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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, for coriander was 2191 mg kg<sup>-1</sup> 3 weeks after planting date, and 23329 mg kg<sup>-1</sup> 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<sup>-2</sup>) were obtained in the second harvest time (Zawiślak and

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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
Р9	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						

 Table 1

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

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  $(12.6 \text{ t ha}^{-1})$  and dry leaf yield  $(7.47 \text{ t ha}^{-1})$  were obtained after full flowering stage (Kizil and Toncer, 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<sup>3</sup> for parsley, dill and mint plants, and it was 111 m<sup>3</sup> 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

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

1max-bachandia110868091	Number	Compound	Rt <sup>a</sup> (min)	RI <sup>a</sup> (exp.)	RI <sup>a</sup> (lit.)	Parsley	Dill	Coriander	Mint
2     Orans     12.12     808     800     P1     CP1       4     Smeen     13.39     899     905     P2     P3       4     Smeen     13.39     899     905     P2     P3     M7       6     Camphene     13.38     944     945     P2     P4     M7       7     Shinnee     13.38     940     900     P4     P15     M72       8     Myrease     15.13     900     900     P4     P15     M7       10     ar-Shinney locatine     15.01     1013     P14     P15     M70       11     ar-Pathankara     15.13     1019     1013     P4     P7     P35     M70       11     ar-Pathankara     15.13     1019     1013     P47     P47     M70     M71     M70       13     ar-Cyman     15.68     1036     1034     P70     D81     M74     M70       14     immenen     17.66     1064     1034     P79     D910     C/4     M74       15     p-Fhellandrene     17.23     1044     1034     P71     P11     M74       17     rare-Solinee hydrate     13.20     1066     1066 <td< td=""><td>1</td><td>trans-2-Hexenal<sup>b</sup></td><td>11.09</td><td>806</td><td>800</td><td></td><td>DV1</td><td></td><td></td></td<>	1	trans-2-Hexenal <sup>b</sup>	11.09	806	800		DV1		
31     shranen     1.19     89     96     97     97     M2     M1       5     shranen     1.38     96     90     P1     93     M2       7     Sahlanen     1.38     96     90     P1     93     M3       7     Sahlanen     1.38     95     97     P2     P3     M3       9     \$\$hrange netrice     1.53     95     90     P4     P4     M3       9     \$\$hrange netrice     1.53     95     90     P4     P4     M3       9     \$\$hrange netrice     1.53     90     90     P4     P4     M3       10     \$\$hrange netrice     1.68     101     103     P4     M3       11     \$\$regen netrice     1.68     104     103     P4     P4       14     Innonen     1.68     104     103     P4     P4       14     Innonen     1.68     104     103     P4     P4       15     Innonen     1.64     104     103     P4     P4       16     Innonen     1.64     104     104     P4     P4       16     Innoh     1.64     104     104     P4     P4<	2	Octane	12.12	808	800			CV1	
4     Samear     1.29     89     80	3	α-Thujene	13.19	899	905		DV2		
5a-Phane1.3.8.906907P1/2P1/2P/2 <td>4</td> <td>Santene</td> <td>13.29</td> <td>879</td> <td>880</td> <td></td> <td></td> <td></td> <td>MV1</td>	4	Santene	13.29	879	880				MV1
6Camphene14.38944945975972PV2PV3PV3PV38Myrcone15.13990990PV3PV3PV3MV310cic3-iteexny isertate15.73990PV3PV3PV3MV311cir1pinene16.8110191013PV7PV8MV612circpinene16.8810311014PV7PV8MV613Pr7PV8DV1MV6MV6MV614circpinene17.2310441034PV7PV8MV1015pi-belandmene17.2310471047PV10DV10CV4MV1016pi-belandmene17.2310641067PV12DV12CV4MV1017Terpisohene hydrate19.43103103103TMV1018Terpisohene hydrate19.43103103TMV12MV1220Undecane19.4311031107TMV12MV1221Linabole19.4211251115P113MV12MV1422rome-hydrateine20.4711251136P114MV1423rome-hydrateine20.4711261137MV14MV1424rome-hydrateine20.4711261137MV14MV1425rome-hydrateine20.4711261147MV14MV1426rome-hydrat	5	α-Pinene	13.58	906	909	PV1	DV3		
7     Subineen     14.89     975     975     P12     DV4     VV       9     \$\beta\$Piresen     15.33     990     970     PV3     DV5     MV4       9     \$\beta\$Piresen     15.23     995     970     PV3     DV5     MV4       11     \$\beta\$Piresen     16.13     1019     1013     PV5     V     MV3       12     \$\beta\$Piresen     16.68     1031     1018     MV7     MV3     MV7       13     \$\beta\$Piresen     17.25     1044     1034     PV3     DV3     CV3     MV3       15     \$\beta\$Piresen     17.25     1044     1034     PV1     DV1     MV1       16     \$\perp\$Piresen     17.25     1044     1034     PV1     DV1     MV1       17     \$\perp\$Piresen     19.66     1067     PV1     DV13     CV3     MV1       17     \$\merp\$Piresen     19.63     1107     1007     PV1     MV13     MV14       18     \$\beta\$Piresen     1103     1103     1103     PV1     MV14       19     1102     1103     1107     PV13     MV14       10     \$\beta\$Piresen     1103     1107     PV14     <	6	Camphene	14.38	944	945				MV2
8Myrene15.13990990PV3PV3VV3MV310cic3-iteequijacetate15.7310081009PV5V2MV311cir5-iteequijacetate15.7010081013PV5V7MV512cir5-iteequijacetate16.8410311013PV7DV8MV713cir5-iteequijacetate16.8410311013PV7DV8MV714cir5-iteequijacetate17.2810441034PV7DV10CV4MV1015promis-focimer17.2810471047PV10DV10CV4MV1016promis-focimer18.2010641066PV11V12V12V1217Terpisolen19.431004107PV12DV12V12V12V11218Terpisolen19.4311031107V13V14V113MV1520tindecane19.4311031107V14V14V11421tindecane20.4711251115P113V14V11422tindecane20.4711241107V14V14V11423tindecane20.4711251135P113V14V11424tindecane20.4711241127V14V14V11425tindecane20.471124120V14V14V1426tindecane20.47124	7	Sabinene	14.89	975	975	PV2	DV4		
9β-Finene15.23950970PV3V60V7MV311a-Phellandrene16.3310191013PV6PV7MV512a-Terpinene16.6810341018MV5MV713p-Cyrattee16.6810341018PV7DV8MV714Linonartee17.2610441039PV8DV10CV3MV815β-Phallandrene17.2310441034PV8DV10CV4MV916mrs/f-Cinnene17.2310441034PV19DV10CV4MV1017mrs/f-Cinnene17.2310441046PV1MV10MV10MV1118Terpinolene19.4310061007PV1V11MV1120Unalexane19.4311031107PV1V11V11V1121Unalexane19.4311021115PV13V11MV1222Nonal20.7411421147MV14MV1424ci-Jamene oxide21.2411321147MV1425mrs-Jamene oxide21.2411231126V14V1426ci-Pamelhalba-2-6-den-124.341231200PV14V1426ci-Pamelhalba-2-6-den-124.34123120PV14V1427mrs-Jamene oxide24.34123120PV14V1427mrs-Jamene Oxide24.3	8	Myrcene	15.13	990	990	PV3	DV5		MV3
10cis.3-Haennyl accitate15.7010081009PV5CV2MV512a "Terpinene16.6810311013	9	$\beta$ -Pinene	15.23	995	990	PV4	DV6		MV4
11     α-Phellandrene     16.68     1031     1018     V7     M7V       13     p-Cymene     16.68     1034     1018     W7     M7V       14     Linonene     17.06     1040     1039     PV8     DV8     M7V       15     β-Phellandrene     17.23     1044     1034     PV9     DV10     CV4       16     mrs/-Colinene     17.23     1044     1047     PV10     DV10     CV4       17     n-Terpinene     18.20     1066     1066     PV11     M7V1       18     mrs-Subinen bydrate     19.00     1084     1097     PV2     DV12     V12       21     Landool     19.82     1103     1107     CV6     M713       23     1.3.8.9-Menthat/Schenel     20.43     1137     1107     CV6     M714       24     ci-Lannonec oxide     21.47     1125     1115     PV13     M714       25     mrs-Subinenec oxide     21.47     1132     1107     M714       26     ci-pomethat/2.64en-1.01     24.40     1133     1147     M715       27     mrs-Subinenec oxide     21.47     1132     1200     PV14       28     aliteher     24.40     1133 </td <td>10</td> <td>cis-3-Hexenyl acetate</td> <td>15.70</td> <td>1008</td> <td>1009</td> <td>PV5</td> <td></td> <td>CV2</td> <td>MV5</td>	10	cis-3-Hexenyl acetate	15.70	1008	1009	PV5		CV2	MV5
12     a Terpine     16.68     1031     1018	11	$\alpha$ -Phellandrene	16.13	1019	1013	PV6	DV7		
13μ-λymene18.8810.3610.34μ-γμ-W	12	α-Terpinene	16.68	1031	1018				MV6
14Landonene17.0010401039PV3DV3CV3MV315β*Phellandrene17.2310441044PV3DV10CV4MV316mars/Polimene18.2010661066PV11MV10MV1018mars/Shinene hydrate19.0010841087WV13CV5MV1018traphnolme19.4410951007PV12DV12MV1220Linadod19.8211031100100V13CV5MV1221Linadod19.82110311071107V13CV5MV1222Nonanal20.0311071107V13CV5MV1224cf-Linnoene oxide21.6711491147W14MV1525cf-g-Menha 2,8-dien-1-023.74119211331147W14MV1526cf-g-Menha 2,8-dien-1-024.74123120PU14W14MV1527mars-p-Menha 2,8-dien-1-024.74123120PU14W14W1428Dil eher24.60129121MV16W14W1429Dil eher24.74123120PU14W14W1420caronel24.74123120PU14W1429Dil eher24.74123120PU14W1420Caronel Mi224.74123120PU14W1420Carone	13	<i>p</i> -Cymene	16.88	1036	1034	PV7	DV8	0110	MV7
15p-relationtrice p-rise17.331044103410741070071007416mark-β-Cinence p-rise17.3810661066PV11MV1017- reprisolance hydrate19.0010841087PV12DV12MV1019Terprisolance hydrate19.4310051007PV12DV12MV1220Undecance19.6311031103CV5MV1221Linalool19.8211031103CV5MV1222Nonanal20.0311071107CV5MV12231.3,8-P-Menthatrice20.7411251115PV13W15MV1524cf-Linonence oxide20.4411331147VMV16MV1725trans-bene oxide20.4711921193DV14W17MV1726cf-p-Menth2-S-dien-1-0124.4011961187DV14W17MV1727trans-concol25.6512201221V14W17MV1828of-cerplael24.671232128DV15CV9MV1931cf-carveol25.6512921287U16W11MV1932cf-acol25.6512921287U16W11MV1933Garvool25.6512921287U16W11MV1934Cf-2-Docenal25.6512921287U16W1235	14	Limonene 0 Dhallan hanna	17.06	1040	1039	PV8	DV9	CV3	MV8
10μπμε/continent1.5.310.710.7PV10PV11MV3017"Terpinolene18.2010661066PV11MV1018trams.shinnen hydrate19.0010841087MV1220Undecane19.6311001100PV12V1221Linalcol19.8211031107PV12V1222Nonanal20.0311071107PV12CV5231.3.8 P-Menthatriene20.7411251115PV13CV524dis-linonene oxide2.1.8711491140MV14MV1526trams-benchast2.5.4411921193MV14MV1526citz-Mentha-2.8-dient-102.5.7411921193MV14MV1527trams-benchast2.4.4011961187PU14MV1528Dill effer2.4.4011961187PU14MV1529af-Terpinol2.5.4312261227MV1830Decanal2.7.5412281237CV10MV1932Carvone2.5.6312201237CV10MV1934E-2-Decenal2.5.4312281237CV10MV19351-Decanol2.5.4312281237CV10MV1936Tradecanal2.6.613001300DV16MV1937Tradecanal2.6.613001301CV12MV19	15	<i>p</i> -Pheliandrene	17.23	1044	1034	PV9	DV10	674	MUO
1programme hydrate19.0010001000PT1Mr11018rams.shinene hydrate19.0010841007PT1Mr11119Tepinolene19.4410051007PT12DY12T21Lindocane19.6311001103V5V5Mr11221Lindol19.8211031103V5V7Mr13222Nonanal20.0311071107V1V7Mr1322313.38ApMenthatriene20.7411251115PT13W124de Linnoene oxide21.6711331147W1Mr1525de p-Menta-2.54 dien 1-0123.7411921133U7Mr1526de p-Menta-2.54 dien 1-0124.4011961167PV14W17227de Graveol24.7712131200PV14W17528Dill eher24.0712241262PV15CV9MV1827de Graveol25.6512201221MV19MV2138Carvone25.45123212841274V14MV173810013051291V16MV21MV2139130713391344V7MV22341-20-anol29.6013051291V16MV21341-20-anol29.6013051291V16MV21341-20-anol29.60130512	10	u Torpinono	17.38	1047	1047	PV10 DV11	DVII		MV9
10Indexample in yunke19.0010.0010.0010.1010.	1/	y-reipinene trans Sabinene hydrate	10.20	1084	1087	PVII			MV10
1111000111200100100100 <td>10</td> <td>Terpipolene</td> <td>19.00</td> <td>1004</td> <td>1007</td> <td>DV12</td> <td>DV12</td> <td></td> <td></td>	10	Terpipolene	19.00	1004	1007	DV12	DV12		
Indication19.82110311031103CV6MV1221Nonanal20.0311071107CV7MV13231.3,8-pMenthatriene20.0411251115PV13MV1424ci-Linnone oxide21.8711491140MV14MV1525ci-p-Mentha-2,8-dien-1-ol23.7411921193MV16MV1726ci-p-Mentha-2,8-dien-1-ol23.7411921193MV16MV1728Dill ther24.4011961187DV14MV1729a Terpinelo24.7412131200PV14MV1830Decanal24.7912141207V14MV1831ci-Carvelo25.6312281221MV1832rars-Garveol25.4312281267CV934E-Decenal27.5412731278CV10351-Decanol28.6413001300DV15CV1036Tridecane29.0613001300DV15MV1237Bornyl acetate30.7013391344CV14MV2238Undecanal26.3613171310CV13MV2239Carvonehyl acetate30.7013391344CV14MV24411-Decanol (tent.)33.8814071386PV15CV1339Carvonehyl acetate34.3914191407CV14MV2445<	20	Undecane	19.63	1100	1100	1 1 12	DV12 DV13	CV5	
22Namal20.031107107107CV7M'13231,3,8,ρ Menthariene20.7411251115PV13MV1424cis-Limonene oxide21.8711491140-MV1425rrans-Limonene oxide22.0411531147-MV1526cis-p-Mentha-2,8-dien-1.0123.0411921193-MV1527rrans-Quentha-2,8-dien-1.0124.0711991196-MV1428Dill efter24.0411061187DV14-MV1729a-Tenpineol24.7412131200PV14-MV1829a-Carveol25.0512201221MV18-MV1831ci-Carveol25.0512201221MV18-MV1932Carvone27.1312641262DV15CV10-34E-Decenal27.5412731278CV10-MV21351-Decenal29.0613001300DV16-MV2136Tridecane29.0613001300DV16-MV2137Bornyl acetate29.0313171310CV12MV2138Udecanal32.4013751371CV12MV2234Decyl acetate3.0314771386CV13-34Decyl acetate3.0314751480-CV16	21	Linalool	19.82	1103	1103		5110	CV6	MV12
231,3,s-p.Menharinen cis-Limonene oxide20.7411251115PV13PV1324cis-Limonene oxide21.8711491140	22	Nonanal	20.03	1107	1107			CV7	MV13
24ci-imonen oxide21.8711491147	23	1,3,8-p-Menthatriene	20.74	1125	1115	PV13			
25rure-imoneneoside22.0411531147	24	<i>cis</i> -Limonene oxide	21.87	1149	1140				MV14
26eys-Mentha-2,8-dien-1-ol23.7411921193MV1627prans-p-Mentha-2,8-dien-1-ol24.7011961187DV14MV1728Dill effer24.7412131207PV14MV1829a "terpineol24.7412131207KV8MV1831de-Carneol25.0512201221MV19MV1932grans-Carveol27.3312641262DV15CV9MV20346.2-Decenal27.5412731278CV11MV20351.Decanal27.6412921287CV11MV21361.Decanal29.0613001300DV16MV2137Borny acetate29.0613051291V16MV2138Undecanal29.0613051371CV13MV2239Garvonenthyl acetate30.7013391344CV12MV2341Dodecanal (tert.)33.881407136CV14MV2342Beburbonene34.3914121407CV15MV2343Dodecanal (tert.)34.3914121407V15MV2344Dodecanal34.3914121407V15MV2345Dodecanal34.3914121407V15MV2345Dodecanal34.3914121407V15MV2345Dodecanal34.3914121407V15MV23 <td>25</td> <td>trans-Limonene oxide</td> <td>22.04</td> <td>1153</td> <td>1147</td> <td></td> <td></td> <td></td> <td>MV15</td>	25	trans-Limonene oxide	22.04	1153	1147				MV15
27rmsp-Memha-2,8-dien-1-ol24.0711991196	26	cis-p-Mentha-2,8-dien-1-ol	23.74	1192	1193				MV16
28Dil eher24.4011961187DV1429a-Terpinelo24.7912131200PV1431cis Carveol24.7912141207CV831cis Carveol25.0512201221MV1932trans-Carveol25.4312281217MV1933Carvone27.1312641262DV15CV9MV2034E-2-Decenal27.5412921287CV11MV1935I-Decanol28.4512921287CV12MV2136I-Decanol29.0613001291DV16MV2137Borny lacetare29.03137130CV12MV2138Undecanal29.031371130CV12MV2139Carvomenhy lacetare32.4013751371CV13MV2341I-Dodecanol (tert.)33.8814071386CV14MV2342 $\beta$ -Bourbonene34.3914121407CV15MV2343Declacatal34.3914121407MV2344Dodecanal36.3714631467V16MV2445Dodecanal36.3714631467V16MV2446Daceanal36.8614751480CV17MV2547C2-Dodecenal36.8614751483CV19MV2648Holoronadendrene36.8614691483CV19 <td>27</td> <td>trans-p-Mentha-2,8-dien-1-ol</td> <td>24.07</td> <td>1199</td> <td>1196</td> <td></td> <td></td> <td></td> <td>MV17</td>	27	trans-p-Mentha-2,8-dien-1-ol	24.07	1199	1196				MV17
29a^-Tepineol24.741213120PV14PV1431Decanal24.791214120KMV1831 <i>dis</i> Carveol25.05120121MV1932mars-Carveol25.4312281217MV1932Carvone27.1312641262DV15C/934E-2-becenal27.5412731278CV10MV2035I-becanol28.4512921287CV10MV2136Tridecane29.0613001300DV16MV2137Bonyl acetate30.7013391344CV12MV2240E-2-beneanl (entr.)38.814071366CV14MV2341I-bodecanol (entr.)34.814121407CV13MV2342β-Bourbonene34.0814121407MV23MV2343Decyl acetate34.3914191402MV23MV2345Dodecanal34.3914191402MV23MV2345Dodecanal34.3914191402MV23MV2347S-2-bodecenal35.6814481455PV15MV2348Decyn aduednene36.8614481455PV15MV2347S-2-bodecenal37.1214631467CV16MV2448Aloromadendenene36.8614481455PV15CV1748Aloromadendenene <td>28</td> <td>Dill ether</td> <td>24.40</td> <td>1196</td> <td>1187</td> <td></td> <td>DV14</td> <td></td> <td></td>	28	Dill ether	24.40	1196	1187		DV14		
30Decanal24.7912141207CV831cix-Carveol25.0512201221MV1932carvone25.4312281212MV1933Carvone27.1312641262DV15CV9MV20351-Decanol28.4512921287CV10T361-Decanol28.4512921287CV10MV20361-Decanol29.0613001300DV16MV2037Bonyl acetate29.0113051291MV2138Undecanal29.0313171310CV12MV2139Carvomenthyl acetate30.7013391344CV13MV20411-Dodecanol (tent.)33.8814071386CV14MV2042β-Bourbonene34.0814121407CV15MV2044β-Carvophyllene34.6814631418MV20MV2045Dodecanal34.3914191407MV20MV2046Dodecanal36.8614751487MV20MV20472-2-Dodecenal36.3714631467CV15MV20472-2-Dodecenal36.3714631467CV16MV2047A-Dodecenal36.8614751489CV19MV2048Alloromadenderne37.4814891468CV19MV2053Germacrene-D38.381502	29	α-Terpineol	24.74	1213	1200	PV14			
31cic Carveol25.0512201221VII8MV1832trans-Carveol25.4312641262DV15CV9MV2034E 2-Decenal27.5412731278CV10VI16VI16351-Decanol28.4512921287CV10VI16VI16VI1636Tridecanal29.0613001300DV16VI12MV2037Bornyl acetate39.0613171310CV12MV2238Undecanal29.6313711310CV13VI1240E-2-Undecenal32.4013391344CV13MV23411-Dodecanol (tent.)33.8814071386CV14MV2342β-Bourbonene34.0814121407CV13MV2343Decyl acetate34.3914191400CV15MV2444β-Caryophyllene34.661418KSMV26MV2445Docyl acetate35.6814481455PV15MV2647Z-Dodecenal37.1214631467CV17MV2548Allomened-merene36.8614681467CV18MV2651E-2-Dodecen-1-ol37.7814551483CV19MV26521-100000000000000000000000000000000000	30	Decanal	24.79	1214	1207			CV8	
32trans-Carveol25.4312281217TotalMV1933Carvoone27.1312641262DV15Cy9MV2034E-2-Decenal27.5412731278CV10Total351-Decanol28.4512921287CV10Total36Tridecane29.061300DV16TotalMV2137Bornyl acetate29.1013051291MV2138Undecanal29.6313171310CV12MV2240E-2-Undecenal32.4013751371CV13MV22411-Dodecanol (tent.)3.8814071386CV14MV2343Decyl acetate34.0814121410CV15MV2344 $\beta$ -Carvophyllene34.6814481455PV15CV16MV2545Dedecanal35.6814481455PV15MV26MV2546Alacomadendrene36.6614751480CV18MV2647Z-2-Dodecenal37.1214801468CV18MV2650e-Humulene37.7814951483CV19MV2651E-2-Dodecen-1-ol37.7814951489CV19MV2653Germacrene-D38.3815021516PV16DV17MV2654Tridecanal38.9715251528PV17CV21T55Horistich39.821532 </td <td>31</td> <td>cis-Carveol</td> <td>25.05</td> <td>1220</td> <td>1221</td> <td></td> <td></td> <td></td> <td>MV18</td>	31	cis-Carveol	25.05	1220	1221				MV18
33Carvone27.312641262DV15CV9MV2034E-2-Decenal27.5412731278CV101365351-Decanol28.4512921287CV1136Tridecane29.0613001300DV16MV2137Bornyl acetate29.031371310CV12MV2238Undecanal29.631371310CV12MV2239Carvomenthyl acetate30.7013391344CV13MV2240E-2-Undecanol (tent.)33.8814071386CV14MV23411-bodecanol (tent.)33.8814071386CV14MV2343Decyl acetate34.0814121410MV24MV2444β-Caryophyllene34.681418MV24MV2445Dodecanal34.3914191420CV16MV2446mars.β-Caryophyllene36.8614751480CV17MV2647Z-2-Dodecenal37.7814891467CV19MV2751E-2-Dodecen-1-ol37.7814951483CV17MV2753Germatcrene-D38.381502158PV16V1754Tridecanal38.9715251528PV17CV2155Myritin39.821532PV18CV2114956Myritin39.821532PV18CV2257E-2-	32	trans-Carveol	25.43	1228	1217				MV19
34 $E2$ -Decenal $27.54$ $1273$ $1278$ $C10$ 35 $1$ -Decanol $28.45$ $1292$ $1287$ $C11$ 36       Tridecane $29.06$ $1300$ $1300$ $DV16$ 37       Bornyl acetate $29.01$ $1305$ $1291$ $CV12$ 38       Undecanal $29.63$ $1371$ $1310$ $CV12$ 39       Carvomenthyl acetate $30.70$ $1339$ $1344$ $CV13$ 40 $E-2$ -Undecenal $32.40$ $1375$ $1371$ $CV13$ $MV22$ 41 $1$ -Dodecanol (tent.) $33.88$ $1412$ $1407$ $CV15$ $MV23$ 42 $\beta$ -Bourbonene $34.08$ $1412$ $1407$ $CV15$ $MV23$ 43       Decyl acetate $34.39$ $1412$ $1407$ $CV15$ $MV24$ 44 $\beta$ -Caryophyllene $36.26$ $1448$ $1455$ $PV15$ $CV17$ 45       Dodecanal $37.37$ $1480$ $1467$ $CV18$ $MV25$ 47 <t< td=""><td>33</td><td>Carvone</td><td>27.13</td><td>1264</td><td>1262</td><td></td><td>DV15</td><td>CV9</td><td>MV20</td></t<>	33	Carvone	27.13	1264	1262		DV15	CV9	MV20
35       1-beand       28.45       1292       1287       0       0       1         36       Tidecane       29.06       1300       1300       DV16       MV21         37       Bornyl acetate       29.00       1305       1291       MV21         38       Undecanal       29.63       1317       1310       CV12       MV21         39       Carvomenthyl acetate       30.70       1339       1344       CV13       MV22         40       E-2-Undecenal       32.40       1375       1371       CV13       MV23         41       1-bodecanol (tent.)       33.88       1407       1386       CV14       MV23         42 $\beta$ -Bourbonene       34.08       1412       1410       CV15       MV24         43       Decyl acetate       34.13       1412       1410       CV16       MV24         44 $\beta$ -Caryophyllene       34.26       1416       1418       CV16       MV24         45       Dodecanal       36.37       1463       1467       CV17       MV25         46 $rarse_i Caryophyllene       36.86       1475       1480       1467       CV18       MV16      5$	34	E-2-Decenal	27.54	1273	1278			CV10	
36Indecade29.0613001300DV1637Borny acetate29.0313171310 $CV12$ 38Undecanal29.6313171310 $CV12$ 39Carvomenthyl acetate30.7013391344 $CV13$ 40E-2-Undecenal32.4013751371 $CV13$ 411-Dodecanol (tent.)33.8814071386 $CV14$ 42 $\beta$ -Bourbonene34.0814121407 $CV15$ 43Decyl acetate34.3314121410 $CV15$ 44 $\beta$ -Caryophyllene35.6814481455PV15 $CV16$ 45Dodecanal36.3714631467 $CV17$ 46trans- $\beta$ -Caryophyllene35.6814481455PV15 $CV18$ 472-2-Dodecenal37.1214801468 $CV17$ $MV25$ 48Alloarmadendrene36.6615021483 $CV19$ $MV27$ 51E-2-Dodecen-1-ol37.7814951483 $CV17$ $MV27$ 521-Dodecanol38.9615221518 $CV20$ 14553Germacrene-D38.3815101516PV16DV1754Mristicin39.8215251528PV17146355Nerolidol39.9215251528PV17146356Mristicin39.8215521524PV18 $CV22$ 55Nerolidol39.821552<	35	1-Decanol	28.45	1292	1287		DUIC	CVII	
$37$ both actuate $29,10$ $130$ $1291$ $1291$ $1291$ $1291$ $38$ Undecanal $29,63$ $1317$ $1310$ $CV12$ $39$ Carvomenthyl acetate $30,70$ $1339$ $1344$ $MV22$ $40$ $E-2$ -Undecenal $32,40$ $1375$ $1371$ $CV13$ $41$ $1$ -bodecanol (tent.) $33.88$ $1407$ $1386$ $CV14$ $42$ $\beta$ -Bourbonene $34.08$ $1412$ $1407$ $MV23$ $43$ Decyl acetate $34.13$ $1412$ $1410$ $CV15$ $44$ $\beta$ -Caryophyllene $34.26$ $1416$ $1418$ $MV24$ $45$ Dodecanal $34.39$ $1419$ $1420$ $CV15$ $46$ $rans-\beta$ -Caryophyllene $35.68$ $1448$ $1455$ $PV15$ $MV27$ $47$ $Z$ -2-Dodecenal $36.37$ $1463$ $1467$ $CV17$ $MV25$ $49$ $E-2$ -Dodecenal $37.12$ $1480$ $1468$ $CV18$ $MV27$ $51$ $e-2$ -Dodecenal-ol $37.78$ $1493$ $1489$ $MV7$ $MV27$ $51$ $E-2$ -Dodecenal-ol $37.78$ $1495$ $1483$ $CV17$ $MV27$ $51$ $E-2$ -Dodecenal $37.78$ $1495$ $1483$ $CV17$ $MV27$ $51$ $F2$ -Dodecanol $38.96$ $1522$ $1518$ $PV17$ $CV21$ $53$ Germacrene-D $38.97$ $1525$ $1528$ $PV17$ $CV22$ $54$ Mrodecanol $38.97$ $1525$ <td>30</td> <td>I ridecane</td> <td>29.06</td> <td>1300</td> <td>1300</td> <td></td> <td>DV16</td> <td></td> <td>M3/01</td>	30	I ridecane	29.06	1300	1300		DV16		M3/01
38Ontectain25.0313171310 $C112$ 39Carvomenthyl acetate30.701339134MV2240E-2-Undecenal32.4013751371CV13411-Dodecanol (tent.)33.8814071386CV1442 $\beta$ Bourbonene34.0814121410CV1543Decyl acetate34.1314121410CV1544 $\beta$ Caryophyllene34.2614161418MV2445Dodecanal36.3714631467CV1646 <i>rans</i> - $\beta$ -Caryophyllene36.6814751480CV1748Alloaromadendrene36.8614751480CV1849E-2-Dodecenal37.1214801468CV1951E-2-Dodecenal37.8815021483CV1951E-2-Dodecenal38.0815021483CV19521-Dodecanol38.0815021483CV1953Germacrene-D38.3815101516PV16DV1754Tridecanal38.9615221528PV17U155Merolidol38.9715251528PV17U156Myristicin39.8215351532PV18DV1859Ptradecanal43.3116321623CV24CV22	3/	Bornyi acetate	29.10	1305	1291			CV12	101 V 2 1
35Cal Volfacenal30.701391347 $MV22$ 40E-2 Undecenal32.4013751371 $CV13$ 411-Dodecanol (tent.)33.8814071386 $CV14$ 42 $\beta$ Bourbonene34.0814121407 $CV13$ 43Decyl acetate34.1314121410 $CV15$ 44 $\beta$ Caryophyllene34.2614161418 $MV24$ 45Dodecanal34.3914191420 $CV16$ 46trans- $\beta$ -Caryophyllene35.6814481455PV15 $MV24$ 47Z-2-Dodecenal36.3714631467 $CV17$ 48Alloaromadendrene36.8614751480 $MV26$ 50 $\alpha$ -Humulene37.4814891489 $MV27$ 51E-2-Dodecenal37.7814951483 $CV19$ 51E-2-Dodecen-1-ol37.7814951485 $CV20$ 53Germacrene-D38.3815101516PV16DV1754Tridecanal38.9615221518 $CV20$ 55Nerolidol38.9715251528PV17 $CV21$ 56Myristicin39.8215351532PV18 $CV22$ 581-Tetradecanal41.5815821571 $CV22$ 59Tardecanal43.3116321623 $CV24$	30	Carvomenthyl acetate	29.03	1317	1310			CV12	MV22
1012-brief of the bar of the	40	F-2-Undecenal	32.40	1375	1371			CV13	101 0 22
111 <th< td=""><td>41</td><td>1-Dodecanol (tent.)</td><td>33.88</td><td>1407</td><td>1386</td><td></td><td></td><td>CV14</td><td></td></th<>	41	1-Dodecanol (tent.)	33.88	1407	1386			CV14	
A3Decyl acetate34.1314121410CV1544 $\beta$ -Caryophyllene34.2614161418MV2445Dodecanal34.3914191420CV1646trans- $\beta$ -Caryophyllene35.6814481455PV15MV2547Z-2-Dodecenal36.3714631467MV2648Alloaromadendrene36.8614751480CV18MV2749E-2-Dodecenal37.1214801468CV18MV2750 $\alpha$ -Humulene37.4814891489CV19MV2751E-2-Dodecen-1-ol37.7814951483CV19MV27521-Dodecanol38.0815021485CV20CV2053Germacrene-D38.3815101516PV16DV17T54Tridecanal38.9715251528PV17CV2155Nerolidol38.9715251532PV18DV1857E-2-Tridecenal41.5815821571CV22581-Tetradecanol42.8116151618CV2359Tetradecanal43.3116321623CV24	42	$\beta$ -Bourbonene	34.08	1412	1407			0111	MV23
44 $\beta$ -Caryophyllene34.2614161418MV2445Dodecanal34.3914191420CV1646 $rans$ - $\beta$ -Caryophyllene35.6814481455PV15MV2547Z-2-Dodecenal36.3714631467CV17MV2648Alloaromadendrene36.8614751480CV18MV2649E-2-Dodecenal37.1214801468CV18MV2550 $\alpha$ -Humulene37.4814891489CV19MV2551E-2-Dodecen-1-ol37.7814951483CV19MV25521-Dodecanol38.0815021485CV20MV2553Germacrene-D38.3815101516PV16DV1754Tridecanal38.9615221518CV2155Nerolidol38.9715251528PV1756Myristicin39.8215351532PV18CV2257E-2-Tridecenal41.5815821571CV22581-Tetradecanol42.8116151618CV2359Tetradecanal43.3116321623CV24	43	Decvl acetate	34.13	1412	1410			CV15	
45Dodecand34.3914191420CV1646trans- $\beta$ -Caryophyllene35.6814481455PV15MV2547Z-2-Dodecenal36.3714631467CV1748Alloaromadendrene36.8614751480MV2649E-2-Dodecenal37.1214801468CV1850 $\alpha$ -Humulene37.4814891489MV2751E-2-Dodecen-1-ol37.7814951483CV19521-Dodecanol38.0815021485CV2053Germacrene-D38.3815101516PV16DV1754Tridecanal38.9615221518CV2155Nerolidol38.9715251528PV1756Myristicin39.8215351532PV18DV1857E-2-Tridecenal41.5815821571CV22581-Tetradecanol42.8116151618CV2359Tetradecanal43.3116321623CV24	44	$\beta$ -Caryophyllene	34.26	1416	1418				MV24
46trans-β-Caryophyllene35.6814481455PV15MV2547Z-2-Dodecenal36.3714631467CV1748Alloaromadendrene36.8614751480MV2649E-2-Dodecenal37.1214801468CV1850α-Humulene37.4814891489CV1951E-2-Dodecen-1-ol37.7814951483CV19521-Dodecanol38.0815021485CV2053Germacrene-D38.3815101516PV16DV1754Tridecanal38.9715251528PV17CV2155Myristicin39.8215351532PV18DV1857E-2-Tridecenal41.5815821571CV22581-tradecanol42.8116151618CV2359Tetradecanal43.3116321623CV24	45	Dodecanal	34.39	1419	1420			CV16	
$47$ Z-2-Dodecenal $36.37$ $1463$ $1467$ CV17 $48$ Alloaromadendrene $36.86$ $1475$ $1480$ $M26$ $49$ E-2-Dodecenal $37.12$ $1480$ $1468$ CV18 $50$ $\alpha$ -Hunulene $37.48$ $1489$ $1489$ $M27$ $51$ E-2-Dodecen-1-ol $37.78$ $1495$ $1483$ CV19 $52$ 1-Dodecanol $38.08$ $1502$ $1485$ CV20 $53$ Germacrene-D $38.38$ $1510$ $1516$ PV16DV17 $54$ Tridecanal $38.96$ $1522$ $1518$ CV21 $55$ Nerolidol $38.97$ $1525$ $1528$ PV17 $56$ Myristicin $39.82$ $1535$ $1532$ PV18CV22 $57$ E-2-Tridecenal $41.58$ $1582$ $1571$ CV22 $58$ 1-tradecanol $42.81$ $1632$ $1623$ CV24	46	trans-β-Caryophyllene	35.68	1448	1455	PV15			MV25
48       Alloaromadendrene       36.86       1475       1480       1480       MV26         49 $E-2$ -Dodecenal       37.12       1480       1468       CV18         50 $\alpha$ -Humulene       37.48       1489       1489       CV19         51 $E-2$ -Dodecen-1-ol       37.78       1495       1483       CV19         52 $1$ -Dodecanol       38.08       1502       1485       CV21         53       Germacrene-D       38.38       1510       1516       PV16       DV17         54       Tridecanal       38.96       1522       1518       CV21       CV21         55       Nerolidol       38.97       1525       1528       PV17       CV21         56       Myristicin       39.82       1535       1532       PV18       CV22         57 $E-2$ -Tridecenal       41.58       1582       1571       CV22         58       1-Tetradecanol       42.81       1615       1618       CV23         59       Tetradecanal       43.31       1632       1623       CV24	47	Z-2-Dodecenal	36.37	1463	1467			CV17	
49       E-2-Dodecenal       37.12       1480       1468       CV18         50 $\alpha$ -Humulene       37.48       1489       1489       M207         51       E-2-Dodecen-1-ol       37.78       1495       1483       CV19         52       1-Dodecanol       38.08       1502       1485       CV19         53       Germacrene-D       38.38       1510       1516       PV16       DV17         54       Tridecanal       38.96       1522       1518       CV21       CV21         55       Nerolidol       38.97       1525       1528       PV17       CV21         56       Myristicin       39.82       1535       1532       PV18       CV22         57       E-2-Tridecenal       41.58       1582       1571       CV22         58       1-Tetradecanol       42.81       1615       1618       CV23         59       Tetradecanal       43.31       1632       1623       CV24	48	Alloaromadendrene	36.86	1475	1480				MV26
$50$ $\alpha$ -Hunulene $37.48$ $1489$ $1489$ $M27$ $51$ $E-2$ -Dodecen-1-ol $37.78$ $1495$ $1483$ $CV19$ $52$ $1$ -Dodecanol $38.08$ $1502$ $1485$ $CV20$ $53$ $6rmacrene-D$ $38.08$ $1510$ $1516$ $PV16$ $DV17$ $54$ Tridecanal $38.96$ $1522$ $1518$ $CV21$ $55$ Nerolidol $38.97$ $1525$ $1528$ $PV17$ $CV21$ $56$ Myristicin $39.82$ $1535$ $1532$ $PV18$ $DV18$ $57$ $E-2$ -Tridecenal $41.58$ $1582$ $1571$ $CV22$ $58$ $1$ -tradecanal $42.81$ $1615$ $1623$ $CV24$	49	E-2-Dodecenal	37.12	1480	1468			CV18	
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 $38.97$ $1525$ $1528$ $PV17$ $CV21$ $56$ 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 $43.31$ $1632$ $1623$ $CV24$	50	α-Humulene	37.48	1489	1489				MV27
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	51	E-2-Dodecen-1-ol	37.78	1495	1483			CV19	
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	52	1-Dodecanol	38.08	1502	1485			CV20	
54         Indecanal         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	53	Germacrene-D	38.38	1510	1516	PV16	DV17	0105	
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	54	Tridecanal	38.96	1522	1518			CV21	
56         Myrnstrum         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	55	Nerolidol	38.97	1525	1528	PV17	DUZO		
5/     E-2-1ridecenal     41.58     1582     15/1     CV22       58     1-Tetradecanol     42.81     1615     1618     CV23       59     Tetradecanal     43.31     1632     1623     CV24	56	Myristicin	39.82	1535	1532	PV18	DV18	01/00	
50         1-retradecanol         42.81         1615         1618         CV23           59         Tetradecanal         43.31         1632         1623         CV24	5/	E-2-Indecenal	41.58	1582	1571			CV22	
59 renauccalial 45.51 1052 1023 CV24	58 50	1-1etradecanol	42.81	1015	1018			CV23	
	57	retradecanal	43.31	1032	1023			GV24	

<sup>a</sup> RT = retention time; RI = retention index; Exp. = experimental and Lit. = Literature.

<sup>b</sup> 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  $\mu$ L of benzyl acetate as an internal standard (1470 mg L<sup>-1</sup>) 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.  $\times$  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<sup>-1</sup> and a total flow of 3.9 mL min<sup>-1</sup> in a split ratio of 1:11 and the following program: (a) 80 °C for 0 min; (b) increase of 3 °C min<sup>-1</sup> from 80 °C to 210 °C and hold for 1 min; (c) increase of 25 °C min<sup>-1</sup> 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 NISTO5 spectral library collection).

$$I = 100 \left[ n + (N - n) \frac{\log t'_r(unknown) - \log t'_r(n)}{\log t'_r(N) - \log t'_r(n)} \right]$$
(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 $(1 \text{ mL min}^{-1})$ . 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: $\alpha$ phellandrene (monoterpenes), α-terpineol (terpenoids), trans-β-caryophyllene (sesquiterpenes), dill ether (terpene ethers), nonanal (aldehydes), myristicin (phenylpropanoids), bornyl acetate (esters), 1decanol (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  $\approx 992$  mg kg<sup>-1</sup> of fresh weight. 1,3,8-*p*-menthatriene,  $\beta$ -phellandrene, myristicin, terpinolene, myrcene, limonene,  $\alpha$ -pinene, and  $\alpha$ -phellandrene formed most of the composition of parsley aroma. Table 3 shows that 4 compounds (1,3,8-*p*-menthatriene,  $\beta$ -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  $\beta$ -phellandrene, 1,3,8-*p*-menthatriene and  $\beta$ -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  $\alpha$ -*p*-dimethylstyrene. Also, López et al. (1999) found that the main constitutes of parsley essential oil were *p*-1,3,8 menthatriene,  $\beta$ -phellandrene and apiole.

Results in Table 3 show that 1,3,8-p-menthatriene (85.8-1026 mg kg<sup>-1</sup>, for P15 and P6 vegetative stages, respectively),  $\beta$ -phellandrene (75.5-637 mg kg<sup>-1</sup>, 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<sup>-1</sup> 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  $\beta$ -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  $\approx 1455 \text{ mg kg}^{-1}$  of fresh weight. The eight main compounds in all samples were:  $\alpha$ -phellandrene, dillether,  $\beta$ -phellandrene, limonene, *p*-cymene, *a*-pinene, trans- $\beta$ -ocimene, and myristicin. In fact,  $\alpha$ -phellandrene, dillether,  $\beta$ -phellandrene were represented 62–92% of the total concentrations of volatiles compounds of dill shoots in all samples (D1-D15). Rådulescu et al. (2010) analyzed the oil of dill leaves and they recorded that the main components in leaves oil were  $\alpha$ -phellandrene (62.71 %), limonene (13.28 %) and dillether (16.42 %). The principal components of dill leaf oil were  $\alpha$ -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:  $\alpha$ -phellandrene (357–1750 mg kg<sup>-1</sup> for D5 and D15 vegetative stages, respectively), dillether (23.8–466 mg kg $^{-1}$ for D5 and D10 vegetative stages, respectively) and  $\beta$ -phellandrene  $(46.1-230 \text{ mg kg}^{-1} \text{ for D5 and D11 vegetative stages, respectively})$  in all harvest times (D1-D15). The highest concentration of  $\alpha$ -phellandrene, dillether and  $\beta$ -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<sup>-1</sup>), 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  $\alpha$ -phellandrene,  $\beta$ -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,

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

Compound	ANOVA <sup>↑</sup>	P1 Concent	P2 tration (mg	P3 kg <sup>-1</sup> )	P4	Р5	Р6	Р7	P8	Р9	P10	P11	P12	P13	P14	P15	P16
PV1	* * *	41.9 <sup>a+</sup>	9.22 <sup>bc</sup>	22.2 <sup>b</sup>	11.0 <sup>bc</sup>	7.49 <sup>bc</sup>	17.6 <sup>bc</sup>	2.70 <sup>c</sup>	12.2 <sup>bc</sup>	13.3 <sup>bc</sup>	11.4 <sup>bc</sup>	24.4 <sup>b</sup>	8.53 <sup>bc</sup>	4.14 <sup>c</sup>	3.16 <sup>c</sup>	2.29 <sup>c</sup>	9.86 <sup>bc</sup>
PV2	***	4.45 <sup>a</sup>	$1.05^{bc}$	1.71 <sup>b</sup>	0.81 <sup>bc</sup>	0.30 <sup>bc</sup>	$1.70^{b}$	0.146 <sup>c</sup>	$1.08^{bc}$	1.00 <sup>bc</sup>	0.69 <sup>bc</sup>	0.84 <sup>bc</sup>	0.38 <sup>bc</sup>	0.34 <sup>bc</sup>	0.08 <sup>c</sup>	0.03 <sup>c</sup>	0.54 <sup>bc</sup>
PV3	***	165 <sup>a</sup>	79.6 <sup>abc</sup>	$100^{abc}$	51.6 <sup>c</sup>	27.7 <sup>c</sup>	$152^{ab}$	21.5 <sup>c</sup>	66.1 <sup>bc</sup>	76.1 <sup>bc</sup>	47.1 <sup>c</sup>	73.4 <sup>bc</sup>	27.1 <sup>c</sup>	28.7 <sup>c</sup>	17.3 <sup>c</sup>	14.4 <sup>c</sup>	24.0 <sup>c</sup>
PV4	***	$22.1^{a}$	3.48 <sup>cd</sup>	9.25 <sup>bc</sup>	4.32 <sup>bcd</sup>	2.62 <sup>cd</sup>	6.64 <sup>bcd</sup>	$0.380^{d}$	3.76 <sup>bcd</sup>	6.07 <sup>bcd</sup>	4.38 <sup>bcd</sup>	10.6 <sup>b</sup>	4.14 <sup>bcd</sup>	1.07 <sup>d</sup>	$1.05^{d}$	0.99 <sup>d</sup>	3.68 <sup>cd</sup>
PV5	***	7.19 <sup>ab</sup>	$8.10^{\mathrm{a}}$	$3.04^{bc}$	$3.52^{abc}$	1.71 <sup>c</sup>	2.96 <sup>bc</sup>	0.556 <sup>c</sup>	0.97 <sup>c</sup>	0.83 <sup>c</sup>	1.18 <sup>c</sup>	0.41 <sup>c</sup>	0.35 <sup>c</sup>	0.34 <sup>c</sup>	$0.02^{c}$	0.17 <sup>c</sup>	0.31 <sup>c</sup>
PV6	***	37.8 <sup>ab</sup>	16.8 <sup>bc</sup>	$25.3^{abc}$	$12.2^{bc}$	6.66 <sup>c</sup>	49.8 <sup>a</sup>	6.19 <sup>c</sup>	16.7 <sup>bc</sup>	15.9 <sup>bc</sup>	15.8 <sup>bc</sup>	17.7 <sup>bc</sup>	$11.0^{bc}$	9.63 <sup>c</sup>	5.59 <sup>c</sup>	3.73 <sup>c</sup>	8.48 <sup>c</sup>
PV7	**	4.54 <sup>ab</sup>	3.09 <sup>ab</sup>	4.62 <sup>ab</sup>	$2.28^{ab}$	$1.48^{b}$	6.19 <sup>a</sup>	$1.06^{b}$	3.95 <sup>ab</sup>	3.25 <sup>ab</sup>	$2.74^{ab}$	2.89 <sup>ab</sup>	1.41 <sup>b</sup>	$1.17^{b}$	$0.74^{b}$	$0.79^{b}$	$1.75^{b}$
PV8	***	59.9 <sup>ab</sup>	30.5 <sup>abcd</sup>	53.7 <sup>abc</sup>	27.9 <sup>abcd</sup>	13.1 <sup>cd</sup>	70.8 <sup>a</sup>	12.0 <sup>cd</sup>	30.0 <sup>abcd</sup>	$27.7^{abcd}$	26.5 <sup>abcd</sup>	32.0 <sup>abcd</sup>	11.3 <sup>cd</sup>	17.7 <sup>bcd</sup>	10.5 <sup>cd</sup>	5.59 <sup>d</sup>	13.7 <sup>cd</sup>
PV9	***	549 <sup>ab</sup>	234 <sup>bc</sup>	$313^{abc}$	169 <sup>c</sup>	103 <sup>c</sup>	637 <sup>a</sup>	95.3°	240 <sup>bc</sup>	$296^{abc}$	$289^{abc}$	$290^{abc}$	122 <sup>c</sup>	140 <sup>c</sup>	88.4 <sup>c</sup>	75.5 <sup>c</sup>	110 <sup>c</sup>
PV10	***	9.94 <sup>ab</sup>	8.59 <sup>ab</sup>	10.0 <sup>ab</sup>	5.34 <sup>ab</sup>	$3.03^{b}$	12.8 <sup>a</sup>	2.43 <sup>b</sup>	6.72 <sup>ab</sup>	6.62 <sup>ab</sup>	3.42 <sup>b</sup>	4.80 <sup>ab</sup>	2.33 <sup>b</sup>	$1.81^{b}$	$1.77^{b}$	$2.26^{b}$	$3.52^{b}$
PV11	**	4.46 <sup>a</sup>	$2.34^{ab}$	3.28 <sup>ab</sup>	1.19 <sup>ab</sup>	0.47 <sup>b</sup>	3.66 <sup>ab</sup>	0.44 <sup>b</sup>	$1.35^{ab}$	0.99 <sup>ab</sup>	0.82 <sup>b</sup>	0.98 <sup>ab</sup>	0.29 <sup>b</sup>	0.61 <sup>b</sup>	0.34 <sup>b</sup>	$0.10^{b}$	$0.30^{b}$
PV12	***	$198^{ab}$	$82.8^{bc}$	$101^{bc}$	55.0 <sup>c</sup>	23.1 <sup>c</sup>	253 <sup>a</sup>	32.5 <sup>c</sup>	50.8 <sup>c</sup>	43.7 <sup>c</sup>	44.3 <sup>c</sup>	51.0 <sup>c</sup>	17.2 <sup>c</sup>	53.3 <sup>c</sup>	26.2 <sup>c</sup>	17.2 <sup>c</sup>	18.9 <sup>c</sup>
PV13	***	870 <sup>ab</sup>	$560^{abc}$	894 <sup>ab</sup>	476 <sup>abc</sup>	$232^{bc}$	$1026^{a}$	$244^{bc}$	391 <sup>abc</sup>	$265^{bc}$	$386^{abc}$	$472^{abc}$	159 <sup>c</sup>	317 <sup>bc</sup>	$206^{bc}$	85.8 <sup>c</sup>	$195^{bc}$
PV14	***	$2.88^{a}$	$0.78^{bc}$	$1.68^{abc}$	0.86 <sup>bc</sup>	0.26 <sup>c</sup>	2.39 <sup>ab</sup>	0.54 <sup>bc</sup>	$2.52^{ab}$	0.61 <sup>bc</sup>	0.34 <sup>c</sup>	$0.12^{c}$	0.40 <sup>c</sup>	0.37 <sup>c</sup>	$0.02^{c}$	0.03 <sup>c</sup>	0.83 <sup>bc</sup>
PV15	***	$3.53^{b}$	$1.62^{b}$	$2.75^{b}$	$1.32^{b}$	0.64 <sup>b</sup>	12.9 <sup>a</sup>	$1.35^{b}$	4.81 <sup>b</sup>	2.53 <sup>b</sup>	$3.08^{b}$	4.58 <sup>b</sup>	1.64 <sup>b</sup>	4.06 <sup>b</sup>	$1.95^{b}$	$0.67^{b}$	$2.20^{b}$
PV16	**	18.4 <sup>ab</sup>	11.4 <sup>abc</sup>	7.89 <sup>abc</sup>	3.67 <sup>bc</sup>	0.95 <sup>c</sup>	20.8 <sup>a</sup>	1.76 <sup>bc</sup>	7.32 <sup>abc</sup>	4.85 <sup>abc</sup>	1.68 <sup>bc</sup>	4.13 <sup>abc</sup>	1.64 <sup>bc</sup>	3.46 <sup>bc</sup>	0.98 <sup>c</sup>	0.50 <sup>c</sup>	1.37 <sup>c</sup>
PV17	***	7.94 <sup>a</sup>	$1.20^{bc}$	$1.14^{bc}$	0.77 <sup>bc</sup>	0.14 <sup>bc</sup>	3.24 <sup>b</sup>	0.15 <sup>bc</sup>	$1.07^{bc}$	0.41 <sup>bc</sup>	0.10 <sup>bc</sup>	0.14 <sup>bc</sup>	0.07 <sup>c</sup>	$0.12^{bc}$	0.00 <sup>c</sup>	0.00 <sup>c</sup>	$0.21^{bc}$
PV18	*	410 <sup>a</sup>	346 <sup>ab</sup>	100 <sup>ab</sup>	90.6 <sup>ab</sup>	45.0 <sup>ab</sup>	263 <sup>ab</sup>	55.8 <sup>ab</sup>	109 <sup>ab</sup>	99.1 <sup>ab</sup>	59.1 <sup>ab</sup>	147 <sup>ab</sup>	45.9 <sup>ab</sup>	$68.2^{ab}$	$32.2^{ab}$	$13.1^{b}$	24.4 <sup>ab</sup>
Total	***	2416 <sup>ab</sup>	1401 <sup>abc</sup>	1654 <sup>abc</sup>	918 <sup>abc</sup>	470 <sup>c</sup>	2543 <sup>a</sup>	479 <sup>c</sup>	949 <sup>abc</sup>	864 <sup>bc</sup>	898 <sup>bc</sup>	1137 <sup>abc</sup>	414 <sup>c</sup>	653 <sup>c</sup>	396 <sup>c</sup>	223 <sup>c</sup>	420 <sup>c</sup>

<sup> $\dagger$ </sup> \*, \*\* and \*\*\*, significant at p < 0.05, 0.01 and 0.001, respectively.

<sup>+</sup> 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<sup>-1</sup>) 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<sup>-1</sup>) 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  $\approx 323$  mg kg<sup>-1</sup> 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<sup>-1</sup> 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<sup>-1</sup> 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 <sup>↑</sup>	D1	D2	D3	D4	D5	D6	D7	D8	D9	D10	D11	D12	D13	D14	D15
		Concentration (mg kg <sup>+</sup> )														
DV1	***	0.24 <sup>d+</sup>	1.22 <sup>cd</sup>	0.06 <sup>d</sup>	0.26 <sup>d</sup>	1.03 <sup>cd</sup>	0.16 <sup>d</sup>	0.92 <sup>cd</sup>	1.59 <sup>cd</sup>	4.77 <sup>abcd</sup>	6.65 <sup>abc</sup>	9.30 <sup>a</sup>	7.50 <sup>ab</sup>	$2.73^{bcd}$	$1.90^{bcd}$	2.23 <sup>bcd</sup>
DV2	***	$5.24^{ab}$	4.26 <sup>abc</sup>	$2.90^{abc}$	$4.42^{abc}$	1.16 <sup>c</sup>	$1.44^{bc}$	$1.63^{bc}$	4.08 <sup>abc</sup>	4.46 <sup>abc</sup>	5.90 <sup>a</sup>	5.69 <sup>a</sup>	$2.99^{abc}$	$1.70^{bc}$	$1.74^{bc}$	6.35 <sup>a</sup>
DV3	***	$28.0^{ab}$	$21.1^{abc}$	$16.2^{abc}$	$22.3^{abc}$	7.66 <sup>c</sup>	8.38 <sup>c</sup>	8.36 <sup>c</sup>	19.5 <sup>abc</sup>	$23.4^{abc}$	30.7 <sup>a</sup>	29.9 <sup>a</sup>	16.9 <sup>abc</sup>	9.53 <sup>bc</sup>	8.46 <sup>c</sup>	32.8 <sup>a</sup>
DV4	***	$2.80^{\mathrm{a}}$	$1.32^{abc}$	0.94 <sup>bc</sup>	$1.73^{abc}$	0.40 <sup>bc</sup>	0.38 <sup>c</sup>	$0.50^{bc}$	$1.43^{abc}$	$1.56^{abc}$	$1.62^{abc}$	$1.72^{abc}$	$0.78^{bc}$	0.38 <sup>c</sup>	0.48 <sup>bc</sup>	$2.12^{ab}$
DV5	***	10.6 <sup>a</sup>	7.73 <sup>ab</sup>	5.79 <sup>ab</sup>	9.13 <sup>ab</sup>	$2.37^{b}$	$2.57^{b}$	$2.95^{b}$	6.82 <sup>ab</sup>	7.77 <sup>ab</sup>	$11.2^{a}$	$11.5^{a}$	5.93 <sup>ab</sup>	$3.35^{b}$	3.11 <sup>b</sup>	$12.2^{\mathrm{a}}$
DV6	***	5.25 <sup>a</sup>	$1.78^{b}$	1.88 <sup>b</sup>	$2.28^{ab}$	0.74 <sup>b</sup>	$0.70^{\rm b}$	0.51 <sup>b</sup>	1.75 <sup>b</sup>	1.44 <sup>b</sup>	1.34 <sup>b</sup>	$1.42^{b}$	$0.89^{b}$	0.44 <sup>b</sup>	$0.29^{b}$	0.86 <sup>b</sup>
DV7	***	$1173^{ab}$	985 <sup>ab</sup>	$730^{b}$	$1028^{ab}$	$352^{b}$	367 <sup>b</sup>	458 <sup>b</sup>	756 <sup>b</sup>	998 <sup>ab</sup>	$1687^{a}$	1735 <sup>a</sup>	1054 <sup>ab</sup>	581 <sup>b</sup>	465 <sup>b</sup>	$1750^{\mathrm{a}}$
DV8	***	$21.6^{bcd}$	$18.2^{bcd}$	7.31 <sup>cd</sup>	$15.2^{bcd}$	18.6 <sup>bcd</sup>	$13.1^{bcd}$	7.40 <sup>cd</sup>	70.5 <sup>a</sup>	64.5 <sup>a</sup>	32.7 <sup>b</sup>	$23.8^{bcd}$	11.0 <sup>cd</sup>	17.9 <sup>bcd</sup>	3.78 <sup>d</sup>	27.3 <sup>bc</sup>
DV9	***	73.7 <sup>ab</sup>	56.4 <sup>abc</sup>	36.8 <sup>abc</sup>	$54.2^{abc}$	17.3 <sup>c</sup>	18.8 <sup>c</sup>	21.8 <sup>c</sup>	41.1 <sup>abc</sup>	$50.2^{abc}$	78.4 <sup>a</sup>	81.4 <sup>a</sup>	45.7 <sup>abc</sup>	25.9 <sup>bc</sup>	$21.0^{\circ}$	$81.3^{a}$
DV10	***	169 <sup>abc</sup>	$139^{abc}$	94.3 <sup>bc</sup>	$141^{abc}$	46.1 <sup>c</sup>	49.2 <sup>c</sup>	58.4 <sup>c</sup>	$111^{abc}$	$137^{abc}$	$223^{ab}$	230 <sup>a</sup>	$132^{abc}$	75.7 <sup>c</sup>	59.0 <sup>c</sup>	$220^{ab}$
DV11	***	2.87 <sup>c</sup>	5.76 <sup>c</sup>	3.15 <sup>c</sup>	7.88 <sup>c</sup>	3.88 <sup>c</sup>	5.55 <sup>c</sup>	6.34 <sup>c</sup>	8.89 <sup>c</sup>	12.0 <sup>bc</sup>	25.0 <sup>ab</sup>	29.7 <sup>a</sup>	$16.3^{abc}$	7.15 <sup>c</sup>	7.39 <sup>c</sup>	$22.6^{ab}$
DV12	***	$2.11^{ab}$	2.45 <sup>ab</sup>	4.49 <sup>a</sup>	4.65 <sup>a</sup>	$2.28^{ab}$	2.69 <sup>ab</sup>	1.43 <sup>b</sup>	1.85 <sup>b</sup>	1.05 <sup>b</sup>	$1.72^{b}$	$1.70^{\rm b}$	$0.55^{b}$	0.34 <sup>b</sup>	$0.32^{b}$	1.36 <sup>b</sup>
DV13	***	47.1 <sup>a</sup>	$28.3^{ab}$	17.3 <sup>b</sup>	19.5 <sup>ab</sup>	6.47 <sup>b</sup>	4.94 <sup>b</sup>	$3.59^{b}$	3.13 <sup>b</sup>	4.11 <sup>b</sup>	$10.5^{b}$	11.9 <sup>b</sup>	$2.70^{b}$	$3.48^{b}$	$1.12^{b}$	2.73 <sup>b</sup>
DV14	***	$38.5^{d}$	101 <sup>d</sup>	$50.7^{d}$	77.0 <sup>d</sup>	$23.8^{d}$	47.9 <sup>d</sup>	$82.2^{d}$	140 <sup>cd</sup>	$373^{ab}$	466 <sup>a</sup>	426 <sup>ab</sup>	$198^{bcd}$	106 <sup>d</sup>	60.0 <sup>d</sup>	$358^{abc}$
DV15	**	14.9 <sup>a</sup>	$0.01^{b}$	$0.01^{b}$	$0.00^{\mathrm{b}}$	$0.00^{\mathrm{b}}$	$0.02^{b}$	$0.53^{b}$	$0.14^{b}$	$0.02^{b}$	$0.01^{b}$	$0.02^{b}$	$0.00^{\mathrm{b}}$	$0.01^{b}$	$0.03^{b}$	0.56 <sup>b</sup>
DV16	***	$12.4^{a}$	5.83 <sup>ab</sup>	$2.60^{b}$	$3.33^{b}$	0.66 <sup>b</sup>	$0.60^{b}$	$0.45^{b}$	$0.63^{b}$	0.69 <sup>b</sup>	$1.11^{b}$	$1.61^{b}$	$0.10^{b}$	$0.17^{b}$	$0.21^{b}$	$0.26^{b}$
DV17	***	3.79 <sup>bcd</sup>	$10.3^{abc}$	5.75 <sup>abcd</sup>	$7.72^{abcd}$	0.42 <sup>d</sup>	4.96 <sup>abcd</sup>	2.01 <sup>cd</sup>	2.49 <sup>cd</sup>	2.77 <sup>bcd</sup>	$13.2^{a}$	11.8 <sup>ab</sup>	5.47 <sup>abcd</sup>	0.21 <sup>d</sup>	1.26 <sup>cd</sup>	3.41 <sup>bcd</sup>
DV18	***	614 <sup>a</sup>	275 <sup>b</sup>	111 <sup>b</sup>	109 <sup>b</sup>	19.2 <sup>b</sup>	$15.0^{b}$	$10.3^{b}$	$33.2^{b}$	19.0 <sup>b</sup>	3.65 <sup>b</sup>	7.39 <sup>b</sup>	$0.00^{\mathrm{b}}$	$0.06^{b}$	0.01 <sup>b</sup>	0.00 <sup>b</sup>
Total	***	2225 <sup>ab</sup>	1665 <sup>abc</sup>	1091 <sup>abc</sup>	1507 <sup>abc</sup>	504 <sup>c</sup>	543 <sup>c</sup>	667 <sup>bc</sup>	1204 <sup>abc</sup>	1706 <sup>abc</sup>	2599 <sup>a</sup>	2619 <sup>a</sup>	1501 <sup>abc</sup>	836 <sup>bc</sup>	635 <sup>c</sup>	2525 <sup>a</sup>

 $^{\dagger}\,$  \*, \*\* 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.

	Volatile composition of	of coriander essentia	l oil changing v	with harvest d	late at different	vegetative stages	of two consecutive seasons
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Compound	ANOVA <sup>↑</sup>	C1 Concentrat	C2 ion (mg k	C3 (g <sup>-1</sup> )	C4	C5	C6	C7	C8	C9	C10	C11	C12	C13	C14	C15
CVI	***	16 6a	10.2bc	21 Jabe	or rabe	16 7bc	12 7ab	E 0.90	21 Aabc	20 Aabc	20 1 abc	o1 oabc	10 0 <sup>bc</sup>	0.176	0.41¢	10 0bc
CV1	***	40.0	19.5 1.76 <sup>b</sup>	21.4 0.24 <sup>b</sup>	20.0 1.00 <sup>b</sup>	10.7	43.7 0 E0 <sup>b</sup>	0.12 <sup>b</sup>	21.4 0 5 2 <sup>b</sup>	20.4 0.02 <sup>b</sup>	1 00 <sup>b</sup>	21.2 1.10 <sup>b</sup>	19.0 0.06 <sup>b</sup>	9.17 0.47 <sup>b</sup>	9.41 0 50 <sup>b</sup>	1 00 <sup>b</sup>
CV2	*	11.4 1.19 <sup>a</sup>	1.70 2.00 <sup>a</sup>	0.24 0.49a	1.02 2.24 <sup>a</sup>	0.04 1.00 <sup>a</sup>	0.30	0.15	0.55	0.95	1.90 0.27 <sup>a</sup>	0.168	0.90 0.00 <sup>a</sup>	0.47 0.00 <sup>a</sup>	0.59	1.09 0.00a
CV3	***	1.13 0.61 <sup>ab</sup>	2.90 0.07 <sup>b</sup>	2.43 0.02 <sup>b</sup>	2.24 0.00 <sup>b</sup>	1.02 0.04 <sup>b</sup>	0.00 0.25 <sup>b</sup>	0.07	0.27 0.26 <sup>b</sup>	0.21 0.47 <sup>b</sup>	0.37 0.20 <sup>b</sup>	0.10 0.12 <sup>b</sup>	0.00 <sup>b</sup>	0.09 0.02 <sup>b</sup>	0.15 0.02 <sup>b</sup>	0.00 1.20a
CV4	***	0.01	0.07	0.05 0 Eocdef	0.00	0.04 0.42 <sup>def</sup>	0.23 1.96 <sup>a</sup>	0.10	0.30 1 = 1 <sup>abc</sup>	1.47 <sup>abcd</sup>	0.20 1.64 <sup>ab</sup>	0.15 1 1 = abcde	0.00	0.02	0.03	1.32 0.25 <sup>ef</sup>
CV5	***	0.92	0.20	0.52 0.40 <sup>bc</sup>	0.78 0.33 <sup>bc</sup>	0.43	1.80 0.05 <sup>b</sup>	0.20 0.00 <sup>c</sup>	1.51 0.00 <sup>bc</sup>	1.4/	1.04 0.02 <sup>c</sup>	1.15 0.15 <sup>c</sup>	0.30 0.00 <sup>c</sup>	0.08	0.03	0.35
CVB	***	4.01 1.40 <sup>a</sup>	0.00	0.40 0.10 <sup>c</sup>	0.23	0.00	0.85	0.09 0.00 <sup>c</sup>	0.22	0.10	0.03 1 = 1 <sup>a</sup>	0.15 0.02 <sup>ab</sup>	0.09 0.10 <sup>c</sup>	0.00	0.00	0.05
CV2	***	1.42 602 <sup>a</sup>	0.05 21.2 <sup>b</sup>	0.12 04.6°	0.05 24.1°	0.01 28.0°	0.10	0.00 17.1°	0.18 E1 1 <sup>c</sup>	0.20 20.0°	1.51 27.0°	0.93 26 4°	0.10 20.0°	0.37 11.0 <sup>c</sup>	0.04 0.41 <sup>c</sup>	0.21 26.4°
CV8	NC	7.02	213	94.0	34.1	28.9	99.5	17.1	51.1	20.9	27.9	20.4	30.9	11.0	0.41	30.4
CV9	IN5	7.93	5./3	8.99	2.70	2.83	2.84	0.10	1.04	0.10	0.03	0.01	0.03	0.00	0.05	0.35
CVID	***	48.1	4.85°	1.03	0.06	0.26	1.1/-	0.11-	2.64	0.42	1.52	0.42	3.40-	0.03-	0.04-	1.49
CVII	***	333-	51.2 <sup>-</sup>	17.8 <sup>-</sup>	5.09°	3.44 <sup>-</sup>	22.1 ···	1.85	0.64 <sup>-</sup>	0.29 <sup>-</sup>	2.46°	5.05°	2.51 <sup>-</sup>	1.95	1.83 <sup>-</sup>	6.5/-
CV12	***	29.8"	9.82 <sup>5</sup>	9.73 <sup>5</sup>	5.60	2.14 <sup>bc</sup>	9.61	1.49°	3.93	3.56	5.05	3.965	5.04	1.64°	2.29**	6.66°°
CV13	***	8.77ª	1.54	0.78	0.10	0.01	0.64 <sup>b</sup>	0.03	0.04 <sup>b</sup>	0.03 <sup>b</sup>	0.23	0.44 <sup>b</sup>	0.40	0.08	0.32	0.75
CV14	***	7.48 <sup>ª</sup>	0.87	0.63 <sup>bc</sup>	0.18 <sup>cu</sup>	0.05 <sup>u</sup>	0.26 <sup>cu</sup>	0.09 <sup>cu</sup>	0.33 <sup>bcu</sup>	0.19 <sup>cu</sup>	0.22 <sup>cu</sup>	0.10 <sup>cu</sup>	0.03 <sup>u</sup>	0.01 <sup>u</sup>	0.01 <sup>u</sup>	0.05 <sup>u</sup>
CV15	***	4.97 <sup>a</sup>	0.09 <sup>b</sup>	0.08 <sup>b</sup>	0.09 <sup>b</sup>	0.01 <sup>b</sup>	0.12 <sup>b</sup>	0.01 <sup>b</sup>	0.01 <sup>b</sup>	0.02 <sup>b</sup>	0.01 <sup>b</sup>	0.02 <sup>b</sup>	0.01 <sup>b</sup>	0.00 <sup>b</sup>	0.01 <sup>b</sup>	0.00 <sup>b</sup>
CV16	***	288 <sup>a</sup>	57.3 <sup>bc</sup>	51.0 <sup>bc</sup>	33.3 <sup>c</sup>	23.5 <sup>c</sup>	105 <sup>b</sup>	14.6 <sup>c</sup>	44.3 <sup>bc</sup>	37.7 <sup>bc</sup>	39.0 <sup>bc</sup>	24.9 <sup>c</sup>	$20.2^{\circ}$	6.87 <sup>c</sup>	8.98 <sup>c</sup>	17.4 <sup>c</sup>
CV17	***	8.64 <sup>a</sup>	1.24 <sup>bc</sup>	$1.53^{b}$	0.65 <sup>bc</sup>	0.11 <sup>c</sup>	0.95 <sup>bc</sup>	0.13 <sup>c</sup>	$0.56^{bc}$	0.19 <sup>c</sup>	0.36 <sup>bc</sup>	0.26 <sup>bc</sup>	0.04 <sup>c</sup>	0.01 <sup>c</sup>	$0.01^{c}$	$0.10^{c}$
CV18	***	389 <sup>a</sup>	94.4 <sup>b</sup>	74.4 <sup>bc</sup>	30.9 <sup>bcd</sup>	14.5 <sup>cd</sup>	69.5 <sup>bcd</sup>	10.1 <sup>d</sup>	49.1 <sup>bcd</sup>	48.6 <sup>bcd</sup>	59.0 <sup>bcd</sup>	41.1 <sup>bcd</sup>	21.3 <sup>cd</sup>	9.24 <sup>d</sup>	$12.2^{cd}$	25.9 <sup>cd</sup>
CV19	***	116 <sup>a</sup>	$21.3^{b}$	$15.2^{bcd}$	6.27 <sup>cde</sup>	1.78 <sup>de</sup>	18.3 <sup>bc</sup>	4.39 <sup>de</sup>	0.64 <sup>e</sup>	0.48 <sup>e</sup>	1.15 <sup>e</sup>	0.97 <sup>e</sup>	$0.02^{e}$	0.01 <sup>e</sup>	$0.02^{\rm e}$	0.04 <sup>e</sup>
CV20	***	46.2 <sup>a</sup>	7.00 <sup>bc</sup>	$8.53^{bc}$	4.27 <sup>c</sup>	1.79 <sup>c</sup>	$15.0^{b}$	0.55 <sup>c</sup>	$0.10^{c}$	0.03 <sup>c</sup>	0.05 <sup>c</sup>	$0.20^{\circ}$	$0.02^{c}$	0.01 <sup>c</sup>	0.14 <sup>c</sup>	$0.01^{\circ}$
CV21	***	$18.2^{a}$	$2.61^{bc}$	5.06 <sup>bc</sup>	4.02 <sup>bc</sup>	$1.86^{bc}$	$8.52^{b}$	0.46 <sup>c</sup>	$1.35^{c}$	$0.82^{c}$	1.19 <sup>c</sup>	$1.20^{c}$	0.80 <sup>c</sup>	0.13 <sup>c</sup>	$1.02^{c}$	1.17 <sup>c</sup>
CV22	***	47.4 <sup>a</sup>	8.16 <sup>bc</sup>	$5.32^{bc}$	2.49 <sup>c</sup>	1.26 <sup>c</sup>	13.6 <sup>b</sup>	1.07 <sup>c</sup>	$5.82^{bc}$	3.89 <sup>c</sup>	5.88 <sup>bc</sup>	4.70 <sup>bc</sup>	4.44 <sup>bc</sup>	2.01 <sup>c</sup>	2.60 <sup>c</sup>	4.54 <sup>bc</sup>
CV23	**	16.1 <sup>b</sup>	$1.00^{b}$	$1.59^{b}$	$0.65^{b}$	0.11 <sup>b</sup>	$1.70^{b}$	$0.12^{b}$	$0.74^{b}$	85.1 <sup>a</sup>	0.30 <sup>b</sup>	0.26 <sup>b</sup>	$0.10^{b}$	$0.02^{b}$	$0.05^{b}$	$0.08^{b}$
CV24	***	63.1 <sup>a</sup>	5.70 <sup>bc</sup>	5.59 <sup>bc</sup>	3.60 <sup>bc</sup>	1.56 <sup>c</sup>	16.5 <sup>b</sup>	0.88 <sup>c</sup>	3.41 <sup>bc</sup>	1.32 <sup>c</sup>	1.80 <sup>c</sup>	2.02 <sup>c</sup>	1.43 <sup>c</sup>	0.31 <sup>c</sup>	0.83 <sup>c</sup>	0.83 <sup>c</sup>
Total	***	2191 <sup>a</sup>	511 <sup>b</sup>	327 <sup>bcd</sup>	164 <sup>cde</sup>	103 <sup>de</sup>	433 <sup>bc</sup>	58.9 <sup>de</sup>	190 <sup>cde</sup>	227 <sup>cde</sup>	182 <sup>cde</sup>	137 <sup>de</sup>	111 <sup>de</sup>	43.5 <sup>e</sup>	50.0 <sup>de</sup>	123 <sup>de</sup>

<sup> $\dagger$ </sup> \*, \*\* 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  $\approx$  6909 mg kg<sup>-1</sup> of fresh weight. Carvone (1465–17092 mg kg<sup>-1</sup> for M7 and M5 vegetative stages, respectively), limonene (282-4525 mg kg<sup>-1</sup> for M7 and M5 vegetative stages, respectively), trans-carveol  $(3.57-322 \text{ mg kg}^{-1} \text{ for M13 and M5 vegetative stages, respectively}),$ *cis*-carveol (17.1–300 mg kg<sup>-1</sup> for M7 and M5 vegetative stages, respectively),  $\beta$ -pinene (9.57–155 mg kg<sup>-1</sup> for M7 and M5 vegetative stages, respectively), santene (8.58-137 mg kg<sup>-1</sup> for M7 and M5 vegetative stages, respectively), trans-caryophyllene (9.35-119 mg kg<sup>-1</sup> for M13 and M1 vegetative stages, respectively), trans- $\beta$ -ocimene  $(6.90-101 \text{ mg kg}^{-1} \text{ for M13 and M5 vegetative stages, respectively})$ and myrcene (4.07–101 mg kg<sup>-1</sup> 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 Dcarvone (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  $(17,092 \text{ mg kg}^{-1})$  and limonene (4525 mg kg<sup>-1</sup>) was found in M5. While, the lowest concentration of carvone (1465 mg kg<sup>-1</sup>) and limonene (282 mg kg<sup>-1</sup>) were obtained from M7. Zawiślak (2011) studied the effect of plant harvest term (midJune-plants in vegetative phase, mid-July-beginning of flowering, midAugust-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<sup>-1</sup>) 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<sup>-1</sup>, for dill, the highest content of volatile compounds was obtained in D15 (19 weeks after planting

Volatile com	position of r	mint essential	l oil changir	a with harves	t date at diff	erent vegetative	stages of two	consecutive seasons
volatile com	position of i	mini essentia	i on changn	ig with haives	t uate at um	ereni vegetative	stages of two	consecutive seasons.

Compound	ANOVA <sup>†</sup>	M1 Concentr	M2 ation (mg l	M3 (xg <sup>-1</sup> )	M4	M5	M6	M7	M8	М9	M10	M11	M12	M13	M14	M15
MV1	***	122 <sup>ab b</sup>	65.3 <sup>cd</sup>	46.8 <sup>cdef</sup>	56.0 <sup>cde</sup>	137 <sup>a</sup>	82.6 <sup>bc</sup>	8.58 <sup>f</sup>	23.1 <sup>def</sup>	23.0 <sup>def</sup>	17.3 <sup>ef</sup>	16.6 <sup>ef</sup>	17.5 <sup>ef</sup>	8.82 <sup>f</sup>	$24.1^{def}$	23.8 <sup>def</sup>
MV2	***	8.55 <sup>bc</sup>	4.89 <sup>cde</sup>	3.84 <sup>def</sup>	5.11 <sup>cd</sup>	13.1 <sup>a</sup>	9.10 <sup>b</sup>	0.95 <sup>ef</sup>	2.20 <sup>def</sup>	2.27 <sup>def</sup>	1.80 <sup>def</sup>	1.44 <sup>def</sup>	1.28 <sup>def</sup>	0.83 <sup>f</sup>	2.76 <sup>def</sup>	3.49 <sup>def</sup>
MV3	***	98.4 <sup>a</sup>	55.7 <sup>bc</sup>	34.8 <sup>cde</sup>	47.0 <sup>bcd</sup>	101 <sup>a</sup>	74.2 <sup>ab</sup>	7.18 <sup>e</sup>	12.6 <sup>de</sup>	13.8 <sup>de</sup>	9.73 <sup>e</sup>	9.62 <sup>e</sup>	8.70 <sup>e</sup>	4.07 <sup>e</sup>	12.5 <sup>de</sup>	13.0 <sup>de</sup>
MV4	***	$137^{ab}$	77.8 <sup>cd</sup>	52.7 <sup>cdef</sup>	63.0 <sup>cde</sup>	155 <sup>a</sup>	95.6 <sup>bc</sup>	9.57 <sup>f</sup>	24.1 <sup>ef</sup>	25.3 <sup>ef</sup>	19.3 <sup>ef</sup>	19.0 <sup>ef</sup>	$21.0^{ef}$	10.4 <sup>f</sup>	30.6 <sup>def</sup>	27.9 <sup>ef</sup>
MV5	***	6.49 <sup>ab</sup>	5.68 <sup>b</sup>	$4.53^{bc}$	6.64 <sup>ab</sup>	$10.5^{\mathrm{a}}$	6.39 <sup>ab</sup>	1.11 <sup>c</sup>	0.58 <sup>c</sup>	0.92 <sup>c</sup>	0.75 <sup>c</sup>	0.63 <sup>c</sup>	0.40 <sup>c</sup>	0.13 <sup>c</sup>	0.84 <sup>c</sup>	0.63 <sup>c</sup>
MV6	**	$11.0^{a}$	6.35 <sup>a</sup>	4.26 <sup>a</sup>	5.10 <sup>a</sup>	$11.2^{a}$	5.46 <sup>a</sup>	0.69 <sup>a</sup>	1.29 <sup>a</sup>	$1.58^{a}$	0.95 <sup>a</sup>	0.68 <sup>a</sup>	11.7 <sup>a</sup>	0.47 <sup>a</sup>	1.37 <sup>a</sup>	$2.37^{a}$
MV7	***	$2.87^{ab}$	$1.94^{abc}$	$1.59^{bc}$	$1.32^{bc}$	3.69 <sup>a</sup>	$1.80^{bc}$	0.31 <sup>c</sup>	0.51 <sup>c</sup>	0.79 <sup>c</sup>	0.40 <sup>c</sup>	0.39 <sup>c</sup>	0.66 <sup>c</sup>	0.42 <sup>c</sup>	1.97 <sup>abc</sup>	$2.74^{ab}$
MV8	***	2902 <sup>b</sup>	2101 <sup>bcd</sup>	1493 <sup>cdef</sup>	1663 <sup>bcde</sup>	4525 <sup>a</sup>	$2527^{bc}$	$282^{f}$	609 <sup>ef</sup>	712 <sup>ef</sup>	498 <sup>ef</sup>	476 <sup>ef</sup>	600 <sup>ef</sup>	317 <sup>f</sup>	805 <sup>def</sup>	722 <sup>ef</sup>
MV9	***	70.3 <sup>ab</sup>	43.2 <sup>bc</sup>	30.7 <sup>cd</sup>	36.6 <sup>cd</sup>	101 <sup>a</sup>	92.4 <sup>a</sup>	9.08 <sup>d</sup>	19.7 <sup>cd</sup>	21.1 <sup>cd</sup>	18.3 <sup>cd</sup>	15.7 <sup>cd</sup>	13.9 <sup>cd</sup>	6.90 <sup>d</sup>	19.7 <sup>cd</sup>	17.9 <sup>cd</sup>
MV10	***	5.70 <sup>ab</sup>	3.99 <sup>abcd</sup>	2.71 <sup>bcd</sup>	2.58 <sup>bcd</sup>	7.24 <sup>a</sup>	5.73 <sup>ab</sup>	0.78 <sup>d</sup>	1.30 <sup>cd</sup>	1.56 <sup>cd</sup>	1.38 <sup>cd</sup>	1.34 <sup>cd</sup>	1.49 <sup>cd</sup>	0.97 <sup>cd</sup>	4.16 <sup>abc</sup>	5.01 <sup>ab</sup>
MV11	***	64.3 <sup>cd</sup>	39.4 <sup>cde</sup>	24.1 <sup>de</sup>	37.4 <sup>cde</sup>	$113^{ab}$	123 <sup>a</sup>	14.5 <sup>e</sup>	35.2 <sup>cde</sup>	44.8 <sup>cde</sup>	33.6 <sup>cde</sup>	33.8 <sup>cde</sup>	33.4 <sup>cde</sup>	19.1 <sup>e</sup>	74.5 <sup>bc</sup>	75.8 <sup>bc</sup>
MV12	***	$37.0^{bc}$	23.7 <sup>bcd</sup>	$17.0^{d}$	23.1 <sup>bcd</sup>	57.5 <sup>a</sup>	38.0 <sup>ab</sup>	5.08 <sup>d</sup>	10.6 <sup>d</sup>	12.4 <sup>d</sup>	10.1 <sup>d</sup>	8.95 <sup>d</sup>	8.89 <sup>d</sup>	4.57 <sup>d</sup>	17.4 <sup>cd</sup>	12.1 <sup>d</sup>
MV13	***	6.88 <sup>a</sup>	4.25 <sup>bc</sup>	$2.25^{cde}$	3.21 <sup>cd</sup>	6.91 <sup>a</sup>	6.15 <sup>ab</sup>	0.75 <sup>de</sup>	1.28 <sup>de</sup>	1.71 <sup>de</sup>	1.37 <sup>de</sup>	1.27 <sup>de</sup>	1.06 <sup>de</sup>	0.63 <sup>e</sup>	$2.20^{cde}$	$2.22^{cde}$
MV14	***	6.96 <sup>ab</sup>	4.95 <sup>bc</sup>	2.69 <sup>cde</sup>	3.87 <sup>bcd</sup>	8.59 <sup>a</sup>	6.83 <sup>ab</sup>	0.61 <sup>e</sup>	1.31 <sup>de</sup>	1.53 <sup>de</sup>	1.09 <sup>de</sup>	0.90 <sup>de</sup>	$1.05^{de}$	$0.82^{de}$	1.59 <sup>de</sup>	1.17 <sup>de</sup>
MV15	***	19.3 <sup>ab</sup>	11.7 <sup>cd</sup>	7.45 <sup>def</sup>	8.45 <sup>cde</sup>	23.7 <sup>a</sup>	14.9 <sup>bc</sup>	$1.50^{f}$	2.88 <sup>ef</sup>	3.05 <sup>ef</sup>	2.05 <sup>ef</sup>	$1.78^{f}$	$2.21^{ef}$	$1.51^{f}$	3.40 <sup>ef</sup>	1.79 <sup>f</sup>
MV16	***	0.00 <sup>e</sup>	0.00 <sup>e</sup>	0.24 <sup>de</sup>	0.45 <sup>de</sup>	0.00 <sup>e</sup>	0.00 <sup>e</sup>	0.00 <sup>e</sup>	6.54 <sup>a</sup>	6.70 <sup>a</sup>	4.30 <sup>b</sup>	$3.52^{bc}$	3.45 <sup>bc</sup>	2.26 <sup>cd</sup>	6.48 <sup>a</sup>	6.45 <sup>a</sup>
MV17	***	16.0 <sup>a</sup>	11.9 <sup>abc</sup>	6.88 <sup>bcd</sup>	6.75 <sup>cd</sup>	16.6 <sup>a</sup>	14.8 <sup>ab</sup>	1.81 <sup>d</sup>	4.54 <sup>cd</sup>	5.34 <sup>cd</sup>	4.80 <sup>cd</sup>	4.44 <sup>cd</sup>	4.39 <sup>cd</sup>	2.94 <sup>d</sup>	$12.3^{abc}$	$11.0^{abc}$
MV18	***	107 <sup>bcde</sup>	59.3 <sup>def</sup>	36.7 <sup>ef</sup>	63.8 <sup>def</sup>	300 <sup>a</sup>	148 <sup>bc</sup>	$17.1^{f}$	67.6 <sup>def</sup>	74.7 <sup>def</sup>	116 <sup>bcd</sup>	87.0 <sup>bcdef</sup>	59.7 <sup>def</sup>	$32.6^{f}$	157 <sup>b</sup>	82.1 <sup>cdef</sup>
MV19	***	$170^{abc}$	$141^{abc}$	79.2 <sup>c</sup>	137 <sup>bc</sup>	322 <sup>a</sup>	$312^{ab}$	26.0 <sup>c</sup>	8.35 <sup>c</sup>	7.47 <sup>c</sup>	4.85 <sup>c</sup>	4.77 <sup>c</sup>	4.82 <sup>c</sup>	3.57 <sup>c</sup>	$8.12^{c}$	8.99 <sup>c</sup>
MV20	***	9745 <sup>b</sup>	8257 <sup>b</sup>	5453 <sup>bc</sup>	6035 <sup>bc</sup>	$17092^{a}$	$10315^{b}$	1465 <sup>c</sup>	2533 <sup>c</sup>	2784 <sup>c</sup>	2021 <sup>c</sup>	1844 <sup>c</sup>	1901 <sup>c</sup>	1071 <sup>c</sup>	2592 <sup>c</sup>	1877 <sup>c</sup>
MV21	***	0.46 <sup>c</sup>	0.13 <sup>c</sup>	0.49 <sup>c</sup>	1.51 <sup>bc</sup>	6.59 <sup>a</sup>	$2.96^{b}$	0.37 <sup>c</sup>	0.54 <sup>c</sup>	0.92 <sup>c</sup>	0.88 <sup>c</sup>	0.67 <sup>c</sup>	0.38 <sup>c</sup>	$0.13^{c}$	$0.72^{\circ}$	0.45 <sup>c</sup>
MV22	***	$14.2^{bc}$	9.09 <sup>c</sup>	$13.0^{bc}$	12.0 <sup>bc</sup>	111 <sup>a</sup>	$37.8^{b}$	8.29 <sup>c</sup>	13.3 <sup>bc</sup>	9.13 <sup>c</sup>	26.6 <sup>bc</sup>	$22.0^{bc}$	19.2 <sup>bc</sup>	4.85 <sup>c</sup>	$30.7^{bc}$	15.5 <sup>bc</sup>
MV23	***	62.1 <sup>a</sup>	38.3 <sup>bc</sup>	21.5 <sup>cde</sup>	28.4 <sup>cd</sup>	59.8 <sup>ab</sup>	52.5 <sup>ab</sup>	6.01 <sup>de</sup>	14.7 <sup>de</sup>	17.4 <sup>cde</sup>	10.6 <sup>de</sup>	7.78 <sup>de</sup>	9.22 <sup>de</sup>	5.56 <sup>e</sup>	15.9 <sup>cde</sup>	14.0 <sup>de</sup>
MV24	***	24.9 <sup>a</sup>	17.3 <sup>b</sup>	9.20 <sup>cd</sup>	9.07 <sup>cd</sup>	$18.8^{ab}$	14.7 <sup>bc</sup>	1.57 <sup>e</sup>	2.24 <sup>e</sup>	3.35 <sup>de</sup>	1.99 <sup>e</sup>	$1.62^{\rm e}$	2.34 <sup>e</sup>	1.39 <sup>e</sup>	3.47 <sup>de</sup>	3.63 <sup>de</sup>
MV25	***	117 <sup>a</sup>	74.4 <sup>abc</sup>	37.1 <sup>bcd</sup>	53.0 <sup>bcd</sup>	99.7 <sup>a</sup>	81.8 <sup>ab</sup>	11.7 <sup>d</sup>	$22.2^{d}$	31.8 <sup>cd</sup>	19.3 <sup>d</sup>	16.3 <sup>d</sup>	$18.3^{d}$	9.35 <sup>d</sup>	26.9 <sup>d</sup>	35.5 <sup>cd</sup>
MV26	***	9.22 <sup>ab</sup>	6.65 <sup>bc</sup>	3.98 <sup>cde</sup>	5.70 <sup>bcd</sup>	$12.5^{a}$	6.55 <sup>bc</sup>	$1.08^{\rm e}$	1.73 <sup>e</sup>	2.61 <sup>de</sup>	0.97 <sup>e</sup>	0.75 <sup>e</sup>	1.21 <sup>e</sup>	0.58 <sup>e</sup>	1.61 <sup>e</sup>	2.56 <sup>de</sup>
MV27	***	21.6 <sup>a</sup>	13.7 <sup>abc</sup>	7.01 <sup>cde</sup>	9.72 <sup>bcd</sup>	$15.7^{ab}$	$14.5^{abc}$	2.08 <sup>de</sup>	3.55 <sup>de</sup>	5.01 <sup>de</sup>	$2.80^{de}$	2.31 <sup>de</sup>	2.50 <sup>de</sup>	1.48 <sup>e</sup>	3.44 <sup>de</sup>	5.09 <sup>de</sup>
Total	***	13785b	11079b	7397 <sup>bc</sup>	8325 <sup>bc</sup>	23329 <sup>a</sup>	14090 <sup>b</sup>	1884 <sup>c</sup>	3423 <sup>c</sup>	3815 <sup>c</sup>	2829 <sup>c</sup>	2583 <sup>c</sup>	2750 <sup>c</sup>	1512 <sup>c</sup>	3861 <sup>c</sup>	2974 <sup>c</sup>

<sup> $\dagger$ </sup> \*, \*\* and \*\*\*, significant at p < 0.05, 0.01 and 0.001, respectively.

<sup>b</sup>  $\ddagger$  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<sup>-1</sup>. 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<sup>-1</sup>), 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<sup>-1</sup>.

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<sup>-1</sup> 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

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.scienta.2020.109336.

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