# The Effect of Particle Board Industry Waste Tar on the Physical and Biological Durability of Wood

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The effect of waste tar from particle board factories was investigated relative to some physical and biological resistance properties of Scots pine (*Pinus sylvestris*) and beech (*Fagus orientalis*) woods. Solutions were prepared by dissolving waste tar in ethanol:toluene (1v:1v) in concentrations of 5%, 10%, 15%, and 20%. These solutions were forced deep into the Scots pine and beech woods under vacuum and pressure (deep treatment, DT). In addition, surface coating (SC) was applied by spreading 96% waste tar on the wood surfaces after treatment. Deep-treated and surface-coated (DT+SC) wood samples were exposed to the wood-decay fungi *Trametes versicolor* L. and *Neolentinus lepideus* Fr. for 12 weeks. At the same time, sample blocks were tested against wood-destroying house borer (*Hylotrupes bajulus*) larvae for 16 weeks. Total phenolic content, water uptake, water-repellent efficiency, and surface contact angle were tested. Although a mean mass loss resulting from *T. versicolor* of 31.1% was seen in the Scots pine control samples, only 3.87% mass loss was found with a concentration of 20% (DT + SC*).* The *H. bajulus* larvae mortality rate was 80% in the Scots pine wood samples deep-treated with 20% waste tar.

*Keywords: Waste tar; Wood protection; Water repellent; Wood decay fungi; Wood destroying insect*

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## INTRODUCTION

In response to increasing demand for particle board, the production of particle board in various countries is also increasing. In the particle board industry, the moisture content must be reduced to 1 to 3% before the particles are glued. For this purpose, the wood particles are placed in dryers that produce the heat through burning solid and liquid fuel in the combustion chambers. Surface sanding powders in particular are widely used for this purpose in factories. However, at high temperatures (1000 °C and above), these powders become carbonized and volatile and are trapped in the vacuum filters. In addition, particles are dried at high temperatures (450 °C) in the drying line. However, with the use of steam, volatile components and fine powders in the particulates are captured and contained in the vacuum filters. Thus, harmful pollutants are prevented from dispersing into the environment. The compounds and carbon coming from both the combustion chamber and the drying unit are trapped by the filters (wet electrostatic precipitator, WESP) during these processes. In the particle board factories, these wastes are collected in the form of molten tar (World Bank 2007). It is estimated that in a factory with a production capacity of 510 thousand $m^3$/year, 800 tons of waste tar are produced in Turkey. An average of 96 million $m^3$/year of particle board is produced worldwide (FAO 2016), and it is estimated that approximately 150 thousand tons of waste tar is produced. The waste tar is generally not evaluated for any purpose or process within the factory and is disposed of by the factories

*via* combustion or by enterprises that charge a fee to dispose of the solid waste. In 2019, the disposal cost of waste tar for factories was 120 $/ton. Disposal of waste, therefore, creates high costs for these factories.

Due to its nature, wood is easily degraded by fungi, termites, and other insects (Lonsdale *et al.* 2008). Many methods have been used in the wood preservation industry to protect wood materials (Richardson 2002; Hill 2006). The copper, chromium, arsenic (CCA) wood preservative has been used frequently due to its high degree of toxicity to fungi and insects (Kartal and Clausen 2001; Mercer and Frostick 2012). As a result of increasing environmental pressure and legal regulations, CCA has been withdrawn from residential applications (Clausen 2004), and environmentally friendly impregnation materials have been investigated (Hsu *et al.* 2009; Brocco *et al.* 2017). Plant-based extracts and tannins with antifungal and insecticidal properties have attracted the most attention (Valette *et al.* 2017). It was stated that the phenolic compounds contained in the extracts and tannins are the responsible for the antifungal properties (Akcay *et al*. 2020). Sen *et al.* (2017) impregnated beech, poplar, and pine wood samples with mimosa, quebracho, and red pine bark extracts and tested their resistance against *Spondylis buprestoides* larvae. Using the same extracts, Tascioglu *et al.* (2012) investigated wood species resistance against *Reticulitermes grassei* termites and later tested the antifungal properties of these extracts (Tascioglu *et al.* 2013). As a result, these extracts and tannins acted as biocides against wood-decay fungi and wood destroying insect larvae. In the literature, the high protective effects of phenolic compounds, benzene and various complex components in the structure of tar oils against biological degradation are discussed in detail (Mazela 2007). Tar and its derivatives have a long history of usage as antifungal agents in wood protection industry. The product application and its positive results were described by Kollmann *et al*. (2012). Tar oil and wood tar application was mentioned in Genesis 6:14 of the Holy Bible (Lutomski 1997). Tar oils have some advantages such as high toxicity to wood-destroying organisms, its insolubility in water, its low cost, its depth penetration to wood, and its oily nature that retards moisture changes. However, there are some disadvantages of tar as well. Fresh tar oil impregnated timber can easily ignite and burn easily by producing dense smoke (Blew 1953).

The present study investigated the possibilities of the waste tar produced in particle board factories being transformed from an expense into a profitable by-product for these enterprises. The effects of waste tar on some physical and biological resistance properties of wood were tested on a laboratory scale.

## EXPERIMENTAL

### Wood Samples and Waste Tar

Scots pine (*Pinus sylvestris*) and beech (*Fagus orientalis*) wood specimens were selected in accordance with EN 113 (1996) and prepared from sapwood in dimensions of $50 \times 15 \times 5$ mm$^3$, $50 \times 25 \times 15$ mm$^3$, $30 \times 30 \times 15$ mm$^3$, and $25 \times 25 \times 25$ mm$^3$ (longitudinal × radial × tangential) for decay tests, larvae tests, water uptake (WA)/water repellent efficiency (WRE), and contact angle test, respectively. Wood species were supplied from a sawmill in Düzce province, Turkey. Both wood species specimens were conditioned at $20 \pm 2$ °C and $65 \pm 2$% relative humidity (RH). Waste tar was obtained from the chip drying section of a particle board factory in Turkey, where it had been covered with wood dust and stored at the factory site. The waste tar (Fig. 1a), consisting of a solid content of 85%,

was stored in the laboratory refrigerator at 5 °C. Experiments were carried out to dissolve the waste tar using hot water, cold water, methanol, ethanol, urea solution, acetone, and toluene solvents. However, it was determined that toluene and ethanol gave better solubility results than the other solvents (Fig. 1b). Therefore, in the current study, an ethanol:toluene (1v:1v) solvent mixture was used.

The impregnation solution was passed through filter paper and prepared at 5, 10, 15, and 20% concentration. In addition, the concentration of the waste tar solution for the surface coating (SC) application was increased to 95% by removing the solvent. The details for each application and process used in the study are given in Table 1.

To determine whether the factor providing the activity in the fungus and insect tests was caused by the solvent or the waste tar, some wood samples were treated with the solvent (SA) and exposed to the fungi and larvae.

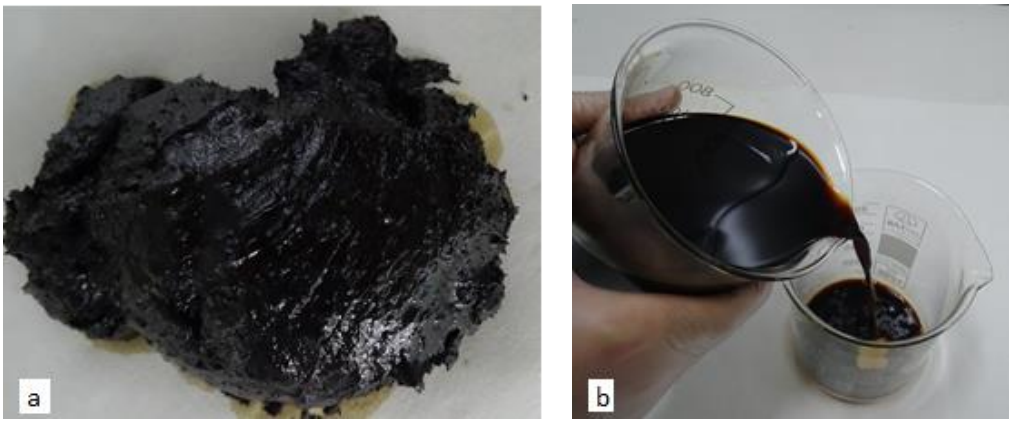


**Fig. 1.** a) Waste tar; b) Waste tar dissolved in toluene and ethanol mixed solvent.

**Table 1.** Details of Tests and Samples Used in the Study

| Tests | Sample Dimensions ($mm^3$) | Number of Replicates | | | | |
|---|---|---|---|---|---|---|
| | | Untreated | Solvent | DT | SC | DT + SC |
| Decay | 50 × 15 × 5 | 12 | 12 | 48 | | 48 |
| Larval test<br>Mature<br>Newly hatched | <br>50 × 25 ×15<br>50 × 25 ×15 | <br>6<br>6 | <br>6<br>6 | <br>18<br>18 | <br>-<br>- | <br>-<br>- |
| Water absorption | 30 × 30 ×15 | 6 | - | 18 | 6 | 18 |
| Water repellent efficiency | 30 × 30 × 15 | 6 | - | 18 | 6 | 18 |
| Contact angle | 25 × 25 × 25 | 6 | - | 6 | - | - |

DT: Deeply treated, SC: Surface coating

## Deep Treatment and Surface Coating Application

Scots pine and beech samples prepared for biological and physical tests were deep-treated (DT) with tar solutions at concentration levels of 5, 10, 15, and 20%. Surface coating was applied using concentrated tar (95%) after deep treatment (DT + SC) in some groups. The DT process was carried out in a cylindrical vacuum-pressure impregnation tank. All samples were dried at 103 ± 2 °C for 24 h, and the dried weights ($M_0$) were recorded before treatment. The test specimens were kept under a vacuum of 0.079 MPa for 20 min. Afterwards, the tar solutions were introduced into the impregnation tank under

vacuum and pressure applied at 1.21 MPa for 20 min. Excess tar solution on the surface of the treated wood samples was wiped off with a paper towel, and the samples were re-weighed ($M_1$). The retention ($R$) amount of the samples was calculated using Eq. 1. All samples were conditioned at 20 ± 2 °C and 65 ± 2 % RH for four weeks after treatment.

$$R = \frac{(M_1 - M_0) \times C}{V} \times 10 \quad (\text{kg/m}^3) \tag{1}$$

In Eq. 1, $M_0$ (g) is the weight before treatment, $M_1$ (g) the weight after treatment, $C$ the solution concentration, and $V$ the volume ($m^3$) of the wood blocks.

## Chemical Analysis

*High performance liquid chromatography (HPLC) analysis*

Phenolic profile analysis was carried out on a Shimadzu 20 A HPLC device (Kyoto, Japan). Chromatographic separations were carried out with an ACE C18 column (250 mm × 4.6 mm × 3 µm) having a protection column (4.0 mm × 10 mm × 2 mm). Phase A (0.1% formic acid water) and phase B (acetonitrile) were used as the mobile phases with the gradient flow. For the phenolic profile, 27 phenolic substances (gallic acid, chlorogenic acid, caffeine, vanillic acid, and rutin, *etc.*) were used.

*Total phenolic content*

In addition to the total number of phenolic compounds determined in the waste tar solution, the total phenolic content in the 1, 4, 16, and 48 h leaching periods was determined for the wood samples after DT application. In addition, the total phenolic content in the leaching water of the untreated Scots pine control samples was also analyzed and compared to the total 48 h leaching period. The NFX 41 568 (2014) standard was taken into consideration in the leaching process. Total phenolic content was determined *via* the Folin-Ciocalteu method (Ainsworth and Gillespie 2007). The results of the three replicates for the liquid samples were expressed as catechin equivalents (CE).

## Physical Tests

*Water absorption (WA) and water-repellent efficiency (WRE)*

A total of 96 samples from DT, SC, and DT + SC applications were used for WA and WRE tests. Control samples having the same annual rings and the samples deep-treated with the waste tar solution (DT) were dried to constant weight at 103 ± 2 °C; their dry weights were determined on a balance having a sensitivity of 0.01 g. Test and control samples were immersed in water with 20 ± 1 °C, and a stone was placed on them to ensure they remained below the water surface. The values of WA of the test and control samples were measured at the end of water holding periods of 2 and 72 h. At the end of each period, the samples were removed from the water container and wiped off with a paper towel. The samples were weighed, and the amount of absorbed water ($A_{bs}$) was recorded. The initial dry-weight test sample ($P_{ao}$) or control sample ($A_o$) and the $A_{bs}$ values were used to calculate the WA according to Eq. 2 for each test and control sample separately for each period (Broda 2018).

$$WA = \frac{A_{bs} - P_{a0}(\text{or } A_0)}{P_{a0}(\text{or } A_0)} \times 100 \tag{2}$$

The WRE value was expressed as a reduction of the *WA* values of the wood samples impregnated with waste tar in different concentrations compared to the control samples. The *WRE* was calculated using Eq. 3 for each test separately for each period,

$$WRE = \frac{(WA_c - WA_t)}{WA_c} \times 100 \quad (3)$$

where $WA_c$ is the *WA* (%) of the control sample at the end of a specified period and $WA_t$ is the *WA* (%) of the test sample at the end of a specified period.

*Surface contact angle measurements*

Contact angle measurements were carried out using the Attention Theta Contact Angle Meter device (Nanoscience, Phoenix, USA). The contact angle was measured at the end of 1 s after 5 µL of Merck water was dropped onto the solid surface. For this purpose, wood samples (untreated and DT) in cubic form (25 × 25 × 25 $mm^3$) were used. Three different measurements were averaged for each surface.

## Biological Tests

*Decay tests*

Decay tests were carried out following the principles specified in EN 113 (1996). Treated Scots pine (*Pinus sylvestris* L.) and beech (*Fagus orientalis* L.) samples having dimensions of 50 × 15 × 5 $mm^3$ were used. A total of 120 samples, 60 for Scots pine and 60 for beech, were used in the decay tests.

White rot fungus of *Trametes versicolor* L. (TV) and brown rot fungus of *Neolentinus lepideus* Fr. (NL) were used. The cultures for the antifungal tests were prepared on a malt extract agar medium and grown in petri dishes. Before the decay tests, the untreated and treated samples were sterilized in an autoclave at 121 ± 2 ºC and 1.1 atm. Any extraction was not seen from the treated wood during the sterilization process. After mycelium development was completed in the petri dishes in a laminar air cabinet, the sterilized samples were placed on the culture media on thin wooden supports to exclude direct contact with fungi. Test samples were kept in an incubator at 25 to 27 ºC and 70 to 80% RH for 12 weeks. At the end of the decay testing, the fungi mycelium were cleaned from the test samples and the weight losses due to the fungi were calculated with Eq. 4,

$$\text{Weight loss } (\%) = [(W_0 - W_1)/W_0] \times 100 \quad (4)$$

where $W_0$ (g) and $W_1$ (g) are the weight before and after the decay test, respectively.

*Larvae tests*

Larvae tests were conducted according to specifications in EN 47 (2016). This method is the criterion for evaluating the effectiveness of the treatment solution against *Hylotrupes bajulus* larvae. In the tests, Scots pine sapwood samples in dimensions of 50 × 25 × 15 $mm^3$ were impregnated with waste tar (solvent, 5, 15, and 20%) and the untreated control samples were used. Six replicates for each group (a total of 30 samples) were tested. Larval tests were performed using six-month-old mature larvae obtained by mating adult female and male *Hylotrupes bajulus* insects under laboratory conditions in the Duzce University Forest Biology and Wood Protection Laboratory. Holes with 4 mm in width and 20 mm in depth were drilled in the tar-treated and untreated wood samples. Larvae were weighed and introduced into the drilled holes in a head-down position and the holes were then plugged with cotton wool. Test specimens were kept in an incubator test cabin at 27 ± 2 °C and 80 ± 2 % RH for 21 weeks. At the end of the test period, all samples were then broken open and the dead and live larvae were noted. Larvae mortality rates were recorded in the control and treated samples. The live larvae were re-weighed, and the larval weight change was obtained *via* Eq. 5,

$$\text{Larvae weight change } (\%) = [(W_0 - W_1)/W_0] \times 100 \quad (5)$$

where $W_0$ is the larvae weight before test, and $W_1$ the weight after larvae test.

The mature larvae and newly hatched larvae were examined, and the mortality rates were compared. Five newly hatched larvae were used for each concentration tested (a total of 25 larvae). Wood samples were kept at 27 ± 2 °C and 80 ± 2% RH for 24 weeks. At the end of the experiment, mortality rates of the larvae were calculated for the newly hatched larvae used in the experiment (Eq. 6).

$$\text{Mortality rate } (\%) = [(N_f/N_t) \times 100 \quad (6)$$

where $N_t$ is the number of total larvae inserted the surfaces of wood, and $N_f$ is the number of dead larvae after the test.

### Statistical Analysis

The data were analyzed at 95% confidence level using SPSS software (IBM Corporation, Armonk, New York, USA). The retention rates of the different tar-treated tree species and weight losses caused by decay fungi were evaluated. All variations were evaluated together and analyzed by multiple variance analysis. When the differences between the factors were found to be statistically significant, the Duncan mean separation test was applied at a level of $\alpha = 0.05$ to compare the averages.

## RESULTS AND DISCUSSION

### Retention Amounts of DT Samples

The retention amounts of the Scots pine and beech wood treated with waste tar solution are shown in Table 2. When the concentration level of waste tar was increased from 5% to 20%, the retention amount increased from 24.9 kg/m$^3$ to 88.1 in Scots pine, from 19.7 to 71.2 in Beech. Retention in the Scots pine was found higher than in beech. Retention results gave values similar to those reported in the literature (Mazela 2007).

**Table 2.** Retention Amounts of DT Samples

| Concentration of Waste Tar | Scots Pine | | Beech | |
|---|---|---|---|---|
| | Mean retention (kg/m$^3$) | Std. dev. | Mean retention (kg/m$^3$) | Std. dev. |
| 5% | 24.9 *a* | 1.5 | 19.7 *a* | 1.3 |
| 10% | 37.2 *b* | 2.3 | 31.1 *b* | 1.7 |
| 15% | 61.3 *c* | 3.7 | 54.4 *c* | 2.8 |
| 20% | 88.1 *d* | 7.0 | 71.2 *d* | 3.8 |

### Chemical Analysis of Waste Tar

The HPLC chemical analysis results of the waste tar are given in Table 3. The active compounds in the waste tar consisted of benzene and its derivatives, which are the main components of lignin and phenolic compounds. The p-xylene content was found at the highest level in the waste tar solution (72.3%), followed by m-xylene (19.1%). Other components were found at low levels.

**Table 3.** GC-MS Results for Waste Tar

| Active Compounds | Rate (%) | Retention Time (min) | Scan number | Area (Ab*s) |
|---|---|---|---|---|
| p-xylene; Benzene, 1,4-dimethyl; p-Dimethylbenzene; p-Xylol; Chromar | 72.32 | 8.938 | 935 | 3045851 |
| Benzene, 1,3-dimethyl-; m-xylene; m-Dimethylbenzene; m-Xylol | 19.07 | 10.588 | 1253 | 803286 |
| 4-(4-Chlorophenyl)-6-phenyl-2-pyridone | 0.36 | 55.797 | 9966 | 15278 |
| Cyclooctasiloxane, hexadecamethyl-; Hexadecamethyl-cyclooctasioxane | 0.31 | 65.448 | 11826 | 13011 |
| (+)1a7,4a,9,10,10a-dodecahydro-1,4a,7-trimethyl-7-vinyl-1-phenanthrencarbal | 3.15 | 93.031 | 17142 | 132750 |
| 9,10-Secochola-5.7.10(19)-triene-3,24-diol,(3.beta.5Z,7E)- (CAS) | 1.19 | 97.311 | 17967 | 50006 |
| 4,5-Methanochrysene; 4,5-methylenechrysene; 4h-cyclopenta(def)chrysene | 1.79 | 99.335 | 18357 | 75517 |
| 2-Hydroxychalcone; omega.-(Salicylidene)acetophenone | 0.35 | 103.994 | 19255 | 14781 |
| Methyl dehydroabietate, Methyl 8.11.13-abietatrien-18-oate | 1.45 | 107.932 | 20014 | 61188 |
| Total | 100 | | | |

The ICP-MS analysis of the waste tar showed that the highest elemental content (11,100 mg/kg) was element K, while element Co (0.01 mg/kg) was the lowest. Moreover, Ca (5790 mg/kg), Na (2100 mg/kg), P (1520 mg/kg), and Mg (1080 mg/kg) were found in significantly higher amounts than the other components. In addition, Ni (8.4 mg/kg), Fe (463 mg/kg), B (130 mg/kg), Cu (82 mg/kg), Mn (153 mg/kg), and Zn (394 mg/kg) were also detected.

In the chemical analyses of waste tar, various active compounds and elements were determined. The antifungal and insecticidal (toxic) effects of aromatic compounds and elements (K, Na, B, Cu, and Zn) identified in waste tar content have been reported in previous studies (Dean 1985; Bergauer *et al*. 2005; Li *et al*. 2012; Akpuaka *et al*. 2013; Hu *et al*. 2013; Suriani 2016).

## Leaching

The total phenolic content was determined in the control and treated samples, which were subjected to leaching over different time periods (Table 4).

**Table 4.** Total Phenolic Content after Leaching

| Total Phenolic Content | mg CE/L Sample |
|---|---|
| Tar solution | 841.53 ± 22.38 |
| Untreated leachate (48 h) | 1.30 ± 0.09 |
| Treated leachate (1 h) | Not detected |
| Treated leachate (4 h) | < 1.00 |
| Treated leachate (16 h) | 6.46 ± 0.37 |
| Treated leachate (48 h) | 16.33 ± 0.42 |

As a result, 842 mg CE/L phenolic substance was found in the tar solution. In the control samples (untreated), 1.3 mg CE/L total phenolic substance was found after 48 h of leaching. Phenolic substances could not be detected or were found in trace amounts after 1 and 4 h of leaching of the treated samples.

As a result of leaching tests, with the increase in leaching time, the total amount of phenolic substances that leached into the water increased. These increases were not significant, however, at the end of 48 h. The phenolic substances accounted for about 2% of the total phenol in the tar solution. This was because the hexadecamethyl siloxane in the structure of the tar inhibited the leaching of phenolic compounds from the wood by forming a barrier on its surface (Mahlberg *et al.* 1998; Denes *et al.* 1999). Moreover, the fact that the tar solution exhibited high water-repellent efficiency was also thought to be an effective factor in the emergence of this finding (See water-repellent activity figures).

## WA and WRE Change

The effects of different concentrations of DT, and DT + SC on WA values are shown in Fig 2. The WA values at 2 and 72 h were determined as 50% and 80%, respectively, for the untreated control samples. The WA values decreased to 20% and lower at 2 h, while rates of 50% and lower were seen at 72 h with the DT application. In this application, with the increase in the concentration level, some decrease was observed in terms of the WA value; however, this decrease was not statistically significant. With the DT + SC application, WA values decreased to 15% and below at 2 h, while they were 45% and lower at 72 h. The lowest WA values were found at 15% and 20% concentration levels with the DT + SC application, a statistically significant difference from all other application levels. Thus, the DT, SC, and DT + SC applications were effective on WA value. However, it was determined that the SC application, in particular, was more effective than the DT application.

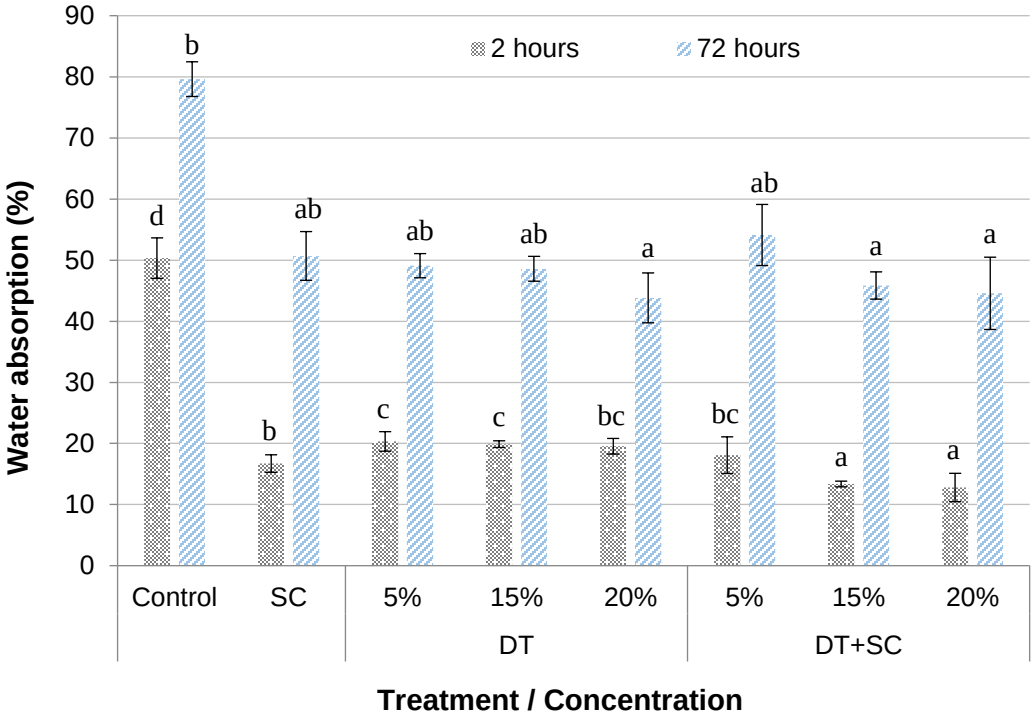


**Fig. 2.** WA values of different tar applications on wood

When the treatment applications were evaluated in terms of water-repellent efficiency (WRE), the highest values were determined in the 20% DT + SC for both the 2 and 72 h immersion times (Fig. 3). The lowest WRE value was found in samples with only the SC application without DT in the 2 h immersion time. However, when the immersion time increased from 2 h to 72 h, the SC application obtained a higher WRE value.

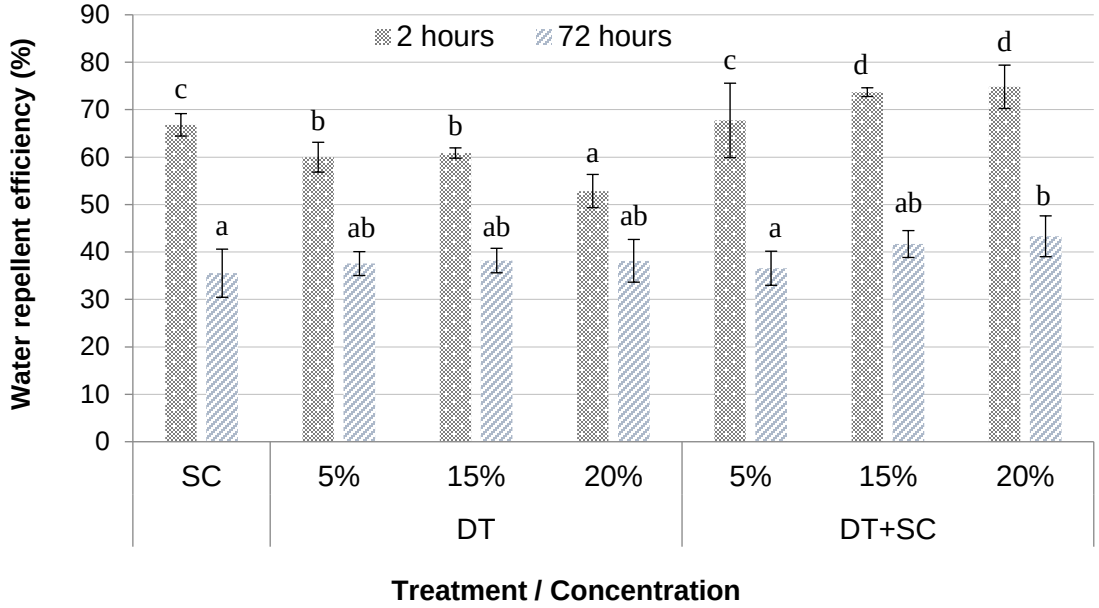


**Fig. 3.** WRE values of treated samples compared to control samples

The WA and WRE tests were performed to determine the effect of waste tar on some physical properties of wood. It was thought that due to extractives in the waste tar, the WA value had been reduced and the WRE increased. In a comparison of the dimensional stabilization of the heartwood and sapwood samples in their study, Bossu *et al.* (2016) found that heartwood is more stable than sapwood. Dimensional stability was higher due to the greater amount of extractives in the heartwood than in the sapwood. Jankowska *et al.* (2017) reported that ethanol-soluble extractives reduced the fiber saturation point to 18.7% for ipé (*Tabebuia* sp.) wood. The WA value is affected by the amount of extractives, oils, the porosity of the wood, tyloses, the ratio of early wood and latewood, ray formation, and intercellular structures of wood (Shukla and Kamdem 2010).

Another factor that affects the WA and WRE is thought to be the hexadecamethyl-cyclooctasioxane content of the waste tar (Table 2). By polymerizing on the wood surface, siloxane-containing hexamethyldisiloxane renders wood hydrophobic. The cross-linking between Si-O-Si and Si-O-C in the structure of the component forms a barrier against water on wood surfaces. Hexamethyldisiloxane has been used effectively in the modification of wood plasma (Kocaefe *et al.* 2015).

There was an increase in WRE after treatment with tar due to the xylene (Table 2), which is also found in the composition of tar. Ramsden *et al.* (1997) investigated the dimensional stabilization and WRE values by modifying Scots pine wood with acetic anhydride in xylene. At the end of the study, the acetic anhydride in the xylene was bound to the hydroxyl groups and changed the structure of the cellulose, hemicellulose, and lignin. The modification of the wood ultimately increased the WRE.

The reason for greater water-repellent activity in SC samples can be explained by the fact that the samples are filled with more impregnation fluid into the cell lumens and a

thin film layer forms on the surface of the cell lumens.

## Contact Angle Measurement

Contact angle measurement was carried out to determine the wettability and water repellency properties of the untreated and treated test samples. The contact angle measurement findings are shown in Fig. 4. Based on the findings, the surface contact angle values of the untreated natural Scots pine wood were between 10° and 14° in all three directions and were quite low compared with the treated samples.

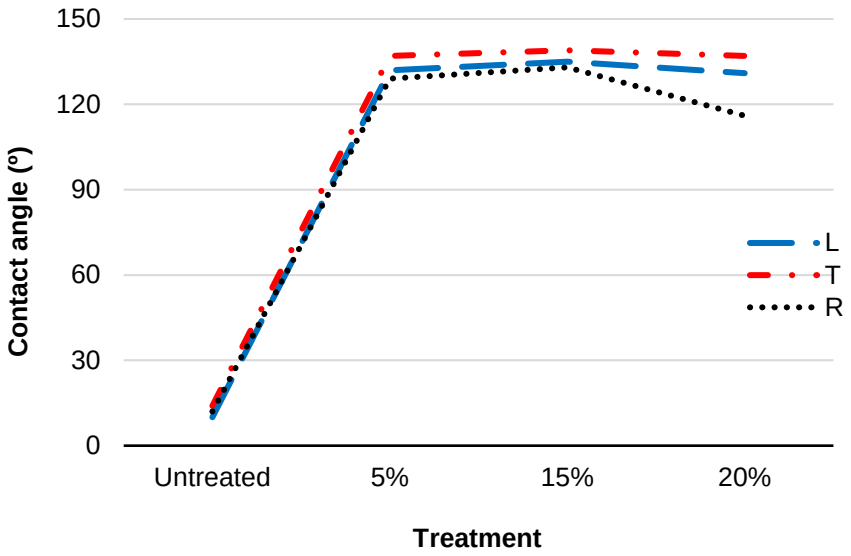


**Fig. 4.** Surface contact angle values of waste tar-treated Scots pine

The contact angle increased significantly in the waste tar treated samples. The contact angle values in the control samples differed according to some previous studies. Previously reported surface contact angle values for unmodified Scots pine wood at the end of wettability tests were between 28° and 46° (Mohammed-Ziegler *et al.* 2006; Petric *et al.* 2007). The contact angle values of Scots pine in the current study were thought to be low because of the predominance of sapwood, high rate of early wood, and low rate of resin in the homogeneous and broad annual ringed wood used.

When contact angle values of the treated sample were investigated, the DT application significantly increased the surface contact angle values. Therefore, the tar solution reduced the wettability of the wood compared with the control. Some modification applications including DMDHEU (dimethyloldihydroxyethylene urea), heat treatment, and silylating agents reduce the wettability of pine wood and increase the contact angles by two-, three-, or four-fold (Mohammed-Ziegler *et al.* 2006; Petric *et al.* 2007). In addition, Yalcin and Ceylan (2017) determined that wettability is reduced by application of herbal extracts and tannins to the wood.

This study determined that for deep treatment the direction of the wood had an important effect, at least relative to the contact angle. The highest contact angle values were recorded on the tangential direction at the three different concentration levels of 5, 10, and 20%. The surface direction (annual ring direction) is an effective factor on wettability as a few other parameters such as the porosity, surface roughness, heterogeneity, surface chemistry, extractives, and acidity of the wood (Shupe *et al.* 1998; Büyüksarı *et al.* 2011;

Yorur *et al*. 2017).

Dimensional stability and resistance to decay fungi can be achieved by the reduction of wettability. Koski (2008) specified that biological resistance increased with the use of water absorption-reducing agents because when the moisture of the wood is kept below levels it is not conducive to the development of decay fungi. Thus, in commercial applications, fungicides and water repellents are used together (Archer and Cui 1997).

According to Rowell and Banks (1985), surfaces having a contact angle of less than 90° are hydrophilic, whereas surfaces with an angle greater than 90° are considered hydrophobic, *i.e.*, water repellent. In the current study, the waste tar can be considered as a water repellent, hydrophobic material because the treated samples yielded contact angle values of 100° to 140°.

## Decay Tests

Table 5 shows the effects of the application of different concentration levels and types of waste tar on white and brown rot fungi. When the effect of the ethanol : toluene (1v:1v) solvent mixture on fungus resistance was investigated, the results showed that weight loss values were very similar to those of the control samples and thus, which indicated that it was not an effective factor on fungus resistance.

**Table 5.** Percentage Weight Losses Caused by Fungus TV and NL According to Wood Species and Application Type

| Application Type | C* | Scots Pine | | Beech | |
|---|---|---|---|---|---|
| | | *T. versicolor* | *N. lepideus* | *T. versicolor* | *N. lepideus* |
| Control | | 31.13$^{f}$ (2.86) | 28.93$^{f}$ (2.20) | 45.40$^{i}$ (1.29) | 43.23$^{g}$ (1.69) |
| (SA) | | 30.45$^{f}$ (3.24) | 30.92$^{f}$ (1.14) | 43.02$^{h}$ (1.58) | 43.08$^{g}$ (1.85) |
| DT | 5% | 22.57$^{e}$ (1.27) | 16.00$^{e}$ (5.56) | 25.48$^{g}$ (2.07) | 25.32$^{e}$ (1.53) |
| | 10% | 18.15$^{d}$ (2.15) | 14.67$^{de}$ (6.02) | 20.18$^{e}$ (1.01) | 31.25$^{f}$ (1.53) |
| | 15% | 13.12$^{c}$ (0.78) | 7.42$^{ab}$ (2.65) | 23.97$^{f}$ (1.17) | 23.37$^{d}$ (1.43) |
| | 20% | 13.25$^{c}$ (0.66) | 7.35$^{ab}$ (1.52) | 16.53$^{bc}$ (0.79) | 19.93$^{c}$ (0.79) |
| DT + SC | 5% | 11.08$^{c}$ (0.38) | 11.55$^{cd}$ (0.60) | 17.87$^{cd}$ (0.80) | 14.02$^{a}$ (2.31) |
| | 10% | 12.77$^{c}$ (0.14) | 9.17$^{bc}$ (1.02) | 18.83$^{de}$ (0.34) | 13.33$^{a}$ (0.70) |
| | 15% | 8.32$^{b}$ (1.57) | 6.72$^{ab}$ (1.84) | 15.17$^{ab}$ (1.73) | 13.60$^{a}$ (0.77) |
| | 20% | 3.87$^{a}$ (2.17) | 4.30$^{a}$ (1.61) | 14.03$^{a}$ (0.23) | 15.77$^{b}$ (0.22) |

There is no difference between the same superscript letters in the same column, *C: Concentration, DT: Deeply treated, DT+SC: Deeply treated + Surface coating, SA: Solvent Application

In the Scots pine decay tests, about 30% of weight losses caused by decay types were found in the untreated and SA samples. In general, weight losses in the DT Scots pine samples were higher than those of the DT + SC samples. This suggested that the application of SC together with DT generated more effective results against decay in the Scots pine wood. The increase in the concentration level of tar in both DT and DT + SC was an effective factor on decay resistance, and with the increasing concentration level, weight losses tended to decrease. No significant differences were found between the fungi species in terms of weigh losses. However, with DT applied, when the concentration level increased to 15%, the weight losses of wood resistant against fungus TV decreased from

30% to 13.12% and resistant against fungus NL fell to 7.42%. A significant reduction in weight loss occurred when the treatment of DT + SC were applied together. The lowest weigh loss was detected at the concentration level of 20% in DT + SC treatment.

The resistance to both fungal species increased by 85% when DT+SC application was compared with the untreated control samples. In the untreated beech samples about 45% of weight loss occurred under white rot, and 43% weight loss occurred under brown rot. With DT applied, the resistance against the fungus increased by 43 to 64% for TV and 40 to 54% for NL compared with the control samples. The weight loss was decreased by 70% for both fungal species after DT + SC treatment.

In general, with the increasing concentration level of waste tar, weight loss in fungal tests and decreases in mortality rate in larval tests were detected under both application types. The antifungal properties of the impregnated samples were thought to be due to the chemical content of the waste tar. Benzene and its derivatives comprised the highest aromatic content in the waste tar, with p-xylene at 72.32% and m-xylene at 19.07%, and both have been reported to show antifungal activity (Bergauer *et al.* 2005; Akpuaka *et al.* 2013; Bayan 2016; Suriani 2016). Bayan (2016) conducted a study with *Cardaria draba* plant extracts and detected high amounts of decane pentanenitrile, nonane, p-xylene, m-xylene, benzene, and 1-ethyl-2-methyl benzene acetaldehyde which are plant extracts with antifungal properties in. It was reported that these compounds exhibited antifungal effects against the plant pathogens *Fusarium oxysporum, Alternaria solani,* and *Verticilium dahliae*. Methyl dehydroabietate (1.45%), which was detected in the waste tar, has an antifungal effect. Burcova *et al*. (2018) reported that even the very low amounts of methyl dehydroabietate in Norwegian spruce extracts showed antifungal effects against *Heterobasidion* spp. fungus.

Hu *et al.* (2013) reported that the high amount (11,100 mg/kg) of potassium salt (K) detected in waste tar when it was synthesized with hinokitiol was effective against *Trametes versicolor* and *Gloeophyllum trabeum* wood-decay fungi, and *Penicillium citrinum, Aspergillus niger,* and *Trichoderma viride* molds. Moreover, Li *et al.* (2012) reported that the salts of sodium (Na) determined in the tar composition (2103 mg/kg) when synthesized with hinokitiol exhibited antifungal activity against *Trametes versicolor* and *Gloeophyllum* spp. Fungi, and *Penicillium citrinum* and *Aspergillus niger* molds. Likewise, Arslan (2015) reported that potassium salts show antifungal properties against plant pathogens. Moreover, potassium salts boost the resistance of plants to fungi. Metals such as copper, boron, zinc, chromium, and their compounds have been evaluated in the wood preservation industry. Copper is needed for the development of fungi. When it is used above the threshold value, however, it prevents the development of wood-decay fungi (Baldrian 2003).

The most efficient protection of Scots pine ensured 20% concentration. However beech wood was not efficiently protected at this concentration. Lower protection of beech wood can be explained by the fact that the density of beech wood is higher than that of Scots pine, which absorbs the tar solution less (Sen et al 2009). The AWPA standard defines that the creosote must have a minimum retention of at least 160 kg/m$^3$ for effective protection in ground contact (Kartal *et al.* 2011). In the present study, the retention value of tar (88.1 kg/ m$^3$) was obtained at the most effective protection level of 20% concentration in Sots pine for brown rot fungus *N. lepideus*. These results indicates that a lower retention level is sufficient to provide protection. Mohan *et al.* (2008) found that the fungicidal activities of both creosote and bio-oil were 80 to 128 kg/m$^3$ and <32 kg m$^3$ for *G. trabeum*

and *T. versicolor* fungi for creosote; and 320 kg /$m^3$ and 64 to 208 kg/$m^3$ for *G. trabeum* and *T. versicolor* fungi for bio-oil.

## Larvae Tests

The mortality rates and weight change of *H. bajulus* larvae are given in Table 6. All mature larvae survived in untreated and solvent-treated samples during the 16 week experiment. The highest larval mortality rate (80%) was obtained at the concentration level of 20% compared with the samples treated with tar solution in different concentrations. When the larvae development rate was investigated, the larvae weights decreased, and the change was detected as 24% at the end of the experiment period. The larval mortality rate was 60% at the concentration levels of 5% and 15%. However, in the samples treated with 20% concentration, larval development was decreased or stopped. The highest larval weight change was detected in the untreated control samples.

**Table 6.** Larval Mortality and Weight Change

| Mature Larvae | | | Newly Hatched Larvae |
|---|---|---|---|
| (DT) | Mortality rate (%) | Larvae weight change (%) | Mortality rate (%) |
| C | 0 | 58 | 10 |
| SA | 0 | 30 | 10 |
| 5% | 60 | 15 | 40 |
| 15% | 60 | -33 | 90 |
| 20% | 80 | -24 | 100 |

DT: Deeply treated, C: Control, SA: Solvent Application

Similar findings were obtained in the tests with mature and newly hatched larvae. A mortality rate of 10% was found in the untreated and solvent-treated Scots pine samples. The mortality rate was 40% in the samples treated with tar at 5% concentration, while the larval mortality rate was 100% when the waste tar concentration reached 20%. In tests with newly hatched larvae, images of live larvae in control, 5% treated samples, and a dead larva in a 20% tar treated sample are given in Fig. 5.

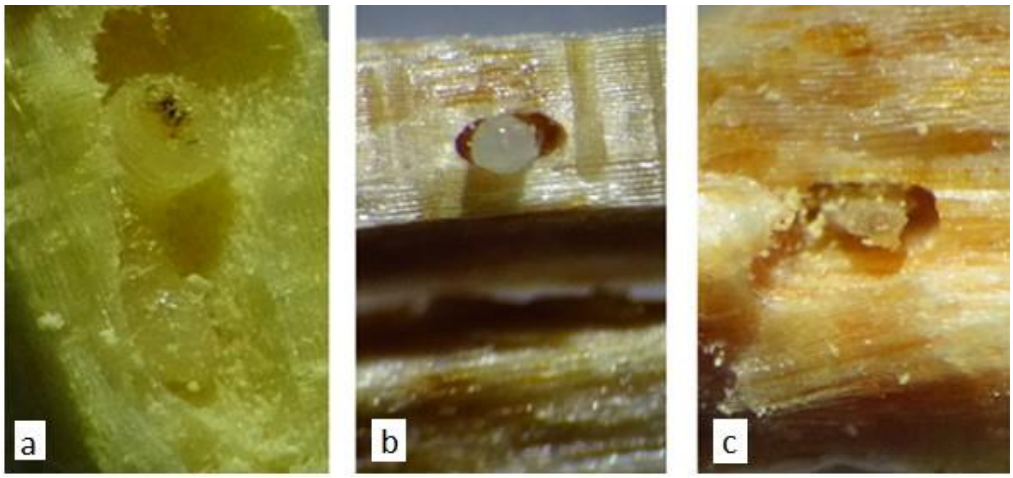


**Fig. 5.** a) Live larva in control sample; b) Live larva in sample treated with 5% tar; c) Dead larva in sample treated with 20% tar

Benzene and its derivatives (p-xylene, m-xylene), which are present in waste tar, have toxic effects on insects, fungi, and bacteria (Dawagreh *et al.* 2001). Moreover, the potassium, sodium content, and salts of these metals in waste tar are used commercially as insecticides, fungicides, herbicides, and algicides in many application areas. These salts are traditionally preferred because of their synergistic effect on insects (US EPA 1982; Baker and Grant 2018).

## CONCLUSIONS

This study investigated the effects of waste tar obtained from particle board plants on some physical properties and the biological resistance of wood.

1. The water uptake (WA) values decreased and the water repellent efficiency (WRE) increased at the end of the 2 and 72 h periods compared to the control samples.
2. After 48 h leaching tests, 2% of the phenolic content of the total phenolic substances was leached from the treated samples.
3. When the surface contact angle values were investigated, the contact angle of the Scots pine increased. The waste tar solution significantly reduced the wettability of the wood compared with the untreated control samples.
4. The waste tar prevented weight losses in Scots pine and beech wood by exhibiting antifungal properties against white and brown rot, TV, and NL fungi, which cause decay in wood.
5. The highest *H. bajulus* mortality rate was obtained at the concentration level of 20% in both mature and newly hatched larvae (80% and 100%, respectively).
6. Thus, waste tar can be used in the wood preservation industry as an effective natural wood protection material.

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