanal, *E*-2-dodecenal, 1-decanol and dodecanal were formed 47–82 % of the total concentration of volatile compounds in coriander oil in all stages of harvest (C1-C15). Nurzyńska-Wierdak (2013) reported that the oil from coriander (*Coriandrum sativum* L.) herb contained the highest amount of aliphatic aldehydes; decanal, *E*-2-dodecanol and *E*-2-decenol had the highest percentages.

The results obtained in the current study show that the lowest concentration of decanal, *E*-2-dodecenal, 1-decanol and dodecanal was 8.41, 9.24, 0.29 and 6.87 mg kg^{-1} in C14, C13, C9 and C13 while, the highest concentration of all these four compounds was found in C1 stage (Table 5). The highest total concentrations of volatile compounds were 2279, 531 and 340 mg kg^{-1} in C1, C2 and C3 vegetative stages, respectively. Ramezani et al. (2009) studied the changes in essential oil content of coriander (*Coriandrum sativum* L.) at different growth stages and they reported that the essential oils at vegetative, full flowering,

Table 4
Volatile composition of dill essential oil changing with harvest date at different vegetative stages of two consecutive seasons.

Compound	ANOVA†	D1	D2	D3	D4	D5	D6	D7	D8	D9	D10	D11	D12	D13	D14	D15
		Concentration (mg kg^{-1})														
DV1	***	0.24^{d‡}	1.22^{cd}	0.06^{d}	0.26^{d}	1.03^{cd}	0.16^{d}	0.92^{cd}	1.59^{cd}	4.77^{abcd}	6.65^{abc}	9.30^{a}	7.50^{ab}	2.73^{bcd}	1.90^{bcd}	2.23^{bcd}
DV2	***	5.24^{ab}	4.26^{abc}	2.90^{abc}	4.42^{abc}	1.16^{c}	1.44^{bc}	1.63^{bc}	4.08^{abc}	4.46^{abc}	5.90^{a}	5.69^{a}	2.99^{abc}	1.70^{bc}	1.74^{bc}	6.35^{a}
DV3	***	28.0^{ab}	21.1^{abc}	16.2^{abc}	22.3^{abc}	7.66^{c}	8.38^{c}	8.36^{c}	19.5^{abc}	23.4^{abc}	30.7^{a}	29.9^{a}	16.9^{abc}	9.53^{bc}	8.46^{c}	32.8^{a}
DV4	***	2.80^{a}	1.32^{abc}	0.94^{bc}	1.73^{abc}	0.40^{bc}	0.38^{c}	0.50^{bc}	1.43^{abc}	1.56^{abc}	1.62^{abc}	1.72^{abc}	0.78^{bc}	0.38^{c}	0.48^{bc}	2.12^{ab}
DV5	***	10.6^{a}	7.73^{ab}	5.79^{ab}	9.13^{ab}	2.37^{b}	2.57^{b}	2.95^{b}	6.82^{ab}	7.77^{ab}	11.2^{a}	11.5^{a}	5.93^{ab}	3.35^{b}	3.11^{b}	12.2^{a}
DV6	***	5.25^{a}	1.78^{b}	1.88^{b}	2.28^{ab}	0.74^{b}	0.70^{b}	0.51^{b}	1.75^{b}	1.44^{b}	1.34^{b}	1.42^{b}	0.89^{b}	0.44^{b}	0.29^{b}	0.86^{b}
DV7	***	1173^{ab}	985^{ab}	730^{b}	1028^{ab}	352^{b}	367^{b}	458^{b}	756^{b}	998^{ab}	1687^{a}	1735^{a}	1054^{ab}	581^{b}	465^{b}	1750^{a}
DV8	***	21.6^{bcd}	18.2^{bcd}	7.31^{cd}	15.2^{bcd}	18.6^{bcd}	13.1^{bcd}	7.40^{cd}	70.5^{a}	64.5^{a}	32.7^{b}	23.8^{bcd}	11.0^{cd}	17.9^{bcd}	3.78^{d}	27.3^{bc}
DV9	***	73.7^{ab}	56.4^{abc}	36.8^{abc}	54.2^{abc}	17.3^{c}	18.8^{c}	21.8^{c}	41.1^{abc}	50.2^{abc}	78.4^{a}	81.4^{a}	45.7^{abc}	25.9^{bc}	21.0^{c}	81.3^{a}
DV10	***	169^{abc}	139^{abc}	94.3^{bc}	141^{abc}	46.1^{c}	49.2^{c}	58.4^{c}	111^{abc}	137^{abc}	223^{ab}	230^{a}	132^{abc}	75.7^{c}	59.0^{c}	220^{ab}
DV11	***	2.87^{c}	5.76^{c}	3.15^{c}	7.88^{c}	3.88^{c}	5.55^{c}	6.34^{c}	8.89^{c}	12.0^{bc}	25.0^{ab}	29.7^{a}	16.3^{abc}	7.15^{c}	7.39^{c}	22.6^{ab}
DV12	***	2.11^{ab}	2.45^{ab}	4.49^{a}	4.65^{a}	2.28^{ab}	2.69^{ab}	1.43^{b}	1.85^{b}	1.05^{b}	1.72^{b}	1.70^{b}	0.55^{b}	0.34^{b}	0.32^{b}	1.36^{b}
DV13	***	47.1^{a}	28.3^{ab}	17.3^{b}	19.5^{ab}	6.47^{b}	4.94^{b}	3.59^{b}	3.13^{b}	4.11^{b}	10.5^{b}	11.9^{b}	2.70^{b}	3.48^{b}	1.12^{b}	2.73^{b}
DV14	***	38.5^{d}	101^{d}	50.7^{d}	77.0^{d}	23.8^{d}	47.9^{d}	82.2^{d}	140^{cd}	373^{ab}	466^{a}	426^{ab}	198^{bcd}	106^{d}	60.0^{d}	358^{abc}
DV15	**	14.9^{a}	0.01^{b}	0.01^{b}	0.00^{b}	0.00^{b}	0.02^{b}	0.53^{b}	0.14^{b}	0.02^{b}	0.01^{b}	0.02^{b}	0.00^{b}	0.01^{b}	0.03^{b}	0.56^{b}
DV16	***	12.4^{a}	5.83^{ab}	2.60^{b}	3.33^{b}	0.66^{b}	0.60^{b}	0.45^{b}	0.63^{b}	0.69^{b}	1.11^{b}	1.61^{b}	0.10^{b}	0.17^{b}	0.21^{b}	0.26^{b}
DV17	***	3.79^{bcd}	10.3^{abc}	5.75^{abcd}	7.72^{abcd}	0.42^{d}	4.96^{abcd}	2.01^{cd}	2.49^{cd}	2.77^{bcd}	13.2^{a}	11.8^{ab}	5.47^{abcd}	0.21^{d}	1.26^{cd}	3.41^{bcd}
DV18	***	614^{a}	275^{b}	111^{b}	109^{b}	19.2^{b}	15.0^{b}	10.3^{b}	33.2^{b}	19.0^{b}	3.65^{b}	7.39^{b}	0.00^{b}	0.06^{b}	0.01^{b}	0.00^{b}
Total	***	2225^{ab}	1665^{abc}	1091^{abc}	1507^{abc}	504^{c}	543^{c}	667^{bc}	1204^{abc}	1706^{abc}	2599^{a}	2619^{a}	1501^{abc}	836^{bc}	635^{c}	2525^{a}

† *, ** and ***, significant at $p < 0.05$, 0.01 and 0.001, respectively.
‡ Values followed by the same letter, within the same column and factor (harvest date), were not significant different ($p < 0.05$), Tukey's multiple-range test.

Table 5
Volatile composition of coriander essential oil changing with harvest date at different vegetative stages of two consecutive seasons.

Compound	ANOVA[†]	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11	C12	C13	C14	C15
		Concentration (mg kg^{-1})														
CV1	***	46.6^{a‡}	19.3^{bc}	21.4^{abc}	25.5^{abc}	16.7^{bc}	43.7^{ab}	5.08^{c}	21.4^{abc}	20.4^{abc}	30.1^{abc}	21.2^{abc}	19.0^{bc}	9.17^{c}	9.41^{c}	18.0^{bc}
CV2	***	11.4^{a}	1.76^{b}	0.24^{b}	1.02^{b}	0.64^{b}	0.50^{b}	0.13^{b}	0.53^{b}	0.93^{b}	1.90^{b}	1.19^{b}	0.96^{b}	0.47^{b}	0.59^{b}	1.09^{b}
CV3	*	1.13^{a}	2.90^{a}	2.43^{a}	2.24^{a}	1.02^{a}	0.86^{a}	0.07^{a}	0.27^{a}	0.21^{a}	0.37^{a}	0.16^{a}	0.00^{a}	0.09^{a}	0.15^{a}	0.00^{a}
CV4	***	0.61^{ab}	0.07^{b}	0.03^{b}	0.00^{b}	0.04^{b}	0.25^{b}	0.10^{b}	0.36^{b}	0.47^{b}	0.20^{b}	0.13^{b}	0.00^{b}	0.02^{b}	0.03^{b}	1.32^{a}
CV5	***	0.92^{abcdef}	0.20^{ef}	0.52^{cdef}	0.78^{bcdef}	0.43^{def}	1.86^{a}	0.26^{ef}	1.51^{abc}	1.47^{abcd}	1.64^{ab}	1.15^{abcde}	0.30^{ef}	0.08^{f}	0.03^{f}	0.35^{ef}
CV6	***	4.61^{a}	0.66^{bc}	0.40^{bc}	0.23^{bc}	0.06^{c}	0.85^{b}	0.09^{c}	0.22^{bc}	0.10^{c}	0.03^{c}	0.15^{c}	0.09^{c}	0.00^{c}	0.00^{c}	0.05^{c}
CV7	***	1.42^{a}	0.05^{c}	0.12^{c}	0.05^{c}	0.01^{c}	0.16^{c}	0.00^{c}	0.18^{c}	0.26^{bc}	1.51^{a}	0.93^{ab}	0.10^{c}	0.37^{bc}	0.04^{c}	0.21^{c}
CV8	***	692^{a}	213^{b}	94.6^{c}	34.1^{c}	28.9^{c}	99.5^{c}	17.1^{c}	51.1^{c}	20.9^{c}	27.9^{c}	26.4^{c}	30.9^{c}	11.0^{c}	8.41^{c}	36.4^{c}
CV9	NS	7.93	5.73	8.99	2.70	2.83	2.84	0.16	1.04	0.10	0.03	0.01	0.03	0.06	0.05	0.35
CV10	***	48.1^{a}	4.85^{b}	1.03^{b}	0.06^{b}	0.26^{b}	1.17^{b}	0.11^{b}	2.64^{b}	0.42^{b}	1.52^{b}	0.42^{b}	3.40^{b}	0.03^{b}	0.04^{b}	1.49^{b}
CV11	***	333^{a}	51.2^{b}	17.8^{c}	5.09^{c}	3.44^{c}	22.1^{bc}	1.85^{c}	0.64^{c}	0.29^{c}	2.46^{c}	5.05^{c}	2.51^{c}	1.95^{c}	1.83^{c}	6.57^{c}
CV12	***	29.8^{a}	9.82^{b}	9.73^{b}	5.60^{bc}	2.14^{bc}	9.61^{b}	1.49^{c}	3.93^{bc}	3.56^{bc}	5.05^{bc}	3.96^{bc}	5.04^{bc}	1.64^{c}	2.29^{bc}	6.66^{bc}
CV13	***	8.77^{a}	1.54^{b}	0.78^{b}	0.10^{b}	0.01^{b}	0.64^{b}	0.03^{b}	0.04^{b}	0.03^{b}	0.23^{b}	0.44^{b}	0.40^{b}	0.08^{b}	0.32^{b}	0.75^{b}
CV14	***	7.48^{a}	0.87^{b}	0.63^{bc}	0.18^{cd}	0.05^{d}	0.26^{cd}	0.09^{cd}	0.33^{bcd}	0.19^{cd}	0.22^{cd}	0.10^{cd}	0.03^{d}	0.01^{d}	0.01^{d}	0.05^{d}
CV15	***	4.97^{a}	0.09^{b}	0.08^{b}	0.09^{b}	0.01^{b}	0.12^{b}	0.01^{b}	0.01^{b}	0.02^{b}	0.01^{b}	0.02^{b}	0.01^{b}	0.00^{b}	0.01^{b}	0.00^{b}
CV16	***	288^{a}	57.3^{bc}	51.0^{bc}	33.3^{c}	23.5^{c}	105^{b}	14.6^{c}	44.3^{bc}	37.7^{bc}	39.0^{bc}	24.9^{c}	20.2^{c}	6.87^{c}	8.98^{c}	17.4^{c}
CV17	***	8.64^{a}	1.24^{bc}	1.53^{b}	0.65^{bc}	0.11^{c}	0.95^{bc}	0.13^{c}	0.56^{bc}	0.19^{c}	0.36^{bc}	0.26^{bc}	0.04^{c}	0.01^{c}	0.01^{c}	0.10^{c}
CV18	***	389^{a}	94.4^{b}	74.4^{bc}	30.9^{bcd}	14.5^{cd}	69.5^{bcd}	10.1^{d}	49.1^{bcd}	48.6^{bcd}	59.0^{bcd}	41.1^{bcd}	21.3^{cd}	9.24^{d}	12.2^{cd}	25.9^{cd}
CV19	***	116^{a}	21.3^{b}	15.2^{bcd}	6.27^{cde}	1.78^{de}	18.3^{bc}	4.39^{de}	0.64^{e}	0.48^{e}	1.15^{e}	0.97^{e}	0.02^{e}	0.01^{e}	0.02^{e}	0.04^{e}
CV20	***	46.2^{a}	7.00^{bc}	8.53^{bc}	4.27^{c}	1.79^{c}	15.0^{b}	0.55^{c}	0.10^{c}	0.03^{c}	0.05^{c}	0.20^{c}	0.02^{c}	0.01^{c}	0.14^{c}	0.01^{c}
CV21	***	18.2^{a}	2.61^{bc}	5.06^{bc}	4.02^{bc}	1.86^{bc}	8.52^{b}	0.46^{c}	1.35^{c}	0.82^{c}	1.19^{c}	1.20^{c}	0.80^{c}	0.13^{c}	1.02^{c}	1.17^{c}
CV22	***	47.4^{a}	8.16^{bc}	5.32^{bc}	2.49^{c}	1.26^{c}	13.6^{b}	1.07^{c}	5.82^{bc}	3.89^{c}	5.88^{bc}	4.70^{bc}	4.44^{bc}	2.01^{c}	2.60^{c}	4.54^{bc}
CV23	**	16.1^{b}	1.00^{b}	1.59^{b}	0.65^{b}	0.11^{b}	1.70^{b}	0.12^{b}	0.74^{b}	85.1^{a}	0.30^{b}	0.26^{b}	0.10^{b}	0.02^{b}	0.05^{b}	0.08^{b}
CV24	***	63.1^{a}	5.70^{bc}	5.59^{bc}	3.60^{bc}	1.56^{c}	16.5^{b}	0.88^{c}	3.41^{bc}	1.32^{c}	1.80^{c}	2.02^{c}	1.43^{c}	0.31^{c}	0.83^{c}	0.83^{c}
Total	***	2191^{a}	511^{b}	327^{bcd}	164^{cde}	103de	433^{bc}	58.9^{de}	190^{cde}	227^{cde}	182^{cde}	137de	111de	43.5^{e}	50.0^{de}	123de

† *, ** and ***, significant at $p < 0.05$, 0.01 and 0.001, respectively.

‡ Values followed by the same letter, within the same column and factor (harvest date), were not significant different ($p < 0.05$), Tukey's multiple-range test.

green fruit (immature) and brown fruit (mature) were 0.14%, 0.23%, 0.37% and 0.31% (w/w) respectively. Naghdi Badi et al. (2004) investigated the effects of harvesting time on herbage yield and quality/quantity of oil in thyme, *Thymus vulgaris* L. and their results reported that the maximum yield and content of oil and thymol can be obtained when thyme harvest at beginning of blooming stage. Similar trend was observed in current results. This last statement showed that the essential oils yield is strongly dependent on both the species and cultural practices, considering that the selection of the proper harvest date is also an agricultural practice.

3.4. Volatile composition of mint oil as affected by harvest date

Based on GC–MS analysis, twenty seven compounds were identified in mint shoots (Table 2) with an average volatile compounds yield of ≈6909 mg kg^{-1} of fresh weight. Carvone (1465–17092 mg kg^{-1} for M7 and M5 vegetative stages, respectively), limonene (282–4525 mg kg^{-1} for M7 and M5 vegetative stages, respectively), *trans*-carveol (3.57–322 mg kg^{-1} for M13 and M5 vegetative stages, respectively), *cis*-carveol (17.1–300 mg kg^{-1} for M7 and M5 vegetative stages, respectively), β-pinene (9.57–155 mg kg^{-1} for M7 and M5 vegetative stages, respectively), santene (8.58–137 mg kg^{-1} for M7 and M5 vegetative stages, respectively), *trans*-caryophyllene (9.35–119 mg kg^{-1} for M13 and M1 vegetative stages, respectively), trans-β-ocimene (6.90–101 mg kg^{-1} for M13 and M5 vegetative stages, respectively) and myrcene (4.07–101 mg kg^{-1} for M13 and M5 vegetative stages, respectively) represented the most abundant in the content of mint volatile compounds in all harvest stages (Table 6), while carvone and limonene were the main volatile compounds of mint shoots (87–94%). Peppermint (*Mentha x piperita* L.) and chocolate mint (*Mentha x piperita* f. citrata 'chocolate') oils were analyzed by Tsai et al. (2013) and they reported that the major components of peppermint essential oil were menthol (30.35%), menthone (21.12%), and trans-carane (10.99%), while for chocolate mint essential oil, the major components were menthol (28.19%), menthone (15.53%) and 1,8-cineole (11.89%). A previous research by de Sousa Barros et al. (2015) reported the chemical composition of different *Mentha* species and recorded that D-carvone (58.79%), and limonene (28.29%) were the main constituents of essential oil of *Mentha aquatica* (lavender mint). The *Mentha spicata*, menthol mint gh variety, had a higher content D-carvone (60.07%) and limonene (19.91%). The *Mentha spicata*, large leaf spearmint variety, and *Mentha spicata*, homegrown mint variety, presented as major components D-carvone (31.35%) and (54.94%), and limonene (22.10%) and (28.81%), respectively. These results are largely consistent with the results obtained in the present study and confirm the variation of the volatile composition according the different species and varieties.

The highest concentration of carvone (17,092 mg kg^{-1}) and limonene (4525 mg kg^{-1}) was found in M5. While, the lowest concentration of carvone (1465 mg kg^{-1}) and limonene (282 mg kg^{-1}) were obtained from M7. Zawiślak (2011) studied the effect of plant harvest term (mid-June–plants in vegetative phase, mid-July–beginning of flowering, mid-August–full blooming, mid-September–after flowering) on the composition of hyssop (*Hyssopus officinalis* L.) oil and results revealed that the highest oil content in fresh raw material was found in the herb collected from plants in full flowering (0.25%) and at the beginning of flowering (0.22 %). There is a large variation of volatile compounds of aromatic herbs composition in relation to harvest time. Moreover, oil content and composition of rosemary (*Rosmarinus officinalis* L.) were influenced by harvesting stage; highest oil yield was found in seed setting stage (110.94 kg ha^{-1}) where, highest oil content (1.17 %) was determined in vegetative stage (Singh and Guleria, 2013).

4. Conclusions

Volatile composition of parsley (*Petroselinum crispum*), dill (*Anethum graveolens*), coriander (*Coriander sativum*) and mint (*Mentha piperita*) was influenced by harvesting date along the different vegetative stages. The highest total concentration of parsley volatile compounds was found in P6 vegetative stage (9 weeks after planting date) with a maximum concentration of 2543 mg kg^{-1}, for dill, the highest content of volatile compounds was obtained in D15 (19 weeks after planting

Table 6
Volatile composition of mint essential oil changing with harvest date at different vegetative stages of two consecutive seasons.

Compound	ANOVA[†]	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	M11	M12	M13	M14	M15
		Concentration (mg kg^{-1})														
MV1	***	122^{ab} [b]	65.3^{cd}	46.8^{cdef}	56.0^{cde}	137^{a}	82.6^{bc}	8.58^{f}	23.1^{def}	23.0^{def}	17.3^{ef}	16.6^{ef}	17.5^{ef}	8.82^{f}	24.1^{def}	23.8^{def}
MV2	***	8.55^{bc}	4.89^{cde}	3.84^{def}	5.11^{cd}	13.1^{a}	9.10^{b}	0.95^{ef}	2.20^{def}	2.27^{def}	1.80^{def}	1.44^{def}	1.28^{def}	0.83^{f}	2.76^{def}	3.49^{def}
MV3	***	98.4^{a}	55.7^{bc}	34.8^{cde}	47.0^{bcd}	101^{a}	74.2^{ab}	7.18^{e}	12.6^{de}	13.8^{de}	9.73^{e}	9.62^{e}	8.70^{e}	4.07^{e}	12.5^{de}	13.0^{de}
MV4	***	137^{ab}	77.8^{cd}	52.7^{cdef}	63.0^{cde}	155^{a}	95.6^{bc}	9.57^{f}	24.1^{ef}	25.3^{ef}	19.3^{ef}	19.0^{ef}	21.0^{ef}	10.4^{f}	30.6^{def}	27.9^{ef}
MV5	***	6.49^{ab}	5.68^{b}	4.53^{bc}	6.64^{ab}	10.5^{a}	6.39^{ab}	1.11^{c}	0.58^{c}	0.92^{c}	0.75^{c}	0.63^{c}	0.40^{c}	0.13^{c}	0.84^{c}	0.63^{c}
MV6	**	11.0^{a}	6.35^{a}	4.26^{a}	5.10^{a}	11.2^{a}	5.46^{a}	0.69^{a}	1.29^{a}	1.58^{a}	0.95^{a}	0.68^{a}	11.7^{a}	0.47^{a}	1.37^{a}	2.37^{a}
MV7	***	2.87^{ab}	1.94^{abc}	1.59^{bc}	1.32^{bc}	3.69^{a}	1.80^{bc}	0.31^{c}	0.51^{c}	0.79^{c}	0.40^{c}	0.39^{c}	0.66^{c}	0.42^{c}	1.97^{abc}	2.74^{ab}
MV8	***	2902^{b}	2101^{bcd}	1493^{cdef}	1663^{bcde}	4525^{a}	2527^{bc}	282^{f}	609^{ef}	712^{ef}	498^{ef}	476^{ef}	600^{ef}	317^{f}	805^{def}	722^{ef}
MV9	***	70.3^{ab}	43.2^{bc}	30.7^{cd}	36.6^{cd}	101^{a}	92.4^{a}	9.08^{d}	19.7^{cd}	21.1^{cd}	18.3^{cd}	15.7^{cd}	13.9^{cd}	6.90^{d}	19.7^{cd}	17.9^{cd}
MV10	***	5.70^{ab}	3.99^{abcd}	2.71^{bcd}	2.58^{bcd}	7.24^{a}	5.73^{ab}	0.78^{d}	1.30^{cd}	1.56^{cd}	1.38^{cd}	1.34^{cd}	1.49^{cd}	0.97^{cd}	4.16^{abc}	5.01^{ab}
MV11	***	64.3^{cd}	39.4^{cde}	24.1^{de}	37.4^{cde}	113^{ab}	123^{a}	14.5^{e}	35.2^{cde}	44.8^{cde}	33.6^{cde}	33.8^{cde}	33.4^{cde}	19.1^{e}	74.5^{bc}	75.8^{bc}
MV12	***	37.0^{bc}	23.7^{bcd}	17.0^{d}	23.1^{bcd}	57.5^{a}	38.0^{ab}	5.08^{d}	10.6^{d}	12.4^{d}	10.1^{d}	8.95^{d}	8.89^{d}	4.57^{d}	17.4^{cd}	12.1^{d}
MV13	***	6.88^{a}	4.25^{bc}	2.25^{cde}	3.21^{cd}	6.91^{a}	6.15^{ab}	0.75^{de}	1.28^{de}	1.71^{de}	1.37^{de}	1.27^{de}	1.06^{de}	0.63^{e}	2.20^{cde}	2.22^{cde}
MV14	***	6.96^{ab}	4.95^{bc}	2.69^{cde}	3.87^{bcd}	8.59^{a}	6.83^{ab}	0.61^{e}	1.31^{de}	1.53^{de}	1.09^{de}	0.90^{de}	1.05^{de}	0.82^{de}	1.59^{de}	1.17^{de}
MV15	***	19.3^{ab}	11.7^{cd}	7.45^{def}	8.45^{cde}	23.7^{a}	14.9^{bc}	1.50^{f}	2.88^{ef}	3.05^{ef}	2.05^{ef}	1.78^{f}	2.21^{ef}	1.51^{f}	3.40^{ef}	1.79^{f}
MV16	***	0.00^{e}	0.00^{e}	0.24^{de}	0.45^{de}	0.00^{e}	0.00^{e}	0.00^{e}	6.54^{a}	6.70^{a}	4.30^{b}	3.52^{bc}	3.45^{bc}	2.26^{cd}	6.48^{a}	6.45^{a}
MV17	***	16.0^{a}	11.9^{abc}	6.88^{bcd}	6.75^{cd}	16.6^{a}	14.8^{ab}	1.81^{d}	4.54^{cd}	5.34^{cd}	4.80^{cd}	4.44^{cd}	4.39^{cd}	2.94^{d}	12.3^{abc}	11.0^{abc}
MV18	***	107^{bcde}	59.3^{def}	36.7^{ef}	63.8^{def}	300^{a}	148^{bc}	17.1^{f}	67.6^{def}	74.7^{def}	116^{bcd}	87.0^{bcdef}	59.7^{def}	32.6^{f}	157^{b}	82.1^{cdef}
MV19	***	170^{abc}	141^{abc}	79.2^{c}	137^{bc}	322^{a}	312^{ab}	26.0^{c}	8.35^{c}	7.47^{c}	4.85^{c}	4.77^{c}	4.82^{c}	3.57^{c}	8.12^{c}	8.99^{c}
MV20	***	9745^{b}	8257^{b}	5453^{bc}	6035^{bc}	17092^{a}	10315^{b}	1465^{c}	2533^{c}	2784^{c}	2021^{c}	1844^{c}	1901^{c}	1071^{c}	2592^{c}	1877^{c}
MV21	***	0.46^{c}	0.13^{c}	0.49^{c}	1.51^{bc}	6.59^{a}	2.96^{b}	0.37^{c}	0.54^{c}	0.92^{c}	0.88^{c}	0.67^{c}	0.38^{c}	0.13^{c}	0.72^{c}	0.45^{c}
MV22	***	14.2^{bc}	9.09^{c}	13.0^{bc}	12.0^{bc}	111^{a}	37.8^{b}	8.29^{c}	13.3^{bc}	9.13^{c}	26.6^{bc}	22.0^{bc}	19.2^{bc}	4.85^{c}	30.7^{bc}	15.5^{bc}
MV23	***	62.1^{a}	38.3^{bc}	21.5^{cde}	28.4^{cd}	59.8^{ab}	52.5^{ab}	6.01^{de}	14.7^{de}	17.4^{cde}	10.6^{de}	7.78^{de}	9.22^{de}	5.56^{e}	15.9^{cde}	14.0^{de}
MV24	***	24.9^{a}	17.3^{b}	9.20^{cd}	9.07^{cd}	18.8^{ab}	14.7^{bc}	1.57^{e}	2.24^{e}	3.35^{de}	1.99^{e}	1.62^{e}	2.34^{e}	1.39^{e}	3.47^{de}	3.63^{de}
MV25	***	117^{a}	74.4^{abc}	37.1^{bcd}	53.0^{bcd}	99.7^{a}	81.8^{ab}	11.7^{d}	22.2^{d}	31.8^{cd}	19.3^{d}	16.3^{d}	18.3^{d}	9.35^{d}	26.9^{d}	35.5^{cd}
MV26	***	9.22^{ab}	6.65^{bc}	3.98^{cde}	5.70^{bcd}	12.5^{a}	6.55^{bc}	1.08^{e}	1.73^{e}	2.61^{de}	0.97^{e}	0.75^{e}	1.21^{e}	0.58^{e}	1.61^{e}	2.56^{de}
MV27	***	21.6^{a}	13.7^{abc}	7.01^{cde}	9.72^{bcd}	15.7^{ab}	14.5^{abc}	2.08^{de}	3.55^{de}	5.01^{de}	2.80^{de}	2.31^{de}	2.50^{de}	1.48^{e}	3.44^{de}	5.09^{de}
Total	***	13785b	11079b	7397^{bc}	8325^{bc}	23329^{a}	14090^{b}	1884^{c}	3423^{c}	3815^{c}	2829^{c}	2583^{c}	2750^{c}	1512^{c}	3861^{c}	2974^{c}

[†] *, ** and ***, significant at $p < 0.05$, 0.01 and 0.001, respectively.
[b] ǂ Values followed by the same letter, within the same column and factor (harvest date), were not significant different ($p < 0.05$), Tukey's multiple-range test.

date) with a total concentration of essential oil composition of 2525 mg kg^{-1}. Coriander showed different behavior and its volatile compounds content was in highest concentration 3 weeks after planting date (C1 vegetative stages with 2191 mg kg^{-1}), while concentration of mint volatile compounds was maximum in M5 vegetative stage (6 weeks after planting date) with a total concentration of 23,329 mg kg^{-1}.

The information reported in current publication provides important and useful information for worldwide farmers since the harvest date according to the content and composition of essential oils has been optimized; however, further investigations must be conducted in order to correlate the highest concentration of essential oils to production in terms of Tn ha^{-1} as well as the sensory quality.

Author statement

Hussein El-Zeddi, Ángel Calín-Sánchez, Luis Noguera-Artiaga, Juan Martínez Tomé, Ángel A. Carbonell-Barrachina planned and designed the experiments; Hussein El-Zaeddi and Ángel Calín-Sánchez performed the experiments; Hussein El-Zeddi, Ángel Calín-Sánchez, Luis Noguera-Artiaga, Juan Martínez Tomé, Ángel A. Carbonell-Barrachina analyzed the data; Hussein El-Zeddi, Ángel Calín-Sánchez, Luis Noguera-Artiaga wrote the manuscript and Hussein El-Zeddi, Ángel Calín-Sánchez, Luis Noguera-Artiaga and Ángel A. Carbonell-Barrachina edited the manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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