

## **ECOTOXICOLOGICAL TESTS OF THE PARTICLEBOARDS CONTAINING RUBBER WASTE**

HELENA HYBSKÁ, MARTINA MORDÁČOVÁ, DAGMAR SAMEŠOVÁ,  
IVETA ČABALOVÁ  
TECHNICAL UNIVERSITY IN ZVOLEN  
SLOVAKIA

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### **ABSTRACT**

The article is focused on the production and environmental evaluation of wood composites using waste rubber in the construction industry. Used aqueous extracts were prepared from the experimental wooden composites with various additions of the waste rubber from tires and waste seals. The pH value and organic pollution (by COD) were determined in the aqueous extracts. The effect on the environmental components (aquatic and terrestrial) was ecotoxicologically tested using the test organisms *Sinapis alba*, *Lemna minor* and *Daphnia magna*. Preliminary acute ecotoxicity tests were performed.

**KEYWORDS:** rubber, tires, wood composites, ecotoxicity, automotive industry.

### **INTRODUCTION**

Wood-plastic composites (WPC) are used in many industries: for outdoor patios (Gardner et al. 2015), as harbour walkways, furniture, highway noise panels, outdoor tables, benches, or chairs (Pritchard 2004). Increasingly, these composites are also used in the automotive industry due to their excellent strength-to-weight ratio as well as stiffness to weight. The main market identified for the application of eco-friendly wood-plastic composites, replacing fiberglass and steel, is automotive components. Composites are used as decorative parts of dashboards, door panels, parcel shelves, seats, backrests, and cabin trim (Bismarc et al. 2006). Intensive research into the production of wood-plastic composites is conducted worldwide. Different materials are tried, as well as the proportions of the main raw materials. Shalbafan et al. (2016) compared the effect of different amounts of expanded polystyrene filler (5, 10 and 15%) on the properties of particleboards. The results showed that the use of polystyrene fillers has a significant effect on flexural properties, internal bonds, edge screw pullout resistance, thickness swelling and water

absorption. The produced wood-plastic composites are characterized by properties such as high resistance and low weight (Silva et al. 2014), and composites that contained more than one type of polymer had high strength (Bhaskar et al. 2021). The properties of the created composites are formed during production. For example, in the production of wood-plastic composites from waste plastics and wood, the maximum improvement in mechanical and physical properties was achieved at a temperature of 185<sup>o</sup> C (Basalp et al. 2020).

The increasing content of the waste from the production of automobiles and from the processing of old vehicles is also related to the constantly growing worldwide production of automobiles. Rubber and plastics represent about 30% of the vehicle's weight. At the end of the car's life, these parts will become waste. It is therefore necessary to look for ways to reuse them, they do not end up in the landfills. Several scientists focused their research on the production of wood composites containing waste tires. Research of Xu et al. (2020) is deal with wood fiber composites containing waste tire powders as functional fillers. Research has shown that it is possible to produce wood fiber composites with rubber filler that have added value and satisfactory properties. The research results of Zhao et al. (2010) showed that the sound-insulating properties of a wood-rubber composite panel from a waste tire are better than those of a commercial composite wood floor and particleboards.

Rubber microparticles can present varying degrees of the chemical toxicity because it depends on the chemical composition, particle type and size or other factors. The cause is possible chemical entrapment which come from the road or other abiotic factors (Sidhu 2021, Laplaca 2021, Šourková et al. 2021).

Sega (2021) deals mainly with the mechanical properties of WPC containing waste (plastics, rubber). Their environmental and ecological impacts are not sufficiently investigated. Wood fiber composites containing waste rubber are used indoors and outdoors. Their properties can change due to external influences (temperature, pressure, humidity, and others). The article traces their impact on the aquatic and terrestrial environment.

Assessing the health safety of construction materials is a current problem, especially with non-traditional construction materials using waste. These construction materials can release harmful and undesirable substances into contact media (water and soil). The leachates of these materials are used to assess the health safety of construction products in terms of their impact on the environment and, consequently, on human health. The issue is serious, which is why we deal with ecotoxicological testing of wood composites with incorporated plastic waste. The work experiment is aimed at assessing the impact of the wood composites prepared with waste from the automotive industry on the aquatic and terrestrial environment.

## **MATERIAL AND METHODS**

Granulate from waste rubber (carpets, isolators) was used in this study. The granulate consists of a mixture of processed materials as polyester, glass fibers, polyurethane, paper, ethylene propylene diene monomer (EPDM). Granulate was sized from 1.0 to 3.0 mm. The granulate from waste tires from the discarded automobiles was produced by AVE

SK-Kechnec plant Slovakia ([www.avesk.sk](http://www.avesk.sk)). The granulate consists of styrene-butadiene rubber (SBR). Granulate from the waste tires had a fraction size of 1.0 – 30 mm.

Wood particles 0.25 – 4.0 mm prepared from fresh spruce logs were processed by Kronospan s.r.o., Zvolen, Slovakia. The particles are commonly used for the core layer and for the single-layer particleboard production. A commercially available urea-formaldehyde (UF) resin Kronores CB 1100 F (Diakol Strážske s.r.o., Strážske, Slovakia) was used.

### **Composites processing**

European standard EN 309 (2005) defines particleboard as a “panel material manufactured under pressure and heat from particles of wood (wood flakes, chips, shavings, saw-dust and similar) and/or other lignocellulosic material in particle form (flax shives, hemp shives, bagasse fragments and similar), with the addition of an adhesive”.

Wood particles were dried to a moisture content of 4%. Particleboards were prepared with dimensions of 360×280×15 mm. The adhesive mixture was prepared with the solid content of 67.1%, viscosity of 460 mPa·s, condensation time of 55 s and a pH value of 8.6. Ammonium nitrate  $\text{NH}_4\text{NO}_3$  (47%) was used as a hardener. Paraffin, used as 35 wt% water emulsion, was applied on the particles in amounts of 0.6%. Such composition of the adhesive mixture was added to particles in an amount of 11 wt%. The moisture content of particles mixed with UF resins was 9.5%.

Particleboards were prepared using the pressing technology, i.e. firstly cold prepressing of particle mats at 1 MPa, followed by hot pressing in the laboratory press (CBJ 10-11, TOS, Rakovník, former Czechoslovakia) at a maximum temperature of the press 230°C, a maximum pressing pressure of 6.50 MPa, and a total pressing time of 356 s which had to be longer than in the conventional production of wood particleboards due to the presence of crushed waste rubber (Štefka 1998). Six boards of each condition were produced.

### **Experimental samples**

Single layer particleboards were made by replacing wood with 10, 15 and 20% waste rubber/seals (marked as sealing) or with 10, 15 and 20% waste tires (marked as tire). Particleboards without added waste was also used for testing as control samples (control). Wood composites were prepared in the laboratories of the Technical University in Zvolen, Faculty of Wood Sciences and Technology, Department of Furniture and Wood Products.

### **Preparation of aqueous extracts**

Aqueous extracts were prepared from each sample by leaching for 24 h and 48 h with demineralized water (a leaching agent) with pH adjusted to a value of 3. The volume of the leaching agent was calculated based on the size of the surface of the wood composites, which had the shape of a cuboid. To maintain the ratio of the values of the surface of the sample and the used demineralized water in the ratio of 1:5, the volume of the treated demineralized water was calculated according to the calculated surface, which was 2950 ml. The dimensions of the composites were the same. Each sample was placed separately in a glass container and a volume

of 2950 ml of extracts was added. After the leaching, samples of the aqueous extracts were taken and used for testing (Hybská and Samešová 2014, TNI CEN/TR 17105).

### Determination of selected indicators: pH, COD<sub>Cr</sub>

The pH value was determined using the WTW Ino Lab pH Level 3. SenTix 81® electrode (STN EN ISO 10523: 2008). By determining the COD<sub>Cr</sub>, the amount of the substances of the organic origin present in the sample is estimated. The process of this method consists in the oxidation of the organic substances found in water by means of potassium dichromate in a strongly acidic environment, which is formed by sulfuric acid during two hours of boiling (STN ISO 15705: 2005).

### Ecotoxicological tests

Tests were used to assess the harmful effects of substances released into water extracts: growth inhibition test with *Lemna minor* (Tab. 1), acute toxicity test on *Daphnia magna* (Tab. 2) and *Sinapis alba* (Tab. 3). Preliminary tests were carried out in all tests (www.alchetron.com, Hybská and Samešová 2015). Test conditions are listed in Tab. 1-3.

Tab. 1: The test conditions for *Lemna minor* (OECD 221, STN EN ISO 20079: 2008).

Test organism	<i>Lemna minor</i>
Biotest conditions	25°C ± 2°C, day and night simulation; continuous lighting with min. intensity 6,500 lux, thermostat ST FOT (Eko Pol Poland)
Control sample	Z-medium (nutrient solution prepared according to the instructions of the supplier CICALA, Třeboň, Czech Republic)
Reference substance	3,5-dichlórphenol, EC <sub>50</sub> = 2.9 mg/l (limit 2.2 – 3.8 mg/l)
Test duration	7 days
Preliminary test	15 leaf/sample
Validity of the test	mean number of leaflets in the control after the end of the test > than eight times at the beginning of the test, pH at the end of the test < than 1.5 compared to the input pH
Monitored response	growth rate (inhibition)

Tab. 2: The test conditions for *Daphnia magna* (OECD 202, STN EN ISO 6341: 1999).

Test organism	<i>Daphnia magna</i> Straus, individuals younger than 24 hours
Biotest conditions	21°C ± 2°C; 7.8 ± 0.2; laboratory conditions
Control sample	diluting water prepared from the solutions of CaCl <sub>2</sub> ·2H <sub>2</sub> O (1), p.a., MgSO <sub>4</sub> ·7H <sub>2</sub> O (2), p.a., NaHCO <sub>3</sub> (3), p.a., KCl (4), p.a.; by the addition of solutions (1) - (4) per 10 ml and adding demineralised water into a volume of 1 liter
Reference substance	K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> , EC <sub>50</sub> = 0.82 mg/l (limit 0.3 – 1.5 mg/l)
Test duration	48 h
Preliminary test	20 daphnia/sample (10 ml), same conditions for a control
Validity of the test	immobilisation ≤ 10 %, change of concentration of dissolved oxygen O <sub>2</sub> ≤ 2 mg/l
Monitored response	% of immobilised individuals

Tab. 3: The test conditions for *Sinapis alba* (STN EN ISO 83 8303: 1999).

Test organism	<i>Sinapis alba</i>
Biotest conditions	20°C ± 1 °C, thermostat TS 606 CZ/2-Var (WTW, Germany).
Control sample	Reconstituted water
Reference substance	K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> , IC <sub>50</sub> , 72 h = 28.5 mg/L (limit 4.1 to 85 mg/L)

Test duration	72 h
Preliminary test	per 30 seeds of <i>S. alba</i> L. in Petri dishes/10 ml sample
Monitored response	Inhibition of growth of root from <i>S. alba</i> compared with the control

## RESULTS AND DISCUSSION

### Growth inhibition test with *Lemna minor*

During the test, the signs of the necrosis or chlorosis were not observed. The growth rate  $\mu$  and its inhibition  $I\mu$  was calculated from the recorded number of the leaves (Tab. 4 and 5).

The results of the preliminary test are positive because the growth rate inhibition was  $\geq 30\%$ . The values of  $I\mu$  in extracts from the wood composites with the addition of the granulate from waste rubber/seals are lower compared to the wood composite without the addition of the waste, but there is no significant difference between them. With increasing addition of the waste to the wood composites,  $I\mu$  decreases (OECD 221, STN EN ISO 20079: 2008). Fořt et al. 2022 and Hybská et al. 2021 also dealt with the effect of the tire extracts on the aquatic environment and confirmed the inhibitory effect on growth rate.

Tab. 4: Basic characteristics of the test using *Lemna minor* in aqueous extracts after 24 h.

Sample composition	$I\mu$ (%)	
	Average	Standard deviation
Control	79.18	2.13
10% sealing	75.14	1.91
15% sealing	75.55	2.33
20% sealing	70.15	1.73
10% tire	78.91	0.71
15% tire	71.55	2.26
20% tire	67.31	1.59

Tab. 5: Basic characteristics of the test using *Lemna minor* in aqueous extracts after 48 h.

Sample composition	$I\mu$ (%)	
	Average	Standard deviation
Control	91.41	2.73
10% sealing	90.96	1.22
15% sealing	79.66	2.72
20% sealing	73.15	2.85
10% tire	90.96	1.22
15% tire	79.66	2.72
20% tire	74.91	2.34

### Inhibition test of *Sinapis alba* root growth

The root growth inhibition (IC) was calculated from the measured lengths of *Sinapis alba* roots compared to the control. The values are in Tab. 6-7.

Tab. 6: Basic characteristics of the bioassays using *Sinapis alba* in aqueous extracts after 24 h.

Sample composition	IC (%)	
	Average	Standard deviation
Control	58.77	5.99

10% sealing	46.26	2.75
15% sealing	40.25	1.42
20% sealing	31.25	0.74
10% tire	51.21	5.82
15% tire	44.26	3.46
20% tire	41.87	6.84

Tab. 7: Basic characteristics of the bioassay using *Sinapis alba* in aqueous extracts after 48 h.

Sample composition	IC (%)	
	Average	Standard deviation
Control	76.23	5.21
10% sealing	61.07	2.10
15% sealing	54.46	2.86
20% sealing	43.74	1.65
10% tire	56.24	2.89
15% tire	43.58	1.92
20% tire	23.09	3.38

The preliminary test shows that the inhibitions of the root growth (compared to the control) with the addition of the wastes are lower than in the samples without the addition of the automotive waste. Also in this test, the influence of the wastes on the reduction of the inhibitory effect on the test organisms was recorded. We assume that a higher addition of the waste to the wood composites causes changes in the composition of the aqueous extracts. The wood content is lower and less water-soluble substances dissolve in the extracts. This is evidenced by the determined  $COD_{Cr}$  values (Tab. 9), with the concentration decreases with the increasing proportion of the waste in wood composites compared to the sample without the addition of the waste. Changes in the content of the organic pollution in extracts due to time have also been recorded. The samples with root growth inhibition  $\geq 30\%$  compared to the control are positive, except for the sample with 20% granulate from the waste tires. Fořt et al. 2022 determined a terrestrial test with *Sinapis alba* to inhibit the aqueous extracts from tires by 31%. Our determined IC values (%) are higher and we assume that the higher inhibition is caused by the substances dissolved in the water extract from the wood (STN EN ISO 83 8303: 1999).

#### Acute toxicity test on *Daphnia magna*

The result of the preliminary test is positive (Tab. 8). During the test, death or immobilization was  $\geq 50\%$  (Hybská and Samešová 2015, OECD 202, STN EN ISO 6341: 1999).

Tab. 8: Immobilization of *Daphnia magna* in 24 h and 48 h aqueous extracts.

Sample composition	Imobilisation (%)	
	for 24 h	for 48 h
Control	100	100
10% sealing	70	70
15% sealing	93	93
20% sealing	100	100
10% tire	90	90
15% tire	100	100
20% tire	87	87

There are several studies (Panko et al. 2013, Wik and Dave 2005) in which the authors also used the test organism *Daphnia magna* in testing the toxicity of tire extracts. Like our testing, these organisms were very sensitive to the composition of the aqueous extracts. These studies recommend using the results from such testing as a basis for the eco-labeling of the automotive tires.

#### Determination of pH and COD<sub>Cr</sub>

From Tab. 9 shows that the pH value in all extracts was acidic. Due to the influence of the addition of the waste to the wood composites and the influence of the leaching time, a slight decrease in acidity was noted. We assume that the higher acidity in the extracts from the particleboards samples is caused by the degradation of acetylated hemicelluloses.

Tab. 9: Selected physico-chemical indicators in aqueous extracts after 24 h and 48 h.

Sample composition	pH		COD <sub>Cr</sub> (mg.l <sup>-1</sup> )	
	for 24 h	for 48 h	for 24 h	for 48 h
Control	3.66	5.11	1655.28	1993.86
10% sealing	4.24	4.87	1053.36	1956.24
15% sealing	4.22	4.69	978.12	1918.62
20% sealing	4.52	4.97	902.88	1918.62
10% tire	4.33	4.57	902.88	1580.04
15% tire	4.42	4.58	865.26	790.02
20% tire	4.12	4.95	865.26	639.54

The highest average COD<sub>Cr</sub> value is in the wood composites without the addition of the waste (particleboards). The addition of the seals and tires in PB caused a reduced content of the substances of the organic origin without the influence of the type of the waste used compared to PB. In the case of PB with the addition of the waste tires after 24 h of leaching, there are up to about 50% less organic substances in the extracts compared to pure PB. In these samples, based on the determined COD<sub>Cr</sub>, the effect of the percentage replacement of the wood with the tire waste in the production of the wood composites was noted. The effect of percentage substitution of wood with plastic waste on the fire properties of chipboard has not been proven (Mancel et al. 2022).

The solubility of any metals contained in the samples containing tires is also affected by the size of the particles, because with a smaller particle size, a greater amount of the elements is leached due to an increase in the surface area. Their higher amount in the aquatic environment can cause a toxic effect for aquatic organisms (Laplaca 2021).

## CONCLUSIONS

During the test with the test organism *Lemna minor*, no signs of necrosis or chlorosis were recorded on its leaves. The preliminary test results were positive and were higher in the 48 h extracts. With increasing addition of the waste to the wood composites, I<sub>μ</sub> decreased. A terrestrial test using *Sinapis alba* revealed an inhibitory effect of the waste on the plant root

growth (compared to the control). We assume that a higher addition of the waste to the wood composites causes changes in the composition of the aqueous extracts. The wood content is lower, and thus less water-soluble substances are leached into the extracts. From a chemical point of the view, wood is composed of the biopolymers of a lignin, cellulose, hemicellulose as the majority organic components. There is more wood in the samples without the addition of the waste, so the values were set higher. Aquatic organisms *Daphnia magna* reacted most sensitively to the composition of the aqueous extracts. All the results were positive, and 100% immobilization was noted in almost all samples. The pH value was acidic in all extracts. Due to the influence of the addition of the waste to the wood composites and the influence of the leaching time, a slight decrease in acidity was noted. We expect that the higher acidity in the extracts from the PB samples was due to the degradation of acetylated hemicelluloses. The highest average value of COD<sub>Cr</sub> was in the wood composites without the addition of the waste (pure PB). The addition of the seals and the tires in PB reduced the content of the substances of the organic origin without the influence of the type of waste used, compared to pure PB.

It can be concluded that the addition of the waste rubber from tires and seals from the automotive industry to the wood composites, even though the test results were positive in most cases, shows in these samples more acceptable results in terms of the impacts on the aquatic and terrestrial environment than wood composites without the addition waste. Because the wood composites have been used in practice for a long time and have the prospect of further use as building materials, the use of such waste from the automotive industry represents a suitable opportunity for their valorization. Wood-based composites are a substitute for solid wood. Their production can contribute significantly to waste recovery, as they usually contain wood waste.

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HELENA HYBSKÁ\*, MARTINA MORDÁČOVÁ, DAGMAR SAMEŠOVÁ  
TECHNICAL UNIVERSITY IN ZVOLEN  
FACULTY OF ECOLOGY AND ENVIRONMENTAL SCIENCES  
DEPARTMENT OF ENVIRONMENTAL ENGINEERING  
T. G. MASARYKA 24, 960 01 ZVOLEN  
SLOVAKIA

\*Corresponding author: [hybska@tuzvo.sk](mailto:hybska@tuzvo.sk)

IVETA ČABALOVÁ  
TECHNICAL UNIVERSITY IN ZVOLEN  
FACULTY OF WOOD SCIENCES AND TECHNOLOGY  
DEPARTMENT OF CHEMISTRY AND CHEMICAL TECHNOLOGIES  
T. G. MASARYKA 24, 960 01 ZVOLEN  
SLOVAKIA