

## Essential Oil Composition of *Tilia platyphyllos* Scop. Collected from Different Regions of Kosovo

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**Abstract:** The aim of this study was to assess variation of the chemical composition of essential oils obtained from inflorescences and leaves of *Tilia platyphyllos* Scop. collected from five different localities in Kosovo. GC-MS/GC-FID analysis revealed the presence of 96 compounds in essential oils obtained from inflorescences of large-leaved lime with the most prominent classes including hydrocarbons, with the respective concentrations in flowers and leaves of 41.7% - 46.2% and 27.5% - 36.8%, respectively, oxygenated sesquiterpenes (12.2%-23.0% and 21.3% -31.2%, in flowers and leaves, respectively) and fatty acids and their derivatives (8.0% - 19.6% and 18.1% - 23.1% in flowers and leaves, respectively) whereas monoterpenes were present in smaller amount. Experimental data showed that large-leaved lime is reach in different groups of volatile compounds, and this species could be further recommended for food and medicinal application.

**Keywords:** Large-leaved lime; essential oil; principal component analysis, © 2020 ACG Publications. All rights reserved.

### 1. Plant Source

Plant material (inflorescences) of the large-leaved lime (*Tilia platyphyllos*, Scop. Malvaceae), were collected from five different localities in Kosovo (Pejë, Gjiilan, Lipjan, Klinë and Suharekë regions) during the period June-July 2016. The plant was identified by Professor asoc. Avni Hajdari, Department of Biology, University of Prishtina. A voucher specimen (Herbarium Number: LEB/2016/1) has been deposited at the Department of Biology University of Prishtina, Kosovo.

### 2. Previous Studies

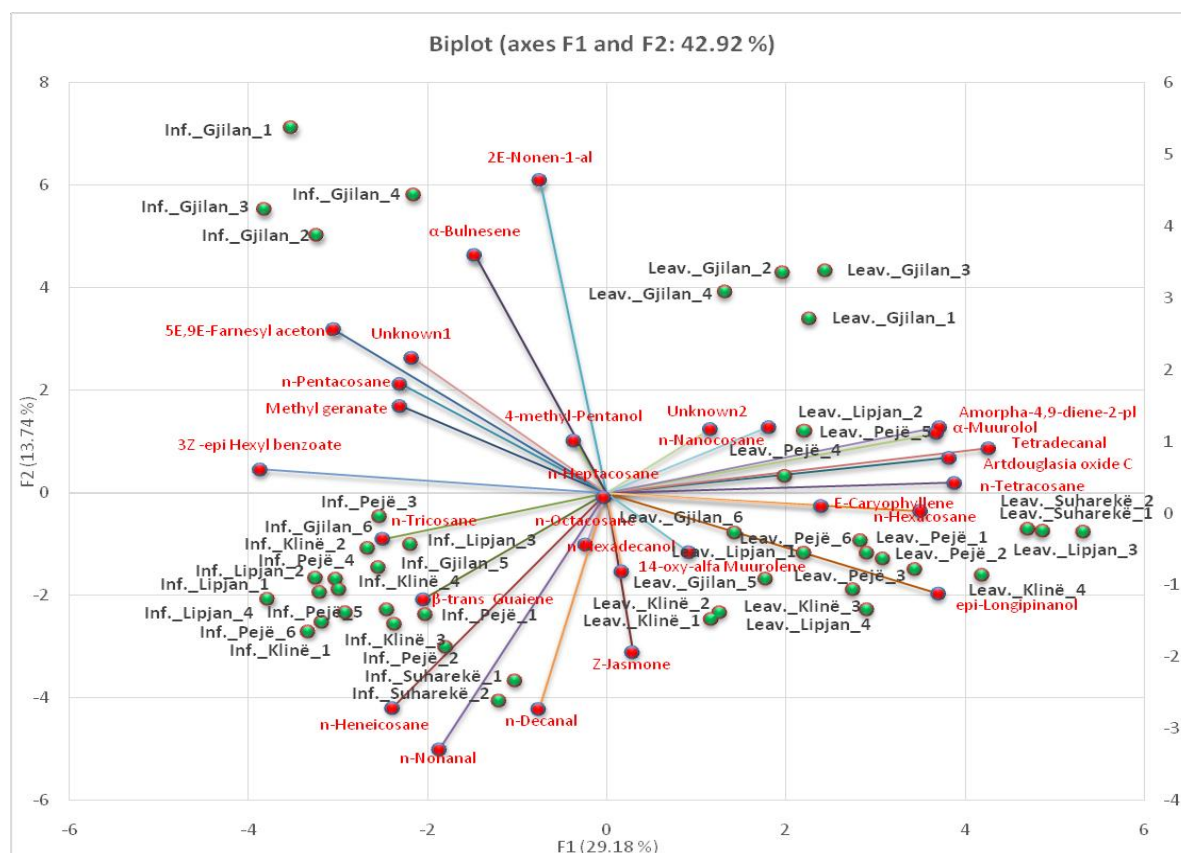
*Tilia platyphyllos* Scop., Malvaceae, known as large-leaved lime or linden, is large-sized deciduous tree, native through Europe. This large-leaved tree prefers warmer climates and can reach 30-40m in height, with a trunk up to around 1m in diameter [1]. Asymmetrical, heart shaped leaves are

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about 9cm long and have pointed tips with upper surface of dark green colour and paler underside. The cream to yellow flowers, are fragrant, have five petals and hang in tiny clusters of 4 to 10 [2]. Large-leaved lime has industrial cosmetics and medicinal importance [3, 4]. In Kosovo large leaved lime was traditionally used for different purposes, including tea preparation (recreational use/ panacea), treatment of sore throat and lung inflammations, abdominal pain and respiratory problems (cough, fever) [5-11]. It has been shown that the principal bioactive compounds of *Tilia platyphyllos* Scop. are mucilaginous substances, flavonoids and essential oils. [4, 11-13]. In a comparative metabolomics study of *Tilia platyphyllos* Scop. bracts totally 504 compounds were detected, including flavonoid glycosides, catechins, procyanidins, quinic acid derivatives and coumarins [14]. Chemical composition of essential oils obtained from linden inflorescences was studied in samples obtained from different region [15-19].

**GC-FID and GC-MS analyses:** GC analyses were performed using of an Agilent 7890A gas chromatography system equipped with flame ionization (FID) detector (Agilent Technologies) using HP-5MS column 30 m x 0.25 mm with 0.25  $\mu$ m film thickness. Helium was used as the carrier gas with an initial flow rate of 0.6 mL/min and subsequently at a constant pressure of 16.6 psi. GC/MS analyses were carried out using an Agilent 7800A GC System coupled with a 575C MSD. The ionisation energy was 70 eV with a mass range of 40-400 m/z. The separation was performed with the same column and temperature program the same as for the analytical GC. Identification of the essential oils components was completed by comparing their Kovats retention indices with those reported in literature [20]. The components were also identified by comparing the mass spectra of each constituent with those stored in the MS library search (NIST 08.L and WILEY MS 9th) and with mass spectra from the literature [20].

**Principal component analysis (PCA)** was used to evaluate whether the identified essential oil components can be useful for reflecting the chemotaxonomy of *Tilia platyphyllos*. Constituents with concentrations higher than 3% (bold in Table 1) of were subjected to statistical analysis.



**Figure 1.** Graph obtained from the Principal Component Analysis of essential oils obtained from inflorescences of *Tilia platyphyllos* from 5 different localities. Inf. - Inflorescences; Leav. - leaves

**Table 1.** The chemical composition of the essential oils obtained from the inflorescences (IF) and leaves (L) of *Tilia platyphyllos*

KI <sup>a</sup>	Compound <sup>b</sup>	Pejë		Gjilan		Lipjan		Klinë		Suharekë	
		IF	L	IF	L	IF	L	IF	L	IF	L
<b>838</b>	<b>4-methylpentanol</b>	-	-	<b>1.42</b>	<b>1.00</b>	<b>1.63</b>	<b>2.50</b>	<b>1.02</b>	<b>0.65</b>	-	<b>0.20</b>
846	<i>E</i> -2-Hexenal	1.15	0.64	0.83	0.47	0.98	0.92	0.74	0.75	0.58	0.37
862	<i>E</i> -2-Hexenol	-	0.05	0.44	0.29	-	0.07	0.03	0.20	0.85	0.46
900	<i>n</i> -Nonane	0.16	0.03	0.19	0.04	0.26	0.14	0.21	0.37	0.17	0.14
902	Heptanal	1.07	0.28	0.80	0.28	0.72	0.51	1.47	0.73	0.84	0.42
960	Benzaldehyde	0.19	0.20	0.06	0.24	0.13	0.21	0.14	0.30	-	0.25
973	Hexanoic acid	0.38	0.24	0.40	0.21	0.49	0.26	0.39	0.36	0.71	0.26
987	<i>E</i> -3-Hexenoic acid	0.18	0.65	0.25	0.96	0.32	1.75	0.41	1.01	0.38	1.41
989	1-Decene	0.41	0.52	0.54	0.64	0.51	1.43	0.66	1.51	0.72	2.36
988	2-Pentylfuran	0.62	0.39	0.50	0.25	0.51	0.29	0.43	0.57	0.48	0.58
1000	<i>n</i> -Decane	0.16	0.16	0.11	0.15	0.24	0.22	0.21	0.39	-	0.20
1009	Hexyl acetate	0.77	0.56	0.52	0.56	0.63	0.69	0.50	0.70	0.42	0.64
1035	<i>E</i> -3-Octen-2-one	-	-	0.46	0.43	0.36	0.52	0.35	0.70	0.20	0.35
1042	Benzeneacetaldehyde	1.80	0.25	1.23	0.37	1.22	0.58	1.68	0.80	1.28	0.56
1068	<i>n</i> -octanol	0.65	0.81	0.91	1.26	0.68	1.41	0.69	1.21	0.49	1.00
1097	2-Nonanol	0.34	0.87	0.40	0.66	0.50	1.90	0.41	0.72	-	0.33
1097	Linalool	1.09	1.35	0.50	0.84	1.40	0.96	1.11	1.71	0.95	2.54
<b>1100</b>	<b><i>n</i>-Nonanal</b>	<b>8.97</b>	<b>4.10</b>	<b>3.30</b>	<b>2.76</b>	<b>8.27</b>	<b>6.15</b>	<b>8.35</b>	<b>6.96</b>	<b>5.40</b>	<b>2.78</b>
<b>1161</b>	<b>2<i>E</i>-Nonen-1-ol</b>	<b>0.62</b>	<b>0.23</b>	<b>4.19</b>	<b>2.22</b>	<b>0.21</b>	<b>0.36</b>	<b>0.68</b>	<b>0.36</b>	-	<b>0.38</b>
1166	Coahuilensol	0.32	0.15	0.30	0.21	0.18	0.27	0.11	0.24	-	0.29
1169	Borneol	0.32	0.11	0.42	0.14	0.55	0.23	0.16	0.05	0.30	0.21
1175	<i>cis</i> -Pinocamphone	0.22	0.05	0.11	0.02	0.16	0.09	0.07	0.12	0.19	0.24
1192	2-Decanone	0.68	0.26	0.71	0.31	0.52	0.24	0.40	0.41	0.42	0.33
1191	Methyl salicylate	0.72	0.23	0.62	0.24	0.54	0.22	0.33	0.34	0.27	0.20
1196	Safranal	-	0.28	-	0.48	-	0.45	0.08	0.44	-	0.74
<b>1201</b>	<b><i>n</i>-Decanal</b>	<b>0.81</b>	<b>0.48</b>	<b>0.44</b>	<b>0.53</b>	<b>0.68</b>	<b>0.65</b>	<b>0.97</b>	<b>0.90</b>	<b>3.00</b>	-
1219	$\beta$ -Cyclocitral	0.46	0.28	0.49	0.28	0.23	0.37	0.26	0.25	-	0.35
1235	3 <i>Z</i> -Hexenyl 3-methylbutanoate	0.20	0.13	0.72	0.23	0.63	0.23	0.26	0.27	-	0.32
1258	Carvenone	1.61	0.05	1.40	0.33	1.38	0.28	0.72	0.39	0.81	0.36
1270	Nonanoic acid	0.52	1.09	0.52	0.39	0.54	0.45	0.46	0.61	0.61	0.78
1305	Undecanal	0.66	0.38	0.73	0.89	0.78	0.94	0.50	0.57	-	0.25
1311	2-Adamantanone	-	0.17	0.37	0.06	0.07	0.13	-	-	-	0.22
<b>1324</b>	<b>Methyl geranate</b>	<b>2.48</b>	<b>1.21</b>	<b>2.38</b>	<b>1.70</b>	<b>1.71</b>	<b>1.10</b>	<b>1.55</b>	<b>1.50</b>	<b>1.58</b>	<b>0.89</b>
1329	<i>Z</i> -Hasmigone	0.27	0.84	0.15	1.08	0.22	1.36	0.42	0.91	0.32	1.19
1349	$\alpha$ -Terpinyl acetate	-	0.08	0.17	0.26	0.03	0.10	0.18	0.79	0.61	1.08
1359	Eugenol	0.23	0.60	0.17	0.09	0.18	-	0.18	0.15	-	0.31
1366	<i>n</i> -decanoic acid	0.24	-	0.19	-	0.16	-	0.14	0.11	0.42	0.26
1380	<i>Z</i> -3-Hexenyl hexanoate	0.39	0.17	0.21	0.09	0.24	0.11	0.27	0.54	0.43	0.79
1384	<i>E</i> - $\beta$ -damascenone	-	0.18	0.15	0.21	-	0.22	0.05	0.18	-	0.33
1391	<i>E</i> -Jasmone	0.18	0.49	0.30	0.56	0.08	0.11	0.05	0.14	-	0.15
<b>1392</b>	<b><i>Z</i>-Jasmone</b>	<b>3.06</b>	<b>1.60</b>	<b>0.65</b>	<b>0.94</b>	<b>2.29</b>	<b>1.81</b>	<b>2.11</b>	<b>1.07</b>	<b>0.77</b>	<b>1.81</b>
1402	$\alpha$ -Funebrene	0.42	0.07	0.57	0.09	0.12	0.16	0.27	1.27	-	0.26

Table 1 continued..

1403	Methyl eugenol	0.25	0.66	0.02	0.01	0.04	0.35	0.12	0.11	-	0.47
1419	<i>E</i> -Caryophyllene	0.18	0.78	0.14	0.74	-	1.11	0.09	0.72	1.07	4.20
1422	$\beta$ -Duprezianene	0.51	0.34	1.08	0.35	0.43	0.35	0.42	0.54	0.42	0.25
1430	<i>E</i> - $\alpha$ -Ionone	0.32	0.09	0.15	0.15	0.38	0.06	0.25	0.32	-	0.21
1441	Aromadendrene	0.15	0.02	0.06	0.32	0.13	0.53	-	0.07	-	-
1446	Seychellene	-	0.41	-	0.35	-	1.47	0.09	0.50	-	1.04
1454	$\alpha$ -Humulene	-	0.98	0.34	0.94	0.71	0.08	0.44	0.11	0.35	0.22
1466	2 <i>E</i> -Dodecenal	0.26	0.15	0.08	0.03	0.13	0.10	0.27	0.38	-	-
1476	$\alpha$ -Neocallitropsene	1.11	0.66	0.34	0.51	0.63	0.20	0.71	0.32	0.88	0.91
1488	<i>E</i> - $\beta$ -Ionone	-	0.15	0.07	0.35	0.04	0.24	0.03	0.10	-	0.49
1493	$\beta$ -etispirene	0.18	1.30	-	1.37	-	1.66	0.20	0.78	0.39	0.86
1500	Isodaucene	-	0.25	0.19	0.23	-	0.32	0.05	0.20	-	0.42
<b>1502</b>	<b><i>trans</i>- <math>\beta</math>-Guaiene</b>	<b>1.30</b>	<b>0.25</b>	<b>1.71</b>	<b>0.70</b>	<b>2.72</b>	<b>0.48</b>	<b>2.64</b>	<b>0.78</b>	-	<b>1.27</b>
<b>1509</b>	<b><math>\alpha</math>-Bulnesene</b>	-	-	<b>4.64</b>	<b>0.38</b>	-	-	-	-	-	-
1512	$\delta$ -Z-Macrocarpene	0.31	0.33	0.35	0.24	0.54	0.56	0.39	0.51	0.33	0.27
1514	$\alpha$ -Cadinene	-	0.39	-	0.42	-	0.45	0.05	0.17	-	0.88
1520	1-endo-Bourbonanol	-	0.37	0.01	0.31	-	0.35	0.07	0.26	0.31	0.52
<b>1523</b>	<b>Artedouglasia oxide C</b>	<b>0.44</b>	<b>1.62</b>	<b>0.56</b>	<b>1.27</b>	<b>0.35</b>	<b>1.50</b>	<b>0.62</b>	<b>1.55</b>	<b>0.37</b>	<b>2.59</b>
1536	Liguloxide	-	1.49	0.07	1.00	-	1.82	0.11	0.54	-	0.90
1545	$\alpha$ -Calacorene	0.16	0.77	0.19	2.16	-	1.63	0.24	0.80	0.26	0.43
<b>1563</b>	<b><i>epi</i>-Longipinanole</b>	<b>1.26</b>	<b>4.05</b>	<b>0.82</b>	<b>2.08</b>	<b>1.74</b>	<b>6.96</b>	<b>2.33</b>	<b>5.42</b>	<b>2.02</b>	<b>7.82</b>
<b>1567</b>	<b>3<i>Z</i> -<i>epi</i> Hexyl benzoate</b>	<b>2.49</b>	<b>0.70</b>	<b>2.84</b>	<b>0.55</b>	<b>3.59</b>	<b>0.48</b>	<b>1.15</b>	<b>0.81</b>	<b>2.28</b>	<b>0.46</b>
1580	<i>n</i> -Hexyl benzoate	-	0.16	-	0.08	-	0.28	0.07	0.37	-	0.14
1587	2 <i>E</i> -Hexenyl benzoate	-	0.35	-	0.31	-	0.09	0.34	0.50	0.32	0.69
	5-oxy-Isobornyl										
1603	isobutanoate	-	0.33	-	0.10	-	0.08	0.05	0.26	0.78	0.48
<b>1612</b>	<b>Tetradecanal</b>	<b>0.45</b>	<b>3.34</b>	<b>0.60</b>	<b>3.72</b>	<b>0.58</b>	<b>4.12</b>	<b>1.34</b>	<b>3.63</b>	<b>0.70</b>	<b>4.67</b>
<b>1646</b>	<b><math>\alpha</math>-Muurolol</b>	<b>0.18</b>	<b>2.58</b>	<b>0.12</b>	<b>1.87</b>	-	<b>2.15</b>	<b>0.52</b>	<b>1.14</b>	<b>0.23</b>	<b>0.92</b>
1654	$\alpha$ -Cadinol	-	0.20	0.24	0.19	0.30	0.13	0.48	0.21	0.26	-
1668	6 <i>Z</i> -Coniferyl alcohol	0.36	0.45	0.29	0.58	0.13	0.41	0.19	0.58	0.35	0.59
1673	5 iso-Cedranol	-	0.56	0.14	0.82	-	0.37	-	-	-	-
1676	Cadalene	-	0.36	-	0.17	-	0.11	0.27	0.07	-	0.26
1700	<i>n</i> -Heptadecane	0.25	0.53	0.09	0.49	0.13	0.90	0.60	1.12	0.71	2.36
<b>1700</b>	<b>Amorpha-4,9-dien-2-ol</b>	<b>0.76</b>	<b>2.76</b>	<b>0.66</b>	<b>2.95</b>	<b>0.88</b>	<b>3.94</b>	<b>0.95</b>	<b>2.23</b>	-	<b>3.49</b>
1713	Cedroxyde	-	0.39	0.02	0.65	-	0.56	0.05	0.22	-	0.40
	<i>E</i> -Sesquilandulyl										
1740	acetate	0.08	0.39	-	0.19	-	0.21	0.04	0.10	0.15	0.27
1768	Cedryl acetate	-	1.01	0.18	1.10	0.48	0.81	0.38	0.53	-	0.95
<b>1768</b>	<b>14-oxy-<math>\alpha</math> Muurolene</b>	<b>1.76</b>	<b>1.09</b>	<b>1.34</b>	<b>2.15</b>	<b>0.86</b>	<b>2.50</b>	<b>1.26</b>	<b>1.11</b>	<b>3.35</b>	<b>1.01</b>
1788	8-Cedren-13-ol, acetate	0.87	0.44	1.12	1.58	0.65	0.72	1.61	0.84	0.47	0.22
	Isopropyl										
1829	tetradecanoate	0.61	0.64	0.73	0.60	0.97	1.15	0.96	1.66	0.64	0.41
	2,7(14),10-										
1845	isabolatriene-1-ol-4-one	0.26	1.64	0.04	0.82	-	0.53	0.04	0.36	0.47	0.70
1847	Phenylethyloctanoate	0.21	0.39	0.21	0.27	0.39	0.27	0.35	0.87	0.67	0.32

Table 1 continued..

1875	<i>n</i> -Hexadecanol	1.32	2.18	1.37	0.81	0.49	0.62	0.90	1.70	1.11	0.66
1900	<i>n</i> -Nanodecane	2.79	1.76	1.22	1.45	2.28	1.01	1.41	0.56	0.26	0.23
1913	5 <i>E</i> ,9 <i>E</i> -Farnesyl acetone	2.42	1.47	3.79	1.07	3.45	0.55	2.46	0.74	11.98	0.56
<b>2100</b>	<b><i>n</i>-Heneicosane</b>	<b>13.50</b>	<b>4.07</b>	<b>3.59</b>	<b>2.10</b>	<b>12.38</b>	<b>2.16</b>	<b>4.92</b>	<b>5.94</b>	<b>9.88</b>	<b>3.91</b>
2300	<i>n</i> -Tricosane	7.25	3.16	5.48	1.73	5.92	0.46	21.76	2.89	5.20	0.93
2400	<i>n</i> -Tetracosane	0.85	1.94	1.10	2.37	1.23	2.75	1.16	2.83	2.84	5.16
<b>2500</b>	<b><i>n</i>-Pentacosane</b>	<b>13.90</b>	<b>10.88</b>	<b>19.61</b>	<b>13.58</b>	<b>14.65</b>	<b>8.91</b>	<b>10.75</b>	<b>9.85</b>	<b>1.06</b>	<b>0.49</b>
2600	<i>n</i> -Hexacosane	0.42	3.71	1.65	2.64	0.41	3.56	0.74	3.62	2.17	6.84
2700	<i>n</i> -Heptacosane	1.14	2.40	1.48	4.21	1.70	0.96	1.00	1.10	2.72	1.81
2725	Unidentified 1	5.87	5.67	5.49	4.96	5.88	2.70	3.48	3.63	-	4.83
2745	Unidentified 2	0.24	1.51	0.35	2.00	0.28	0.92	0.18	0.32	1.33	0.49
2800	<i>n</i> -Octacosane	1.74	2.74	2.96	2.26	1.36	1.55	0.98	1.38	6.47	1.77
<b>2900</b>	<b><i>n</i>-Nanocosane</b>	<b>1.49</b>	<b>4.91</b>	<b>3.66</b>	<b>4.96</b>	<b>2.80</b>	<b>3.43</b>	<b>1.54</b>	<b>3.37</b>	<b>13.98</b>	<b>3.69</b>
<b>Total (%)</b>		<b>99.9</b>	<b>100</b>	<b>100.0</b>	<b>100</b>	<b>99.9</b>	<b>100</b>	<b>100</b>	<b>99.9</b>	<b>99.9</b>	<b>100</b>

<sup>a</sup>Kovats indices calculated against a C9- C28n-alkanes mixture on the HP5 MS column. <sup>b</sup>Compounds are listed in order of elution from a HP-5MS column. The percentage for each population represents the mean values of n calculated samples (n=3-5 samples). Boldface marked compounds (with concentrations higher than 3%) were chosen for PCA statistical analyses. ” –“ < 0.1%. inflorescences (IF) and leaves (L)

In this study, ninety-six compounds were separated (Table 1) from the essential oils obtained from inflorescences of *Tilia platyphyllos*, from which ninety-four were identified, while two compounds were not identified. (Supporting information, Figures S1 and S2). The most abundant classes of compounds were hydrocarbons with the respective concentrations in inflorescences and leaves of: 41.68 - 46.18% and 27.48 - 36.81%; followed by oxygenated sesquiterpenes (12.19 - 23.05% and 21.29 - 31.19% in inflorescences and leaves, respectively), fatty acids and their derivatives (8.03 - 19.61% and 18.05 - 23.1% in inflorescences and leaves, respectively); sesquiterpenes (3.44 - 9.42% and 6.04 - 10.84% in inflorescences and leaves, respectively) and oxygenated monoterpenes (4.23% - 6.18 % and 3.88 - 7.22% in inflorescences and leaves, respectively) (Table 1). The results of our study were in accordance with studies performed elsewhere [16, 17, 19]. In this work the total phenolic and flavonoid contents and antioxidant activity of methanol extract of *Tilia platyphyllos* was studied as well (Supporting information, Table 1 and 2).

The two-dimensional axis system of the PCA (Figure 1) shows the presence of two principal groups of compounds clustered based on the plant organs from which volatile compounds have been extracted, while the origin of plant material played a smaller role in the variability of the chemical composition of essential oils.

## Supporting Information

Supporting Information accompanies this paper on <http://www.acgpubs.org/journal/records-of-natural-products>

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