Identification of Acetone Extractable Component from Iranian Elm (*Ulmus glabra* huds) Wood

Majid Kiaei, [a,*] and Milad Tajik[b]

This study was carried out for identification of chemical compounds in acetone extractives of elm wood. Wood samples were selected from Arasbaran forest, Iran. Extracts from the elmwood *(Ulmus glabra*) were obtained using acetone solvent (according to the TAPPI standards). BSTFA reagent was added to extractive soluble in acetone and then samples were kept in Ben Marry Bath at 70 °C for an hour. Then chemical components of acetone-extraction were analyzed by gas chromatography mass spectroscopy. The most important identified component chemicals were bis(2-ethylehxl) phthalate (93.40%), 4-(N-Isopropylamino)-6-phenyl (1.89%) and 1-naphthalenol, 5,6,7,8-tetrahydro (1.80%). Chemical composition of elm wood was compared to other Iranian hardwood species.

Keywords: Ulmus glabra huds*; Gas chromatography mass spectroscopy (GC/MS).*

*Contact information: a: Department of Wood and Paper Science and Technology, Chalous Branch, Islamic Azad University, Chalous, Iran; b: Young Researchers and Elite Club, Chalous Branch, Islamic Azad University, Chalous, Iran and M.sc student, Department of pulp and paper, Energy and New Technology Faculty, Shahid Beheshti University; *Corresponding author: mjd_kia59@yahoo.com*

INTRODUCTION

Extractives can protect wood from decay, add color and odor to wood, and enhance the strength properties of the wood. Extractives may also inhibit setting of concrete, glues, and finishes and cause problems in paper making (Hillis 1987; Amusant *et al.* 2007; Hashemi *et al.* 2009).

Most extractives are organic substances soluble in organic solvents, including polyphenols, terpenoids, fats, waxes, organic acids, complex polysaccharides, and nitrogenous compounds (Hillis 1987). Some of them are unsaponifiable (hydrocarbons, sterols, various alcohols, aldehydes, *etc*.), and some are saponifiable (fats, sterols, esters). The amount of extractives and their composition vary with respect to the botanical families, wood species, growth region, wood tissue and extraction methods. The wood species is probably the most important factor that affects the extractive content and component. Usually a higher extractive content is found in hardwoods than in softwoods (Hillis 1987).

One of the most important factors affecting the natural durability is wood extractives. In durable wood species, phenolic compounds, resin acids, terpenoids, and tropolons have antifungal activity. Likewise the flavonoids, quinines, sesquiterpenoids and stilbene have anti-termite activity. The presence of any of the listed compounds can impart natural durability of wood (Jorbandian and Farahani 2012). More research can help in the identification of natural and environmentally friendly compounds to act as effective preservatives and enhance the wood durability.

Ulmus glabra is one of the most important hardwood species of Iranian forests. It is distributed from the Gorgan region (in the northeast) to Arasbaran site (in the northwest). Due to development of Dutch elm disease in their habitats, countless numbers of this species have died, and its presence in the forest ecosystems is in danger of being eliminated. The mean of wood oven-dry density, basic density, and volumetric shrinkage of elm wood were determined 656, 575 Kg/m^{-3} and 12.39 %, respectively. There also is positive relationship between wood density with swelling and shrinkage of elm wood in the Arasbaran forest in Iran (Kiaei and Samariha 2011).

There are no studies on different properties of elm wood (*Ulmus glabra* huds) in Iran. Therefore, the goal of this work was to determine the wood extractive chemical components by gas chromatography mass spectroscopy (GC-MS) for elm wood (*Ulmus glabra*).

EXPERIMENTAL

Materials

Wood samples were naturally collected from elm trees (*Ulmus glabra*) from Arasbaran forest (Ardebil region), Iran. Three trees were randomly selected, and a disc was cut at breast height from each of them. The annual rainfall and annual average temperature (1976-2008) was 450 mm and 7 to 15 °C, respectively.

Methods

Wood samples for chemical analysis were converted to wood flour according to the TAPPI T 257. The extractive materials were extracted with the use of acetone solvent according to the TAPPI standard T 204-om-97. In order to identify components of each extract, about 1 mg of solid obtained from extraction was mixed with 30 mL of BSTFA [bis (trimethylsilyl) trifluoroacetamide] + 1% TMCS (trimethylchlorosilane) reagent and about 15 mL of pyridine inside a tube test. The samples were kept in a Ben Marry Bath at 70 ^{o}C for an hour, and then they were analyzed by using GC-MS on an HP 6890 Gas Chromatograph (Agilent technology, USA), equipped with a split/split less injector and a 5973 Mass Selective Detector (MSD). The oven column was programmed as follows: Chromatography was performed on a HP-5MS capillary column (SGE, 30 m, 0.25 mm), using helium as the carrier gas with 1 mL/min speed and temperature program between 60 and 260 °C, with a temperature increase rate 6 °C/min. To identifying the compounds, a GC diagram, which shows abundance and retention time of each compound, and calculation of quartz index and Adams table were used,

$$I = \frac{100n+100\ (t_{rx}-t_{rn})}{t_{rn+1}-t_{rn}} \quad (1)$$

where I is the quartz index, n is the carbon number of normal Alcan, $t_{\text{rn+1}}$ is the retention time of the unknown compound, and t_{rn} is the retention time of normal Alcan.

Finally, the acetone extractable component from Iranian elm wood was compared to other Iranian hardwood species such as walnut wood (Hashemi *et al*. 2009), black locust (Hashemi *et al.* 2011), and *Morus alba* wood (Sadeghifar *et al.* 2011). Elm wood and all of the other mentioned species belong to ring porous species in the vessel classification.

RESULTS AND DISCUSSION

Table 1 and Fig. 1 represent the chemical composition of the acetone extracts of elm wood. The average of acetone extractive was 1.2% for elm wood. As can be seen from these tables, 17 chemical compounds were characterized with different percentage in elm wood. The major compounds are as follows: bis (2-ethylehxl) phthalate (93.40%), 4-(N-isopropylamino)-6-phenyl (1.89%), 1-naphthalenol, 5,6,7,8-tetrahydro (1.80%), 5-isopropyl-2-(phenyl) pyrimidin (0.60%), and tetradecanal (0.59%).

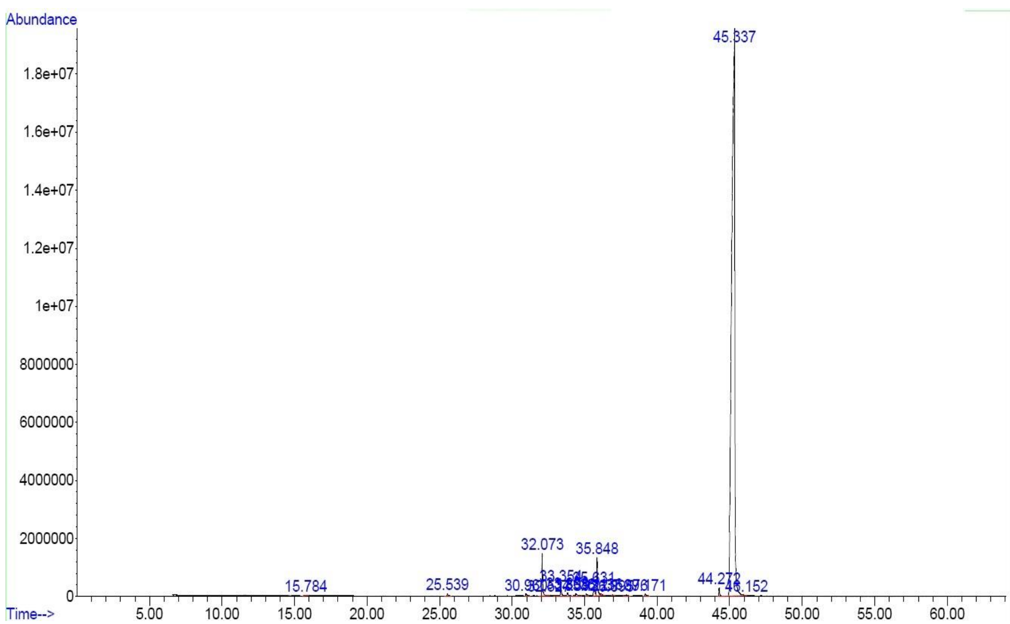

Fig. 1. Elm wood extractives GC-MS chromatogram

This result showed that extracts with solvent acetone contain high amounts of Bis (2-ethylehxl) phthalate (93.40%). This compound (Fig. 2) is an organic compound with the formula C_6H_4 $(C_8H_{17}COO)_2$.

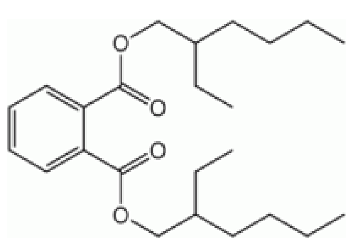

Fig. 2. Formula of Bis (2-ethylehxl) phthalate.

This compound is the most common of the class of phthalate plasticizers (Fig. 2). The major use of bis (2-ethylehxl) phthalate is in the production of PVC and vinyl chloride resins, where it is added to plastics to make them flexible (Gillum *et al.* 2009).

Table 1. Elm wood extractive in acetone solvent (Rt: retention time, KI: quartz index)

Rt	Compounds	Total	KI
15.785	Dihydro carvone	0.05	1201
25.54	Phenol, 2,4-bis (1,1-dimethylethyl)	0.12	1424
30.96	7-propylfuro [3, 2-b] pyridine	0.14	1552
32.07	1-naphthalenol, 5, 6,7,8-tetrahydro	1.80	1582
32.52	5,9-undecadien-1-OL, 2,6,10-trimethyl	0.02	1591
32.354	Tetradecane	0.59	1613
33.81	Pentanoic acid, 3, 7-dimethyl-6-octenylester	0.22	1621
34.389	8-methoxy-1-acetonaphthone	0.27	1640
35.120	Hexadecanoic acid	0.13	1663
35.63	5-Isopropyl-2-(phenyl) pyrimidin	0.60	1672
35.851	4-(N-Isopropylamino)-6-phenyl	1.89	1679
36.136	1,3,5-Triazine -2,4-diamine	0.14	1684
36.893	1-hydroxyspiro	0.04	1704
44.273	1,2-Benzendicarboxylic acid	0.44	1911
39.169	Quinindoline	0.14	1767
45.334	Bis(2-ethylhexyl) phthalate	93.40	1942
46.149	Phtalic acid, 2-hexylester	0.01	1966

These components of elmwood were compared with extractive component of other species such as walnut wood (Hashemi *et al.* 2009), black locust (Hashemi *et al.* 2011), and *Morus alba* wood (Sadeghifar *et al.* 2011). Although these wood species are ring porous species, there are no shared components between the studied species and walnut wood. A shared component that was found between *Ulmus glabra* and black locust wood (Fig. 3), is called hexadecanoic acid. The content of this compound in black locust (0.31%) is higher than elmwood (0.13%). In addition, tetradecane is a shared compound between studied wood species and *Morus alba* wood (Fig. 4). This compound in *Morus alba* (2.31%) is higher compared than in the studied species (0.59%). The most abundant extractives compositions were (23 S)-ethylcholest-5-en-3, beta-o1 (18.33%), n-decane (50.78%) and gallic acid (44.57%) in black locust, *Morus alba* and walnut wood, respectively (Hashemi *et al.* 2009; Hashemi *et al.* 2011; Sadeghifar *et al.* 2011). The type of solvent, extraction method, site variation, and wood species are important reasons in chemical composition differences among hardwood species.

Fig. 3. The value of Hexadocanoic in elm wood and black locuts wood.

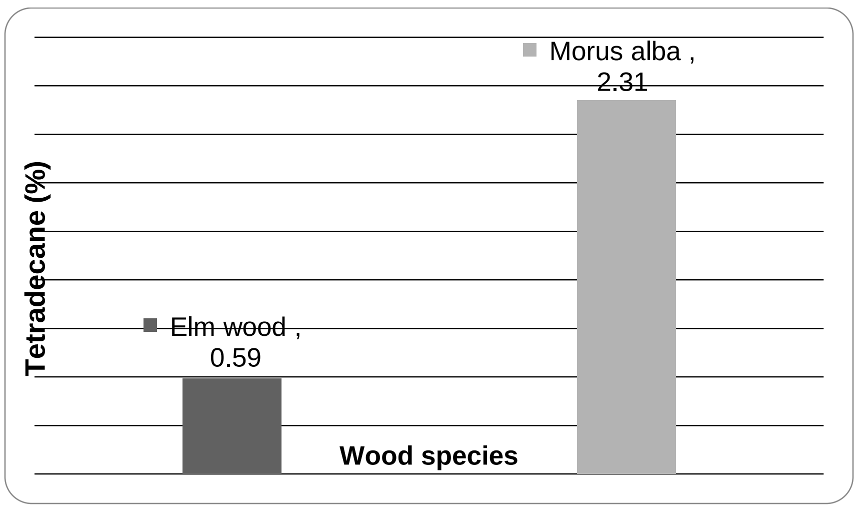

Fig. 4. The value of Tetradecane in elm wood and *Morus alba* wood.

CONCLUSIONS

The chemical components of acetone extractive were identified for elm wood by gas chromatography mass spectroscopy (GC-MS). Five chemical components were found: bis(2-ethylehxl) phthalate (93.40%), 4-(N-isopropylamino)-6-phenyl (1.89%), 1-naphthalenol, 5,6,7,8-tetrahydro (1.80%), 5-isopropyl-2-(phenyl) pyrimidin (0.60%), and tetradecanal (0.59%) were 98.28% of total extractive components. These components play an important role in wood natural durability.

REFERENCES CITED

Amusant, N., Moretti, C., Richard, B., and Prost, E. (2007). "Chemical compounds from *Eperua falcata* and *Eperua grandiflora* heartwood and their biological activities against wood destroying fungus (*Coriolus versicolor*)," *Holz Roh Werkst* 65(1), 23-28.

Jorbandian, A., and Farahani, M. R. M. (2012). "Introduce of some effective extractives compounds in natural durability of wood," *Journal of Conservation and Utilization of Natural Resources* 1(2), 105-109.

Gillum N., Karabekian Z., Swift, L. M., Brown, R. P., Kay, M. W., and Sarvazyan, N. (2009). "Clinically relevant concentrations of di (2-ethylhexyl) phthalate (DEHP) uncouple cardiac syncytium," *Toxicology and Applied Pharmacology* 236(1), 25-38.

Hashemi, S. K. H., Javadi, M. R., and Miri, A. (2011). "Identification of chemical compounds in ethanolic extractives of black locust heartwood by GC/MS method," *Journal of Sciences and Techniques in Natural Resources* 6(3), 63-75.

Hashemi, S. K. H., Parsapajouh, D., Khademi-eslam, H., Mirshokraeai, S. A., and Hemmasi A. H. (2009). "Identification of chemical compounds within north of iran's walnut heart wood extractives by GC/MS method," *Iranian Journal of Agriculture Science* 12(4), 939.

Hilis, W. E. (1987). *Heartwood and Tree Exudates*, Springer-verlag, Berlin, Germany, 26 pp.

Kiaei, M., and Samariha, A. (2011). "Wood density and shrinkage of *Ulmus glabra* in northwestern of Iran," *American-Eurasian Journal of Agriculture and Environment Science* 11(2), 57-260.

Sadeghifar, H., Sheikh, A., Khalilzadeh, A., and Ebadi, A. G. (2011). "Heartwood extractives of Iranian *Morus alba* wood," *J. Chem. Soc. Pak.* 33(1), 104-106.

TAPPI (Technical Association of the Pulp and Paper Industry). (1994). *TAPPI Test Methods*, TAPPI Press, Atlanta.

Article submitted: October 15, 2013; Peer review completed: December 7, 2013; Revised version received and accepted: December 15, 2013; Published: December 25, 2013.