Ciência Rural, Santa Maria, v.51:11, e20200979, 2021 http://doi.org/10.1590/0103-8478cr20200979

ISSNe 1678-4596
BIOLOGY

Seasonal variation in essential oil content and chemical profile of mint in southeast of Brazil

Joyce Pereira Alvarenga[1] Andreísa Flores Braga[1] Fernanda Ventorim Pacheco[1] Alexandre Alves de Carvalho[1] José Eduardo Brasil Pereira Pinto[1] Suzan Kelly Vilela Bertolucci[2*]

[1]Laboratório de Cultura de Tecidos Vegetais e Plantas Medicinais, Departamento de Agricultura, Universidade Federal de Lavras (UFLA), Lavras, MG, Brasil.
[2]Laboratório de Fitoquímica e Plantas Medicinais, Departamento de Agricultura, Universidade Federal de Lavras (UFLA), 37200-900, Lavras, MG, Brasil. E-mail: suzan@ufla.br. *Corresponding author.

ABSTRACT: *This study evaluated the influence of seasonality in essential oil (EO) chemical composition of Mentha x piperita L. and Mentha viridis L. grown in southeast of Brazil. Leaves were collected from November 2011 until August 2013, in the middle of the following seasons: Spring (November), Summer (February), Autumn (May) and Winter (August). EO extraction was carried out by hydrodistillation in Clevenger apparatus for 1 hour. Chemical composition of EO was analyzed by GC-FID and GC-MS. The EO content and its chemical composition were influenced by seasonality in both species. The highest EO content was obtained for M. x piperita during spring (4.26%) and for M. viridis during spring (3.30%) and summer (3.70%). Mentha x piperita increased menthol (16.31 to 41.26%), neomenthol (3.02 to 6.39%) and menthone (5.56 to 41.58%) contents during spring and summer, respectively. Whereas, M. viridis EO content did not show a quality response pattern in relation to seasonality. Therefore, harvest time for M. x piperita is recommended during spring, and for M. viridis is recommended during spring and summer.*
Key words: *medicinal plant, Mentha x piperita, Mentha viridis, menthol, linalool.*

Variação sazonal no teor e perfil químico do óleo essencial de menta no sudeste do Brasil

RESUMO: *O estudo teve como objetivo avaliar a influência da sazonalidade na composição química do óleo essencial (OE) de Mentha x piperita L. e Mentha viridis L. cultivadas no sudeste do Brasil. As folhas foram coletadas de novembro de 2011 até agosto de 2013, em meados das seguintes estações: primavera (novembro), verão (fevereiro), outono (maio) e inverno (agosto). A extração do OE foi realizada por hidrodestilação em aparelho de Clevenger por uma hora. A composição química do EO foi analisada por GC-FID and GC-MS. O teor de OE e sua composição química foram influenciados pela sazonalidade em ambas as espécies. O maior teor de OE foi obtido para M. x piperita na primavera (4,26%) e para M. viridis na primavera (3,30%) e verão (3,70%). Mentha x piperita aumentou o conteúdo de mentol (16,31 a 41,26%), neomentol (3,02 a 6,39%) e mentona (5,56 a 41,58%) durante a primavera e o verão, respectivamente. Já o conteúdo de OE de M. viridis não apresentou padrão de resposta de qualidade em relação à sazonalidade. Portanto, a colheita para M. x piperita é recomendada durante a primavera, e para M. viridis é recomendada durante a primavera e verão.*
Palavras-chave: *planta medicinal, Mentha x piperita, Mentha viridis, mentol, linalol.*

INTRODUCTION

Species from *Mentha* genus (Lamiaceae) are globally known for their medicinal and aromatic properties, which promote a greater demand of raw materials. Mint species are broadly used in traditional medicine, but also have a great economic importance. Their essential oil (EO) has antimicrobial, antioxidant, larvicidal and cytotoxic activities (GONÇALVES et al., 2009; MKADDEM et al., 2009; RAMOS et al., 2017). Those EOs are widely explored in food industry, pharmaceutics, cosmetics and perfumery (POOVAIAH et al., 2006; KIZIL et al., 2010). Additionally, the use of EO has been increased by different means, including its use in agriculture as botanical insecticides; and also as food preservative and disinfectants in food industry (ANUAR et al., 2019; CAMPOS et al., 2019; FALCÓ et al., 2019).

The EO chemical composition of *Mentha x piperita* is constituted by oxygenated monoterpenes such as menthol, menthone, menthofuran, menthyl acetate and 1,8-cineole (KIZIL et al., 2010; RAMOS et al., 2017). It can also be reported in its composition limonene, carvone, neoisomenthol, β-caryophyllene,

Received 11.05.2020 Approved 03.15.2021 Returned by the author 05.12.21
CR-2020-0979.R1

myrcene, α-pinene and β-pinene (GONÇALVES et al., 2009). In the EO of *M. viridis* is also reported a rich composition of monoterpenes, particularly limonene, carvone, 1,8-cineole, terpinen-4-ol and α-terpineol (KUMAR et al., 2011).

Qualitative and quantitative determination of those monoterpenes regulates the quality and commercial value of EOs. For instance, a high quality EO of *M.* x *piperita* consists predominantly of menthol (30-55%), a moderate content of its precursor menthone (14-32%) and a low content of pulegone (<4%), menthofuran (1-9%) and menthyl acetate (2.8-10%) (BEHN et al., 2010). When working with medicinal plants, is important to know that the EO chemical composition can change significantly due to different variables; such as environmental conditions and plant phenology. Once its composition change, the biological activity can also change (EHLERT et al., 2013; SILVA et al., 2015; LEMOS et al., 2017). Hence, this is an essential subject that needs to be assessed for EO production and its application. Studies have been performed to demonstrate the seasonal influences on biosynthesis and chemical composition of EO (LEMOS et al., 2017; GAD et al., 2019). Including differences observed in the genus *Mentha* in different regions of the world (HUSSAIN et al., 2010; SANTOS et al., 2012; NIKŠIĆ et al., 2014; ZOUARI-BOUASSIDA et al., 2018)

Brazil is a large country with very diverse geographical areas, thus varying environmental conditions. Therefore, seasonal differences will affect the EO production by medicinal plants. Relevant studies verify these effects on EO content and quality, contributing to a better understanding of the EO production and obtaining quality plant material (VERMA et al., 2014). In this way, this study to evaluated the effect of seasonality on EO content and chemical composition of *M.* x *piperita* and *M. viridis* in the southeast region of Brazil.

MATERIALS AND METHODS

The experiment was performed over the span of two years (from November 2011 until August 2013), in Lavras, Minas Gerais, Brazil (21º 14'S and 45º 00'W, 918 m altitude). The weather is classified as type Cwa, with dry winter and prevalent rainfall during summer (JÚNIOR et al., 2012). Figure 1 shows the climatic data observed during the period of the experiment.

Plantlets of *M.* x *piperita* and *M. viridis* were obtained by microcuttings (4 - 6 cm long), which were grown in trays with commercial substrate Hortplant® in a greenhouse, with constant irrigation. The donor plants were identified by the botanist Dr. Manuel Losada Gavilanes. After 30 days, the plantlets were transplanted to 6x1.20 m beds, previously fertilized with poultry manure. The exsiccates of *M. viridis* (Mentol Mint CEN47647) and *Mentha* x *piperita* (cv. Grapefruit Mint CEN47639 X cv. Mint Persian Field CEN47646) are deposited in the herbarium of the National Center for Genetic Resources and Biotechnology - CENARGEN / EMBRAPA , Brasilia DF.

The experiment was conducted during two consecutive years, with a single harvest at 8:00 am per season in each year. EOs were obtained from fresh leaves for both species. Leaves were harvested randomly in the middle of each season: November/2011 (spring), February/2012 (summer), May/2012 (autumn) and August/2012 (winter); second year: November/2012 (spring), February/2012 (summer), May/2013 (autumn) and August/2013 (winter).

EO extraction was performed by hydrodistillation for 90 minutes using the Clevenger apparatus (GONÇALVES et al., 2009; SILVA et al., 2019) and 4 extractions per treatment was done. The EO content was determined by the dry leaf mass to EO mass (%) ratio. Quantitative analysis of the EO was performed by gas chromatography coupled with a flame ionization detector (GC-FID) in an Agilent® 7890A system equipped with DB-Wax capillary column (30 m length x 0.25 mm internal diameter × film thickness of 0.25 μm) (Agilent J&W). The technical parameters of the chemical analysis were adjusted according to SILVA et al. (2019) and OLIVEIRA et al. (2021). Helium was used as carrier gas at 1.0 mL/min, temperatures of the injector and detector were maintained at 220 °C and 240 °C, respectively. The initial oven temperature was set in 60 °C, isothermal for 2 min, followed by a temperature ramp of 3 °C/min to 190 °C, followed by a ramp of 10 °C/min to 230 °C. The oil was diluted in ethyl acetate (1% v/v) and automatically injected into the chromatograph using volume of 1.0 μL injection in split injection mode at a ratio of 50:1. The analysis was performed in triplicate (n=3) and the results expressed by mean of the percentage of normalized area relative to the chromatographic peaks ± standard deviation. The EO qualitative analyzes were performed by gas chromatography coupled to mass selective detector (GC-MS), using a Agilent® 5975C equipment operated by electronic impact ionization at 70eV in scan mode at a speed 1.0 scan/s, with acquisition of a mass range of 40-

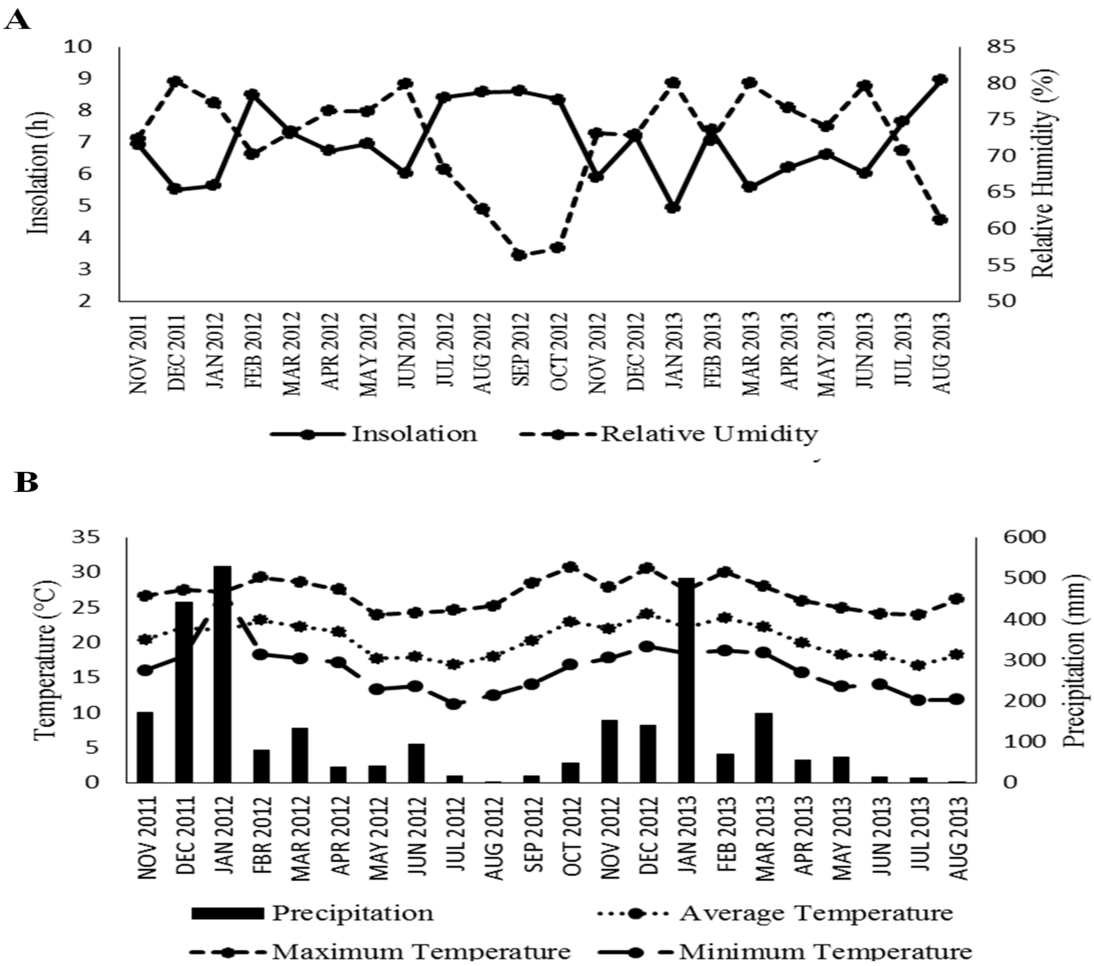

Figure 1 - Regional climate variation during the experimental period (from November 2011 to August 2013): (A) Insolation and relative humidity data; (B) Precipitation and temperature data. Source: Climatological Station of Universidade Federal de Lavras.

400*m/z*. The chromatographic conditions were the same used in quantitative analysis.

The components were identified by comparing their retention index with tabulated data and the GC-MS obtained mass spectra to the library database NIST/ EPA/ NIH. Linear retention indices were calculated by injection of a series of *n*-alkanes solution (C8-C20) (Sigma Chemical Co., St. Louis, MO) in the same column and condition as above, using the equation proposed by VAN DEN DOOL & KRATZ (1963).

The software SISVAR® (FERREIRA, 2011) was used for the statistical analysis. Tukey test at 5% probability was used to compare the means. Standardized data of three replicate (compound contents of *M.* x *piperita* and *M. viridis*) were subjected to multivariate analysis. Principal component analysis (PCA) was presented in graphic biplot using Statistica software, version 13.5 (StatSoft, Tulsa, OK). The identified chemical compounds and respective contents were transformed by the PCA in orthogonal latent variables, which are linear combinations of all original variables (BARTHOLOMEW, 2010).

RESULTS AND DISCUSSION

The seasonality influenced both mint species EO content (Figure 2). The highest EO

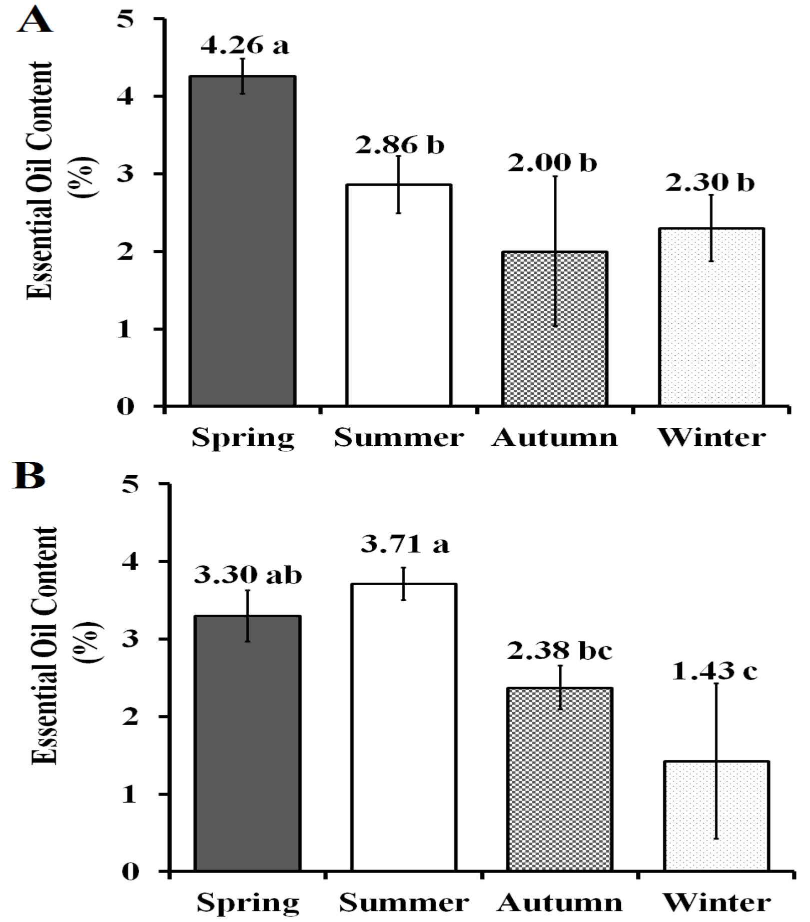

Figure 2 - Essential oil content from Mentha x piperita (A) and Mentha viridis (B) harvested in different seasons (spring, summer, autumn and winter). Means (±standard deviation) followed by the same letter do not differ in Tukey's test (P>0.05).

content for *M.* x *piperita* was obtained during spring (4.26%) and for *M. viridis* during spring (3.30%) and summer (3.70%) harvests. Similar results were obtained in different studies, where it reported a higher EO content during spring and summer seasons, in contrast to autumn and winter (DESCHAMPS et al., 2008; SANTOS et al., 2012; LEMOS et al., 2017). During spring (September to December) and summer (December to March) higher insolation (incidence of UV radiation), temperatures and precipitations were observed compared to autumn (March to June) and winter (June to September) (Figure 1). Such conditions may provide an increase in the photosynthetic activity and growth of species, contributing with a greater amount of carbon skeletons to produce secondary metabolites (SANTOS et al., 2012). According to SINGH & SHARMA (2015), the role of light and temperature in modulating a range of terpenoids and the corresponding transcripts has been reported, but there is no universal behavior and it varies depending upon the type of metabolites as well as plant species.

Additionally, these findings may be associated with species phenology, which is highly influenced by seasons. During spring and summer harvest, for both years, *M.* x *piperita* and *M. viridis* were in the bloom stage. Some authors claim that mint species tend to produce more EO content during this phase (DESCHAMPS et al., 2008; HUSSAIN et al., 2010; SANTOS et al., 2012; ZOUARI-BOUASSIDA et al., 2018). Since mono- and sesquiterpenes compounds present in EOs play an important role in attraction of pollinators and protection against photo-oxidative and oxidative stress, insects and thermo-protection (PAOLINI et al., 2010; REHMAN et al., 2016). Such event can be explained by the need to increase the production of secondary metabolites for reproduction and protection during this period.

The chemical composition of the EOs of *M.* x *piperita* and *M. viridis* are shown in tables 1 and 2, respectively. A range of 68.12 to 96.17% of total components was identified for both EO samples. Oxigenated monoterpene was the most abundant class of volatile compounds in the analyzed samples, ranging from 66.91 to 92.13% for *M.* x *piperita* and 78.76 to 88.47% for *M. viridis*. Many of these molecules have an especially important biological activity, being important the antimicrobial, antifungal, antioxidant, anti-inflammatory, insecticide, analgesic, anticancer and cytotoxic (NORIEGA, 2020). Results showed that EO composition of *M. piperita* and *M. viridis* were influenced qualitatively and quantitatively according to the season and year harvest period (Table 1 and 2). The major compounds identified in *M.* x *piperita* EO were menthol (16.31 - 41.26%) and menthone (5.56 - 41.58%), and in *M. viridis* EO were linalool (45.18 - 52.23%) and carvone (13.20 to 21.28%). According to NORIEGA (2020), menthol is one of the most used compounds in the food, cosmetic, pharmaceutical industries, and pesticides, and its aromatic properties are very well known. Menthone is used as perfume and flavor compositions, linalool in perfumery instead of Bergamot or French lavender oil, since it has similar odor and carvone as flavor in liqueurs, perfumery and oral hygiene products (GRACINDO et al., 2006).

Concerning qualitative analysis, in the EO of *M.* x *piperita* 1,8-cineole was not detected during spring and summer of the second year, while menthofuran was detected just during spring of the first year; and summer and winter of the second year. For *M. viridis* EO

Table 1 - Seasonal variation of the chemical composition of *Mentha* x *piperita* L. essential oil growth in southeast of Brazil in two consecutive years (November 2011 to August 2013).

Compounds	IR[a]	%Area ± SD							
		-----------Spring----------		----------Summer-----		-----------Autumn---------		----------Winter-------	
		1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year
Limonene	1202	1.04±0.17	3.51±0.49	1.10±0.10	2.87±0.52	0.67±0.21	1.21±0.57	1.06±0.21	1.23±0.28
1,8-Cineole	1213	4.38±0.31	nd	4.20±0.61	nd	3.49±0.62	0.29±0.05	1.20±0.25	0.12±0.04
Menthone	1460	19.00±1.76	41.58±1.78	17.01±2.63	17.49±1.47	5.56±1.86	8.90±1.90	18.57±1.30	8.56±1.41
Menthofuran	1478	3.14±0.39	nd	nd	11.46±0.75	nd	nd	nd	4.13±6.95
iso-Menthone	1487	2.67±0.09	4.51±0.12	2.32±0.11	2.37±0.12	1.56±0.57	1.38±0.42	2.45±0.85	0.66±0.12
Neomenthol acetate	1527	0.69±0.10	0.55±0.15	1.63±0.10	0.39±0.06	2.06±0.12	1.19±0.30	1.73±0.08	4.36±0.21
Menthyl acetate	1561	10.96±1.28	5.06±0.95	18.75±0.74	6.27±0.78	27.18±2.15	18.97±5.72	27.92±1.16	52.24±2.43
iso-Menthol acetate	1576	0.52±0.06	0.26±0.04	0.90±0.03	0.36±0.04	1.13±0.10	0.66±0.19	1.16±0.06	1.67±0.07
Neomenthol	1599	5.66±0.18	3.59±0.23	6.39±0.41	4.18±0.10	5.22±0.15	3.91±0.45	4.41±0.09	3.02±0.18
Neoisomenthol	1638	0.15±0.02	2.34±0.15	0.79±0.20	2.66±0.21	1.02±0.27	2.63±0.89	1.71±0.19	0.27±0.03
Menthol	1645	41.26±0.95	29.55±0.19	38.88±2.16	40.39±0.47	35.36±0.73	28.98±3.69	30.99±0.16	16.31±1.80
Hydrogenated monoterpenes		1.04	3.51	1.10	2.87	0.67	1.21	1.06	1.23
Oxygenated monoterpenes		90.32	88.68	91.88	86.81	82.57	66.91	90.14	92.13
Total identified (%)		91.36	92.19	92.98	89.68	83.24	68.12	91.2	93.36

[a]Retention index on n-alkane series (C_8-C_{20}) DB-Wax column in order of elution. SD: standard deviation (*n*=3), nd: not detected.

Table 2 - Seasonal variation of the chemical composition of *Mentha viridis* L. essential oil growth in southeast of Brazil in two consecutive years (November 2011 to August 2013).

Compounds	IRa	%Area ± SD							
		----------Spring-----------		-----------Summer----------		----------Autumn----------		----------Winter----------	
		1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year
Limonene	1202	0.24±0.08	0.69±0.06	0.93±0.18	0.55±0.15	0.34±0.24	0.67±0.33	0.47±0.36	nd
1,8-Cineole	1213	0.81±0.14	1.81±0.06	1.77±0.08	1.56±0.20	0.94±0.43	1.85±0.37	0.71±0.34	0.58±0.24
γ-Terpinene	1245	0.40±0.07	0.65±0.09	0.54±0.12	0.82±0.19	0.28±0.13	0.56±0.20	0.30±0.15	0.13±0.04
3-Octanone	1255	1.01±0.19	1.50±0.11	1.13±0.28	1.42±0.18	0.79±0.33	2.38±0.57	1.04±0.45	0.39±0.10
3-Octanol	1401	6.94±0.41	7.36±0.65	9.41±1.08	6.99±0.43	9.32±0.30	6.00±0.43	3.44±0.27	4.91±0.80
trans-Sabinene hydrate	1469	8.42±0.87	9.91±0.26	11.17±0.70	9.53±0.44	11.99±0.25	10.86±0.60	11.11±0.46	11.22±1.00
3Z-Hexenyl valerate	1489	0.81±0.05	0.73±0.02	0.76±0.03	0.81±0.01	0.89±0.02	0.77±0.03	0.7±0.05	1.04±0.04
Linalool	1555	52.23±0.63	48.08±0.49	49.29±1.49	49.07±0.45	49.88±0.92	47.32±0.78	49.94±1.00	45.18±1.26
β-Caryophyllene	1589	2.27±0.31	1.66±0.24	1.49±0.49	1.74±0.33	1.54±0.11	1.89±0.29	1.75±0.16	2.25±0.46
Terpinen-4-ol	1602	4.79±0.54	3.84±0.23	3.64±0.57	4.37±0.28	3.14±0.12	3.28±0.39	3.34±0.20	4.13±0.22
p-Menth-1-en-8-ol	1697	2.19±0.12	1.26±0.02	1.45±0.21	1.25±0.07	1.51±0.11	1.21±0.05	2.09±0.09	1.49±0.14
Carvone	1724	15.51±0.25	13.86±0.37	13.20±1.25	15.06±0.93	14.86±0.72	15.78±0.47	21.28±0.92	18.81±0.81
Hydrogenated monoterpenes		0.64	1.34	1.47	1.37	0.62	1.23	0.77	0.13
Oxygenated monoterpenes		83.95	78.76	80.52	80.84	82.32	80.3	88.47	81.41
Hydrogenated sesquiterpenes		2.27	1.66	1.49	1.74	1.54	1.89	1.75	2.25
Others compoundsb		8.76	9.59	11.3	9.22	11	9.15	5.18	6.34
Total identified (%)		95.62	91.35	94.78	93.17	95.48	92.57	96.17	90.13

[a]Retention index on n-alkane series (C_8-C_{20}) DB-Wax column in order of elution. SD: standard deviation (n=3), nd: not detected. [b]Alcohol, ketone and aliphatic ester.

composition the constituent limonene was not detected during winter season in the second year.

For *M.* x *piperita* constituents, the highest and lowest value reported for menthol was during spring of the first year and winter of the second year, respectively (Table 1 and Figure 3). While for menthone the highest value was found during spring of the second year and the lowest was found during autumn of the first year. According to TSASI et al. (2017), the essential oil content in the varieties of *Ocimum basilicum* L. decreased gradually after the successive harvests. Despite this observation, the individual components like linalool and eugenol seem to follow an opposite pattern. The most expressive season variation for *M.* x *piperita* constituents was observed for menthyl acetate content. There was a decrease in menthyl acetate from the first to the second year, respectively during spring (10.96% and 5.00%), summer (18.75% and 6.27%) and autumn (27.18% and 18.97%). Conversely, menthyl acetate content increased during winter comparing the first (27.92%) to the second year (52.24%), reaching the highest content of menthyl acetate for all samples.

Depending on environmental conditions, menthone may be reduced to menthol by menthone reductase enzyme or oxidized to menthofuran, and the product of menthol acetylation is menthyl acetate (CROTEAU et al., 2005; GRULOVA et al., 2015). An inverse relationship between menthol and menthone (an increase in menthol and a decrease in menthone content) occurs only when the plant is in the juvenile stage (SANTOS et al., 2012). Such a

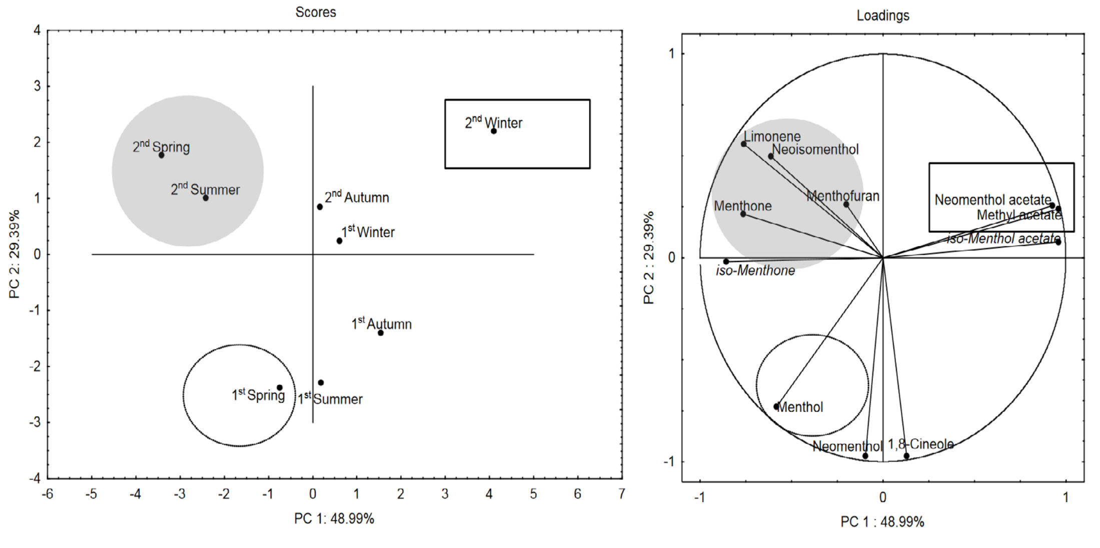

Figure 3 - Principal component analysis of the compounds of M. x piperita harvested in different seasons (spring, summer, autumn and winter).

pattern was observed in our results, since menthol content was higher in the first year (Table 1 and Figure 3). Furthermore, the synthesis of menthone reductase enzyme is most pronounced during the bloom stage (MONTEIRO et al., 2011). This was reflected in our results when species were in bloom stage during spring and summer; where menthone content was higher during this period in the second year (Table 1 and Figure 3). Additionally, the results indicate that the effect of seasonality in the EO chemical composition is also related to its influence on the development of the species. Monoterpenes accumulation in *M.* x *piperita* is closely related to plant development stages, and both yield and composition of their essential oils are propitious by the time of flowering, which occurs mainly in warmer seasons and with higher precipitation (GRULOVA et al., 2015). Additionally, the reduction of menthyl acetate during spring and summer season allows a high quality EO. High values of menthyl acetate, present in the EOs of *Mentha spp*., is believed to cause oxidation and organoleptic interference on the EOs by changing their quality and pharmacological function (SANTOS et al., 2012). The qualitative and quantitative compositions of essential oil determine the quality and commercial value. Hence, according to our results harvesting this species during spring and summer will provide a lower content in menthyl acetate and a higher content in menthol in EO.

Mentha viridis EO also shows seasonal and annual variations (Table 2 and Figure 4). The great variation was observed in linalool (45.18 - 52.23%), carvone (13.20 to 21.28%), *trans*-sabinene hydrate (8.42 - 11.99%), 3-octanol (3.44 to 9.41%) and terpinen-4-ol (3.14 - 4.79%) (Table 2). SILVA et al. (2015) reported similar contents of these volatile compounds in the essential oil from *M. viridis*. Linalool and carvone were the ones with a bigger range in values, compared to the others (Table 2). The lower values reported for linalool were during autumn and winter, both in the second year. While for carvone, its accumulation was greater during winter season, independent of the year (Figure 4).

From all major constituents, linalool is the major compound reported in *M. viridis* EO; once its content reaches 50% of the entire EO composition. So, for *M. viridis* species will be considerable to harvest leaves for EO extraction during the seasons when linalool content is higher. Hence, according to our results the best period would be during spring,

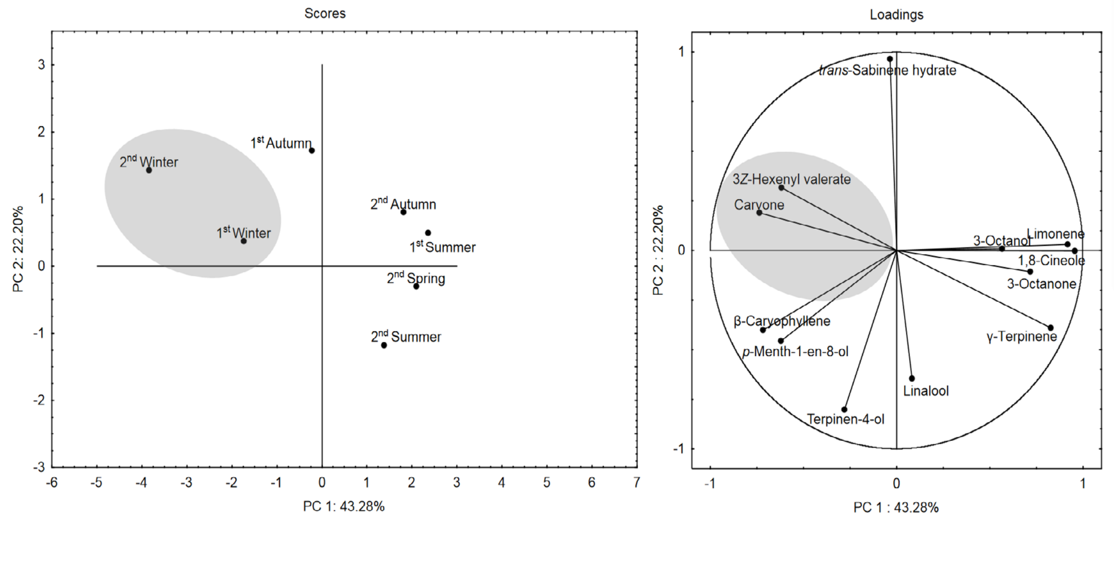

Figure 4 - Principal component analysis of the compounds of M. viridis harvested in different seasons (spring, summer, autumn and winter).

which coincides with the higher EO content achieved in this study for this species.

CONCLUSION

Seasonal variation influences the EO content and chemical composition of *Mentha* x *piperita* and *M. viridis*. The highest content was obtained for *M.* x *piperita* during spring and for *M. viridis* during spring and summer. Changes in chemical composition during the seasons were more pronounced for *M.* x *piperita* than for *M. viridis*. Oxigenated monoterpene was the most abundant class of volatile compounds in both species. The major compounds identified were menthol and menthone for *M.* x *piperita*, and linalool and carvone for *M. viridis*. Menthol and menthone contents were increased in *Mentha* x *piperita* during spring and summer. In *Mentha viridis*, lower linalool content was reported during autumn and winter, and the higher accumulation of carvone was during winter season.

ACKNOWLEDGEMENTS

The authors are grateful to Neha Babar from the University of Toronto for her contribution on English review. They acknowledge the financial support of the project, study and productivity scholarships to Fundação de Amparo à Pesquisa do Estado de Minas Gerais (FAPEMIG), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) and Coordenação de Aperfeiçoamento de Pessoal Nível Superior (CAPES).

DECLARATION OF CONFLICT OF INTEREST

The authors declare no conflict of interest. The founding sponsors had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

AUTHORS' CONTRIBUTIONS

JPA performed the experiments and wrote the manuscript. SKVB and JEBPP designed the study, supervised the laboratory work and contributed to critical reading of the manuscript. AFB and FVP performed the experiments. AAC analyzed the data. All the authors have read the final manuscript and approved the submission.

REFERENCES

ANUAR, N. A. F. M., et al. A review on natural-based active compounds delivery system and its potential in food preservative application. **IOP Conference Series: Earth and Environmental Science**, v.269, p.012010. 2019. Available from: <http://dx.doi.org/10.1088/1755-1315/269/1/012010>. Accessed: Jun. 15, 2020. doi: <https://doi.org/10.1088/1755-1315/269/1/012010>.

BARTHOLOMEW, D. J. Principal components analysis. In: P. Peterson, E. Baker, et al (Ed.). **International Encyclopedia of**

Education (Third Edition). Oxford: Elsevier, 2010. Principal Components Analysis, p.374-377.

BEHN, H., et al. Ultraviolet-B and photosynthetically Active Radiation Interactively Affect Yield and Pattern of Monoterpenes in Leaves of Peppermint (*Mentha* × *piperita* L.). **Journal of Agricultural and Food Chemistry**, v.58, n.12, p.7361-7367. 2010. Available from: <https://doi.org/10.1021/jf9046072>. Accessed: Jul. 7, 2018. doi: <https://doi.org/10.1021/jf9046072>.

CAMPOS, E. V. R., et al. Use of botanical insecticides for sustainable agriculture: Future perspectives. **Ecological Indicators**, v.105, p.483-495. 2019. Available from: <http://www.sciencedirect.com/science/article/pii/S1470160X18302917>. Accessed: Jul. 5, 2020. doi: <https://doi.org/10.1016/j.ecolind.2018.04.038>.

CROTEAU, R. B., et al. (−)-Menthol biosynthesis and molecular genetics. **Naturwissenschaften**, v.92, n.12, p.562. 2005. Available from: <https://doi.org/10.1007/s00114-005-0055-0>. Accessed: Jul. 10, 2019. doi: <https://doi.org/10.1007/s00114-005-0055-0>.

DESCHAMPS, C., et al. Avaliação sazonal do rendimento de óleo essencial em espécies de menta. **Ciência e Agrotecnologia**, v.32, p.725-730. 2008. Available from: <http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1413-70542008000300004&nrm=iso>. Accessed: Jul. 10, 2019. doi: <http://dx.doi.org/10.1590/S1413-70542008000300004>.

EHLERT, P. A. D., et al. Influência do horário de colheita sobre o rendimento e composição do óleo essencial de erva-cidreira brasileira [*Lippia alba* (Mill.) N. E. Br.]. **Revista Brasileira de Plantas Medicinais**, v.15, p.72-77. 2013. Available from: <http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1516-05722013000100010&nrm=iso>. Accessed: Jul. 10, 2018. doi: <http://dx.doi.org/10.1590/S1516-05722013000100010>.

FALCÓ, I., et al. Sanitizing food contact surfaces by the use of essential oils. **Innovative Food Science and Emerging Technologies**, v.51, p.220-228. 2019. Available from: <http://www.sciencedirect.com/science/article/pii/S1466856417313486>. Accessed: Jul. 12, 2020. <doi: https://doi.org/10.1016/j.ifset.2018.02.013>.

FERREIRA, D. F. Sisvar: a computer statistical analysis system. **Ciência e Agrotecnologia**, v.35, p.1039-1042. 2011. Available from: <http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1413-70542011000600001&nrm=iso>. Accessed: Jul. 5, 2018. doi: <https://doi.org/10.1590/S1413-70542011000600001>.

GAD, H. A., et al. Phytochemical profiling and seasonal variation of essential oils of three *Callistemon* species cultivated in Egypt. **PLOS ONE**, v.14, n.7, p.e0219571. 2019. Available from: <https://doi.org/10.1371/journal.pone.0219571>. Accessed: Jul. 12, 2020. doi: <https://doi.org/10.1371/journal.pone.0219571>.

GONÇALVES, R. S., et al. Antioxidant properties of essential oils from *Mentha* species evidenced by electrochemical methods. **Revista Brasileira de Plantas Medicinais**, v.11, p.372-382. 2009. Available from: <http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1516-05722009000400004&nrm=iso>. Accessed: Feb. 8, 2019. doi: <https://doi.org/10.1590/S1516-05722009000400004>.

GRACINDO, L., et al. Chemical characterization of mint (*Mentha* spp.) germplasm at Federal District, Brazil. **Revista Brasileira de Plantas Medicinais**, v.8, p.5-9. 2006. Available from. doi.

GRULOVA, D., et al. Seasonal variability of the main components in essential oil of *Mentha*×*piperita* L. **Journal of the Science of Food and Agriculture**, v.95, n.3, p.621-627. 2015. Available from: <https://doi.org/10.1002/jsfa.6802>. Accessed: Jul. 17, 2019. doi: <https://doi.org/10.1002/jsfa.6802>.

HUSSAIN, A. I., et al. Seasonal variation in content, chemical composition and antimicrobial and cytotoxic activities of essential oils from four *Mentha* species. **Journal of the Science of Food and Agriculture**, v.90, n.11, p.1827-1836. 2010. Available from: <https://doi.org/10.1002/jsfa.4021>. Accessed: Jan. 10, 2019. doi: <https://doi.org/10.1002/jsfa.4021>.

JÚNIOR, A. D. S., et al. Application of the Köppen classification for climatic zoning in the state of Minas Gerais, Brazil. **Theoretical and Applied Climatology**, v.108, n.1, p.1-7. 2012. Available from: <https://doi.org/10.1007/s00704-011-0507-8>. Accessed: Jul. 9, 2018. doi: <https://doi.org/10.1007/s00704-011-0507-8>.

KIZIL, S., et al. Mineral content, essential oil components and biological activity of two mentha species (*M. piperita* L., *M. spicata* L.). **Turkish Journal of Field Crops**, v.15, n.2, p.148-153. 2010. Available from: Jul. 5, 2018.

KUMAR, P., et al. Insecticidal properties of *Mentha* species: A review. **Industrial Crops and Products**, v.34, n.1, p.802-817. 2011. Available from: <http://www.sciencedirect.com/science/article/pii/S0926669011000604>. Accessed: Jan. 7, 2018. doi: <https://doi.org/10.1016/j.indcrop.2011.02.019>.

LEMOS, M. F., et al. Seasonal variation affects the composition and antibacterial and antioxidant activities of *Thymus vulgaris*. **Industrial Crops and Products**, v.95, p.543-548. 2017. Available from: <http://www.sciencedirect.com/science/article/pii/S092666901630752X>. Accessed: Jan. 12, 2018. doi: <https://doi.org/10.1016/j.indcrop.2016.11.008>.

MKADDEM, M., et al. Chemical Composition and Antimicrobial and Antioxidant Activities of *Mentha* (*longifolia*L. and*viridis*) Essential Oils. **Journal of Food Science**, v.74, n.7, p.M358-M363. 2009. Available from: <https://onlinelibrary.wiley.com/doi/abs/10.1111/j.1750-3841.2009.01272.x>. Accessed: Jul. 10, 2019. doi: <https://doi.org/10.1111/j.1750-3841.2009.01272.x>.

MONTEIRO, R., et al. Desenvolvimento vegetativo de *Mentha campestris* Schur e produção de mentol em diferentes espaçamentos de plantio e épocas de colheita. **Revista Brasileira de Plantas Medicinais**, v.13, p.401-407. 2011. Available from: <http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1516-05722011000400005&nrm=iso>. Accessed: Jul. 10, 2019. doi: <http://dx.doi.org/10.1590/S1516-05722011000400005>.

NIKŠIĆ, H., et al. Seasonal variation in content and chemical composition of essential oils from leaves of *Mentha longifolia* Huds.(Lamiaceae). **Bulletin of the Chemists and Technologists of Bosnia and Herzegovina**, v.43, p.29-34. 2014. Available from: Jul. 10, 2018. doi.

NORIEGA, P. Terpenes in Essential Oils: Bioactivity and Applications. In: S. Perveen (Ed.). **Terpenes and Terpenoids**. London: IntechOpen, v.8, 2020. Terpenes in Essential Oils: Bioactivity and Applications, p.32.

OLIVEIRA, T. D., et al. The effect of alternative membrane system, sucrose, and culture methods under photosynthetic photon flux on growth and volatile compounds of mint *in vitro*. **In Vitro Cellular**

& Developmental Biology - Plant. 2021. Available from: <https://doi.org/10.1007/s11627-020-10147-z>. Accessed. doi: <https://doi.org/10.1007/s11627-020-10147-z>.

PAOLINI, J., et al. Chemical composition, intraspecies variation and seasonal variation in essential oils of *Calendula arvensis* L. **Biochemical Systematics and Ecology**, v.38, n.5, p.865-874. 2010. Available from: <http://www.sciencedirect.com/science/article/pii/S0305197810001389>. Accessed: Jul. 15, 2018. doi: <https://doi.org/10.1016/j.bse.2010.07.009>.

POOVAIAH, C. R., et al. Adventitious shoot regeneration of scotch spearmint (*mentha* x *gracilis* Sole). **In Vitro Cellular & Developmental Biology - Plant**, v.42, n.4, p.354-358. 2006. Available from: <https://doi.org/10.1079/IVP2006783>. Accessed: Jul. 9, 2019. doi: <https://doi.org/10.1079/ivp2006783>.

RAMOS, R. D. S., et al. Chemical Composition and *In Vitro* Antioxidant, Cytotoxic, Antimicrobial, and Larvicidal Activities of the Essential Oil of *Mentha piperita* L. (Lamiaceae). **The Scientific World Journal**, v.2017, p.4927214. 2017. Available from: <https://doi.org/10.1155/2017/4927214>. Accessed: Jul. 10, 2018. doi: 10.1155/2017/4927214.

REHMAN, R., et al. Biosynthesis of essential oils in aromatic plants: A review. **Food Reviews International**, v.32, n.2, p.117-160. 2016. Available from: <https://doi.org/10.1080/87559129.2015.1057841>. Accessed: Mar. 17, 2019. doi: <https://doi.org/10.1080/87559129.2015.1057841>.

SANTOS, V. M. C. S., et al. Seasonal variation of vegetative growth, essential oil yield and composition of menthol mint genotypes at southern Brazil. **Bioscience Journal**, v.28, n.5. 2012. Available from: <http://www.seer.ufu.br/index.php/biosciencejournal/article/view/13942>. Accessed: May. 16, 2019.

SILVA, D. M. D., et al. Evaluation of vegetative growth, chemical composition, and antioxidant capacity of essential oil of peppermint under water regimes. **Journal of Agricultural Science**, v.11, n.4. 2019. Available from. doi: <https://doi.org/10.5539/jas.v11n4p197>.

SILVA, L. F., et al. Chemical characterization, antibacterial and antioxidant activities of essential oils of *Mentha viridis* L. and *Mentha pulegium* L.(L). **American Journal of Plant Sciences**, v.6, n.05, p.666. 2015. Available from: <https://doi.org/10.4236/ajps.2015.65072>. Accessed: Jul. 10, 2018. doi: <https://doi.org/10.4236/ajps.2015.65072>.

SINGH, B.; R. A. SHARMA. Plant terpenes: defense responses, phylogenetic analysis, regulation and clinical applications. **3 Biotech**, v.5, n.2, p.129-151. 2015. Available from: <https://doi.org/10.1007/s13205-014-0220-2>. Accessed. doi: 10.1007/s13205-014-0220-2.

TSASI, G., et al. The Effect of Harvesting on the Composition of Essential Oils from Five Varieties of *Ocimum basilicum* L. Cultivated in the Island of Kefalonia, Greece. **Plants** v.6, n.3, p.41. 2017. Available from: <https://pubmed.ncbi.nlm.nih.gov/28927018>. Accessed: Jun. 5, 2018. <doi: https://doi.org/10.3390/plants6030041>.

VAN DEN DOOL, H.; P. DEC. KRATZ. A generalization of the retention index system including linear temperature programmed gas—liquid partition chromatography. **Journal of Chromatography A**, v.11, p.463-471. 1963. Available from: <http://www.sciencedirect.com/science/article/pii/S002196730180947X>. Accessed: May. 10, 2018. doi: <https://doi.org/10.1016/S0021-9673(01)80947-X>.

VERMA, R. S., et al. Essential oil composition of *Aegle marmelos* (L.) Correa: chemotypic and seasonal variations. **Journal of the Science of Food and Agriculture**, v.94, n.9, p.1904-1913. 2014. Available from: <https://doi.org/10.1002/jsfa.6510>. Accessed: Jul. 18, 2018. doi: <https://doi.org/10.1002/jsfa.6510>.

ZOUARI-BOUASSIDA, K., et al. Seasonal Variation in Essential Oils Composition and the Biological and Pharmaceutical Protective Effects of *Mentha longifolia* Leaves Grown in Tunisia. **BioMed Research International** v.2018, p.7856517. 2018. Available from: <https://doi.org/10.1155/2018/7856517>. Accessed: Jun. 8, 2019. doi: <https://doi.org/10.1155/2018/7856517>.