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Influence of urban environment on chemical composition of *Tilia cordata* essential oil

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The composition of essential oils of *Tilia cordata* blossom collected from ecologically clean localities and urban environment (Vilnius city) was analyzed by GC-MS. The main differences in the essential oils were revealed by the analysis of oxygenated compounds (monoterpenes, sesquiterpenes, aliphatics and others). The essential oils of linden blossom from the clean localities contained 47.3–55.0% of oxygenated constituents, while the blossom oils from trees growing on the tile-covered pavements contained only 20.6–27.7% of these compounds. The major constituents determined in linden blossom oils were 3-p-menthene, nonanal, nonanoic acid, α-cadinene, hexahydrofarnesyl acetone, kaurene, aliphatic hydrocarbons ($C_{20}H_{42}$ – $C_{24}H_{50}$). The identified compounds (169 out of 250) made up 73.4–97.7% of the oils.

Key words: *Tilia cordata* Mill., Tiliaceae, composition of essential oils, urban environment

INTRODUCTION

Linden (Tiliaceae) genus comprises more than 60 species spread in temperate and subtropical climatic zones of the Northern hemisphere. *Tilia cordata* Mill. is the solely self-growing species in Lithuania, and there are more than 30 species are introduced [1, 2].

T. cordata mostly is cultivated in city parks, roadsides and homesteads. It is also suitable for hedges and alleys. The blossom is valuable for apiculture and pharmacology. Its wood can be used for domestic needs. About 80% of these lindens in the Lithuanian towns are grown on green grass and tile-covered pavements. These lindens are the most resistant to biotic and abiotic factors in an urban environment. However, due to the anthropogenic activity and meteorological conditions, lindens are affected by various, mostly fungal, pathogenics [3]. An early (starting already in June) leaf necrosis is observed owing to an extremely small vital area, compressed soil, bad ventilation of roots, lack of microelements and salt sprinkle in winter. Lindens do not come into bloom at all or just formed bracts fall down. If lindens have wounded roots that are not able to supply enough water and mineral substances, the branches in the second and third rows usually are blasted by frost or dried off by drought. Sometimes healthy leaves or even nectarous blossom can still form on healthier branches of unhealthy trees. About 60% of lindens growing in Vilnius streets are mechanically wounded. Vermin and fungous diseases affect the leaves and sometimes the blossom [4]. There is no data concerning the essential oils from linden growing in different environments in Lithuania.

More than 100 compounds were identified in the essential oils from linden blossom growing in Poland [5]. Among the main compounds there were aliphatic hydrocarbons, aliphatic acids, linalool, nonanal, hexahydrofarnesyl acetone, damascenone, p-cymenene, germacrene D and farnesyl acetone. Linden ether and cis-rose oxide were also found in an extract obtained from the blossom of *T. cordata* growing in Germany [6]. The main compounds of the dried blossom oils of *T. cordata* obtained by headspace analysis [7] were p-cymene, fenchone, α- and β-thujones, camphor, anethole and menthone, while those of the living blossom were limonene, p-cymene, δ-3-carene, germacrene D, α-phellan-

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drene, farnesol and δ-cadinene [8]. The extracts prepared from the nectar and from ripe honey of linden blossom contained hydrocarbons, ethers, aldehydes, acids and aromatic compounds [9]. Terpenoid compounds germacrene D, dehydroaromadendrene, β-cubebene and longicyclene were detected in *Tilia americana* roots by headspace SPME/gas chromatography/mass spectrometry [10].

The aim of this work is to determine the differences among the compositions of the essential oils from linden trees growing in ecologically clean localities and urban environment.

EXPERIMENTAL

The blossoms were collected from linden (*T cordata*) growing on green grass and tile-covered pavements in July 3–12, 2006: A – Kaišiadorys district (green grass, blight), B – Vingis park in Vilnius (green grass, luxuriant foliage), C – Žirmūnų street in Vilnius (green grass, blight, vermin, fungous diseases, salt sprinkle in winter), D – Čiurlionio street in Vilnius (vermin, tile-covered pavement, salt sprinkle in winter), E – Antakalnio street in Vilnius (tile-covered pavement, blight, leaf-beetle, fungous diseases, salt sprinkle in winter), F – Vrublevskio street in Vilnius (tile-covered pavement, blight, leaf-beetle, fungous diseases, salt sprinkle in winter). Fungal diseases were identified according to [11, 12, 13, 14].

The essential oils were prepared by hydrodistillation (2 h) of 40–50 g of air-dried blossom. The ratio of the blossom and water was 1:10. The essential oils were collected in 2 ml of hexane–diethyl ether mixture (1:1). The analysis of the essential oils was carried out by GC and GC/MS. The separation was performed on a DB-5 silica capillary column (50 m × 0.32 mm, film thickness 0.25 µm) using an HP 5890II chromatograph equipped with FID. The GC oven temperature was programmed as follows: from 60 °C (isothermal for 1 min) increased to 160 °C at a rate of 5 °C/min and to 250 °C at a rate of 10 °C/min. The final temperature was kept for 10 min. The temperatures of the injector and detector were 250 °C. The flow rate of a carrier gas (helium) was 1 ml/min. The analysis by GC/MS was carried out with an HP 5890 gas chromatograph equipped with an HP 5971 mass selective detector and an HP 7673 split / splitless injector. Mass spectra in the electron mode were generated at 70 eV. The percentage composition of the essential oils was computed from GC peak areas without correction factors. The qualitative analysis was based on the comparison of the retention indexes and the mass spectra with corresponding data in the literature [15] and computer mass spectra libraries (Wiley and NBS 54K).

RESULTS AND DISCUSSION

Linden (*T. cordata*) tree blossoms were collected from six trees. Two trees were from ecologically clean habitats (A and B), the C tree was growing on green grass near a street and the other three trees (D, E and F) were growing on a tile-covered pavement. The essential oils of linden tree blossoms were obtained by hydrodistillation. All the essential oils contained more than 250 constituents. One hundred and sixty nine compounds were identified and comprised 73.4–97.7% of the oils. A large part of the constituents was the same (Table) as in neighbouring Poland [5]. The essential oils under study from the clean habitats (A and B) contained 97 of the same compounds, while the oils from the polluted areas (C, D, E and F) contained only 29 of the same compounds.

The oils D–F from the polluted localities contained only 20.6–27.7% of oxygenated compounds, while in the oils A and B obtained from the clean habitats included 47.3–55.0% of these compounds (Table). The essential oils

Table. **Chemical composition (%) of linden (*Tilia cordata* Mill.) blossom essential oils**

Compounds	RI	A	B	C	D	E	F
Hexanal	802	t	0.2	0.4	–	0.4	0.2
(2E)-Hexenal	855	0.1	1.1	0.6	–	0.3	–
(3Z)-Hexenol	859	–	0.2	0.1	0.1	0.4	t
Hexanol	871	0.2	1.4	0.5	0.1	–	–
Heptanal	902	0.1	0.4	0.6	0.5	0.4	0.2
α-Pinene	939	0.1	0.1	–	0.1	0.1	0.1
Camphene	954	t	t	–	t	t	t
Benzaldehyde	960	0.2	0.1	0.1	0.1	0.3	0.3
1-Octen-3-ol	979	0.3	t	0.5	–	t	0.3
6-Methyl-5-hepten-2-one	986	0.1	0.1	0.4	0.2	0.3	0.3
3-p-Menthene	988	2.4	2.1	0.2	0.2	0.4	2.0
2-Pentylfuran	990	0.4	0.1	–	0.2	0.4	0.3
α-Terpinene	1017	0.5	0.9	–	0.1	0.5	–
p-Cymene	1025	0.2	0.3	–	0.2	0.4	–
Limonene	1029	0.5	0.4	–	1.0	0.6	t
(Z)-β-ocimene	1037	0.2	0.1	–	–	0.1	–

Table (continued)

Hexanoic acid	1038	0.1	0.1	0.1	0.1	0.1	0.1
Benzene acetaldehyde	1042	0.7	0.2	0.7	0.4	–	0.7
(E)-β-Ocimene	1050	0.2	0.3	–	0.7	0.4	–
γ-Terpinene	1060	0.1	0.3	–	0.2	0.2	0.1
trans-Linalool oxide	1073	1.3	–	0.3	0.1	–	0.6
cis-Linalool oxide	1087	0.9	–	0.1	0.1	–	0.2
Terpinolene	1089	0.2	0.1	–	0.2	–	–
Dehydrolinalool	1091	–	–	0.1	–	0.1	0.1
p-Cymenene	1091	0.4	1.5	0.7	1.0	0.2	0.5
Linalool	1097	0.6	0.5	0.3	0.4	0.2	1.8
Hotrienol	1098	2.4	0.1	0.6	–	0.2	1.3
Nonanal	1101	2.7	3.0	2.7	0.5	0.4	–
Phenyl ethyl alcohol	1107	–	–	0.4	–	–	0.6
cis-Rose oxide	1108	0.1	0.1	0.1	–	0.1	–
1,3,8-p-Menthatriene	1110	0.1	0.2	–	0.2	0.2	0.1
trans-p-Mentha-2,8-dien-1-ol	1123	0.4	0.3	–	0.1	0.2	–
trans-Rose oxide	1126	t	t	–	0.1	0.1	–
cis-Limonene oxide	1137	–	–	–	0.1	–	–
p-Mentha-1,5,8-triene	1137	0.1	–	–	–	0.2	0.1
cis-p-Mentha-2,8-dien-1-ol	1138	0.3	0.2	0.9	0.1	0.2	–
trans-Pinocarveol	1139	–	0.1	–	t	–	–
cis-Verbenol	1141	t	t	–	0.4	0.5	0.1
trans-Verbenol	1145	0.1	0.1	–	0.3	0.3	0.1
Camphor	1146	0.1	–	–	–	–	t
(2Z)-Nonen-1-al	1149	–	0.1	0.1	–	0.1	–
(Z)-Tagetone	1152	–	–	0.2	–	–	–
Citronellal	1153	0.1	0.1	–	–	–	–
Nerol oxide	1158	0.2	–	–	–	–	–
(2E)-Nonen-1-al	1162	0.2	–	0.1	0.2	–	–
Borneol	1169	0.6	0.1	0.3	–	0.2	0.6
Nonanol	1169	–	–	–	–	0.1	0.1
p-Mentha-1,5-dien-8-ol	1170	–	0.1	0.4	0.3	0.4	–
Terpinen-4-ol	1177	0.4	1.3	0.6	0.9	0.6	0.6
p-Cymen-8-ol	1183	0.4	1.1	1.3	1.4	0.7	0.4
Dill ether	1187	0.1	0.1	–	–	0.2	0.1
α-Terpineol	1189	0.2	0.4	–	–	0.2	0.5
Methyl salicylate	1192	0.7	1.0	0.3	0.8	0.3	1.1
2-Decanone	1192	0.1	–	–	–	–	–
(4Z)-Decenal	1194	–	–	0.1	–	–	0.5
cis-Piperitol	1196	0.4	–	0.1	0.4	–	0.2
Safranal	1197	–	0.5	0.4	0.4	0.6	–
(4E)-Decenal	1197	0.1	0.2	t	–	–	–
Verbenone	1205	–	0.4	–	0.3	0.4	–
trans-Piperitol	1208	0.3	0.4	0.2	–	–	0.1
trans-Carveol	1217	0.3	0.2	–	–	0.2	–
Citronellol	1226	–	0.2	–	–	0.1	0.1
(Z)-Ocimenone	1229	0.2	0.4	0.1	0.4	0.3	0.2
cis-Carveol	1229	–	0.1	–	–	0.1	–
Nerol	1230	–	t	–	–	0.1	0.1
Thymol methyl ether	1235	–	0.1	–	–	–	–
(E)-Ocimenone	1238	0.1	1.1	0.1	0.3	t	–
Neral	1238	–	0.2	–	–	–	0.2
Cumin aldehyde	1242	0.1	–	–	–	–	–
Carvone	1243	0.1	0.3	–	–	0.1	–
Linden ether	1247	0.1	0.1	–	–	0.1	–
p-Anis aldehyde	1250	–	–	–	–	t	–

Table (continued)

Geraniol	1253	0.3	–	–	–	–	–
(Z)-Anethole	1253	–	0.3	–	–	0.4	–
Methyl citronellate	1261	–	–	0.1	–	t	–
(2E)-Decenal	1264	–	–	0.2	–	–	–
Nonanoic acid	1271	13.1	2.3	4.5	2.6	3.1	3.7
α-Terpinen-7-al	1285	–	–	–	0.4	0.1	–
Bornyl acetate	1289	0.1	0.1	–	0.1	–	–
Thymol	1290	0.1	0.1	0.1	t	0.1	0.1
p-Cymen-7-ol	1291	0.1	0.1		t	0.1	0.1
(2E,4Z)-Decadienal	1293	–	0.3	0.3	–	0.1	–
Tridecane	1300	–	0.2	–	–	–	0.2
Eugenol	1359	0.5	0.1	2.4	0.2	0.1	0.2
γ-Nonalactone	1361	0.6	0.2	–	0.1	t	0.1
(Z)-β-Damascenone	1364	0.6	0.2	0.5	–	0.1	0.3
α-Copaene	1377	0.1	0.1	–	t	–	–
Geranyl acetate	1381	0.6	–	–	0.2	–	–
Decanoic acid	1384	0.4	0.5	0.1	0.3	0.1	0.2
(E)-β-Damascenone	1385	0.3	0.5	0.3	–	0.1	0.2
β-Bourbonene	1388	0.4	0.2	–	–	0.1	–
β-Elemene	1391	–	0.3	–	–	–	–
Tetradecane	1400	0.5	–	–	t	t	–
Methyl eugenol	1404	–	0.6	–	–	0.1	–
(Z)-Isoeugenol	1407	1.0	–	–	–	–	–
Dodecanal	1409	–	0.3	0.2	–	–	–
β-Caryophylene	1419	0.8	4.0	–	0.2	0.2	0.3
(E)-α-Ionone	1430	–	–	–	t	0.1	t
γ-Elemene	1437	–	–	–	0.3	–	–
(Z)-β-Farnesene	1443	t	t	–	–	0.5	–
Geranyl acetone	1455	0.9	7.4	3.3	0.8	–	0.9
α-Humulene	1455	0.1	0.1	–	–	0.2	–
(E)-β-Farnesene	1457	t	0.1	0.2	–	0.1	–
Dodecanol	1471	–	3.8	0.2	–	–	0.1
trans-Cadina-1(6),4-diene	1477	0.3	0.1	–	0.1	–	–
γ-Muurolene	1480	–	–	–	0.1	–	–
Germacrene D	1485	0.7	0.6	0.6	0.6	–	–
(E)-β-Ionone	1489	0.3	1.3	0.3	0.4	0.3	0.3
trans-Muurola-1(14),5-diene	1494	1.3	2.5	0.2	0.1	0.2	–
epi-Cubebol	1494	0.4	0.9	–	0.2	–	0.1
Benzyl tiglate	1498	–	0.9	–	–	0.1	–
Pentadecane	1500	–	0.3	0.1	–	–	–
α-Muurolene	1500	0.8	0.3	–	–	t	0.1
(E,E)-α-Farnesene	1506	1.2	1.7	0.2	1.0	0.2	–
Tridecanal	1510	0.2	–	0.1	–	–	–
γ-Cadinene	1514	–	–	–	0.4	–	–
Cubebol	1515	1.3	1.1	–	0.2	–	–
Myristicin	1519	–	0.9	–	–	–	–
β-Sesquiphellandrene	1523	0.4	1.3	-	-	-	-
δ-Cadinene	1523	1.8	2.4	0.2	1.5	0.1	–
Citronellyl butanoate	1532	0.1	0.2	–	–	–	–
(Z)-Nerolidol	1533	0.6	1.2	0.2	–	0.1	–
trans-Cadina-1(2),4-diene	1535	0.3	3.4	–	0.2	–	–
α-Cadinene	1539	0.1	0.1	–	0.1	–	–
α-Calacorene	1546	0.2	0.4	–	0.1	–	t
Elemol	1550	0.1	–	–	0.2	–	–
cis-Cadinene ether	1554	0.1	–	–	–	–	–
(E)-Nerolidol	1563	0.2	0.4	0.4	0.1	0.2	0.1

Table (continued)

β-Calacorene	1566	–	–	–	0.1	–	–
(3Z)-Hexenyl benzoate	1567	0.2	2.4	0.5	0.5	0.2	0.4
Tridecanol	1572	–	–	–	–	–	0.2
Hexyl benzoate	1580	0.2	1.4	0.3	–	0.1	0.1
Caryophylene oxide	1583	–	–	–	0.3	0.1	–
2-Phenyl ethyl tiglate	1585	–	0.5	–	–	0.1	–
Hexadecane	1600	0.1	0.5	0.4	–	0.2	–
Humulene epoxide II	1608	0.1	0.4	–	0.3	–	0.1
Tetradecanal	1613	0.5	0.9	0.3	0.1	0.2	0.3
Isopropyl laurate	1617	–	–	–	–	0.2	0.1
1-epi-Cubenol	1629	1.3	0.1	–	0.4	–	0.2
epi-α-Cadinol	1640	0.5	0.1	–	1.4	–	–
epi-α-Muurolol	1642	0.5	–	0.4	0.5	–	0.1
α-Muurolol	1646	0.4	–	0.2	0.4	–	0.1
β-Eudesmol	1651	–	–	0.2	0.1	–	–
α-Cadinol	1654	0.4	0.2	1.5	2.4	–	0.2
14-hydroxy-9-epi-(E)-Caryophylene	1670	0.8	0.1	–	0.2	–	–
Tetradecanol	1673	0.2	0.1	–	0.1	0.4	0.4
Eudesma-4(15),7-dien-1-β-ol	1688	0.5	–	–	–	–	0.1
Heptadecane	1700	0.2	–	2.9	–	0.1	0.2
Pentadecanal	1715	1.6	–	1.0	1.2	0.8	0.9
(E,Z)-Farnesol	1746	0.2	0.2	0.3	0.3	0.2	–
Benzyl benzoate	1760	0.1	–	0.3	–	–	–
Pentadecanol	1774	–	–	–	0.3	0.3	0.3
Octadecane	1800	0.3	0.5	0.3	0.3	0.2	0.1
2-Ethylhexyl salicylate	1807	0.3	–	–	–	–	0.2
Hexadecanal	1819	0.3	–	0.3	0.1	–	0.1
(2Z,6E)-Farnesyl acetate	1822	1.1	0.3	–	0.9	0.2	0.2
Isopropyl tetradecanoate	1830	–	–	–	–	0.4	–
Hexahydrofarnesyl acetone	1843	2.8	2.6	6.0	1.6	2.6	3.2
Hexadecanol	1876	0.2	–	0.3	0.2	–	0.3
Nonadecane	1900	0.2	–	–	1.6	1.1	7.4
Farnesyl acetone	1927	1.6	1.3	3.6	1.4	1.8	1.8
Hexadecanoic acid	1972	2.6	0.3	0.3	0.4	0.5	0.9
Eicosane	2000	0.4	5.1	4.9	7.7	0.3	4.0
Kaurene	2043	1.2	5.5	1.1	7.5	26.3	–
Heicosane	2100	5.4	7.9	11.0	5.2	2.8	16.0
9,12-Octadecadienoic acid	2130	1.3	–	–	–	–	2.5
Docosane	2200	2.7	1.3	10.8	4.6	11.3	4.6
Tricosane	2300	10.5	1.1	6.4	12.2	9.6	6.5
Total		90.4	97.7	82.7	76.0	79.8	73.4
Aliphatic hydrocarbons		20.3	16.9	36.8	31.6	25.6	39.0
Oxygenated aliphatics		27.6	15.6	14.4	7.1	8.5	13.3
Monoterpene hydrocarbons		4.8	4.0	0.7	3.5	2.5	0.7
Oxygenated monoterpenes		11.9	16.4	9.7	7.6	6.6	8.4
Sesquiterpene hydrocarbons		8.4	17.6	1.7	5.2	1.9	0.7
Oxygenated sesquiterpenes		15.5	15.3	13.0	12.6	5.5	6.5
Oxygenated aromatics		2.1	8.3	3.9	1.5	1.5	2.1
Aliphatic acids		17.5	3.2	5.0	3.4	3.8	7.4
Oxygenated hydrocarbons		55.0	47.3	37.1	27.3	20.6	28.2

Notes: RI - retention indexes on nonpolar column; t - traces; A–F indicated localities.

of linden growing on green grass in the polluted localities (C) contained 36.8% of the oxygenated compounds. The reactions of oxidation in the polluted localities were reduced nearly twice. The pollution of the streets and bad ventilation of the roots inhibited the production or activity of oxidation enzymes in the linden tree blossom. The same correlation was noticed for oxygenated aliphatics (from 7.1–12.8% to 15.6–27.6%), oxygenated monoterpenes (from 6.6–8.4% to 11.9–16.4%) and oxygenated sesquiterpenes (from 5.5–12.6% to 15.3–15.5%).

The influence of environmental pollution increases in the following sequence: B (green grass-plot + luxuriant foliage) > A (green grass-plot + blight) > C (green grass-plot – vermin + fungous diseases + salt sprinkle in winter). At the same time the amount of sesquiterpene hydrocarbons in B, A and C essential oils (17.6, 8.4, and 1.7%, respectively) were decreasing, while the amounts of aliphatic hydrocarbons (16.9, 20.3 and 36.8%, respectively) were increasing. The essential oil A was rich in nonanoic acid (Table, 13.1%). These compounds possessed strong antifeedant properties against *Hylobius abietis,* according to earlier investigations [16]. The same properties were characteristic of decanoic acid [16], which was found in all the essential oils under study (Table, ≤0.5%).

The amounts of aliphatic alcohols, aldehydes and esters in the essential oil of blossoms increased (3.7 > 9.1 > 12.4) with improving conditions (from a pavement covered to green grass and ecologically clean environment, respectively) of linden growth.

The main constituents in the essential oils were 3-p-menthene, nonanal, nonanoic acid, hexahydrofarnesyl acetone, farnesyl acetone, kaurene, heicosane, docosane and tricosane (Table). Only one linden blossom essential oil – E from a polluted street contained a large amount of diterpene kaurene (26.3%).

CONCLUSIONS

The investigated linden (*T. cordata*) blossom oils from Vilnius city (five samples) and from Kaišiadorys district (one sample) contained about 250 constituents. One hundred and sixty nine compounds were identified and made up 73.4–97.7% of the oils. The blossom oils collected from linden growing on green grass in ecologically clean localities contained 47.3–55.0% of oxygenated compounds, and it was nearly twice as much as compared to the amount found in the oils obtained from lindens growing on tile-covered pavements (20.6–28.2%).

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URBANISTINĖS APLINKOS ĮTAKA *TILIA CORDATA* ETERINIŲ ALIEJŲ CHEMINEI SUDĖČIAI

S a n t r a u k a

Liepų (*Tilia cordata* Mill.) žiedai 2006 metais surinkti ekologiškai švariose vietovėse ir Vilniaus mieste. Hidrodistiliacijos būdu gauti eteriniai aliejai analizuoti dujų chromatografijos ir dujų chromatografijos/masių spektrometrijos metodais. Eteriniuose aliejuose labiausiai skiriasi oksiduotų junginių (monoterpenų, seskviterpenų, alifatinių ir kt.) kiekiai. Liepų, augančių švarioje aplinkoje, žiedų aliejuose rasta 47,3–55,0% minėtų junginių. Oksiduotų junginių kiekis aliejuose sumažėja beveik dvigubai, kai liepos auga plytelėmis dengtuose šaligatviuose (20,6–27,7%). Daugiausia liepų žiedų aliejuose rasta 3-p-menteno, nonanalio, nonano rūgšties, α-kadineno, heksahidrofarnezilacetono, kaureno, alifatinių angliavandenilių ($C_{20}H_{42}$ – $C_{24}H_{50}$). Identifikuoti junginiai (169 iš 250) sudarė 73,4–97,7% eterinių aliejų.