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Determination of modulus of elasticity and bending strength of wood material impregnated with nanoparticle silicon dioxide (SiO_2)

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Abstract: The purpose of this study is to determine the effects of the nano-particulate silicon dioxide (SiO_2) material on the bending strength and modulus of elasticity in bending of some wood species. Oriental beech (*Fagus orientalis* L.) and sessile oak (*Quercus petrea* L.) woods species which are commonly used in the furniture industry were used in this study. Impregnation was carried out with the preparation of SiO_2 at concentrations of 1% and 3% according to ASTM-D 1413-76 (1976) standards. According to the results, while solution concentration was increasing in both wood species, total retention (kg/m^3) and percentage retention (%) values increased; air dry density (12%), bending strength and modulus of elasticity in bending decreased. The maximum mean values of bending strength and modulus of elasticity was in control samples of oriental beech and the minimum were in sessile oak wood with 3% concentration. These results can be related to the fact that the higher density of oriental beech wood (0.630-0.685 g/cm³). In both species of wood, impregnation with SiO_2 resulted loss of about 3-5% in bending strength and 0.6-9% in modulus of elasticity.
Keywords: Impregnation, Silicon dioxide (SiO_2), Bending strength, Modulus of elasticity in bending

Nanoparçacık silisyum dioksit (SiO_2) ile emprenye edilmiş ahşap malzemenin elastikiyet modülü ve eğilme dayanımı tayini

Özet: Bu çalışmanın amacı, nano tanecikli silisyum dioksit (SiO_2) maddesinin bazı odun türlerinde eğilme direnci ve eğilmede elastikiyet modülüne etkilerini belirlemektir. Odun türü olarak mobilya endüstrisinde yaygın olarak kullanılan doğu kayını (*Fagus orientalis* L.) ve sapsız meşe (*Quercus petrea* L.) odunları tercih edilmiştir. ASTM-D 1413–76 (1976) esaslarına göre %1 ve %3 konsantrasyonlar hazırlanmış olan SiO_2 maddesi ile emprenye işlemi gerçekleştirilmiştir. Deney sonuçlarına göre; her iki ağaç türünde de çözelti konsantrasyonu artarken toplam retensiyon (kg/m^3) ve yüzdesel retensiyon değerleri artmış; hava kurusu yoğunluk (%12), eğilme direnci ve elastikiyet modülü değerleri azalmıştır. En yüksek ortalama eğilme direnci ve eğilmede elastikiyet modülü değerleri doğu kayını kontrol örneklerinde ve en düşük %3 konsantrasyonlu sapsız meşe odunundadır. Bu sonuçlar, doğu kayını odununun ağacının yoğunluğunun (0.630-0.685 g/cm³) daha yüksek olması ile ilgili olabilir. Her iki odun türünde de SiO_2 ile emprenye uygulamaları eğilme direncini %3-5, elastikiyet modülünü %0.6-9 oranında azaltmıştır.
Anahtar kelimeler: Emprenye, Silisyum dioksit (SiO_2), Eğilme direnci, Eğilmede elastikiyet modülü

1. Introduction

The wood material which stays in natural conditions can get destroyed in under 5 years (except some certain wood species) (Ors and Keskin, 2001; Ozcifci, 2009; Tan and Peker, 2015a; Tan and Peker, 2015b; Keskin and Daglioglu, 2016). The service life of wood can increase with impregnation with wood preservative materials (Archer and Lebow, 2006; Hill, 2006; Tondi et al., 2012, Sandberg et al., 2017, Yasar and Altunok, 2019). Impregnation is the process of penetrating wood with substances with different contents with the purpose of preventing rotting, burning and dimensional work in wood material (Kurtoglu, 2000; Hill, 2006, Ayar, 2008; Akgul and Apay, 2014a; Akgul and Apay, 2014b; Esteves et al., 2014. Sandberg et al., 2017).

The effectiveness of impregnation process depends on toxicity of preservative substance, penetration depth and retention amount, anatomic structure of the wood, slitting and drying processes conducted before impregnation (Bozkurt et al., 1993; Yalinkilic et al., 1996; Baysal, 2003; Baysal et al., 2003; Archer and Lebow, 2006; Tan and Peker, 2015a; Tan and Peker, 2015b). However, it is expected that today's wood preservatives do not harm the humans and the environment (Zabel and Morrell, 1992; Reinprecht, 2016). EPA, has proposed label changes of some wood preservatives (chromated arsenicals, pentachlorophenol and creosote) to avoid creating human health and environmental concerns (US. EPA, 2016).

A decline was found in the bending strength values of the beech and spruce woods impregnated with water-soluble salts (Kollman, 1959). In the study which Stabnikov (1957) performed, 10-22% increase in bending strength of pine, spruce, fir, beech and poplar woods impregnated with anthracene was reported. Salty impregnation substances

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were identified to reduce bending strength by 2.9-16% (Wazny, 1973). While the bending strength of the wood material impregnated with CCA was found to decrease about 12%; a decline of 5% was observed in test samples impregnated with ACQ (Temiz et al., 2014).

The impregnation process with vacuum pressure by using Tanalith C and Vascol WR impregnation materials has been stated to decrease bending strength, to increase the specific weight, to reduce hardness in scots pine and to raise it in Calabrian pine (Gur, 2003). Certain effects of impregnation materials on the mechanical features of scots pine was investigated. In samples impregnated with Wolmanit CX-8 of 2.8% and Tanalith E-3491 of 2%, modulus of elasticity increased more in comparison with control samples; however, samples impregnated with other chemicals were detected to remain the same or decrease. In samples impregnated with Wolmanit CX-8 of 2.8% and ACQ-1900 of 7%, bending strength was found to increase in comparison with control samples, the samples impregnated with other chemicals were found to remain the same or decrease (Yildiz et al., 2003). Mountain ash was impregnated with boron compounds, Vascol, Azure, Immersol-Aqua and Tanalith-E with double vacuum method and then effects of impregnation substances on compressive strength, bending strength and modulus of elasticity in bending were investigated. According to test results, the highest bending strength values were found in samples impregnated with Tanalith-E (Keskin et al., 2013).

A rise in density and thermal conductivity values was observed in poplar, linden, and chestnut woods impregnated with borax and boric acid and a decline was observed in the bending strength and modulus of elasticity values (Aytaskin, 2009). The heat treatments usually reduce the mechanical properties of wood (Srinivas and Pandey, 2012; Bal and Bektas, 2013; Percin and Altunok, 2017). That waste boron oil decreases the mechanical properties of wood material by 2-5% is found to be statistically insignificant (Ozcifci, 2009). A decrease in bending and compressive strength of scots pine and Oriental beech wood impregnated with boron compounds was stated and an increase was stated in deterioration strength (Simsek, 2009). In beech wood samples impregnated with certain tannins and boron compounds, a decline in bending strength and modulus of elasticity was determined (Adanur, 2015). Since boron compounds have a crystalline structure, they cause some decrease in bending strength parallel to grain and modulus of elasticity after they were penetrated wood material, nevertheless this ratio was reported to be too little (Cicek, 2015).

The nanotechnology has been successfully applied in many fields (coating, rubber, adhesives, and plastics) (Wu et al., 2005; Ozcifci et al., 2018). In recent years, increasing interest in nanotechnology has offered alternative use of nanoparticles to improve properties of wood (Moon et al., 2006). Nano-SiO_2 is an active nanoparticle against fire and weather conditions that can create a large surface area (Devi and Maji, 2013; Yan et al., 2015; Reinprecht, 2016).

In a study, the leachate amount was lower in wood impregnated with nano-TiO_2 with respect to control samples. (Marzbani and Mohammadnia-afrouz, 2014). The results of the study investigating effect of nano-TiO_2 and nano-SiO_2 on bonding performance and structural properties of PVAc, showed that thermal stability of PVAc blends was largely improved (Bardak et al., 2016).

The impact of nanofillers (TiO_2, SiO_2 and nanoclay) were also reported to be effective in improving flame retardancy, water resistance, anti-swelling efficiency in the wood polymer nanocomposite (WPNC) simul wood (Bombex ceiba, L.) (Devi and Maji, 2013).

The effects of SiO_2 and TiO_2 nanoparticles at different concentrations on the mechanical and morphological characteristics of polypropylene nano composites were investigated. The increase in nanoparticles from 0 to 3% resulted in increased mechanical strength, but the addition of more nano-filler resulted in a significant reduction in mechanical strength (Ismaeilimoghadam et al., 2016).

The effect of SiO_2 and TiO_2 nanoparticles on the bending strength of poly (methyl methacrylate) acrylic resins was examined, the maximum bending strength (43.5 MPa) belongs to the control group. In addition, as the amount of SiO_2 increased (0.5-1%), this value decreased (Sodagar et al., 2013).

Nano-SiO_2 has been added to different materials to increase their strength, flexibility, aging resistance and mechanical properties in many researches (Bauer et al., 1996; Wang and Cheng, 2002; Lei et al., 2006; Chaichana et al., 2007; Jo et al., 2007; Flores et al., 2010).

This study was conducted with the intent of determination of changes occurring in bending strength and modulus of elasticity in bending values after the protective process with silicon dioxide (SiO_2) of oriental beech (*Fagus orientalis* L.) and sessile oak (*Quercus petraea* L.) which are widely used in the furniture industry.

2. Material and methods

2.1. Material

In the study, oriental beech *(Fagus orientalis* L.*)* and sessile oak *(Quercus petraea* L.*)* woods, which are widely used in Turkish woodworking industry, were chosen as test samples. Wood materials were supplied from lumber managements located in Karabuk province Yenice district with random selection method and during selection lumbers were paid attention to be flawless, with smooth fibres, not decayed, without reaction wood and not damaged by fungi and pests.

SiO_2 substance utilized in impregnation process in pulverulent state was obtained from TIMED (Timed, 2018). Silicon dioxide nanoparticles are commonly used in areas like agriculture, textile, electronics, cosmetics, paint industry and medicine (Comelekoglu et al., 2017).

2.2. Method

Wood samples were cut up in dimensions of 5x5x80 cm according to TS 2470, 1976 principles, they were kept in the conditioning chamber, which was in conditions of temperature of 20±2°C and relative humidity of 65±5%, until they reached to a constant weight and then they were impregnated with vacuum method. After the impregnated samples were waited in a setting with air circulation for 15-20 days for evaporation of the solvent, samples were kept until they reach to the 12% humidity in a temperature of 20±2°C and in 65±5% relative humidity. Total of 60 test samples (2x3x10) were prepared, containing two wood material species (beech and oak), two experiment type

(bending strength and modulus of elasticity) + 1 control and 10 recurrences.

As the impregnation method, vacuum-pressure method (empty cell method), one of the methods to exert pressure, was applied. The impregnation process was conducted according to ASTM D 1413-76, 1976. Accordingly, the samples, which will be impregnated, were left at diffusion inside a solution under 4 atmospheric pressure for 60 minutes after a pre-vacuum for 60 minutes. To avoid post impregnation deformations occurring because of fast evaporation, samples were kept in the conditioning chamber until they reached to an air-dry state gradually. The weights of test samples measured at an analytic scale with a sensitivity of 0.001 g, total retention (adhesion) amount (R, kg/m³) and percentage retention (R, %) was calculated through the following equations.

$$R = \frac{G \times C}{V} \times 10^3 \; g/cm^3 \quad (1)$$

Here;
G= T2-T1
T1 = Pre-impregnation weight (g)
T2 = Post-impregnation sample weight (g)
V = Sample size (cm^3)
C = Solution concentration (%)

$$R\ (\%) = \frac{M_{oes} - M_{oeö}}{M_{oeö}} \quad (2)$$

M_{oes}= Oven dry weight after impregnation (g).
$M_{oeö}$= Oven dry weight before impregnation (g)

Solution and treatment temperature were applied at 20 ± 2 ˚C during the impregnation application. After impregnation, the specimens weighed and dimensioned at full age were conditioned until relative humidity of 60±3% and humidity of 20±2 % at 20 ± 2 ˚C.

Air dry and oven dry density of the wood materials used in the preparation of test sample were determined in accordance with TS 2472, 1972. In compliance with this; samples, which were prepared at dimensions of 20x30x30 mm for air dry density specification, were kept in the temperature of 20±2 °C and relative humidity of 65 ± 5% till they reached to a constant weight. At this state, their weights were measured in an analytic scale with a sensitivity of ±0.01g and their dimensions were measured with a digital caliper (±0.01 mm) following this, their sizes and air-dry densities (δ_{12}) were calculated with the following equation.

$$\delta_{12} = \frac{M_{12}}{V_{12}} \; g/cm^3 \quad (3)$$

M_{12}: Air dry weight (g)
V_{12}: Air dry volume/size (cm^3)

The samples used for determination of bending strength and modulus of elasticity were prepared with respect to TS EN 326-1, 1999. TS 2474, 1976 principles were followed for bending strength at 3 point, the principles stated in TS 2478, 1976 was complied with in modulus of elasticity in bending. According to these standards, the samples were prepared in dimensions of 20x20x360 mm for each of two experimental groups (Figure 1).

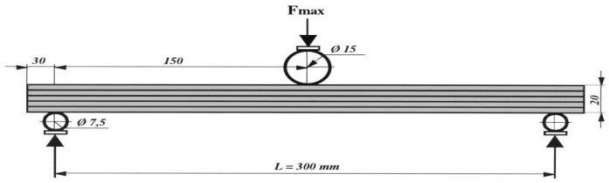

Figure 1. Bending Strength and Elasticity Modulus in Bending Test (Keskin, 2003)

Experiments were conducted in a computer driven static test machine with 50 KN capacity. In experiments, the load implemented at the exact center of test specimens. The maximum force (Fmax) in the instant of failure for the bending strength (σ_e) was calculated with the formula below.

$$\sigma_e = \frac{3Fmax.L}{2b\,h^2} \; (N/mm^2) \quad (4)$$

Here;
L: The gap between fulcrums (mm)
b: section width (mm)
h: section height (mm) dir.

In determination of modulus of elasticity, the samples, which were used in bending strength, were utilized. The strength difference applied to elastic deformation area (ΔF) was measured with bending amount difference in samples (Δf) and modulus of elasticity (E) by following this equation:

$$E = \frac{\Delta F L^3}{4b\,h^3 \Delta f} \; (N/mm^2) \quad (5)$$

Here;
ΔF: The force equal to the difference between the arithmetic means of lower and upper limits of the load at elastic deformation area (N)
L: The gap between fulcrums (mm)
Δf: The difference between arithmetic means of results belonging to deflection at net bending area and deflections at lower and upper limits of the load (mm);

b: section width (mm)
h: section height (mm).

Statistical evaluation of the test results were analyzed with SPSS 22.0 (2013) statistical package software. In case of mutual interactions of sources of variance being significant according to (P< 0.05) for which factors are the differences important was identified by Duncan test.

3. Results and discussion

Air dry and oven dry density values are given in Table 1. When the Table 1 is examined, the maximum value for air-dry and oven-dry density was in beech wood control samples (0.685-0,630 g/cm^3), the minimum was in oak wood with silicon dioxide solution of 1% (0.658-0,584 g/cm^3). The variety n air dry density values of impregnated wood material can be stated to originate from solution concentration, chemical compounds, participation level of earlywood and latewood, annual ring width, cell lumen diameter, cell wall thickness and air void ratio depending on the anatomic structure of wood materials.

Ors et al., (1999) stated that the oven dry and air-dry density values of impregnated samples assigned higher values in comparison with control samples, and oven dry and air-dry density values of Oriental Beech samples were higher than the Calabrian pine wood samples. As the solution concentration (0-6%) increased in boron compounds, retention ratio and amount increased, too. Therefore, the densities of wood material showed an increase in parallel with the solution concentration (Adanur, 2015). Retention values (total and percentage) of wood samples are given in Table 2.

As in the literature and shown in Table 2, retention amount increased with the increase in solution concentration (Ors et al., 2005; Bal, 2006; Atar and Keskin, 2007; Toker, 2007; Tan and Peker, 2015a; Adanur, 2015; Aydin, 2015). Accordingly, the highest retention amount was attained as in beech wood samples treated with solution of 3% silicon dioxide (36 kg/m^3), the lowest retention amount was in oak wood samples treated with a silicon dioxide solution of 1% (3.6 kg/m^3). The maximum retention rate was obtained in beech wood with 3% solution concentration (0.06%), the minimum retention rate was in beech wood with 1% solution concentration of oak (0.01%). This situation can stem from reasons like solution characteristic, wood species, high permeability of beech wood etc. In this study, silicon dioxide concentration increases in direct proportion to retention amount, too.

Toker, (2007) impregnated Oriental beech woods with borax and boric acid in various concentrations in the study he conducted. He determined retention values as 4.95 kg/m^3 in 1% boric acid concentration, as 13.86 kg/m^3 in boric acid in concentration of 3%, as 26.69 kg/m^3 in 5% boric acid concentration, as 5.03 kg/m^3 in 1% borax, as 15.20 kg/m^3 in 3%, borax and as 25.22 kg/m^3 in 5% borax concentration. In the beech impregnated with Tanalith-E, the retention rate was 2.11%, total retention rate was 9.90 km/m^3 while in Scots pine, % retention rate was 1.60% and total retention was 4.85 km/m^3 (Peker et al., 1999). In fir wood, retention rate was 12 kg/m³ in borax and 12 kg/m³ in boric acid at vacuum pressure method for borax and boric acid (Atar and Keskin, 2007).

The bending strengths of test samples impregnated with silicon dioxide (SiO_2) are given in Table 3. The results of variance analysis conducted to determine differences between bending strength values are shown in Table 4.

As shown in Table 3, bending strength values decreased with the increase in solution concentration. Accordingly, the highest average bending strength values was attained in beech wood control samples (124 N/mm^2), the lowest values in oak wood treated with solution of 3% silicon dioxide (106.6 N/mm^2).

According to Table 4, except the binary interaction of wood species and solution concentration, comparison values on bending strength was found to be statistically significant with margin of error ($P<0.05$). Duncan test results of bending strength values with respect to solution concentration are given in Table 5.

As shown in Table 5, as solution concentration was increasing, bending strength decreased. Therefore, the highest bending strength value was obtained as 118.1 N/mm^2 in control samples. The lowest bending strength change was identified as 111.45 N/mm^2 in samples impregnated in 3% concentration. T test results of bending strength values of test samples impregnated with silicon dioxide (SiO_2) according to wood species are shown in Table 6.

In accordance with Table 6, the bending strength values was obtained in beech as 120.17 N/mm^2 and in oak as 109.27 N/mm^2. Keskin and Daglioglu, (2016) stated that the bending strength of impregnated wood materials with Tanalith-E was lower at a rate of 6.83% in Oriental beech, at 5.12% in ash and at 5.93% scotch pine wood with respect to control samples.

The modulus of elasticity in bending values and statistical rates of impregnated wood materials and control samples are given in Table 7. The variance analysis results conducted to determine differences modulus of elasticity in bending values are shown in Table 8.

Table 1. Air dry and oven dry density values

Wood material	Solution concentration (%)	Air dry density (g/cm³)	Oven dry density (g/cm³)
Beech	Control	0.685	0.630
	1	0.674	0.620
	3	0.678	0.625
Oak	Control	0.665	0.590
	1	0.658	0.584
	3	0.660	0.587

Table 2. Total and percentage retention (%) values of wood samples

Wood material	Solution concentration (%)	Total retention (kg/m³)	Retention (%)
Beech	1	7.2	0.02
	3	36	0.06
Oak	1	3.6	0.01
	3	21.6	0.04

Table 3. Descriptive statistics of bending strength values [N/mm^2]

Wood species	Solution concentration (%)	Xmin	Xmax	Xavg	SE
Beech	Control	122	125	124	4.03
	1	118	121	120	3.46
	3	114	118	116	3.09
Oak	Control	110	113	112	1.81
	1	106	110	108	1.05
	3	104	108	106	1.26

SE: Standart Error

Table 4. Variance Analysis of bending strength values.

Source	Sum of squares	Degrees of freedom	Mean of squares	F Value	P
Wood Species (A)	1782.150	1	1782.150	244.440	0.000*
Solution Concentration (B)	442.633	2	221.317	30.356	0.000*
Interaction A*B	11.700	2	5.850	0.802	0.454
Error	393.700	54	7.291		
Total	792225.000	60			

Table 5. Duncan test results of bending strength according to solution concentration [N/mm^2].

Solution concentration (%)	Xavg	Homogenous Group
Control	118.10	A
1	114.60	B
3	111.45	C

Xavg: Average Values

Table 6. Bending Strength T test results according to wood species [N/mm^2]

Wood species	Xavg	SD	SE	P
Beech	120.17	4.684	0.855	0.000*
Oak	109.27	2.709	0.494	0.000*

SD: Standart Deviation, SE: Standard Error

Table 7. Statistical analysis of modulus of elasticity values in bending [N/mm^2]

Wood species	Solution concentration (%)	Xmin	Xmax	Xavg	SE
Beech	Control	12974	13364	13169	410.78
	1	12888	13278	13083	269.56
	3	12569	12960	12765	230.97
Oak	Control	12406	12796	12601	283.47
	1	11705	12095	11900	291.21
	3	11331	11721	11526	328.21

SE: Standart Error

Table 8. Variance Analysis of modulus of elasticity values in bending

Source	Sum of squares	Degrees of freedom	Mean of squares	F Value	P
Wood species (A)	14891198.017	1	14891198.017	157.403	0.000*
Solution concentration (B)	5474393.233	2	2737196.617	28.933	0.000*
Mutual interaction A*B	1385110.433	2	692555.217	7.320	0.002*
Error	5108685.300	54	94605.283		
Total	9413588053.000	60			

As shown in Table 7, modulus of elasticity in bending values decreased with the increase in solution concentration. Accordingly, the highest average modulus of elasticity in bending value was attained in beech wood control samples (13169.2 N/mm^2), the lowest values were in oak wood treated with solution of 3% silicon dioxide (11526.8 N/mm^2).

According to Table 8, all interactions of wood species and solution concentration, on modulus of elasticity in bending were found be significant (P<0.05). Duncan test results of modulus of elasticity in bending are shown in Table 9 according to the solution concentration.

When Table 9 is analyzed, modulus of elasticity values decreased with the increase in the solution concentration. Accordingly, the maximum modulus of elasticity in bending value was obtained as 12885.3 N/mm^2 in control samples. The minimum modulus of elasticity in bending amount was obtained as 12145.9 N/mm^2 in samples impregnated with concentration of 3% in terms of solution concentration. T test results of modulus of elasticity in bending values of test samples impregnated with silicon dioxide (SiO_2) according to wood species are shown in Table 10.

Table 9. Duncan test results with modulus of elasticity in bending according to solution concentration [N/mm^2]

Solution concentration (%)	Xavg	Homogenous group
Control	12885.30	A
1	12492.25	B
3	12145.90	C

Xavg: Average values

Table 10. T test results of modulus of elasticity in bending according to wood species [N/mm^2]

Wood species	Xavg	SD	SE	P
Beech	13006.00	350.39	63.97	0.000*
Oak	12009.63	538.44	98.31	0.000*

SD: Standart Deviation, SE: Standard Error, P≤0.05

According to Table 10, the modulus of elasticity values were obtained in beech wood as 13006 N/mm^2, and in oak as 12009.63 N/mm^2. With respect to impregnated wood material, modulus of elasticity in bending values of non-impregnated control samples were found to be higher than oak wood with a ratio of 7.15% and ash wood with a ratio of 6.58%. Impregnation substance affects the modulus of elasticity in bending values of oak and ash wood adversely (Keskin and Daglioglu, 2016).

4. Conclusion

This study is to determine the effects of the nano-particulate silicon dioxide (SiO_2) material on the bending strength and modulus of elasticity in bending of oriental beech and sessile oak woods. While impregnation with the application of silicon dioxide revealed favorable results on total retention (kg/m^3), percentage retention (%) and air-dry density values, it yielded adverse results on bending strength and modulus of elasticity.

Retention values are compared with the literature, it demonstrated positiveness and parallelism. The highest total retention value was obtained in beech wood with 3% silicon dioxide solution. The highest % retention value achieved at beech wood with 3% solution concentration.

The air-dry density value was identified to be highest in beech wood control samples, to be lowest in oak wood processed with a solution concentration of 1%. The oven dry density value was found to be highest in beech wood control samples, lowest in oak wood treated with a solution of 1% concentration.

The average value for bending strength occurred highest in beech wood control samples, the lowest was in oak wood treated with a solution concentration of 3%. The bending strength of impregnated wood materials with was lower at a rate of %3-6 in oriental beech, at 3-5 % in oak wood with respect to control samples. The modulus of elasticity of impregnated wood materials with was lower at a rate of 0.6-

3% in oriental beech, at 5.5-9% in oak wood with respect to control samples.

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