WOOD RESEARCH
59 (1): 2014
177-190

INFLUENCE OF URBAN ENVIRONMENT ORIGINATED HEAVY METAL POLLUTION ON THE EXTRACTIVES AND MINERAL SUBSTANCES CONTENT IN BARK AND WOOD OF OAK (*QUERCUS ROBUR* L.)

Donata Krutul, Tomasz Zielenkiewicz, Janusz Zawadzki,
Andrzej Radomski, Andrzej Antczak, Michal Drożdżek
Warsaw University of Life Sciences, Faculty of Wood Technology
Department of Wood Science and Wood Protection
Warsaw, Poland

(Received April 2012)

ABSTRACT

Extractives and mineral substances contents were analyzed in oak wood and bark (*Quercus robur* L.) which was sampled from the polluted environment. Results were compared with corresponding values in bark and wood of oak from unpolluted environment. Samples were collected from butt-end, middle and top section of three trunks of cca. 100 years old oaks which were grown in polluted environment. Pollution originated from "Żerań" heat and power plant and "ArcelorMittal" foundry. Three trunks of cca. 100 years old oaks which were grown in unpolluted environment in Mazury-Podlasie region were examined for comparison.

200 mm thick disks were cut off from each mentioned above part of trunks. Five zones were distinguished on the each cross-section: Pith adjacent wood, heartwood, sapwood adjacent heartwood, sapwood and bark. Following analyses were performed in samples taken from all zones: extractives determination, mineral substances determination and content analysis of: Nitrogen (N_2) (Kjeldahl method), calcium (Ca), magnesium (Mg), iron (Fe), copper (Cu) (ICP-AES technique), potassium (K), lead (Pb), cadmium (Cd) (GFAAS technique).

Our results show that environmental pollution significantly influences content of examined substances. It causes the increase of extractives content in wood, and decrease of the extractives content in bark. Nitrogen content is generally lower and analyzed metals contents are higher in polluted wood in relation to unpolluted one.

KEYWORDS: Oak wood, bark, extractives, mineral substances, heavy metals.

INTRODUCTION

Wood extractives are chemically differential and their distribution in wood and bark depends on their function in plant. Some of them, with phenolic character, makes wood resistant to fungus and insects, others causes corrosive effect. Tannins are basic extractives both in wood and bark of oak.

Extractives content is higher in the middle and top part of a tree than in butt-end (Krutul and Buzak 1986; Krutul and Makowski 2004). This phenomenon takes place in wood from the youngest age class (20-30 years) as well as older (80, 90, 100 and 160 years). Extractives content on the trunk cross-section decreases in the direction from pith to perimeter, apart from the tree habitat. According to Adamopoulos et al. (2005), heartwood of Black locust (*Robinia pseudoacacia* L.) contains 43 % more extractives in relation to sapwood. Extractives content in bark is higher than in wood and it is also (like in wood) lower in the butt-end (Krutul and Sacharczuk 1997, Passialis et al. 2008).

Mineral substances content in wood is low, but their role in wood tissue forming is significant. Mineral substances content in wood of one species is variable (Fengel and Wegener 1984, Loto and Fakankun 1989, Pereira 1988, Rademacher et al. 1986).

Ash content which testifies to mineral substances content, varies from 0.3 to 1.2 % of dry mass of wood gained from temperate climatic zone (Fengel and Wegener 1984). Passialis et al. (2008) stated that wood of Black locust (*Robinia pseudoacacia* L.) gained in Greece, Bulgaria and Hungary contains 0.72 – 1.24 % of ash (sapwood), 0.47 – 0.71 % (heartwood juvenile), 0.34 – 0.89 % (heartwood mature). Ash content in bark varies from 7.24 to 8.56 %.

Distribution of mineral substances on the trunk cross- and longitudinal-section depends on wood species, age and tree habitat, what is confirmed in number of papers (Rademacher et al. 1986, 1988, Eckstein 1988, Krause and Eckstein 1992, Denne and Dodd 1981, Viqourocix et al. 1998, Krutul 1995, 1996). Significant difference in mineral substances content were stated for butt-end and top part of scots pine and oak trees (90 years old). This content is higher in top part of a trunk (Krutul 1996, 1998, Krutul and Sacharczuk 1997).

Variations of bark chemical composition on the cross- and longitudinal-section of the trunk are more significant than in wood (for instance, almost ten times higher ash content, what means much more quantity of macro- and microelements). Content of calcium and sulphur in pine bark (*Pinus sylvestris* L.) is five times higher in relation to sapwood adjacent heartwood and eight times higher than in sapwood (Krutul et al. 1999).

Rademacher et al. (1986) analyzed distribution of macro- and microelements in spruce wood on the trunk cross-section, in sapwood, heartwood and bark separately. Their results show that analyzed elements contents are several times higher in bark.

Results of such experiments may show some trends in particular elements distribution on the trunk cross- and longitudinal-section, because there are many factors which influence content of these elements in wood as well as in bark. Analysis becomes much more complicated if factors causing environmental pollution must be taken into account.

Environmental pollution does not influence distribution of structural wood components (lignin, cellulose, hemicelluloses) on the trunk cross- and longitudinal section and in bark. It does influence the content and distribution of elements classified as macro-, micro- and trace- elements, apart from the wood species (Krutul et al. 2006, 2010, Watmough et al. 1998, Watmough and Hutchinson 1999 and 2002).

Presence of heavy metals, especially lead, is the measure of industrial environmental pollution (Gulson et al. 1981). Fuels combustion in the industry, transport as well as in house hearths is the main source of heavy metals pollution. The content of heavy metals is raised in humus in forests growing in urban environment (Pouyat and McDonnel 1991, Pouyat et al. 1995). The content of particular elements in wood and bark of different species was discussed in number of papers (Alvarado et al. 1993, Bartholomay et al. 1997, Eklund 1995, Watmough et al. 1998, Warmough and Hutchinson 1999, Krutul and Makowski 1995, Krutul et al. 2006).

Environmental pollution also influences extractives and mineral substances content variations in wood and bark and roots. For example, nitrogen content on the trunk cross-section in common maple (*Acer platanoides* L.) wood is lower in perimeter adjacent wood (0.1 %) in relation to bark (0.7 %) and roots (0.2 %) (Krutul and Makowski 2005). Raised contents of calcium, chlorine, potassium, magnesium and sodium in contaminated wood were also stated by these authors. Especially chlorine content is very high in perimeter adjacent wood and equals 1320 – 1340 $mg.kg^{-1}$. It is twice higher in relation to wood gained from unpolluted environment. Raised concentrations of lead (24 $mg.kg^{-1}$) and strontium (cca. 158 $mg.kg^{-1}$) were specified in norway maple bark from polluted area. High contents of iron (455 $mg.kg^{-1}$ in bark and 330 $mg.kg^{-1}$ in roots), aluminium (300 vin bark and 290-376 $mg.kg^{-1}$ in roots) were also stated (Krutul and Makowski 2005).

The aim of this paper is to analyze variations in contents of extractives, mineral substances and some elements (N, Ca, K, Mg, Fe, Cu, Pb, Cd) on the cross- and longitudinal-section of oak trunks (*Quercus robur* L.) gained from polluted area, and comparison with results for oak wood samples from unpolluted environment. "Żerań" heat and power plant and "ArcelorMittal" foundry are the source of pollution.

MATERIAL AND METHODS

Extractives and mineral substances content analyses were made on the cross- and longitudinal sections of three cca. 100 years old oak trunks (*Quercus robur* L.) They were gained from Jabłonna forest inspectorate, Zegrze forest district. Examined trees were growing in fresh mixed forest, on the fertile, dusty-loamy soil. There were also analyzed samples from three oak (*Quercus robur* L.) trunks (90-100 years old) gained from unpolluted area of Mazury-Podlasie II and Baltic I regions junction. Examined trees were grown on podzol in Stępniewo forest district, II bonitation. Wood disks of 200 mm height were sampled from both polluted and unpolluted trunks. Disks were collected from butt-end, half height and top part of each tree. Disks were divided into several zones on their cross-sections: Sapwood, sapwood adjacent heartwood, heartwood, pith adjacent heartwood and bark.

Sapwood adjacent heartwood was defined as wood in 5 mm range from sapwood and heartwood frontier. Other samples were gained from each zone middle part all around the trunk cross-section using drill. Obtained samples were fractionated - the fraction passing 0.6 mm and remaining on 0.49 mm mesh sieve was taken for extractives analysis.

Extractives content were determined using ethanol-benzene (1:1) mixture (Kačík and Solár 1999) in Soxhlet apparatus. Error of this determination equals 0.01 and variation coefficient was 0.01 – 0.04. Mineral substances content was determined in muffle furnace at the temperature of 600°C, until the constant mass was reached. Determination error is equal to 0.02 and variation coefficient differs from 0.01 to 0.05.

Micro and macro-elements were analyzed as follows: Known mass of dry ash obtained by combustion of 2 g wood sample was placed in 50 cm^3 flask. Then 5 cm^3 of 10 % hydrochloric acid was added and flask was filled with redistilled water to the volume of 50 cm^3. ICP-AES technique was applied to determine the content of calcium, magnesium, iron and copper. GFAAS was used to analyze potassium, lead and cadmium. Nitrogen content was measured with Kjeldahl method.

RESULTS AND DISCUSSION

Extractives content in pith adjacent zone of oak wood (*Quercus robur* L.) is higher in relation to sapwood along trunk height apart from the habitat. It is presented in Fig. 1a, b. Extractives content in sapwood adjacent heartwood is higher than in sapwood and heartwood. This content in sapwood adjacent heartwood, heartwood and pith adjacent heartwood from oak trunks collected from polluted environment is higher in relation to corresponding wood zones obtained from unpolluted area. On the oak trunk cross-section (samples both from polluted and unpolluted area) it increases in the direction from perimeter to pith. Variations in samples from unpolluted environment are much more regular. Content of extractives in bark is 13 % higher in butt-end, 36 % higher in the middle part and 12 % higher in the top of the trunk from unpolluted area, in relation to the trunk from polluted one.

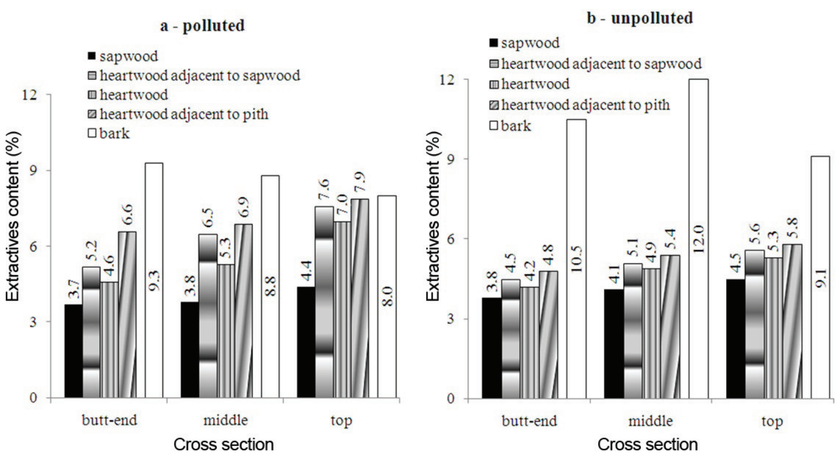

Fig. 1: Extractives content in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

According to Krutul et al. (2006) the extractives content in scots pine bark (*Pinus sylvestris* L.) in the distance of 1 km and 21 km from heat and power plant "Kozienice" is lower in relation to bark obtained from unpolluted environment. Krutul and Buzak (1986) stated that content of extractives in oak wood (*Quercus petraea*) (80-years old trunks) is higher in the middle and top part of a trunk than in the butt-end. These observations are consistent with presented in current paper results, both for polluted and unpolluted environment originated samples.

Mineral substances content in sapwood is higher in relation to sapwood adjacent heartwood, heartwood and pith adjacent heartwood, apart from the habitat. It is presented in the Fig. 2a, b. Mineral substances content in oak sapwood from polluted environment in relation to samples from unpolluted area. In butt-end section it is 12 and 25 % higher than in (respectively) middle and top part of a trunk. Higher mineral substances content was determined in all zones of oak wood in samples collected from unpolluted environment than in polluted one.

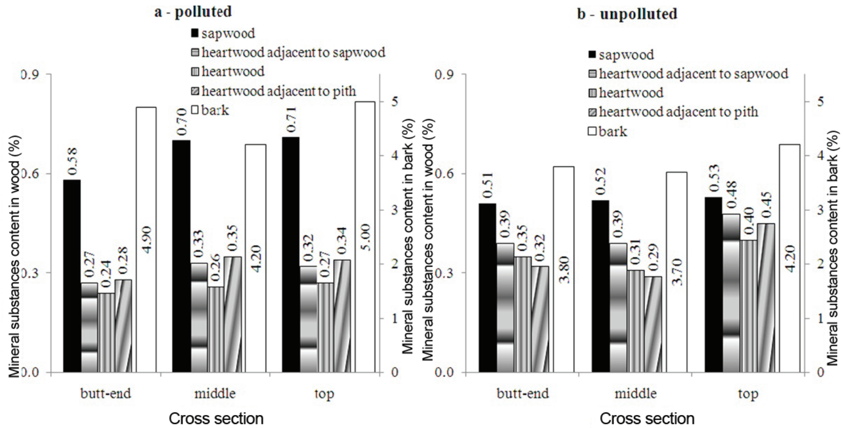

Fig. 2: Mineral substances content in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

Mineral substances content is always higher in bark than in wood, apart from the habitat. Bark from the top part of a trunk contain higher amount of mineral substances in relation to bark taken from the butt-end and middle part. These results are consistent with the data from Krutul and Makowski (2004) which relate to mineral substances content in bark and wood of oak trunks (78 and 66 years old) gained from unpolluted environment.

Nitrogen is the fourth element in biomass with respect to contents, after carbon, oxygen and hydrogen. It is essential element to aminoacids and both pyrimidine and purine bases forming (also proteins, nucleotides, nucleic acids). Nitrogen takes a part in almost every biochemical reaction in living organisms. That is why the nitrogen content in wood and bark samples collected from polluted and unpolluted area was compared.

Oak wood gained from unpolluted environment contains higher amount of nitrogen in relation to polluted samples. It is presented in the Fig. 3a, b. In sapwood from unpolluted area nitrogen content in the butt-end is 45 % higher, in the middle part – 25 % higher and in the top of a trunk it is 55 % higher in relation to polluted trunks. In other wood zones similar differences are also observed.

Nitrogen content is higher in bark than in wood samples both from polluted and unpolluted area. Differences are more significant in case of samples from polluted environment.

Calcium is the element collected by plants from the soil in the soluble form as Ca^{2+}, only when pH is low. Its function consist on many enzymes activity regulation (for instance: ATP, amylase, phospholipase). It easy joins with polysaccharides (cellulose, pectins, polygalacturonic acid), causing the decrease of cell wall flexibility. Calcium is the macroelement which, with potassium, is in the highest amount in plants. Its content in unpolluted samples is the highest in pith adjacent heartwood and decreases in the direction to perimeter. Apart from wood zone, calcium content is higher in the middle and top part of a trunk in relation to the butt-end section (Fig. 4a, b).

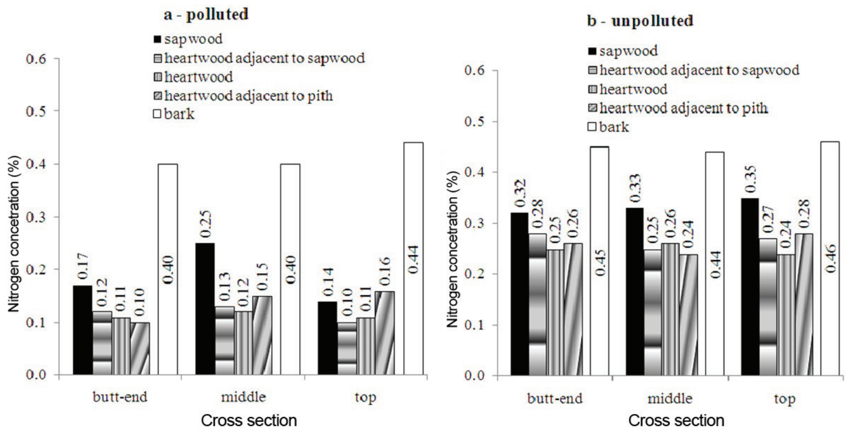

Fig. 3: Nitrogen (N) concentration in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) upolluted environment.*

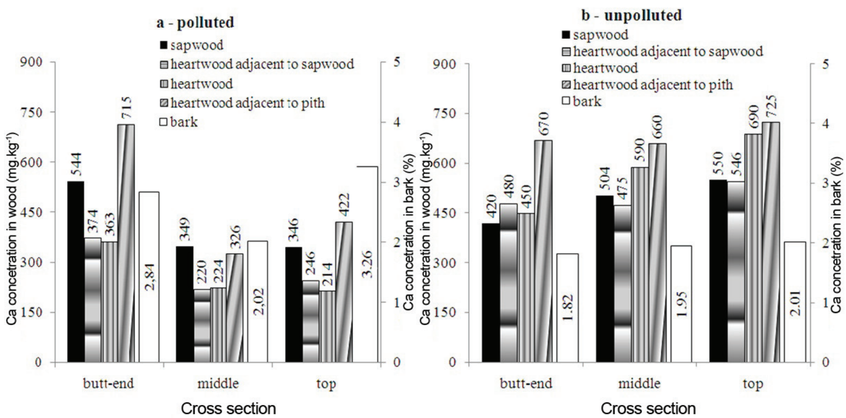

Fig. 4: Calcium (Ca) concentration in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

In wood gained from polluted environment calcium content is higher in pith adjacent heartwood and sapwood in relation to the rest of wood zones. Butt-end section contains higher amount of calcium in comparison to the middle and top part of a trunk.

Bark from oak trees which were grown in polluted area contains higher amount of calcium in relation to unpolluted ones. Calcium content in polluted samples is 35 % higher in the butt-end, 40 % higher in the top part and about 3 % in the middle part of a trunk. At the same time, calcium content in examined bark is several times higher in comparison with wood, what is compatible to results of Krutul and Sacharczuk (1997).

According to Rademacher et al. (1986), calcium content in spruce (*Picea abies*) bark taken from significantly polluted environment is cca. 7.5 % higher in relation to samples from middle and low polluted areas.

Watmough et al. (1998) examined distribution and content of calcium on cross-sections of sugar maple (*Acer saccharum*). Their data shows that calcium content decreases in the direction from pith to perimeter. Depending on the habitat, this content vary from 1600-1800 $mg.kg^{-1}$ in pith adjacent wood and from 800 to 1000 $mg.kg^{-1}$ in perimeter adjacent wood. It is almost twice higher than oak wood analyzed in current paper.

Our results show that the environmental pollution definitely influences distribution and content of calcium in the cross- and longitudinal-section of oak wood, as well as its content in bark along the trunk.

Potassium is absorbed by trees in the form of K^+. It is very mobile ion which activates more than 50 enzymes. Potassium is the essential element for proper plant development. The highest content of this element is in sapwood, apart from the habitat (Fig. 5a, b). It is higher in sapwood collected from polluted environment – 35 % higher in the butt-end, 15 % higher in the middle and about 20 % higher in the top section of a trunk (in relation to unpolluted samples). Potassium content in other wood zones is several times lower than in sapwood (in both kinds of samples). According to Krutul and Sacharczuk (1997) and Krutul and Makowski (2004), oak sapwood (collected from 20, 40, 100 and 120 year old trunks) contains much more potassium than heartwood.

Potassium content in bark taken from polluted environment is 15 % higher in the butt-end, 7 % higher in the middle and 40 % higher in the top part of a trunk, in comparison to bark collected from unpolluted environment. It is shown in the Fig. 5a, b. Potassium content in the top part of a trunk is 20 % higher (polluted environment) than in sapwood. In comparison, potassium content in the bark of Norway maple from polluted environment is about 55 % higher in relation to sapwood and 30 % higher in relation to heartwood (according to Krutul et al. 2006).

Summarizing, the environmental pollution influences distribution and content of potassium in wood and bark on the cross- and longitudinal section of a trunk, apart from factors causing this pollution.

Magnesium content in wood and bark is far lower than calcium and potassium, apart from the habitat. Magnesium being present in wood is the very strong hydrated bivalent ion (Mg^{2+}) which relocate mainly through xylem. It is found in chlorophyll – its content in leaves depends on, among others, exposition. In cell walls magnesium bonds with pectins. It demonstrates adversarial activity against K^+ and NH_4^+ ions, reducing their uptake.

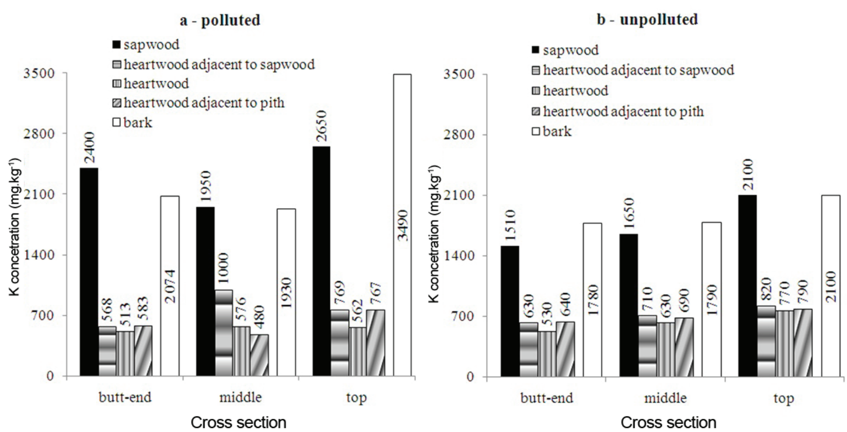

Fig. 5: Potassium (K) concentration in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

Magnesium content is the highest in oak sapwood (in relation to other examined zones), apart from the habitat. It is presented in the Fig. 6a, b. Sapwood collected from unpolluted environment contains more magnesium in relation to sapwood from polluted area. This difference decreases with the trunk height. Magnesium content in bark from polluted environment is 35 % higher in the butt-end and middle section, and about 13 % higher in the top part of a trunk, in comparison to unpolluted bark samples. Bark contains several times higher amount of magnesium in relation to all wood zones. It is consistent with the data of Krutul and Sacharczuk (1997).

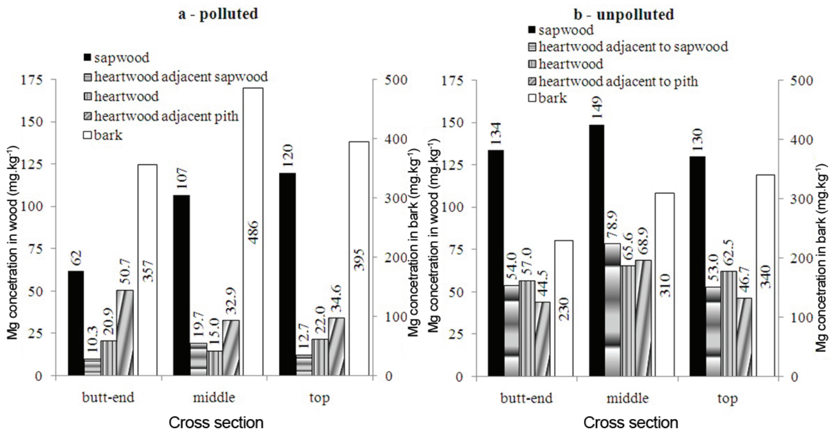

Fig. 6: Magnesium (Mg) concentration in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

Elements such as: Iron, copper, manganese, molybdenum, zinc and nickel are classified as heavy metals and microelements in wood chemistry.

Iron relocates in plants through xylem as the ion compounds with citrates or other anions. Plants collect iron when pH of the soil is low, just like in case of calcium. When soil contains high amount of calcium, iron transform to the sort inaccessible for plants. This element participate in redox reactions as the electrons carrier or it can create an ion bridge between the enzyme and substrate. The catalysis of chlorophyll and some proteins is especially significant function of iron. The highest content of iron was stated in leaves. It is essential element for proper plant development.

Fig. 7a, b show that oak wood gained from polluted environment contains almost twice higher amount of iron than wood from unpolluted area. Iron content in bark is also higher in samples from polluted environment.

Krutul et al. (2007) were analyzed 120-year old oak (*Quercus petraea* Liebl.) wood which was sampled in unpolluted environment. Iron content in heartwood (79 $mg.kg^{-1}$) was higher than in sapwood ($mg.kg^{-1}$). This value for bark was $mg.kg^{-1}$ Krutul and Sacharczuk (1997) were examined 70 year old oak (*Quercus robur* L.) trunks from unpolluted environment. Iron content in heartwood varied from 10.3 to 12.3 $mg.kg^{-1}$, in sapwood from 7.5 to 7.9 $mg.kg^{-1}$, in bark from the butt-end section – 178 $mg.kg^{-1}$ and in bark from three-quarters of height – 143 $mg.kg^{-1}$. Presented data point that iron content may vary a lot apart from the environmental pollution. It means that changes in iron concentration observed in current paper don't have to be caused by pollution.

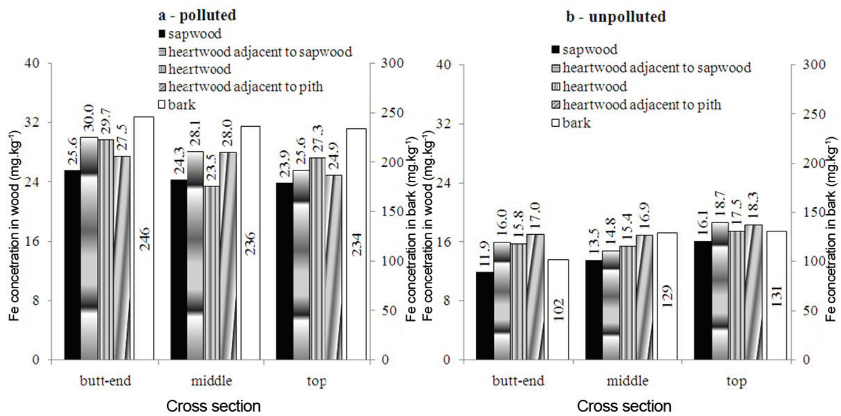

Fig. 7: Iron (Fe) concentration in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

Lead, mercury, cadmium and copper are the most toxic elements for plants. Their harmful acting is connected with the raised concentration in cells (Kopcewicz and Lewak 1998).

Copper is an element which is found in enzymes participating in redox reactions. It easily creates bonding with amino-acids and proteins, also takes a part in cell wall lignification. But the excess of this element leads to intoxication causing the iron deficiency, lipids peroxidation and roots growth inhibition.

Copper content is lower both in wood and bark in relation to iron. Copper content in wood and bark gained from polluted area is 30 to 50 % higher than in unpolluted samples. It is presented in Fig. 8a, b. According to Watmough et al. (1998), copper content in xylem of sugar maple (*Acer saccharum*) formed from 1916 to 1993 in the urban environment (city centre) is higher in relation to samples collected from rural woodlands. Samples from the urban area contains 0.3-3.5 mg.kg^{-1} of copper in the annual ring formed in 1916, and 1-5 mg.kg^{-1} of copper in the annual ring created in 1993. Krutul et al. (2007) stated that copper content in oak (*Quercus petraea* Liebl.) wood from unpolluted environment equals to 2.8 mg.kg-1 in sapwood, 2.5 mg.kg^{-1} in heartwood and 6.4 mg.kg^{-1} in bark.

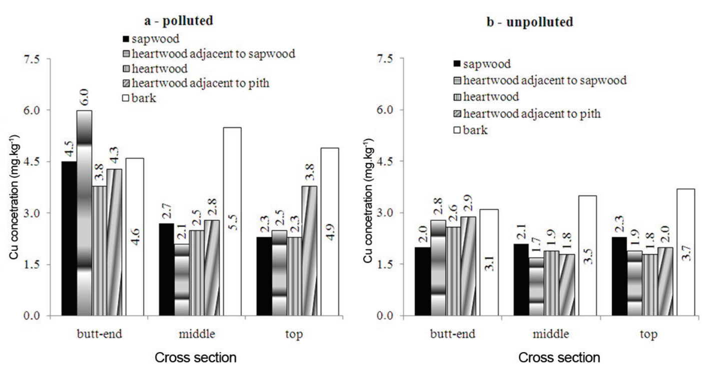

Fig. 8: Copper (Cu) concentration in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

Summarizing, copper content in wood above 5 mg.kg^{-1} seems to be the measure of environmental pollution.

Lead is not the essential element for proper plant development. Its presence testifies the environmental pollution. The highest lead content on the oak wood cross-section is stated in heartwood adjacent to sapwood. Only in the butt-end section it is higher in pith-adjacent heartwood. Watmough and Hutchinson (2002) stated that there is increased lead content in the frontier of pine heartwood and sapwood. The highest lead content is in the butt-end section of the pine trunk and it decreases in the direction to the top part. It is consistent with the results in the current paper.

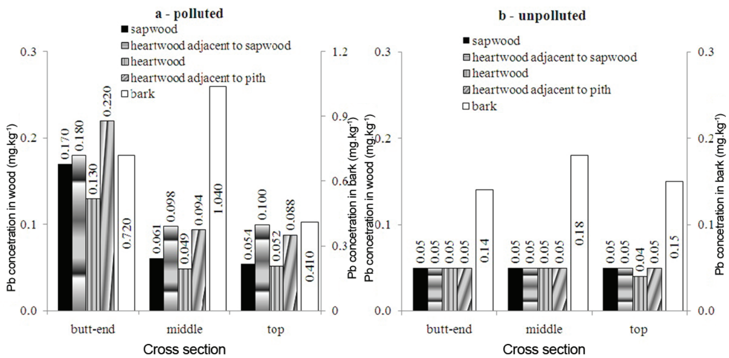

Fig. 9: Lead (Pb) concentration in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

Many other authors state that trace elements accumulate in the frontier of sapwood and heartwood (Okada et al. 1993a, b, Chun and Hui-Yi 1992). Also lead content in heartwood adjacent to sapwood in analyzed oak wood is higher than in other examined zones. It is presented in the Fig. 9a, b. According to Patrick et al. (2006), lead content in heartwood of more than 80-years old oak stays in the range of 0.03 – 1.77 mg.kg^{-1}.

Lead content in bark from polluted environment is several times higher in comparison to wood. It is also several times higher than in bark taken from unpolluted trunks. Oak wood gained from unpolluted area contains marginal or trace amounts of lead. It is compatible to the results of Krutul and Sacharczuk (1997) and Krutul and Makowski (2004). According to Watmough et al. (1998), lead content in annual rings of sugar maple (*Acer saccharum*) varies from 0.5 to 1.5 mg.kg^{-1}, depending on the environmental pollution.

The environmental pollution definitely influences lead content in wood and bark. Lead content may be an indicator of the pollution with taking into consideration factors causing contamination.

Fig. 10 presents cadmium content in examined trunks. It varies from 0.011 to 0.035 mg.kg^{-1} and is quite similar for both polluted and unpolluted samples. According to Watmough et al. (1998), wood of sugar maple (*Acer saccharum*) contains from 0.03 to 0.19 mg.kg^{-1} of cadmium.

Cadmium content in bark gained from polluted environment varies from 0.080 to 0.099 mg.kg^{-1}, which is almost twice higher in relation to bark taken from unpolluted trunks.

For comparison, cadmium content in oak wood (*Quercus petraea* Liebl.) from unpolluted area equals 0.021 mg.kg^{-1} in sapwood, 0.014 mg.kg^{-1} in heartwood and 0.15 mg.kg^{-1} in bark (according to Krutul et al. 2007).

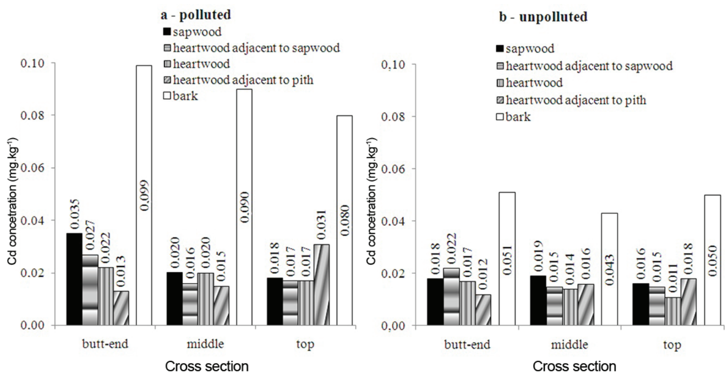

Fig. 10: Cadmium (Cd) concentration in bark and wood of oak (Quercus robur L.*) sampled from a) polluted, b) unpolluted environment.*

Presented data show that cadmium is not a good indicator of pollution in environment chosen for sampling.

CONCLUSIONS

Environmental pollution influences content of most examined substances and elements.

Extractives content on the cross-sections of analyzed trunks increases in the direction from perimeter to pith, apart from the habitat. However, concentration gradient is much more regular in samples from unpolluted area. Oak heartwood contains more extractives in case of polluted samples, while in sapwood and bark it is inversely. Sapwood and bark from polluted trunks contains more mineral substances in relation to unpolluted ones.

Nitrogen content in oak wood gained from unpolluted environment is higher than in samples from polluted area. It is higher in bark than in wood both from polluted and unpolluted samples.

Pollution influences the distribution and content of calcium and potassium in oak wood and bark on the cross- and longitudinal section.

Magnesium content is higher in sapwood from unpolluted area than in samples collected from polluted one, but differences in this element concentration decreases in the direction from the butt-end to the top part of a trunk. Bark gained from polluted area contains more magnesium than bark from unpolluted environment.

Iron content, both in wood and bark, just depends on tree habitat but not on the environmental pollution. Copper and cadmium content also cannot be treated as the pollution indicator.

Lead content in bark and wood is higher in samples from polluted environment. This element may be a good indicator of the pollution.

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Donata Krutul, Tomasz Zielenkiewicz, Janusz Zawadzki
Andrzej Radomski, Andrzej Antczak, Michal Drożdżek
Warsaw University of Life Sciences
Faculty of Wood Technology
Department of Wood Science and Wood Protection
Nowoursynowska 159,
02-787 Warsaw
Poland
Phone.: 0048225938642
Corresponding author: tomasz_zielenkiewicz@sggw.pl