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Essential oil content and components of *Ferulago angulate* (Schltdl.)Boiss affected by foliar application of some important micronutrient

Mehrab Yadegari*

Research Center of Nutrition and Organic Products (R.C.N.O.P), Shahrekord Branch, Islamic Azad University, Shahrekord, Iran;

***Email:** mehrabyadegari@gmail.com

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ABSTRACT

Background & Aim: *Ferulago angulate* (Schltdl.) Boiss belongs to the family Apiaceae, is one of the important and endangered endemic species in Iran. The present study was conducted to investigate the effects of micronutrient elements on the content and composition of essential oil (EOs) of *F. angulate* shoots in southwestern Iran (Shahrekord) in 2022 and 2023.

Experimental: Four foliar fertilizers including Fe, Cu, Zn and Mn were applied in 20, 40 and 60 mgL^{-1} in RCBD design by factorial layout and 3 replications. The seeds of *F. angulate* were obtained from Forest and Rangeland Institute, Iran. The EOs content and composition were determined by Clevenger apparatus and GC-MS analysis, respectively.

Results: Results obtained from gas chromatography/mass spectrometry (GC-MS) showed 15 components in EOs. Micronutrients used significantly affected the EOs content/ composition of *F. angulate.* In two years, the highest content of EOs (0.59-0.68%) was obtained in plants treated with 40 mgL^{-1} of micronutrients ($Fe_2Cu_2Zn_2Mn_2$) and the lowest content (0.37-0.41%) obtained in control plants, however the plants treated by 60 $mg.L^{-1}$ of micronutrients in most characters showed the same group with control plants. The most important chemical compounds related to *F. angulate* EOs quality including alpha-pinene (20.13-35.88%), alpha–thujene (12.67-18.14%), cis-ocimene (11.41-22.01%) from monoterpene hydrocarbons category and 4-thujanol (10.54-1.01 %) from oxygenated monoterpenes category were identified. Alpha-pinene belonging to monoterpene hydrocarbons was the predominant constituent of the EOs of *F. angulate.*

Recommended applications/industries: The application of micronutrients with a concentration of 40 mgL-1 may be a good strategy to improve the EOs quantity and quality of *F. angulate* in cold and semi-arid climates.

1. Introduction

The genus *Ferulago* is a member of the Apiaceae (Umbelliferae) family and consists of 49 species throughout the world. In traditional medicine, *Ferulago* species have been utilized as sedatives, carminatives, aphrodisiacs, for increasing milk secretion and for the treatment of bronchitis, skin diseases, and eye diseases (Selcuk and Dikpinar, 2021). This genus is common throughout the Mediterranean area, particularly western Iran, Turkey, Iraq, Greece, Serbia, and Macedonia. Plants of this genus have been used since ancient times as spices and salad, as a tonic and as remedies for digestive pains and hemorrhoids, mainly in Turkey and Iran, and these properties have been confirmed by numerous pharmacological studies. These plants, therefore, possessed antibacterial, antifungal, antioxidant, anticoagulant and anti- inflammatory properties, hepatoprotective and nephroprotective effects and, lastly, insecticidal activities (Badalamenti *et al.,* 2021). Due to their multiple properties, the chemical compositions and biological activities have been extensively studied taking into consideration the different parts of the plants (roots, stems, fruits and leaves). Most of the therapeutic effects may be due to the presence of various phytoconstituents such as coumarins, EOs, terpenoids and flavonoids (Razavi *et al.,* 2015). The *Ferulago* genus, rich in secondary metabolites and various biological activities, seems to be a potential source for the development of new drugs and for application in the agro-food field (Selcuk and Dikpinar, 2021). The major compounds in this plant are flavonoids, sesquiterpene aryl esters, phenolic acids, aromatic, and sitosterols isolated from the *Ferulago* species have been assessed phytochemically. Furanocoumarins are the major phytochemical group in the *Ferulago* genus. Antibacterial and antifungal activities of *Ferulago* spp. have been previously investigated, and their significant inhibitory effects against microorganisms have been reported (Shahbazi *et al.,* 2016; Badalamenti *et al.,* 2023). *F. angulate* commonly known as Chavil in Persian is an aromatic plant growing in the west of Iran. It is a perennial glabrous herb with 40–100 cm height, cylindrical dichotomously branched stem, shortly petiolate, pinnate-sect, terminal segment, linear-oblong, acute leaves and yellowish, synflorescence corymbose-paniculiform flowers (Mozaffarian, 2008).

The aerial parts of *F. angulate* have been used as a food and flavouring agent. In Iran, the aerial parts of *F. angulate*, are used in meat, dairy, and ghee oil as a natural food preservative. *Ferulago* species have been used for treating ulcers, snakebites, intestinal worms and haemorrhoids (Golfakhrabadi *et al.,* 2015). The essential oils (EOs) extracted of *F. angulate*were traditionally used to treat bacterial and fungal infections in the people of Iran for several centuries (Azarbani *et al.,* 2023). The previous studies showed that the EOs *F. angulate* was characterized by large amounts of monoterpene hydrocarbon. Research on EOs of *F. angulate* showed that the major constituents were Z-beta-ocimene, bornyl acetate, δ-terpinolene, sabinene (Razavi *et al.,* 2015; Ghasemi Pirbalouti *et al.,* 2016); alpha-pinene, sabinene, (Z)-beta-ocimene, p-cymene, alpha-phellandrene, beta-phellandrene (Moghaddam *et al.,* 2018; Golfakhrabadi *et al.,* 2015); Cis-beta-ocimene, alpha-pinene, alpha-phellandrene (Mumivand *et al.,* 2019); alpha-pinene, alpha-thujene, alpha-phellandrene, cis-ocimene, beta-phellandrene, beta-ocimene (Shahbazi, 2016; Safari *et al.,* 2019). alpha-pinene, bornyl acetate, terpinolene, octane, beta-pinene, alpha-phellandrene, dodecane, germacren-d, caryophyllene oxide (Azarbani *et al.,* 2023).

When nutrient deficiency cannot be corrected through soil application, foliar nutrition is adopted as an alternate method (Marschner, 1995). It has been shown that micronutrients such as Fe, Mn, Zn and Cu are necessary for plant intensification in much lower amounts for plant intensification than those of the primary nutrients (Bilal *et al.,* 2020). Four important micronutrients used in medicinal plants are Fe, Cu, Mn and Cu. Iron (Fe) is one of the four essential nutrient elements needed by plants and is a key element in cytochrome structure. In addition, plants treated with this micronutrient produce more yield (Majeed *et al.,* 2020). Copper (Cu) is another essential microelement in higher plants as it occurs as part of the prosthetic groups of several enzymes. Zinc (Zn) is a building block of many proteins and an important chemical element in biological activity. Zn acts on enzymatic activation and cell division, so it has been shown that its deficiency causes cell damage, low protein and carbohydrate synthesis, impaired growth and development, and low crop yields (Alamer *et al.,* 2020; Cakmak *et al.,* 2017; Figueiredo *et al.,* 2017). Manganese (Mn) is involved in many biochemical functions, primarily acting as an activator of enzymes such as dehydrogenases and decarboxylases involved in respiration, amino acid and lignin synthesis, and hormone concentrations (Alejandro *et al.,* 2020). Foliar application of these micronutrients have important effects in morpho-physiological attributes such as chlorophyll, phenol and relative water content that made more EOs content and composition in *Rosa damascena* (Yadegari, 2023), *Satureja* (Bani Taba *et al.,* 2022), *Melissa officinalis* (Yadegari, 2017b), *Carthamus tinctorius* (Galavi *et al.,* 2012), *Calendula officinalis* L. *Borago officinalis*, *Alyssum desertorum* and *Thymus vulgaris* (Yadegari, 2015; 2017a), *Anethum graveolens* (Rostaei *et al.,* 2018), *Matricaria chamomilla* (Nasiri *et al.,* 2010) and *Coriandrum sativum* (Said-Al Ahl and Omer, 2009).

Foliar fertilization is particularly useful to meet the basic needs of plants for one or more micro- or macro-nutrients, especially trace minerals. It also helps correct deficiencies, strengthen weak or damaged crops, and enhance growth (Aziz *et al.*, 2019). As far as I found there has been no comprehensive study on the foliar application of important micronutrients on content and compositions of *F. angulate* Eos grown in the Iran. Hence, here I report the volatile oil compositions of *F. angulate* from the west of Iran. Additionally, I evaluate the comparitions between content and the main compositions of EOs of this plant. The aim of this research was to determine the effects of foliar applications of iron, zinc, copper and manganese on EOs content and composition in *F. angulate* Boiss. to introduce the best combination of these micronutrients for better yield in this multipurpose plant.

2. Materials and Methods

2.1. Plant material and fertilizers

Four foliar fertilizers including Librel Fe-Lo, Librel Cu, Librel Zn and Librel Mn were applied and all of them are mineral fertilizers. Librel Fe-Lo contains 13.2% chelated iron, Librel Zn is a foliar fertilizer that contains 14% Zinc in chelated form, Librel Cu has 14% copper in chelated form and Librel Mn contains of 13% Mn chelated with EDTA (England and Germany). Theses fertilizers were sprayed at three concentrations (for example Fe_1, Fe_2 and Fe_3 indicates the concentration of 20, 40 and 60 $mgL^{-1}$of Fe, respectively). The concentrations were similar in other micronutrients. The foliar application was done in three stages once every 10 days (before harvest) in the early morning. The control plants had no treatment with micronutrient. For soil analysis, the soil samples were taken from three randomly selected sites in each plot from 0-15 and 15-30 cm of depth. The samples were homogenized, mixed and passed through a 2 mm filter for determination of soil physical and chemical characteristics. Soil moisture was measured by a TDR device (PMS-714, Lutron, Taiwan) following the manufacturers' protocol.

2.2. Experimental conditions

This study was conducted from spring (May) 2022 to fall (September) 2023 at the Research Farm of Islamic Azad University, Shahrekord Branch, Iran. Based on the Köppen climate classification, the climate of the area of study is classified as cold and semiarid. The present study was conducted in a randomized complete block design (RCBD) with three replications. In each year, treatments were applied in stage of V_4-V_8 and the sampling was performed in flowering stage. The soil (typic calci xerocrepts) physical and chemical properties and climatic properties of the region are listed in Table 1 and 2, respectively. The top-soil of the experimental plot area was kept moist throughout the growing season when necessary. The aerial parts and inflorescences of *F. angulate* were hand-harvested at the flowering and then were dried in the shade at room temperature (25±4°C) for two weeks with the moisture content fixed at around 14 to 16%. The samples were ground to fine powder using a micro hammer mill and passed through a sieve (mesh 20). The EOs was extracted from 100 g of powdered tissue by hydro-distillation method using the Clevenger apparatus (Ashk-e-Shishe Co., Tehran, Iran) with 500 mL water for 3 h according to the British Pharmacopoeia.

Table 1. Physico-chemical properties of research farm in two years.

Characters	Year	
	2022	2023
N_{total} (%)	0.18	0.25
Organic matter (%)	0.75	0.83
pH	7.82	7.73
P ($mg·kg^{-1}$)	16.44	15.55
K ($mg·kg^{-1}$)	312	308
Ca ($mg·kg^{-1}$)	3.45	3.78
Mn ($mg·kg^{-1}$)	8.44	8.34
Fe ($mg·kg^{-1}$)	1.02	1.91
Cu ($mg·kg^{-1}$)	0.55	0.43
Zn ($mg·kg^{-1}$)	0.82	0.79
EC ($dS·m^{-1}$)	0.62	0.65
Texture	Clay loam	Clay loam

Table 2. Climatic properties of research farm.

Average annual precipitation (mm)	Average of annual temperature (ºC)	Average maximum temperature (ºC)	Average minimum temperature (ºC)	Height (m)	Latitude and longitude
331	11.7	23.9	-1.8	2060	32019/N-50051/E

2.3. Plant material and agronomic practices

The seeds of *F. angulate* Boiss. (Apiaceae) were obtained from Forest and Rangeland Institute, Iran. Firstly, the seeds were sterilized and sown on May 2022. After about 45-50 days from sowing, when the seedlings had 4-6 true leaves with 8-10 cm tall, they were planted in the experimental field. The dimensions of each experimental plot were 4.0 × 3.0 m and the distance between replicates was 2 m. No inorganic fertilizer and systemic pesticide were used during the experiment, and weeds control was done manually.

2.4. Essential oil extraction

EOs content was determined by distilling shoots in the Clevenger apparatus. The content of 100 g of shoots was placed in 6 L Clevenger- type distillation apparatus and distilled for 5 h with 3 L of pure water. The oil content of *F. angulate* obtained at the end of distillation and the percentage of EOs (w/w) was calculated by considering the oil density of 0.858. All the EOs samples were dried over sodium sulphate and stored at 4°C until gas chromatography–mass spectrometry (GC–MS) analysis. GC analysis was done on an Agilent Technologies 7890 GC equipped with FID and a HP-5MS 5% capillary column. The carrier gas was helium (99.99% purity) at a flow rate of 0.8 mL/min. Initial column temperature was 60°C and programmed to increase at 4°C/min to 280°C. The split ratio was 40:1. The injector temperature was set at 300°C and 0.1 mL of each sample was injected manually in the split mode. GC–MS analyses were carried out on a Thermo Finnigan Trace 2000 GC-MS system equipped with an HP-5MS capillary column (30 m × 0.25 mm i.d., film thickness 0.25 μm). The oven temperature was held at 120°C for 5 min and then programmed to reach 280°C at a rate of 10°C/min. The detector temperature was 260°C and the injector temperature was 260°C. The compositions of the EOs were identified by comparison of their retention indices relative to a series of *n*-alkanes (C7-C24), retention times and mass spectra with those of authentic samples in Wiley library (Adams, 2007).

2.5. Data Analysis

After Bartlett test, all data were subjected to ANOVA and simple Pearson correlation indices using the statistical computer package SAS v.11 and treatment means separated using LSD's multiple range test at P<0.05 and P<0.01 levels.

3. Results and discussion

The results showed that the effects of foliar-spraying on EOs content was significant (P≤0.01) (Table 3). The results obtained from GC-MS indicated the presence of 15 components in the Eos of *F. angulate* and significant differences in chemical compositions of EOs were observed between treatments (Table 3). The maximum EOs content was obtained in the foliar-sprayings of 40 mgL^{-1} (0.59-0.68%), whereas the minimum (0.37-0.41%) was observed in the control (Tables 4-9). In present study, the use of 40 mgL^{-1} improved the content of *F. angulate* EOs. According to the results of the chemical analysis of the EOs from *F. angulate* by GC/MS, the most important chemical compounds related to the quality of *F. angulate* EOs including alpha-thujene (12.67-18.14%), alpha-pinene (20.13-35.88%), beta-phellandrene (1.11-7.12%), cis-ocimene (11.41-22.01%) and 4-thujanol (1.01-10.54%).were identified (Tables 4-9). alpha-pinene belonging to monoterpene hydrocarbons was the predominant constituent of the *F. angulate* EOs (Tables 4-9). The simple effects of the foliar application of each micronutrients were significant on the percentages of some of the major compounds of the EOs (Table 3). In general, the foliar application of micronutrients in 40 mgL^{-1} increased the percentage of almost all of the compounds compared to the control (Tables 4-9). The interaction effects of micronutrients (Fe, Zn, Cu and Mn) on the concentrations of constituents of EOs such as alpha-thujene, alpha-pinene, beta-phellandrene, cis-ocimene and 4-thujanol ($P \leq 0.01$) was significant (Table 3). The highest levels of alpha-thujene, alpha-pinene, beta-phellandrene, cis-ocimene and 4-thujanol were obtained under 40 mgL^{-1} treatments (18.14, 35.88, 7.12, 22.01 and 10.54%, respectively) (Tables 4-9).

According to the biennial results of the chemical analysis of the EOs from *F. angulate* by GC/MS, the most important chemical compounds that determine the quality of *F. angulate* EOs including alpha-thujene, alpha-pinene, beta-phellandrene, cis-ocimene and 4-thujanol were identified. By comparing the data of two years, there were positive correlations between EOs content and main components and a high correlation was observed between EOs content and alpha-thujene, alpha-pinene, beta-myrcene, alpha-phellandrene, beta-phellandrene, cis-ocimene, beta-ocimene and sabinene (Table 10).

Table 3. Complex analysis of variance of variation of EOs content and main compositions in *F. angulate* by different micronutrients.

SOV^{z}	df^{y}	EOs	4-thujanol	Cis-ocimene	beta-phellandrene	alpha-phellandrene	beta-myrcene	alpha-pinene	alpha-Thujene
Year(Y)	1	6.62^{ns}	12.5**	0.007^{ns}	25.2**	5.56^{ns}	1.21**	3.44**	14.4**
R/Y	4	8.7	1.4	0.009	1.3	6.7	1.1	0.32	0.4
Copper (Cu)	3	27.5**	0.5^{ns}	1.11**	1.1^{ns}	14.4**	12.9**	1.8^{ns}	1.3^{ns}
Manganese (Mn)	3	4.1^{ns}	11.2**	0.02^{ns}	12.2**	12.6**	14.6**	1.8^{ns}	2.1^{ns}
Iron (Fe)	3	42.8**	12.9**	1.24**	14.4^{ns}	32.8**	16.8**	15.9**	22.4**
Zinc (Zn)	3	33.6**	14.2**	1.05**	11.1**	22.6**	8.5**	11.3**	11.9**
Cu × Mn	9	15.9**	25.2**	0.98**	0.84**	2.9**	7.1**	108**	12.8**
Cu × Fe	9	17.8**	32.4**	0.89**	0.92**	6.8**	6.4**	9.9**	10.4**
Cu × Zn	9	22.1**	21.8**	0.21**	0.77**	3.1**	5.3**	8.8**	12.1**
Mn × Fe	9	26.9**	27.1**	0.59**	0.92**	9.9**	7.9**	7.7**	14.1**
Mn × Zn	9	24.8**	12.1**	0.39**	0.88**	2.8**	8.9**	6.7**	10.9**
Fe × Zn	9	18.1**	8.4**	0.64**	17.8**	1.98**	5.9**	8.5**	9.2*
Cu × Mn × Fe	27	21.5**	5.1**	0.42**	0.91**	1.5**	3.4**	0.94^{ns}	10.8**
Cu × Mn × Zn	27	14.8**	4.6**	0.82**	0.99**	1.8**	4.1**	1.2^{ns}	1.9^{ns}
Cu × Fe × Zn	27	15.4**	4.22**	0.43**	0.2^{ns}	1.4**	0.44^{ns}	0.2^{ns}	0.7^{ns}
Fe × Zn × Mn	27	12.6**	5.6**	0.012^{ns}	0.1^{ns}	1.6**	0.57^{ns}	0.1^{ns}	0.8^{ns}
Cu × Zn × Mn × Fe	81	1.7^{ns}	0.28^{ns}	0.02^{ns}	0.11^{ns}	0.22^{ns}	0.21^{ns}	0.8^{ns}	0.9^{ns}
T(Cu,Zn,Mn,Fe)×Y	255	0.55^{ns}	0.33^{ns}	0.001^{ns}	0.12^{ns}	0.22^{ns}	0.32^{ns}	0.43^{ns}	0.28^{ns}
E	1020	1.9	0.45	0.02	0.11	0.3	0.55	0.88	2.23
	CV^{x}	6.5	4.54	5.65	8.8	7.7	6.5	10.1	11.1

^{z} SOV: source of variation, ^{y}df: degree of freedom, ^{x}CV: coefficient of variation, *, ** significant at P=0.05 and P=0.01 probability levels, respectively.

Table 3. Continued complex analysis of variance of variation of EOs content and main compositions in *F. angulate* by different micronutrients

SOV^{z}	df^{y}	Henicosane	Nonadecane	Bicyclo-germacrene	Naphthalene methanol	Bornyl acetate	Verbenone	Sabinene	beta-ocimene
Year(Y)	1	0.005^{ns}	2.2**	24.2**	11.99^{ns}	0.88^{ns}	12.55**	0.99^{ns}	1.7**
R/Y	4	0.07	0.3	1.2	11.14	1.7	0.5	1.1	0.1
Copper (Cu)	3	1.71**	20.1**	12.5**	0.32^{ns}	1.6^{ns}	1.5^{ns}	0.69^{ns}	11.1**
Manganese (Mn)	3	1.65**	16.4**	13.1**	25.1**	1.8**	24.8**	1.1^{ns}	15.8**
Iron (Fe)	3	125**	21.4**	14.8**	26.4**	22.9**	22.9**	31.4**	14.5**
Zinc (Zn)	3	1.36**	18.1**	10.6**	20.9**	17.5**	26.9**	22.9**	15.3**
Cu × Mn	9	0.99**	11.4**	11.9**	16.8**	14.1**	14.8**	23.8**	10.9**
Cu × Fe	9	0.89**	10.9**	12.8**	13.4**	10.4**	15.9**	22.4**	9.8**
Cu × Zn	9	1.1**	12.7**	9.1**	14.1**	11.3**	12.8**	14.1**	11.1**
Mn × Fe	9	1.08**	8.1**	8.9**	12.1**	13.9**	17.7**	15.1**	10.7**
Mn × Zn	9	1.07**	7.7**	9.8**	10.9**	12.9**	13.7**	18.9**	8.8**
Fe × Zn	9	0.88**	6.8**	9.5**	9.2**	10.9**	10.5**	12.2**	9.1**
Cu × Mn × Fe	27	0.59**	5.9**	4.4**	8.8**	9.4**	0.61^{ns}	11.8**	4.5**
Cu × Mn × Zn	27	0.66**	6.6**	5.8**	7.9**	8.1**	0.58^{ns}	10.9**	8.1**
Cu × Fe × Zn	27	0.61**	0.2^{ns}	6.4**	0.48^{ns}	0.22^{ns}	0.52^{ns}	0.99^{ns}	7.4**
Fe × Zn × Mn	27	0.45^{ns}	0.41^{ns}	5.6**	0.35^{ns}	0.28^{ns}	0.41^{ns}	0.67^{ns}	6.4**
Cu × Zn × Mn × Fe	81	0.012^{ns}	0.32^{ns}	0.38^{ns}	0.24^{ns}	0.19^{ns}	0.48^{ns}	0.59^{ns}	0.68^{ns}
T(Cu,Zn,Mn,Fe)×Y	255	0.061^{ns}	0.11^{ns}	0.46^{ns}	0.41^{ns}	0.22^{ns}	0.44^{ns}	0.66^{ns}	0.42^{ns}
E	1020	0.11	0.55	1.1	0.91	1.02	1.44	1.53	0.72
	CV^{x}	8.8	9.9	5.8	3.9	5.15	6.12	6.18	5.5

^{z} SOV: source of variation, ^{y}df: degree of freedom, ^{x}CV: coefficient of variation, *, ** significant at P=0.05 and P=0.01 probability levels, respectively.

Table 4. Means of EOs content and main constituents (%) in *F. angulate* plants affected by micronutrients (20 mgL^{-1}) concentration (1st year).

Compound	RI [z]	Fe	Zn	Cu	Mn	Fe×Zn	Cu×Fe	Mn×Fe	Cu×Zn
alpha-Thujene	926	12.61±0.5[y]	12.43±0.4	12.22±0.3	12.55±0.2	12.81±0.2	13.01±0.3	13.09±0.2	13.02±0.2
alpha-pinene	937	22.21±0.5	21.42±0.4	21.55±0.4	21.66±0.5	22.33±0.6	22.41±0.7	23.66±0.5	23.55±0.6
beta-myrcene	986	1.81±0.01	1.75±0.1	1.66±0.01	1.63±0.02	1.93±0.08	1.91±0.01	1.88±0.01	1.91±0.01
alpha-phellandrene	1003	1.99±0.01	1.26±0.01	1.24±0.03	1.21±0.02	2.01±0.01	2.11±0.01	2.08±0.02	2.11±0.02
beta -phellandrene	1027	3.31±0.01	3.26±0.01	3.24±0.03	3.21±0.02	3.34±0.01	3.37±0.01	4.35±0.02	3.29±0.02
Cis-ocimene	1031	13.18±0.8	12.8±0.8	12.2±0.7	12.1±0.6	13.3±0.8	13.5±0.6	13.83±0.8	12.9±0.9
4-thujanol	1041	7.88±0.01	7.77±0.01	7.66±0.03	7.7±0.02	8.67±0.01	8.12±0.01	8.19±0.02	8.11±0.01
beta -ocimene	1053	3.88±0.01	3.77±0.01	3.65±0.02	3.58±0.02	4.01±0.01	4.11±0.01	4.14±0.02	4.19±0.3
Sabinene	1192	1.65±0.01	1.63±0.01	1.61±0.03	1.59±0.02	1.67±0.01	1.69±0.01	1.71±0.02	1.63±0.02
Verbenone	1204	6.69±0.01	6.65±0.03	6.61±0.02	6.51±0.1	6.71±0.06	6.73±0.03	6.74±0.02	6.69±0.02
Bornyl acetate	1219	3.29±0.01	3.26±0.01	3.25±0.02	3.21±0.02	3.36±0.01	3.39±0.01	3.45±0.02	3.41±0.03
Naphthalenemethanol	1468	1.11±0.01	1.09±0.01	1.07±0.02	1.02±0.01	1.13±0.02	1.14±0.01	1.16±0.02	1.15±0.03
Bicyclo-germacrene	1491	0.89±0.01	0.92±0.02	0.91±0.01	0.88±0.01	0.95±0.01	0.99±0.01	1.01±0.01	1.03±0.1
Nonadecane	1900	1.88±0.01	1.78±0.01	1.61±0.02	1.63±0.01	1.91±0.02	1.95±0.01	1.99±0.01	1.92±0.1
Henicosane	2109	0.88±0.02	0.78±0.01	0.61±0.02	0.63±0.01	0.91±0.02	0.95±0.01	1.02±0.01	1.14±0.1
Monoterpene hydrocarbons		60.64±1.8	58.32±1.9	57.37±1.7	57.53±1.5	61.4±1.6	62.11±1.4	64.74±1.8	62.6±1.4
Oxygenated monoterpenes		14.57±0.2	14.42±0.3	14.27±0.2	14.21±0.3	15.38±0.2	14.85±0.3	14.93±0.2	14.8±0.2
Sesquiterpenes		0.89±0.01	0.92±0.02	0.91±0.01	0.88±0.01	0.95±0.01	0.99±0.01	1.01±0.01	1.03±0.1
Acetate esters terpenoids		3.29±0.01	3.26±0.01	3.25±0.02	3.21±0.02	3.36±0.01	3.39±0.01	3.45±0.02	3.41±0.03
Polycyclic Aromatic Hydrocarbons		1.11±0.01	1.09±0.01	1.07±0.02	1.02±0.01	1.13±0.02	1.14±0.01	1.16±0.02	1.15±0.03
Hydrocarbons Alkanes		2.76±0.03	2.56±0.02	2.22±0.01	2.26±0.02	2.82±0.01	2.9±0.02	3.01±0.03	3.06±0.02
EOs content (w/w%, g/100g fresh weight basis)		0.41±0.01	0.40±0.01	0.40±0.02	0.39±0.01	0.42±0.02	0.42±0.01	0.41±0.01	0.43±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation ($P<0.05$)

Table 4. Continued means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (20 mgL^{-1}) concentration (1st year).

Compound	RI [z]	Mn×Zn	Cu×Mn	Cu×Fe×Zn	Fe×Zn×Mn	Cu×Mn×Fe	Cu×Mn×Zn	Cu×Zn×Mn ×Fe	Control
alpha-Thujene	926	13.61±0.5[y]	13.43±0.4	14.12±0.3	14.23±0.2	14.81±0.2	14.55±0.3	13.32±0.2	13.12±0.2
alpha-pinene	937	24.11±0.5	24.42±0.4	25.55±0.4	25.66±0.5	27.02±0.6	26.66±0.7	28.43±0.5	20.41±0.6
beta -myrcene	986	1.88±0.01	1.93±0.01	1.98±0.03	1.99±0.02	2.01±0.01	2.03±0.01	2.05±0.02	1.43±0.01
alpha-phellandrene	1003	1.88±0.01	1.94±0.01	2.11±0.01	2.14±0.02	2.22±0.01	2.15±0.01	2.31±0.02	1.51±0.01
beta -phellandrene	1027	3.12±0.01	3.26±0.01	3.33±0.04	3.32±0.02	3.36±0.01	3.29±0.01	4.01±0.01	3.12±0.02
Cis-ocimene	1031	13.18±0.6	13.21±0.5	14.22±0.5	13.25±0.6	14.12±0.6	13.89±0.5	15.22±0.6	11.41±0.6
4-thujanol	1041	8.21±0.01	8.12±0.01	8.66±0.03	8.73±0.02	8.92±0.01	8.45±0.01	9.11±0.02	8.03±0.01
beta -ocimene	1053	4.22±0.01	4.26±0.01	4.45±0.02	4.33±0.02	4.39±0.01	4.42±0.01	4.55±0.02	3.14±0.03
Sabinene	1192	1.66±0.01	1.69±0.01	1.71±0.03	1.73±0.02	1.81±0.01	1.84±0.01	1.99±0.02	1.58±0.01
Verbenone	1204	6.71±0.01	6.73±0.01	6.76±0.02	6.81±0.02	6.85±0.01	6.77±0.01	7.11±0.02	6.47±0.01
Bornyl acetate	1219	3.45±0.01	3.48±0.01	3.49±0.02	3.37±0.02	3.55±0.01	3.99±0.01	4.99±0.02	3.12±0.01
Naphthalenemethanol	1468	1.21±0.01	1.22±0.01	1.24±0.02	1.27±0.01	1.66±0.02	1.55±0.01	1.99±0.02	0.99±0.01
Bicyclo-germacrene	1491	1.07±0.01	1.09±0.02	1.11±0.01	1.15±0.01	1.21±0.04	1.23±0.03	2.11±0.01	0.79±0.01
Nonadecane	1900	1.98±0.01	2.01±0.01	2.05±0.02	2.08±0.01	2.39±0.02	2.15±0.01	0.71±0.01	5.45±0.01
Henicosane	2109	1.15±0.01	1.17±0.01	1.22±0.02	1.27±0.01	1.23±0.02	1.29±0.01	0.51±0.01	2.56±0.01
Monoterpene hydrocarbons		63.66±1.5	64.14±1.3	67.47±1.5	66.65±1.4	69.74±1.5	68.83±1.3	71.88±1.5	55.72±1.1
Oxygenated monoterpenes		14.9±0.04	14.85±0.0	15.42±0.03	15.54±0.04	15.77±0.05	15.22±0.05	16.22±0.04	14.5±0.02
Sesquiterpenes		1.07±0.01	1.09±0.02	1.11±0.01	1.15±0.01	1.21±0.04	1.23±0.03	2.11±0.01	0.79±0.01
Acetate esters terpenoids		3.45±0.01	3.48±0.01	3.49±0.02	3.37±0.02	3.55±0.01	3.99±0.01	4.99±0.02	3.12±0.01
Polycyclic Aromatic Hydrocarbons		1.21±0.01	1.22±0.01	1.24±0.02	1.27±0.01	1.66±0.02	1.55±0.01	1.99±0.02	0.99±0.01
Hydrocarbons Alkanes		3.13±0.02	3.18±0.03	3.27±0.02	3.35±0.03	3.62±0.02	3.44±0.04	1.22±0.02	8.01±0.01
EOs content (w/w%, g/100g fresh weight basis)		0.44±0.01	0.44±0.01	0.45±0.02	0.46±0.01	0.47±0.02	0.46±0.01	0.49±0.01	0.37±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation ($P<0.05$).

Table 5. Means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (20 mgL^{-1}) concentration (2^{nd} year)

Compound	RI [z]	Fe	Zn	Cu	Mn	Fe×Zn	Cu×Fe	Mn×Fe	Cu×Zn
alpha-Thujene	926	13.91±0.7[y]	13.84±0.5	13.82±0.6	13.77±0.6	15.99±0.5	14.85±0.5	14.12±0.6	14.14±0.5
alpha-pinene	937	22.11±0.9	21.9±0.8	21.5±0.7	21.53±0.9	23.44±0.6	24.6±0.5	24.43±0.5	23.8±0.6
beta -myrcene	986	1.65±0.01	1.63±0.01	1.62±0.03	1.58±0.02	1.75±0.01	1.82±0.01	1.86±0.02	1.89±0.02
alpha-phellandrene	1003	2.22±0.01	2.17±0.02	2.14±0.04	2.12±0.04	2.29±0.08	2.27±0.03	2.31±0.03	2.25±0.02
beta -phellandrene	1027	3.66±0.02	3.63±0.01	3.61±0.04	3.58±0.02	3.74±0.01	3.77±0.01	3.81±0.01	3.72±0.02
Cis-ocimene	1031	14.18±0.8	13.21±0.9	13.22±0.6	13.25±0.6	14.77±0.9	14.9±0.8	14.22±0.6	14.7±0.5
4-thujanol	1041	8.77±0.01	8.72±0.01	8.66±0.03	7.91±0.02	9.99±0.01	9.88±0.01	9.66±0.02	9.89±0.01
beta -ocimene	1053	4.66±0.01	4.71±0.01	4.12±0.02	4.33±0.02	4.44±0.01	4.52±0.01	4.99±0.02	4.71±0.03
Sabinene	1192	1.89±0.01	1.85±0.01	1.81±0.03	1.83±0.02	1.91±0.01	1.93±0.01	1.94±0.02	1.89±0.02
Verbenone	1204	6.94±0.01	6.92±0.01	7.89±0.02	6.81±0.02	7.96±0.01	6.98±0.01	7.99±0.02	6.94±0.01
Bornyl acetate	1219	3.61±0.01	3.58±0.01	3.55±0.02	3.49±0.02	3.65±0.01	3.68±0.01	3.71±0.02	3.61±0.03
Naphthalenemethanol	1468	1.19±0.01	1.17±0.01	1.14±0.02	1.15±0.01	1.21±0.02	1.23±0.01	1.26±0.02	1.31±0.03
Bicyclo-germacrene	1491	1.26±0.01	1.24±0.02	1.21±0.01	1.15±0.01	1.28±0.04	1.31±0.03	1.33±0.01	1.32±0.02
Nonadecane	1900	1.39±0.01	1.32±0.01	1.28±0.02	1.22±0.01	1.44±0.02	1.48±0.01	1.53±0.01	1.51±0.02
Henicosane	2109	0.88±0.01	0.78±0.01	0.66±0.02	0.81±0.01	0.91±0.02	0.97±0.01	0.91±0.01	0.92±0.02
Monoterpene hydrocarbons		64.28±1.6	62.94±1.4	61.84±1.5	61.99±1.3	68.33±1.5	68.66±1.6	67.68±1.4	67.11±1.3
Oxygenated monoterpenes		15.71±0.6	15.64±0.4	16.55±0.5	14.72±0.6	17.95±0.8	16.86±0.6	17.65±0.8	16.83±0.6
Sesquiterpenes		1.26±0.01	1.24±0.02	1.21±0.01	1.15±0.01	1.28±0.04	1.31±0.03	1.33±0.01	1.32±0.02
Acetate esters terpenoids		3.61±0.01	3.58±0.01	3.55±0.02	3.49±0.02	3.65±0.01	3.68±0.01	3.71±0.02	3.61±0.03
Polycyclic Aromatic Hydrocarbons		1.19±0.01	1.17±0.01	1.14±0.02	1.15±0.01	1.21±0.02	1.23±0.01	1.26±0.02	1.31±0.03
Hydrocarbons Alkanes		2.27±0.01	2.1±0.02	1.94±0.03	2.03±0.01	2.35±0.02	2.45±0.02	2.44±0.01	2.43±0.02
EOs content (w/w%, g/100g fresh weight basis)		0.44±0.02	0.43±0.01	0.42±0.02	0.42±0.01	0.45±0.02	0.45±0.01	0.45±0.01	0.46±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation (P<0.05).

Table 5. Continued means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (20 mgL^{-1}) concentration (2^{nd} year).

Compound	RI [z]	Mn×Zn	Cu×Mn	Cu×Fe×Zn	Fe×Zn×Mn	Cu×Mn×Fe	Cu×Mn×Zn	Cu×Zn×Mn×Fe	Control
alpha-Thujene	926	14.17±0.6[y]	14.15±0.5	14.23±0.6	13.33±0.5	13.44±0.6	14.47±0.5	14.51±0.5	13.67±0.7
alpha-pinene	937	24.11±0.9	24.42±0.8	26.55±0.9	27.53±0.7	28.44±0.9	28.6±0.8	29.43±0.8	21.12±0.7
beta -myrcene	986	1.83±0.01	1.81±0.01	1.93±0.03	1.94±0.02	1.98±0.01	2.03±0.01	2.09±0.02	1.54±0.02
alpha-phellandrene	1003	2.23±0.01	2.21±0.01	2.25±0.03	2.27±0.02	2.29±0.01	2.31±0.01	2.33±0.02	1.66±0.02
beta -phellandrene	1027	3.57±0.04	3.59±0.01	3.81±0.04	3.83±0.02	3.79±0.01	3.89±0.01	4.15±0.01	3.55±0.02
Cis-ocimene	1031	14.18±0.9	13.21±0.7	13.22±0.5	14.25±0.5	14.77±0.6	15.11±0.7	15.32±0.8	11.71±0.8
4-thujanol	1041	8.89±0.01	8.91±0.01	8.66±0.03	9.01±0.02	9.03±0.01	8.99±0.01	9.22±0.02	8.29±0.01
beta -ocimene	1053	4.88±0.01	4.93±0.01	4.77±0.02	4.66±0.02	4.82±0.01	4.88±0.01	5.01±0.02	3.57±0.03
Sabinene	1192	1.91±0.01	1.93±0.01	1.95±0.03	1.99±0.02	2.01±0.01	2.06±0.01	2.14±0.02	1.71±0.02
Verbenone	1204	6.98±0.01	6.99±0.01	7.01±0.02	7.03±0.02	7.07±0.01	7.18±0.01	7.23±0.02	6.79±0.01
Bornyl acetate	1219	3.77±0.01	3.81±0.01	4.01±0.02	4.22±0.02	3.99±0.01	4.56±0.01	2.11±0.02	3.46±0.02
Naphthalenemethanol	1468	1.33±0.05	1.35±0.01	1.36±0.02	1.82±0.01	1.44±0.02	1.79±0.01	2.11±0.02	1.12±0.01
Bicyclo-germacrene	1491	1.44±0.04	1.55±0.02	1.69±0.01	1.78±0.01	1.71±0.04	0.31±0.03	0.21±0.01	1.12±0.01
Nonadecane	1900	1.56±0.06	1.62±0.01	1.61±0.02	2.17±0.01	2.01±0.02	0.81±0.01	0.98±0.01	5.99±0.02
Henicosane	2109	1.11±0.02	1.23±0.01	1.34±0.02	1.45±0.01	1.65±0.02	0.54±0.01	0.93±0.01	5.01±0.02
Monoterpene hydrocarbons		66.88±1.6	66.25±1.2	68.71±1.5	69.8±1.2	71.54±1.7	73.35±1.6	74.98±1.6	58.53±1.2
Oxygenated monoterpenes		15.87±0.2	15.9±0.1	15.67±0.3	16.04±0.2	17.01±0.1	16.17±0.3	16.45±0.2	15.08±0.1
Sesquiterpenes		1.44±0.04	1.55±0.02	1.69±0.01	1.78±0.01	1.71±0.04	0.31±0.03	0.21±0.01	1.12±0.01
Acetate esters terpenoids		3.77±0.01	3.81±0.01	4.01±0.02	4.22±0.02	3.99±0.01	4.56±0.01	2.11±0.02	3.46±0.02
Polycyclic Aromatic Hydrocarbons		1.33±0.05	1.35±0.01	1.36±0.02	1.82±0.01	1.44±0.02	1.79±0.01	2.11±0.02	1.12±0.01
Hydrocarbons Alkanes		3.01±0.01	2.85±0.02	2.95±0.01	3.62±0.03	3.66±0.04	1.35±0.03	1.91±0.04	11.01±0.06
EOs content (w/w%, g/100g fresh weight basis)		0.46±0.02	0.47±0.01	0.47±0.02	0.47±0.01	0.48±0.02	0.48±0.01	0.48±0.01	0.41±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation (P<0.05).

Table 6. Means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (40 mgL^{-1}) concentration and control plants (1st year).

Compound	RI ^{z}	Fe	Zn	Cu	Mn	Fe×Zn	Cu×Fe	Mn×Fe	Cu×Zn
alpha-Thujene	926	14.33±0.5^{y}	14.09±0.8	14.08±0.5	14.12±0.6	13.33±0.7	13.39±0.5	13.44±0.6	14.22±0.5
alpha-pinene	937	29.11±0.9	27.9±0.9	27.6±0.8	27.1±0.9	30.1±0.8	30.3±0.9	30.4±0.7	29.8±0.8
beta -myrcene	986	2.23±0.01	2.19±0.01	2.18±0.03	2.15±0.02	2.26±0.01	2.29±0.01	2.33±0.02	2.25±0.02
alpha-phellandrene	1003	2.77±0.1	2.64±0.1	2.59±0.1	2.61±0.1	2.65±0.08	2.77±0.1	2.82±0.1	2.55±0.1
beta -phellandrene	1027	4.35±0.1	4.12±0.1	3.99±0.1	4.02±0.1	4.38±0.1	4.44±0.05	4.55±0.05	4.66±0.1
Cis-ocimene	1031	16.18±0.9	15.1±0.8	15.2±0.9	14.9±0.6	16.4±0.8	16.9±0.9	16.22±0.8	16.7±0.7
4-thujanol	1041	6.88±0.01	6.92±0.01	6.66±0.03	6.74±0.02	6.92±0.01	6.84±0.01	6.99±0.02	6.81±0.01
beta -ocimene	1053	4.98±0.01	4.77±0.01	4.65±0.02	4.51±0.02	4.99±0.01	5.05±0.01	5.09±0.02	5.01±0.03
Sabinene	1192	2.59±0.01	2.51±0.01	2.48±0.03	2.44±0.02	2.61±0.01	2.63±0.01	2.65±0.02	2.71±0.02
Verbenone	1204	5.45±0.01	5.41±0.01	5.33±0.02	5.22±0.02	4.55±0.01	4.61±0.01	4.65±0.02	4.71±0.01
Bornyl acetate	1219	3.99±0.01	3.88±0.01	3.74±0.02	3.58±0.02	4.01±0.01	4.04±0.01	4.19±0.02	4.05±0.03
Naphthalenemethanol	1468	1.19±0.1	1.14±0.05	1.12±0.05	1.09±0.07	1.32±0.07	1.38±0.08	1.42±0.07	1.39±0.07
Bicyclo-germacrene	1491	2.88±0.1	2.55±0.05	2.44±0.07	2.39±0.08	2.89±0.09	2.94±0.06	2.97±0.08	2.01±0.1
Nonadecane	1900	0.56±0.1	0.33±0.06	0.21±0.07	0.59±0.06	0.61±0.03	0.81±0.08	0.71±0.07	0.82±0.1
Henicosane	2109	0.91±0.1	0.78±0.06	0.61±0.06	0.63±0.06	0.92±0.07	0.95±0.06	0.94±0.05	0.92±0.1
Monoterpene hydrocarbons		76.54±1.6	73.32±1.3	72.77±1.2	71.4±1.5	76.72±1.6	77.71±1.7	77.5±1.6	77.9±1.9
Oxygenated monoterpenes		12.33±0.5	12.33±0.4	13.99±0.7	12.96±0.6	13.47±0.8	13.45±0.5	11.64±0.4	11.52±0.5
Sesquiterpenes		2.88±0.1	2.55±0.05	2.44±0.07	2.39±0.08	2.89±0.09	2.94±0.06	2.97±0.08	2.01±0.1
Acetate esters terpenoids		3.99±0.01	3.88±0.01	3.74±0.02	3.58±0.02	4.01±0.01	4.04±0.01	4.19±0.02	4.05±0.03
Polycyclic Aromatic Hydrocarbons		1.19±0.1	1.14±0.05	1.12±0.05	1.09±0.07	1.32±0.07	1.38±0.08	1.42±0.07	1.39±0.07
Hydrocarbons Alkanes		1.47±0.1	1.11±0.2	0.82±0.1	1.22±0.2	1.53±0.3	1.76±0.2	1.65±0.1	1.74±0.5
EOs content (w/w%, g/100g fresh weight basis)		0.46±0.01	0.45±0.01	0.45±0.02	0.45±0.01	0.47±0.02	0.47±0.01	0.48±0.01	0.48±0.02

^{z} RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. ^{y} Values are means of triplicates ± standard deviation (P<0.05).

Table 6. Continued means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (40 mgL^{-1}) concentration and control plants (1st year).

Compound	RI ^{z}	Mn×Zn	Cu×Mn	Cu×Fe×Zn	Fe×Zn×Mn	Cu×Mn×Fe	Cu×Mn×Zn	Cu×Zn×Mn×Fe
alpha-Thujene	926	14.44±0.8^{y}	14.31±0.9	14.29±0.8	14.33±0.9	14.54±0.7	14.31±0.6	16.12±0.6
alpha-pinene	937	31.11±0.9	30.42±0.9	32.55±0.8	33.53±0.8	33.44±0.9	33.11±0.9	34.51±0.8
beta -myrcene	986	0.56±0.01	0.63±0.01	0.78±0.03	0.73±0.02	0.69±0.01	0.65±0.01	0.81±0.02
alpha-phellandrene	1003	0.28±0.01	0.23±0.01	0.69±0.03	0.75±0.02	0.77±0.01	0.33±0.01	0.12±0.02
beta -phellandrene	1027	1.55±0.1	1.64±0.06	1.11±0.06	1.23±0.06	1.44±0.05	1.24±0.05	1.55±0.08
Cis-ocimene	1031	16.18±0.7	17.21±0.9	17.22±0.9	18.25±0.8	18.77±0.9	17.9±0.8	19.11±0.9
4-thujanol	1041	3.98±0.01	3.88±0.01	3.66±0.03	5.01±0.02	4.12±0.01	4.03±0.01	5.12±0.02
beta -ocimene	1053	5.07±0.01	5.11±0.01	5.15±0.02	5.99±0.02	5.31±0.01	5.77±0.01	5.41±0.02
Sabinene	1192	2.88±0.01	2.91±0.01	2.94±0.03	3.15±0.02	3.12±0.01	3.18±0.01	3.55±0.02
Verbenone	1204	4.78±0.01	4.81±0.01	4.85±0.02	5.11±0.02	5.11±0.01	5.01±0.01	5.44±0.02
Bornyl acetate	1219	5.11±0.01	5.16±0.01	5.19±0.02	5.99±0.02	5.11±0.01	5.78±0.01	6.51±0.02
Naphthalenemethanol	1468	2.42±0.1	2.45±0.05	2.49±0.05	2.72±0.06	2.73±0.06	2.66±0.05	3.21±0.05
Bicyclo-germacrene	1491	1.02±0.1	1.04±0.05	1.08±0.05	1.11±0.06	1.14±0.07	1.09±0.08	1.21±0.05
Nonadecane	1900	0.89±0.1	0.01±0.05	0.03±0.05	0.08±0.06	0.44±0.06	0.22±0.05	0.45±0.06
Henicosane	2109	0.01±0.1	0.07±0.06	0.09±0.08	0.11±0.08	0.22±0.05	0.09±0.06	0.56±0.05
Monoterpene hydrocarbons		72.07±1.5	72.46±1.2	74.73±1.5	77.96±1.1	78.08±1.2	76.49±1.5	81.18±1.8
Oxygenated monoterpenes		4.78±0.01	4.81±0.01	4.85±0.02	5.11±0.02	5.11±0.01	5.01±0.01	5.44±0.02
Sesquiterpenes		1.02±0.1	1.04±0.05	1.08±0.05	1.11±0.06	1.14±0.07	1.09±0.08	1.21±0.05
Acetate esters terpenoids		5.11±0.01	5.16±0.01	5.19±0.02	5.99±0.02	5.11±0.01	5.78±0.01	6.51±0.02
Polycyclic Aromatic Hydrocarbons		2.42±0.1	2.45±0.05	2.49±0.05	2.72±0.06	2.73±0.06	2.66±0.05	3.21±0.05
Hydrocarbons Alkanes		0.90±0.01	0.08±0.01	0.12±0.01	0.19±0.01	0.66±0.02	0.31±0.03	1.01±0.01
EOs content (w/w%, g/100g fresh weight basis)		0.49±0.02	0.48±0.01	0.50±0.02	0.51±0.01	0.57±0.02	0.55±0.01	0.59±0.01

^{z} RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. ^{y} Values are means of triplicates ± standard deviation (P<0.05).

Table 7. Means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (40 mgL^{-1}) concentration and control plants (2^{nd} year).

Compound	RI [z]	Fe	Zn	Cu	Mn	Fe×Zn	Cu×Fe	Mn×Fe	Cu×Zn
alpha-Thujene	926	15.77±0.9[y]	15.12±0.8	14.22±0.8	14.31±0.8	14.26±0.9	15.39±0.7	15.51±0.6	16.22±0.9
alpha-pinene	937	32.11±0.9	31.9±0.9	31.5±0.8	31.3±0.8	33.4±0.9	33.6±0.9	32.43±1.1	31.8±0.9
beta -myrcene	986	2.77±0.01	2.65±0.01	2.61±0.03	2.55±0.02	2.81±0.01	2.83±0.01	2.84±0.02	2.73±0.02
alpha-phellandrene	1003	2.99±0.1	2.87±0.1	2.82±0.1	2.79±0.2	3.11±0.08	3.15±0.1	3.16±0.1	3.01±0.1
beta -phellandrene	1027	5.57±0.1	5.59±0.08	4.61±0.08	4.63±0.06	6.64±0.08	6.59±0.09	6.93±0.07	6.59±0.1
Cis-ocimene	1031	17.18±0.9	16.1±0.9	15.8±0.8	15.5±0.8	17.7±0.9	17.9±0.6	17.22±0.8	17.4±0.9
4-thujanol	1041	3.89±0.01	3.77±0.01	2.66±0.03	3.67±0.02	3.9±0.01	2.11±0.01	2.15±0.02	2.19±0.01
beta -ocimene	1053	5.01±0.01	4.88±0.01	4.66±0.02	4.71±0.02	5.11±0.01	5.52±0.01	5.33±0.02	5.21±0.03
Sabinene	1192	2.88±0.01	2.77±0.01	2.66±0.03	2.55±0.02	2.91±0.01	2.94±0.01	2.95±0.02	2.88±0.02
Verbenone	1204	2.66±0.01	3.55±0.01	2.34±0.02	2.31±0.02	3.69±0.01	2.73±0.01	2.75±0.02	2.71±0.01
Bornyl acetate	1219	2.99±0.01	2.81±0.01	2.73±0.02	2.55±0.02	1.01±0.01	1.09±0.01	1.19±0.02	1.02±0.03
Naphthalenemethanol	1468	0.14±0.02	0.18±0.01	0.15±0.02	0.14±0.01	0.15±0.02	0.19±0.01	0.22±0.02	0.18±0.03
Bicyclo-germacrene	1491	2.45±0.01	2.33±0.02	2.28±0.01	2.17±0.01	2.48±0.04	2.49±0.03	2.55±0.01	2.51±0.1
Nonadecane	1900	0.33±0.03	0.21±0.01	0.11±0.02	0.07±0.01	0.45±0.02	0.81±0.01	0.71±0.01	0.82±0.03
Henicosane	2109	0.34±0.01	0.25±0.01	0.12±0.02	0.09±0.01	0.44±0.02	0.47±0.01	0.55±0.01	0.59±0.01
Monoterpene hydrocarbons		84.28±1.6	81.88±1.2	78.88±1.4	78.34±1.2	85.94±1.6	87.92±1.2	86.37±1.6	85.84±1.2
Oxygenated monoterpenes		6.55±0.3	7.32±0.4	5.01±0.01	5.98±0.2	7.59±0.3	4.84±0.2	4.90±0.3	4.90±0.2
Sesquiterpenes		2.45±0.01	2.33±0.02	2.28±0.01	2.17±0.01	2.48±0.04	2.49±0.03	2.55±0.01	2.51±0.1
Acetate esters terpenoids		2.99±0.01	2.81±0.01	2.73±0.02	2.55±0.02	1.01±0.01	1.09±0.01	1.19±0.02	1.02±0.03
Polycyclic Aromatic Hydrocarbons		0.14±0.02	0.18±0.01	0.15±0.02	0.14±0.01	0.15±0.02	0.19±0.01	0.22±0.02	0.18±0.03
Hydrocarbons Alkanes		0.67±0.01	0.46±0.01	0.23±0.01	0.16±0.01	0.89±0.01	1.28±0.04	1.26±0.03	1.41±0.05
EOs content (w/w%, g/100g fresh weight basis)		0.59±0.02	0.58±0.01	0.58±0.02	0.58±0.01	0.60±0.02	0.61±0.01	0.61±0.01	0.61±0.02

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation (P<0.05).

Table 7. Continued means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (40 mgL^{-1}) concentration and control plants (2^{nd} year).

Compound	RI [z]	Mn×Zn	Cu×Mn	Cu×Fe×Zn	Fe×Zn×Mn	Cu×Mn×Fe	Cu×Mn×Zn	Cu×Zn×Mn ×Fe
alpha-Thujene	926	16.11±0.6[y]	16.18±0.7	17.12±0.5	17.34±0.9	17.55±0.8	17.21±0.9	18.14±0.8
alpha-pinene	937	31.11±1.1	31.42±1.1	33.55±1.2	34.53±1.4	34.44±1.2	33.6±1.5	34.88±1.5
beta -myrcene	986	2.66±0.01	2.71±0.01	2.89±0.03	2.81±0.02	2.85±0.01	2.82±0.01	2.12±0.02
alpha-phellandrene	1003	3.03±0.01	3.06±0.01	3.19±0.03	3.22±0.02	3.31±0.01	3.25±0.01	3.44±0.02
beta -phellandrene	1027	5.57±0.1	6.11±0.1	6.61±0.08	6.63±0.09	6.64±0.08	6.23±0.08	6.12±0.07
Cis-ocimene	1031	17.18±1.1	18.21±1.2	18.22±0.9	18.25±0.9	19.77±1.1	18.9±0.9	19.01±1.1
4-thujanol	1041	3.15±0.01	3.22±0.01	3.36±0.03	2.66±0.02	1.91±0.01	1.11±0.01	1.54±0.02
beta -ocimene	1053	5.41±0.01	5.43±0.01	5.55±0.02	5.57±0.02	5.63±0.01	5.33±0.01	5.89±0.02
Sabinene	1192	2.93±0.01	2.91±0.01	3.01±0.03	3.09±0.02	3.18±0.01	3.29±0.01	3.69±0.02
Verbenone	1204	2.75±0.01	2.77±0.01	2.81±0.02	3.83±0.02	2.11±0.01	2.99±0.01	1.59±0.02
Bornyl acetate	1219	1.18±0.01	1.22±0.01	1.31±0.02	1.33±0.02	1.39±0.01	1.55±0.01	0.87±0.02
Naphthalenemethanol	1468	0.26±0.05	0.36±0.01	0.44±0.02	0.51±0.01	0.99±0.02	0.88±0.01	0.51±0.02
Bicyclo-germacrene	1491	0.53±0.05	0.56±0.02	0.62±0.01	0.72±0.01	0.81±0.04	0.76±0.03	0.01±0.01
Nonadecane	1900	0.56±0.07	0.61±0.01	0.64±0.02	0.99±0.01	0.88±0.02	0.31±0.01	0.99±0.01
Henicosane	2109	0.88±0.04	0.11±0.01	0.61±0.02	0.63±0.01	0.91±0.02	0.82±0.01	0.01±0.01
Monoterpene hydrocarbons		84.01±1.8	86.03±1.4	90.13±1.6	91.44±1.1	93.37±1.4	90.63±1.2	93.29±1.1
Oxygenated monoterpenes		5.9±0.2	5.99±0.3	6.17±0.2	5.49±0.4	5.02±0.3	5.1±0.4	3.13±0.2
Sesquiterpenes		0.53±0.05	0.56±0.02	0.62±0.01	0.72±0.01	0.81±0.04	0.76±0.03	0.11±0.01
Acetate esters terpenoids		1.18±0.01	1.22±0.01	1.31±0.02	1.33±0.02	1.39±0.01	1.55±0.01	0.87±0.02
Polycyclic Aromatic Hydrocarbons		0.26±0.05	0.36±0.01	0.44±0.02	0.51±0.01	0.99±0.02	0.88±0.01	0.51±0.02
Hydrocarbons Alkanes		1.44±0.05	0.77±0.02	1.25±0.06	1.62±0.05	1.79±0.06	1.13±0.04	1.01±0.01
EOs content (w/w%, g/100g fresh weight basis)		0.62±0.01	0.62±0.01	0.63±0.02	0.63±0.01	0.64±0.02	0.65±0.01	0.68±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation (P<0.05).

Table 8. Means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (60 mgL^{-1}) concentration (1st year).

Compound	RI [z]	Fe	Zn	Cu	Mn	Fe×Zn	Cu×Fe	Mn×Fe	Cu×Zn
alpha-Thujene	926	18.9±1.2[y]	18.7±1.4	17.5±1.1	18.2±0.9	18.1±0.8	17.6±0.9	18.39±0.8	19.4±0.9
alpha-pinene	937	23.4±1.1	23.2±1.2	22.5±1.1	22.3±1.2	21.9±0.9	21.6±0.8	20.43±0.9	21.8±1.1
beta -myrcene	986	1.88±0.01	1.77±0.01	1.66±0.03	1.58±0.02	1.82±0.01	1.81±0.01	1.79±0.02	1.78±0.02
alpha-phellandrene	1003	1.71±0.01	1.69±0.01	1.67±0.03	1.59±0.02	1.67±0.01	1.62±0.01	1.59±0.02	1.58±0.02
beta -phellandrene	1027	5.35±0.06	5.12±0.01	5.99±0.04	5.02±0.02	4.89±0.01	4.78±0.01	4.31±0.01	4.66±0.07
Cis-ocimene	1031	15.77±0.9	14.66±0.7	14.81±0.8	14.5±0.9	13.7±0.8	13.9±0.7	12.22±0.7	13.7±0.8
4-thujanol	1041	3.88±0.01	3.77±0.1	4.66±0.0	3.17±0.2	4.99±0.08	3.88±0.09	2.66±0.08	3.65±0.08
beta -ocimene	1053	5.03±0.01	4.88±0.01	4.73±0.02	4.61±0.02	4.55±0.01	4.66±0.01	3.15±0.02	3.55±0.03
Sabinene	1192	2.01±0.01	1.99±0.01	1.89±0.03	1.79±0.02	1.69±0.01	1.73±0.01	1.71±0.02	1.75±0.02
Verbenone	1204	3.15±0.01	3.14±0.01	3.11±0.02	2.08±0.02	3.17±0.01	2.13±0.01	3.12±0.02	3.17±0.01
Bornyl acetate	1219	4.43±0.01	4.35±0.01	4.22±0.02	4.15±0.02	4.11±0.01	4.05±0.01	4.01±0.02	4.08±0.03
Naphthalenemethanol	1468	1.67±0.05	1.69±0.01	1.71±0.02	1.72±0.01	1.55±0.02	1.51±0.01	1.49±0.02	1.52±0.03
Bicyclo-germacrene	1491	1.88±0.04	1.76±0.02	1.66±0.01	1.58±0.01	1.91±0.04	1.94±0.03	1.88±0.01	1.82±0.03
Nonadecane	1900	1.56±0.03	1.63±0.01	1.61±0.02	1.63±0.01	1.72±0.02	1.81±0.01	1.75±0.01	1.73±0.02
Henicosane	2109	1.32±0.05	1.26±0.01	1.22±0.02	1.11±0.01	1.22±0.02	1.33±0.01	1.17±0.01	1.08±0.06
Monoterpene hydrocarbons		74.05±1.5	72.01±1.2	70.75±1.8	69.59±1.3	68.32±1.4	67.7±1.2	63.59±1.4	68.22±1.2
Oxygenated monoterpenes		7.03±0.3	6.91±0.4	7.77±0.2	5.25±0.3	8.16±0.5	6.01±0.3	5.78±0.2	6.82±0.3
Sesquiterpenes		1.88±0.04	1.76±0.02	1.66±0.01	1.58±0.01	1.91±0.04	1.94±0.03	1.88±0.01	1.82±0.03
Acetate esters terpenoids		4.43±0.01	4.35±0.01	4.22±0.02	4.15±0.02	4.11±0.01	4.05±0.01	4.01±0.02	4.08±0.03
Polycyclic Aromatic Hydrocarbons		1.67±0.05	1.69±0.01	1.71±0.02	1.72±0.01	1.55±0.02	1.51±0.01	1.49±0.02	1.52±0.03
Hydrocarbons Alkanes		2.88±0.05	2.89±0.04	2.83±0.03	2.74±0.02	2.94±0.04	3.14±0.05	2.92±0.02	2.81±0.01
EOs content (w/w%, g/100g fresh weight basis)		0.44±0.02	0.43±0.01	0.41±0.02	0.42±0.01	0.40±0.02	0.39±0.01	0.38±0.01	0.40±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation (P<0.05).

Table 8. Continued means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (60 mgL^{-1}) concentration (1st year)

Compound	RI [z]	Mn×Zn	Cu×Mn	Cu×Fe×Zn	Fe×Zn×Mn	Cu×Mn×Fe	Cu×Mn×Zn	Cu×Zn×Mn ×Fe
alpha-Thujene	926	18.32±1.1[y]	18.35±0.9	18.29±0.8	19.25±0.9	18.22±0.8	17.57±0.9	13.11±0.5
alpha-pinene	937	21.11±1.2	21.42±1.2	22.55±0.9	22.53±0.8	21.44±1.1	20.64±1.1	19.43±0.9
beta -myrcene	986	1.67±0.01	1.77±0.01	1.66±0.03	1.59±0.02	1.56±0.01	1.53±0.01	1.45±0.02
alpha-phellandrene	1003	1.63±0.01	1.65±0.01	1.62±0.03	1.58±0.02	1.56±0.01	1.57±0.01	1.52±0.02
beta -phellandrene	1027	4.45±0.06	4.33±0.01	4.23±0.04	4.23±0.02	3.44±0.01	3.49±0.01	3.13±0.01
Cis-ocimene	1031	13.18±0.9	12.21±0.8	13.22±0.9	12.25±0.9	12.77±1.1	12.9±1.2	11.43±1.1
4-thujanol	1041	3.51±0.01	3.33±0.01	1.22±0.03	1.11±0.02	1.09±0.01	2.01±0.01	1.01±0.02
beta -ocimene	1053	3.42±0.01	3.66±0.01	3.55±0.02	3.44±0.02	3.61±0.01	3.52±0.01	3.12±0.02
Sabinene	1192	2.01±0.01	1.92±0.01	1.89±0.03	1.83±0.02	1.77±0.01	1.66±0.01	1.56±0.02
Verbenone	1204	3.15±0.01	3.11±0.01	2.06±0.02	3.05±0.02	3.16±0.01	2.78±0.01	2.45±0.02
Bornyl acetate	1219	3.98±0.01	3.89±0.01	3.77±0.02	3.65±0.02	3.41±0.01	3.53±0.01	3.11±0.02
Naphthalenemethanol	1468	3.48±0.07	3.44±0.01	3.41±0.02	3.37±0.01	3.33±0.02	3.39±0.01	3.01±0.02
Bicyclo-germacrene	1491	1.55±0.07	1.44±0.02	1.37±0.01	1.22±0.01	0.99±0.04	1.01±0.03	0.78±0.01
Nonadecane	1900	4.56±0.05	4.62±0.01	3.59±0.02	3.61±0.01	4.44±0.02	3.12±0.01	3.99±0.01
Henicosane	2109	4.09±0.05	3.01±0.01	3.02±0.02	3.01±0.01	3.08±0.02	3.95±0.01	4.54±0.01
Monoterpene hydrocarbons		65.79±1.4	65.31±1.2	65.39±1.4	66.7±1.7	64.37±1.2	62.88±1.1	54.75±1.4
Oxygenated monoterpenes		6.66±0.3	6.44±0.2	3.28±0.3	4.16±0.2	4.25±0.1	4.79±0.2	3.46±0.1
Sesquiterpenes		1.55±0.07	1.44±0.02	1.37±0.01	1.22±0.01	0.99±0.04	1.01±0.03	0.78±0.01
Acetate esters terpenoids		3.98±0.01	3.89±0.01	3.77±0.02	3.65±0.02	3.41±0.01	3.53±0.01	3.11±0.02
Polycyclic Aromatic Hydrocarbons		4.48±0.07	3.44±0.01	3.41±0.02	3.37±0.01	3.33±0.02	3.39±0.01	3.01±0.02
Hydrocarbons Alkanes		8.65±0.3	7.63±0.2	6.61±0.1	6.62±0.2	7.52±0.2	7.07±0.3	8.53±0.2
EOs content (w/w%, g/100g fresh weight basis)		0.39±0.01	0.40±0.01	0.41±0.02	0.40±0.01	0.39±0.02	0.39±0.01	0.38±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation (P<0.05).

Table 9. Means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (60 mgL^{-1}) concentration (2nd year).

Compound	RI [z]	Fe	Zn	Cu	Mn	Fe×Zn	Cu×Fe	Mn×Fe	Cu×Zn
alpha-Thujene	926	14.12±0.9[y]	14.03±0.8	14.05±0.9	13.09±0.8	13.02±0.9	13.99±0.7	14.82±0.7	14.75±0.8
alpha-pinene	937	23.11±1.1	23.1±1.2	23.5±1.1	23.3±1.1	22.9±1.2	22.6±1.1	22.43±1.2	22.8±0.9
beta -myrcene	986	1.91±0.01	1.88±0.01	1.76±0.03	1.87±0.02	1.77±0.01	1.72±0.01	1.71±0.02	1.69±0.02
alpha-phellandrene	1003	2.01±0.01	1.89±0.01	1.91±0.03	1.85±0.02	1.87±0.01	1.79±0.01	1.75±0.02	1.78±0.02
beta -phellandrene	1027	4.57±0.07	4.34±0.01	4.61±0.04	4.63±0.02	4.64±0.01	4.59±0.01	4.94±0.01	4.59±0.07
Cis-ocimene	1031	16.18±0.9	16.1±0.8	15.2±0.9	15.5±0.8	15.7±0.7	14.9±0.9	14.22±0.8	14.7±0.9
4-thujanol	1041	10.12±0.01	10.11±0.5	10.08±0.5	9.02±0.08	9.91±0.07	9.88±0.09	8.79±0.08	8.68±0.09
beta -ocimene	1053	5.11±0.01	5.08±0.01	5.01±0.02	5.04±0.02	4.88±0.01	4.79±0.01	4.66±0.02	4.82±0.03
Sabinene	1192	2.12±0.01	2.08±0.01	2.03±0.03	2.01±0.02	1.99±0.01	1.89±0.01	2.01±0.02	2.04±0.02
Verbenone	1204	7.15±0.01	7.13±0.01	7.08±0.02	8.03±0.02	7.19±0.01	7.11±0.01	7.07±0.02	8.01±0.01
Bornyl acetate	1219	5.22±0.01	5.12±0.01	5.06±0.02	5.02±0.02	5.14±0.01	5.12±0.01	4.99±0.02	5.01±0.03
Naphthalenemethanol	1468	1.89±0.05	1.78±0.01	1.77±0.02	1.69±0.01	1.65±0.02	1.61±0.01	1.58±0.02	1.62±0.03
Bicyclo-germacrene	1491	1.91±0.06	1.89±0.02	1.77±0.01	1.66±0.01	1.55±0.04	1.44±0.03	1.39±0.01	1.47±0.05
Nonadecane	1900	1.44±0.04	1.35±0.01	1.31±0.02	1.28±0.01	1.47±0.02	1.46±0.01	1.48±0.01	1.33±0.05
Henicosane	2109	1.33±0.04	1.21±0.01	1.19±0.02	1.12±0.01	1.09±0.02	1.11±0.01	1.13±0.01	1.17±0.04
Monoterpene hydrocarbons		69.13±1.3	68.5±1.1	68.07±1.2	67.29±1.1	66.77±1.3	66.27±1.1	66.54±1.2	67.17±1.1
Oxygenated monoterpenes		17.27±0.6	17.24±0.3	17.16±0.2	17.05±0.3	17.01±0.2	16.99±0.5	15.86±0.3	16.69±0.4
Sesquiterpenes		1.91±0.06	1.89±0.02	1.77±0.01	1.66±0.01	1.55±0.04	1.44±0.03	1.39±0.01	1.47±0.05
Acetate esters terpenoids		5.22±0.01	5.12±0.01	5.06±0.02	5.02±0.02	5.14±0.01	5.12±0.01	4.99±0.02	5.01±0.03
Polycyclic Aromatic Hydrocarbons		1.89±0.05	1.78±0.01	1.77±0.02	1.69±0.01	1.65±0.02	1.61±0.01	1.58±0.02	1.62±0.03
Hydrocarbons Alkanes		2.77±0.01	2.56±0.01	2.51±0.01	2.41±0.01	2.56±0.01	2.57±0.02	2.61±0.01	2.51±0.01
EOs content (w/w%, g/100g fresh weight basis)		0.51±0.01	0.50±0.01	0.50±0.02	0.49±0.01	0.48±0.02	0.50±0.01	0.48±0.01	0.48±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation ($P<0.05$).

Table 9. Continued means of EOs content and composition (%) in *F. angulate* plants affected by micronutrients (60 mgL^{-1}) concentration (2nd year)

Compound	RI [z]	Mn×Zn	Cu×Mn	Cu×Fe×Zn	Fe×Zn×Mn	Cu×Mn×Fe	Cu×Mn×Zn	Cu×Zn×Mn×Fe
alpha-Thujene	926	13.71±0.6[y]	13.79±0.7	12.81±0.8	13.81±0.7	13.77±0.6	14.72±0.7	12.18±0.7
alpha-pinene	937	22.11±1.1	22.42±1.1	22.55±0.9	22.53±0.9	21.95±0.8	21.44±0.8	20.13±0.9
beta -myrcene	986	1.71±0.01	1.68±0.01	1.61±0.03	1.62±0.02	1.59±0.01	1.57±0.01	1.55±0.02
alpha-phellandrene	1003	1.89±0.01	1.84±0.01	1.82±0.03	1.75±0.02	1.73±0.01	1.74±0.01	1.65±0.02
beta -phellandrene	1027	4.57±0.04	4.59±0.01	4.11±0.04	4.16±0.02	4.12±0.01	4.09±0.01	3.56±0.01
Cis-ocimene	1031	14.18±1.2	13.21±1.1	13.22±1.2	12.25±0.9	12.77±0.8	12.9±0.9	11.72±0.8
4-thujanol	1041	9.55±0.01	9.33±0.01	9.22±0.03	8.12±0.02	8.99±0.01	8.81±0.01	8.28±0.02
beta -ocimene	1053	4.55±0.01	4.34±0.01	4.12±0.02	4.09±0.02	4.02±0.01	3.89±0.01	3.55±0.02
Sabinene	1192	2.02±0.01	2.01±0.01	1.99±0.03	1.89±0.02	1.88±0.01	1.91±0.01	1.69±0.02
Verbenone	1204	7.05±0.01	7.06±0.01	7.03±0.02	6.99±0.02	6.93±0.01	6.96±0.01	5.78±0.02
Bornyl acetate	1219	4.78±0.01	4.65±0.01	4.55±0.02	4.43±0.02	4.21±0.01	4.22±0.01	3.45±0.02
Naphthalenemethanol	1468	1.67±0.04	1.55±0.01	1.48±0.02	1.44±0.01	1.38±0.02	1.39±0.01	1.13±0.02
Bicyclo-germacrene	1491	1.33±0.05	1.25±0.02	1.22±0.01	1.17±0.01	1.11±0.04	1.15±0.03	1.01±0.01
Nonadecane	1900	5.31±0.03	5.21±0.01	5.27±0.02	5.25±0.01	5.22±0.02	5.19±0.01	5.81±0.01
Henicosane	2109	4.09±0.07	4.11±0.01	4.06±0.02	4.05±0.01	4.91±0.02	4.95±0.01	4.99±0.01
Monoterpene hydrocarbons		64.74±1.4	63.88±1.2	62.23±1.5	62.1±1.1	61.83±1.2	62.26±1.4	56.03±0.9
Oxygenated monoterpenes		16.6±0.8	16.39±0.4	16.25±0.4	15.11±0.3	15.92±0.5	15.77±0.4	14.06±0.5
Sesquiterpenes		1.33±0.05	1.25±0.02	1.22±0.01	1.17±0.01	1.11±0.04	1.15±0.03	1.01±0.01
Acetate esters terpenoids		4.78±0.01	4.65±0.01	4.55±0.02	4.43±0.02	4.21±0.01	4.22±0.01	3.45±0.02
Polycyclic Aromatic Hydrocarbons		1.67±0.04	1.55±0.01	1.48±0.02	1.44±0.01	1.38±0.02	1.39±0.01	1.13±0.02
Hydrocarbons Alkanes		9.40±0.04	9.32±0.03	9.33±0.04	9.30±0.05	10.13±0.03	10.14±0.04	10.81±0.03
EOs content (w/w%, g/100g fresh weight basis)		0.47±0.01	0.46±0.01	0.47±0.02	0.45±0.01	0.45±0.02	0.44±0.01	0.42±0.01

[z] RI: Retention Indices, as determined with FID and HP-5MS 5% capillary column using a series of the standards of C7-C30 saturated n-alkanes. [y] Values are means of triplicates ± standard deviation ($P<0.05$).

Table 10. Simple correlation between content and main compositions of EOs of *F. angulate* plants under application of tested micronutrients in two year.

Year	Verbenone (10)	4-thojanol (9)	Nonadecane (8)	Bornyl acetate (7)	beta-ocimene (6)	Cis-ocimene (5)	beta-phellandrene (4)	alpha-pinene (3)	alpha-Thujene (2)	EOs (1)	
2022	-	-	-	-	-		-	-	-	1	1
	-	-	-	-	-		-	-	1	0.95**	2
	-	-	-	-	-		-	1	0.75**	0.64*	3
	-	-	-	-	-		1	0.8**	0.52**	0.5**	4
	-	-	-	-	-	1	0.66**	0.71**	0.55**	0.9**	5
	-	-	-	-	1	0.54**	0.95**	0.47**	0.64**	0.68*	6
	-	-	-	1	0.95**	0.78**	0.66**	0.82**	0.6**	0.4*	7
	-	-	1	0.69**	0.48**	0.48**	0.86**	0.67**	0.72**	-0.8**	8
	-	1	0.86**	0.53**	0.81**	0.81**	0.68**	0.79**	0.65**	0.75**	9
	1	0.77**	0.99**	0.86**	0.66**	0.81**	0.84**	0.68**	0.69**	0.55**	10
2023	-	-	-	-	-	-	-	-	-	1	1
	-	-	-	-	-	-	-	-	1	0.84**	2
	-	-	-	-	-	-	-	1	0.9**	0.66**	3
	-	-	-	-	-	-	1	0.62**	0.59**	0.51**	4
	-	-	-	-	-	1	0.3	0.75**	0.55**	0.82**	5
	-	-	-	-	1	0.79**	0.42**	0.45**	0.71**	0.5**	6
	-	-	-	1	0.68**	0.95**	0.76**	0.73**	0.62**	0.4**	7
	-	-	1	0.77**	0.95**	0.77**	0.66**	0.63**	0.7**	-0.9**	8
	-	1	0.67**	0.72**	0.88**	0.81**	0.88**	0.65**	0.66**	0.45**	9
	1	0.81**	0.77**	0.86**	0.53**	0.73**	0.66**	0.55**	0.83**	0.65**	10

*, ** significant at P=0.05 and P=0.01 probability levels, respectively.

In this study, the quality of EOs, which is expressed as a percentage of the chemical compounds, showed a significant increase under concentration of micronutrients. According to obtained results, application of micronutrients significantly affected the EOs content/composition of *F. angulate*, however the assessed traits showed slightly variation during the studied years. In most of measured characters, plants treated with 20 and 40 mgL^{-1} were in the same group. Amounts of some characters in plants treated with 60 mgL^{-1} were less than control plants. The main constituents of EOs were alpha-thujene, alpha-pinene, beta-phellandrene, cis-ocimene (monoterpene hydrocarbons) and 4-thujanol (oxygenated monoterpenes) that made the most components of EOs plants. Applied combination of micronutrients showed better effectiveness on EOs content and composition and the highest EOs content was obtained from the treated plants by 20 and 40 mgL^{-1} of these micronutrients. Although in some main treatments such as foliar application of Cu and Mn, no significant difference in chemical compositions of *F. angulate* EOs was found, however, in combined treatments there were differences in most compositions of EOs and in this regard, the $Fe_2Cu_2Mn_2Zn_2$ treatment was the best treatment. The main components in the EOs of all plants treated by micronutrients included alpha-thujene, alpha-pinene, beta-myrcene, alpha-phellandrene, beta-phellandrene, cis-ocimene, beta-ocimene, sabinene (monoterpene hydrocarbons), 4-thujanol, verbenone (oxygenated monoterpenes), bicyclo-germacrene (sesquiterpenes), bornyl acetate (acetate esters terpenoids), naphthalenemethanol (polycyclic aromatic hydrocarbons) and nonadecane and henicosane (hydrocarbons alkanes). The most amounts in some of constituents such as alpha-thujene, alpha-pinene, beta-myrcene, alpha-phellandrene, beta-phellandrene, cis-ocimene, beta-ocimene and sabinene were made by plants that treated by 40 mgL^{-1} of Fe, Zn and Mn but in another components of EOs, the most amounts were obtained by 40 mgL^{-1} of Fe, Zn, Mn and Cu. Treatments with 60 mgL^{-1} of micronutrients in many of constituents had the same group with control plants and the lowest amount of components made by this treatment. In some cases such as the lowest amounts of alpha-thujene, alpha-pinene, beta-myrcene, alpha-phellandrene, beta-phellandrene, cis-ocimene, beta-ocimene and sabinene made by Mn or Cu in 60 mgL^{-1}, but generally the control plants and plants treated with combination of 60 $mgL^{-1}$of Fe, Mn, Zn and Cu in two cultivation seasons made the lowest levels of EOs content and almost of compositions.

The most amounts of constituents belonging of hydrocarbons alkanes category obtained by combination of 60 $mgL^{-1}$of Fe, Mn, Zn and Cu and control. Application of micronutrients with the concentration of 20 mgL^{-1} improved most of the content and compositions of components but at higher concentrations (i.e. 60 mgL^{-1}), the content and compositions in all treated plants were decreased. The mean content (%) of many chemical compositions in *F. angulate* were lower than those of the control treatment when the plants were sprayed with concentrations of 60 mgL^{-1}. It seems that the content and composition of EOs were more affected by Zn and Fe compared to other micronutrients.

It has been shown that micronutrients of Fe, Cu, Zn and Mn help to increase better and more absorption of nutrients by influencing enzyme activities (Marschner, 1995; Pradhan *et al.*, 2017). Copper deficiency limits the activity of many plant enzymes, including ascorbate oxidase, phenolase, cytochrome oxidase, diamine oxidase, plastocyanin, and superoxide dismutase. Oxidation–reduction cycling between Cu(I) and Cu(II) oxidation states is required during single electron transfer reactions in copper-containing enzymes and proteins (Barker and Pilbeam, 2007). Iron is limited largely by diffusion in the soil solution, and thus the absorption is highly dependent on root activity and growth. In terms of fertilizers for terrestrial plants, iron deficiency usually comes about because of alkaline pH in the soil, and supply of iron salts to the soil would have no effect. Foliar application of iron-cheletes can be effective. Therefore, the usual way in which lime-induced chlorosis is alleviated is by supply of iron chelates to the foliage (Barker and Pilbeam, 2007; Pradhan *et al.*, 2017). Manganese involved in many biochemical functions, primarily acting as an activator of enzymes such as dehydrogenases, transferases, hydroxylases, and decarboxylases involved in respiration, amino acid and lignin synthesis, and hormone concentrations, but in some cases it may be replaced by other metal ions (Marschner, 1995). Zinc is an integral component of enzyme structures and coordinated to four ligands in enzymes with catalytic functions. Three of them are amino acids, with histidine being the most frequent, followed by glutamine and asparagine (Marschner, 1995; Pradhan *et al.*, 2017).

As a result, these positive impacts of the micronutrients could lead to the improvement of photosynthetic rate, biomass production and yield of aerial parts of medicinal plants (Hamedi *et al.*, 2020; Yadegari, 2023). In the current study it was found that the EOs content is in a yield ranging from 0.37 to 0.68% (w/w) made by control plants and plants treated with 40 mgL^{-1} of micronutrients ($Fe_2Zn_2Mn_2Cu_2$), respectively. The main reasons for this enhancement in the EOs content by applying micronutrients can be related to the balance between absorption of the essential elements in the root environment, increasing the rate of photosynthesis, stimulating the vital enzymes, activating plant growth regulators (PGR) production as inducing signal for terpenes biosynthesis (Pradhan *et al.*, 2017). In this research use of 40 mgL^{-1} of Fe, Zn, Mn and Cu increased EOs content from 0.37% to 59% in first year and from 0.41% to 68% in second year with an increase of more than 60%. The findings of this research showed that by increasing EOs content, the main compositions such as alpha-thujene, alpha-pinene, beta-phellandrene, cis-ocimene, beta-ocimene, 4-thujanol and verbenon in *F. angulate* plants treated with micronutrients in two years, increased.It has been shown that the percentage of the main compounds of EOs, is the main factor determining the quality of the EOs. It has been reported that among these compounds, monoterpene hydrocarbons such as alpha-thujene, alpha-pinene, beta-phellandrene, cis-ocimene and beta-ocimene improve the quality of the EOs of *F. angulate* (Azarbani *et al.*, 2023; Ghasemi Pirbalouti *et al.*, 2016). Maintaining the balance between nutrients and soil fertility is critical in sustainable soil management. Organic, biological and chemical fertilizers return nutrients consumed by plants to the soil. According to the results of the present study, the availability of optimum amounts of micronutrients provided the necessary nutrients for producing higher content and composition of EOs of *F. angulate*. EOs composition of *F. angulate* varies depending on the variety, climatic conditions, and nutritional status of the plant and soil. According to reported literature alpha-pinene, beta-pinene, beta-ocimene, bornyl acetate, thujanol, δ-terpinolene, sabinene, verbenone, alpha-phellandrene, beta-phellandrene, Cis-beta-ocimene and alpha-thujene, (Golfakhrabadi *et al.*, 2015; Razavi *et al.*, 2015; Ghasemi Pirbalouti *et al.*, 2016; Moghaddam *et al.*, 2018; Safari *et al.*, 2019; Mumivand *et al.*, 2019). A combination of the four micronutrients had a greater effect than a single micronutrient. Thus, the foliar application of 40 $mg.l^{-1}$ Fe, Cu, Mn and Zn was the most effective treatment compared to other treatments. The amount (percentage) of the main components in plants treated with 40 mgL^{-1} of micronutrients was twice more than those of the control plants.

Exogenous micronutrients affect respiration, photosynthesis, carbohydrate assimilation, and amino acids biosynthesis. These processes usually are simultaneous with the changes in the content of intermediate compounds and the activity of involved enzymes in the primary and secondary metabolism of plants (Marschner, 1995). Therefore, induced variations in the plant's physiological behaviour by micronutrients determine the quality of produced secondary metabolites. EOs belong to the group of terpenes and glucose is an essential precursor in the synthesis of terpenoids, especially monoterpenes. Therefore photosynthesis and photosynthetic products directly determine the biosynthesis of EOs (Bohlman and Keeling, 2008). A sufficient supply of nutrient elements in plant in response to exogenous micronutrients affects the biosynthesis of involved substrates and enzymes in terpenoids biosynthesis (Aghaei *et al.*, 2021; Pradhan *et al.*, 2017). For instance, providing a sufficient amount of micronutrients such as magnesium may affect the activity of geranyl diphosphate synthase, which requires this element for its activity (Chiyaneh *et al.*, 2022). EOs content is directly correlated with the main compounds such as alpha-thujene, alpha-pinene, beta-phellandrene, cis-ocimene, beta-ocimene, 4-thujanol and verbenon. In the present study based on GC and GC-MS results, monoterpene hydrocarbons represented by alpha-thujene, alpha-pinene, beta-myrcene, alpha-phellandrene, beta-phellandrene, cis-ocimene, beta-ocimene and sabinene, made more 55% of compounds of EOs, and oxygenated monoterpenes represented by 4-thujanol and verbenone made more than 4% of compounds of EOs of treated plants. In all of treated plants, the most compound belongs of monoterpenes hydrocarbons and oxygenated monoterpens, were alpha-pinene and 4-thujanol, respectively. The main compounds of EOs of treated plants by 40- mgL^{-1} of micronutrients were more than other treated plants, especially monoterpene hydrocarbons in compounds were more than other categories and otherwise the hydrocarbons alkanes such as nonadecane and henicosane increased in plants treated by 60 mgL^{-1} of micronutrients. Treatment of $Fe_2Cu_2Zn_2Mn_2$ in this research made the most alpha-thujene, alpha-pinene, beta-phellandrene, cis-ocimene, beta-ocimene such as monoterpene hydrocarbons and 4-thujanol and verbenone belong to the category of oxygenated monoterpene. Also treatment of $Fe_2Cu_2Zn_2Mn_1$ and $Fe_2Cu_1Zn_2Mn_2$ in most of main compounds were in the same group with $Fe_2Cu_2Zn_2Mn_2$. It seems that the role of iron and zinc are more important than other micronutrients that reported in pervious researches (Yadegari, 2023; Bilal *et al.*, 2020; Hamedi *et al.*, 2020). The results of previous investigation by Azarbani *et al.* (2023) showed that the EOs of *F. angulate* mainly consisted of alpha-pinene, bornyl acetate, beta-pinene and alpha-phellandrene. The results of another study indicated that the major components of volatile oil from *F. angulate* were cis-beta-ocimene, alpha-pinene, alpha-phellandrene, alpha-thujene, alpha-phellandrene, cis-ocimene and beta-phellandrene (Safari *et al.*, 2019; Mumivand *et al.*, 2019). The upper range of micronutrients (i.e. 60 mg.l^{-1}) decreased the content of the EOs of *F. angulate* but the composition of the hydrocarbons alkanes category increased. Control plants made better amounts of many EOs components of *F. angulate* than those treated plants with 60- mg.l^{-1} concentrations of Fe, Cu, Mn and Zn. In most treatments, the combination of $Fe_2Cu_2Mn_2Zn_2$, $Fe_2Cu_3Mn_3Zn_2$ and $Fe_2Cu_2Mn_2Zn_2$ made the maximum amount of EOs. However, $Fe_2Cu_2Mn_2Zn_2$ was the best combination. The increase in the EOs content in the plants treated with 20 and 40 mg.l^{-1} may be due to its role in enhancing the overall growth of aerial parts. In addition, active substances such as volatile oils are made by plants due to the plant's adaptation to biological and abiotic stresses, and the signals of these stresses act as elicitors for the plant cell (Azarbani *et al.*, 2023; Badalamenti *et al.*, 2023).

In some EOs compositions, control plants were similar to plants treated with $Fe_3Cu_3Mn_3Zn_3$, $Fe_3Cu_3Mn_2Zn_3$ and $Fe_2Cu_2Mn_3Zn_3$. It seems for toxicities of upper concentrations of Fe, Cu, Mn and Zn (i.e. 60 mg.l^{-1}), the content of components obtained at higher concentrations of micronutrients was similar to control plants. It was clear from the presented data that the highest levels of the four foliar fertilizers were more effective than the lower levels, and Librel Zn and Fe fertilizers were superior over other micronutrients. However, the highest EOs percentage was found with $Fe_2Cu_2Mn_2Zn_2$. Similar to the results obtained in this studyregarding *F. angulate*, the beneficial effect of micronutrients (Fe, Zn, Cu and Mn) and production of higher EOs content were reported by other researchers in *Rosa damascena* (Yadegari, 2023), *Satureja* sp. (Bani Taba *et al.*, 2022), *Melissa officinalis* (Yadegari, 2017b), *Carthamus tinctorius* (Galavi *et al.*, 2012), *Calendula officinalis* L., *Borago officinalis*, *Alyssum desertorum* and *Thymus vulgaris* (Yadegari, 2015 2017a), *Anethum graveolens* (Rostaei *et al.*, 2018) and *Matricaria chamomilla* (Nasiri *et al.*, 2010).

Results of this research indicated that foliar application of micronutrients resulted in higher EOs content in the shoots of *F. angulate* plants than the control plants. In the present study, the effect of micronutrients on the EOs content and composition was determined over two consecutive years. The EOs yield increased with Fe, Cu, Zn and Mn applications because of a significant rise in dry matter and the number of flowers (data not published)**.** It was revealed that Fe, Cu, Zn and Mn are beneficial for *F. angulate* plants with concentrations of 40 mg.l^{-1}or lower, and can result in more content of EOs up to 40%. Fe, Cu, Zn and Mn have immediate impacts on the growth and development of plants. There are still many unanswered questions about the mechanism of Fe,Cu,Zn and Mn in enhancing yield and its components for *F. angulate* plants. One possibility is that the foliar applied Fe, Cu, Zn and Mn can affect absorbtion of other minerals and then increase shoot dry matter and finally the EOs yield in plants increased (Alamer *et al.*, 2020; Alejandro *et al.*, 2020; Aziz *et al.*, 2019). It was determined in this study that control plants with no foliar treatment experienced better growth than the plants with a higher concentration of the micronutrients. Combinations of micronutrients with an optimum concentration (i.e. 40 mgL^{-1}) had the best effect. However, combinations of micronutrients of a concentration higher than 40 mgL^{-1} (i.e. 60 mgL^{-1}) had more reducing effects than single micronutrients. These results reflect the role of the simultaneous application of an optimum concentration of the four foliar fertilizers in improving the total EOs in medicinal plants. Micronutrients of higher than 40 mg.l^{-1} concentration especially in three micronutrient- or in four micronutrient-combinations reduced the content and composition of the EOs. Generally, the production of the secondary metabolites and the chemical compositions of the plant EOs is influenced by genetic factors, ecological, soil conditions, management (sowing to harvesting and post-harvesting processes) and their interactions (Shahbazi, 2016; Ghasemi *et al.*, 2016; Hamedi *et al.*, 2020).

4. Conclusion

F. angulate plants treated with 40 mgL^{-1} of iron, zinc, manganese and copper in the chelate formula, produced higher content and composition of the EOs. It could be concluded from the results that iron, zinc, manganese and copper fertilization had significant effects on the measured characters as well as the chemical composition of the EOs of *F. angulate* plants. Also, the combined application of micronutrient fertilizers had a more pronounced effect in comparison with the individual use of the micronutrients. This study provided some useful information about the impact of foliar application of micronutrients where soils have undesirable characteristics and chemical properties in particular. The main constituents of the volatile oils of *F. angulate* were alpha-thujene, alpha-pinene, beta-myrcene, alpha-phellandrene, beta-phellandrene, cis-ocimene, beta-ocimene and sabinene (54-93%). In this study, the highest content of EOs and the percentages of monoterpene hydrocarbons in the EOs were observed in the herbs under treatments of 40 mgL^{-1} of micronutrients however the combination of 40 mgL^{-1} of iron, zinc and copper was in the same group. Finally, the use of 40 mgL^{-1} of micronutrients (Fe, Zn, Cu and Mn) is recommended for stabilizing the quantitative and qualitative yield of *F. angulate* in same climates.

5. References

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