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Physical, Mechanical and Biological Properties of Beech and Fir Wood Modified with Gamma-aminopropyltriethoxysilane

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Abstract − Silanes have potential for modifying wood materials to improving their properties. The aim of this study was to determine the physical and mechanical properties of beech (*Fagus orientalis* Lipsky) and fir (*Abies bornmülleriana* Mattf.) wood samples impregnated with gamma-aminopropyltriethoxysilane (APTES) as well as their performance against decay fungi. Beech and fir wood samples were impregnated with APTES at 20% and 40% concentrations. Control and treated samples were subjected to water uptake, bending strength and thermogravimetric analysis (TGA) tests as well as exposure to the white-rot fungus *Trametes versicolor* and the brown-rot fungus *Coniophora puteana*. Weight percentage gain (WPG) was higher in fir samples than in beech samples after APTES treatment. Water uptake was higher in fir samples than beech for control and impregnated samples. Water uptake decreased with higher APTES concentrations. APTES treatment slightly increased modulus of rupture (MOR) and modulus of elasticity (MOE). Mass loss caused by decay fungi was much lower in APTES treated timbers.

Keywords − *Wood modification, gamma-aminopropyltriethoxysilane, water uptake, bending strength, decay resistance*

Gama-aminopropiltrietoksisilan ile Modifiye Edilmiş Kayın ve Göknar Odunlarının Fiziksel, Mekanik ve Biyolojik Özellikleri

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Research Article

Öz − Silanların ahşap malzemeyi modifiye etme ve özelliklerini iyileştirme potansiyeline sahip olduğu bilinmektedir. Bu çalışmanın amacı gama-aminopropiltrietoksisilan (APTES) ile emprenye edilmiş kayın ve göknar odun örneklerinin fiziksel ve mekanik özellikleri ile birlikte çürüklük mantarlarına karşı performansını belirlemekti. Kayın ve göknar odun örneklerine APTES ile %20 ve %40 konsantrasyonlarda emprenye işlemi yapılmıştır. Kontrol ve emprenye edilmiş örnekler beyaz çürüklük mantarı *Trametes versicolor* ve kahverengi çürüklük mantarı *Coniophora puteana*'nın yanı sıra su alımı, eğilme mukavemeti ve termogravimetrik analize (TGA) tabi tutuldu. Yüzde ağırlık artışı, APTES ile emprenye işleminden sonar göknar örneklerinde kayın örneklerinden daha yüksekti. Kontrol ve emprenyeli örneklerde, kayın oduna göre göknar örneklerinde su alımı daha yüksek elde edilmiştir. Daha yüksek APTES konsantrasyonunun su alımını azalttığı bulunmuştur. Odunların APTES ile emprenyesi sonucunda eğilme direnci ve elastikiyet modülünü artırdığı tespit edilmiştir. Çürüklük mantarlarının neden olduğu kütle kaybı APTES emprenyesiyle önemli ölçüde iyileştirildi.

Anahtar Kelimeler − *Odun modifikasyonu, gama- aminopropiltrietoksisilan, su alımı, eğilme direnci, çürüklük direnci*

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1. Introduction

Wood has many excellent properties (carbon neutrality, high strength to weight ratio, aesthetic appearance, etc.), but is prone to dimensional changes with varying moisture regimes, is sensitive to ultraviolet light damage and is susceptible to biological degradation when wetted above the fiber saturation point (Zabel and Morrell, 2020). Conventional preservative treatments can protect wood from biological degradation, while water repellents can reduce moisture fluctuations that lead to physical damage. However, many timber users are eager to find non-biocidal methods for wood protection.

An alternative approach for protecting wood is to modify the chemical structure of the wood to reduce the hygroscopicity and rendering it less susceptible to dimensional changes and biological attack (Hill, 2006). Wood modification can take many forms including thermal modification, acetylation and furfurylation. All of these processes modify the hygroscopicity of the timber, making it less likely to experience the swelling and shrinkage that induces physical damage. The sharply reduced moisture uptake also reduces the risk of biological attack. While these are the most commonly studied modification processes, there are a variety of other strategies for non-biocidal wood protection including silanes. Silanes are silicon-based systems with the potential to include four functional groups that can be either organic or inorganic. Functional groups can impart water repellency, antimicrobial activity, compatibility between hygroscopic and hydrophobic materials and a range of other properties. Silanes provide considerable opportunities for improving wood properties without using biocides.

Silanes are used in plastics, textiles, buildings and papers as modification agents. They are utilized, for instance, to hydrophobize ceramics, create scratch-resistant surfaces, for soil proofing and anti-graffiti coatings, and improve adhesion between organic and inorganic materials (Donath et al. 2004). Silane modification has been shown to impart water repellency of wood (Tshabalala and Gangstad, 2003; Donath et al., 2007), increase biocide effectiveness and enhance fire retardancy (Saka et al., 2001). Unlike other water repellents such as oils or waxes, silanes do not strongly affect the sorption properties of wood. For example, tetraalkoxysilane treatment improved dimensional stability especially when hydrolysis and condensation of silanes was directed into the cell wall (Mai and Militz, 2004). Organo-silanes are bifunctional molecules containing three silicon functional alkoxy groups, mostly methoxy and ethoxy groups, and an organo-functional group. These materials can increase hydrophobicity or form covalent bonds with cell wall polymers.

Acylalkoxysilanes and arylalkoxysilanes have been used to achieve hydrophobization of ligno-cellulose materials. For example, a mixture of alkyl triethoxysilanes and dialkylthiethoxysilanes increased water repellency in wood compared to conventional silanes. The alkoxysilanes used also achieved some penetration of the wood cell wall, but further studies are needed to polymerize oligomers in the wood cell wall to create more substantial moisture resistance (Panov and Terziev, 2009). Hochmanska et al., (2014) reported that silane-modified wood exhibited improved hydrophobicity and resistance to UV degradation. Solvent-borne formulation with 5% aminoethylaminopropyltrimethoxysilane (AEAPTMOS) and an alkyd emulsion containing 5% methyltrimethoxysilane (MTMOS) provided the best protection against the accelerated weathering. Laboratory decay tests of amino-functional silane treated blocks in a separate study showed excellent efficacy against the brown rot fungus *Coniophora puteana* up to 18 weeks, while amino oligomeric silane systems limited decay by the white rot fungus *Trametes versicolor* over a 6-week period. However, the alkyl groups had little effect on water uptake of the wood (Donath et al., 2006).

Scots pine and beech wood samples impregnated with organo-silicones provided protection against *Postia placenta* and *C. puteana* fungi even at low low weight gains. Beech specimens were also partially protected against *C. puteana* and *T. versicolor*. Full protection was provided by some silicones against *C. puteana* for scots pine sapwood and *T. versicolor* for beech. The most promising products used solvent-based blends of the alkoxysilanes methyltrimethoxysilane (MTM) and octyltriethoxysilane (OTES) or a water-based micro-emulsion of polydimethylsiloxane (PDMS) and triethoxysilane (TES) at 20 and 30 % weight gains for Scots pine or 30 and 40 % weight gains for beech wood (De vetter et al., 2009).

The previous studies illustrate the potential for using silanes to reduce hydrophobicity, but there are other potential silanes worthy of study. Gamma-aminopropyltriethoxysilane (APTES) has been used to bond organics to metal oxides and has the potential to react with the wood to improve the physical and mechanical properties as well as enhance decay resistance. The objective of this study was to explore the potential effects of APTES on a softwood and a hardwood.

2. Materials and Methods

Freshly sawn beech (*Fagus orientalis* Lipsky) and fir (*Abies nordmanniana subsp. bornmulleriana* boards, free of defects, were obtained from a sawmill in Bartin, Turkey. The boards were cut into test specimens measuring 10 by 10 by 10 mm long for water uptake tests, 10 by 10 by 180 mm long for mechanical properties assessments, and 5 by 15 by 30 mm long for decay tests. The samples were oven-dried (103 °C) and weighed prior to testing. The dimensions of the water absorption samples were measured using digital calipers.

Gamma-aminopropyltriethoxysilane (APTES) was provided in liquid form from Azelis (Istanbul, Turkey). APTES was a liquid form, clear and colorless with a specific gravity of 0.95, silane coupling agent has the following structural formula: $H_2NCH_2CH_2CH_2Si(OCH_2CH_3)_3$. Merck malt extract agar with pH: 5.8 was used.

2.1. Impregnation

The wood samples were leached in 500 ml ethanol for 24 hours prior to impregnation. ASBESTO 20 or 40% (v/v) solution in ethanol was prepared. The specimens were submerged in the appropriate solution and subjected to a 30-minute vacuum (0.08 MPa) followed by 1 hour of pressure at 0.5 MPa. The samples were kept in the oven at 40°C for 2 days, and then they were subjected to the temperature of 103°C for 24 hours for curing. Weight Percentage Gain (WPG) was calculated based on the dry state of the samples before and after impregnation according to equation.

$$WPG\ (\%) = ((W_t - W_0)/W_0) \times 100 \tag{2.1}$$

where W_0 and W_t are the oven dry weights before and after treatment. A total of 60, 60, and 36 samples were treated with each solution for water uptake, mechanical properties and decay tests, respectively.

2.2 Water Uptake

Wood samples treated with 20 or 40 % APTES were immersed in distilled water in separate glass jars at room conditions for 6, 24, 48, 72 and 120 hours. Ten replicates were used for each experiment and untreated samples. The specimens were weighed, and their dimensions were measured using digital caliper without changing the water at each time point. The difference between wet weight and original oven dry weight was used to calculate net water uptake and the differences in dimensions between the pre-and post-soaking measurements were used to determine % swelling.

At the end of the water immersion period, the blocks were redried at 103°C and weighed. The oven dry mass difference before and after the water immersion was used to calculate % leaching loss.

2.3 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) of untreated and treated samples was performed with a Netzsch TG 209 F1 Iris TGA (NETZSCH Group Selb, Germany). About 2 mg of wood sample was placed on an alumina crucible dish (17 mm diameter) that was heated from 25°C to 500°C at a rate of 15°C min^{-1} under nitrogen atmosphere. The resulting curves were examined for differences between the treated and untreated samples.

2.4 Bending Strength and Modulus of Elasticity

The effect of treatment on flexural properties was assessed on 10 samples of each of the three groups (untreated, 20 % APTES and 40 % APTES) that were conditioned to constant weight at 20°C and 65% RH.

The samples were tested to failure on a Universal Testing Machine (U-TEST) with a 100kN max. load cell. The samples (10x10x200 mm) were loaded at a rate of 5 mm/mm and load and deflection were continuously recorded to failure. The resulting data were used to calculate modulus of elasticity (MOE) and modulus of rupture (MOR).

2.5 Decay tests

Untreated and treated blocks for the decay tests were oven-dried (103°C) and weighed to obtain a starting weight. For the agar media test according to principles of EN 113, malt extract agar solution of 4.8% concentration and samples were sterilized in an autoclave at a pressure of about 0.1 MPa at 121 °C for 20 min. The decay chambers were Petri dishes containing 4,8% malt extract agar inoculated with either *Trametes versicolor* (L: Fr.) Pilat. (Mad-697) or *Coniophora puteana* (Schum.: Fr.) P. Karst (Mad-515). The fungi were incubated until the agar surface was covered, then each plate received an untreated control and an APTES modified sample. The beech blocks were exposed to *T. versicolor* while the fir samples were exposed to *C. puteana*. The plates were incubated for 8 weeks at 25 ± 2°C and 75 ± 5% relative humidity. At the end of the test, the blocks were removed, scraped clean of any adhering mycelium and oven dried at 103°C before being weighed. Mass before and after fungal exposure was used as the measure of decay resistance. Each treatment was replicated on 6 blocks per fungus.

3. Results and Discussion

3.1. Water Absorption

Table 1

Effect of APTES modification on density, weight percent gain, swelling, and leaching mass loss of beech and fir blocks

Wood species	APTES Level (%)	Oven density (g/cm^3)	WPG (%)	Swelling (%)	Leaching mass loss (%)
Beech	Control	0,60±0,01	-	18,01±0,68	2,32±1,12
	20	0,65±0,01	9,04±0,73	18,55±1,04	6,71±0,91
	40	0,70±0,02	17,72±1,51	18,97±0,73	14,47±0,85
Fir	Control	0,39±0,03	-	11,68±0,88	0,71±1,14
	20	0,51±0,04	15,14±2,09	14,92±1,59	9,53±1,25
	40	0,57±0,05	31,66±4,89	15,33±1,78	19,98±2,91

APTES impregnation was associated with increased density and weight percent gain (WPG) for both species, but the effect was much greater in fir which increased 67.5 and 78.7 % more than beech samples treated with 20 or 40 % APTES, respectively (Table 1). APTES treated fir samples also tended to experience the greatest mass gains, perhaps reflecting their higher initial void volume, which created more capacity for cell lumen filling.

APTES treatment had no noticeable effect on swelling of beech, regardless of concentration. These results suggest that treatment had very little direct interaction with the cellulose hydroxyls. Conversely, APTES treated fir blocks swelled 27.7 % and 31.3 % more than the untreated controls for the 20 % and 40 % concentrations, respectively. These results suggest that APTES interacted with the wood cell walls in fir to render the cellulose more rather than less susceptible to swelling.

Leaching losses from the untreated beech controls were slightly greater than those for fir, but generally low. Leaching losses almost doubled for the 20 % APTES treated beech and then increased by 6.7 times the control for the 40 % treatment level. Leaching losses for the 20 and 40 % APTES treatment levels were proportional to the increased concentration. Leaching losses for the untreated fir control were lower than those for the beech; however, losses from the 20 and 40 % APTES treatments were 13.4 and 28.1 times those for the untreated control. Leaching losses were again nearly proportional to the increase in APTES concentration. These results suggest that a considerable amount of the APTES remained unreacted within either the lumen or the wood cell wall. These materials would then be expected to leach out in service. These results suggest that the use of lower APTES concentration might produce the same effects. Sebe et al. (2004) observed the highest leaching after 4 days of water soaking. Maximum silane loss obtained with n-propyltrimethoxysilane (PTMS) was 2 % after 14 days water soaking. According to Babicka et al. (2018), FTIR study showed that aminosilanes were leached from cellulose after water extraction. Wood impregnated with various tetra-alkoxysilanes with acetic acid as a catalyst showed stability against leaching with water. (Donath et al. 2004; Saka et al. 1992).

Water uptake tended to be lower in untreated beech than in fir, reflecting the greater void volume of the fir (Figures 1, 2). Most of the water uptake occurred within the first 6 hours of immersion and then rose more slowly with continued immersion. APTES impregnation reduced water uptake for both species and the effect was greater at the higher treatment concentration.

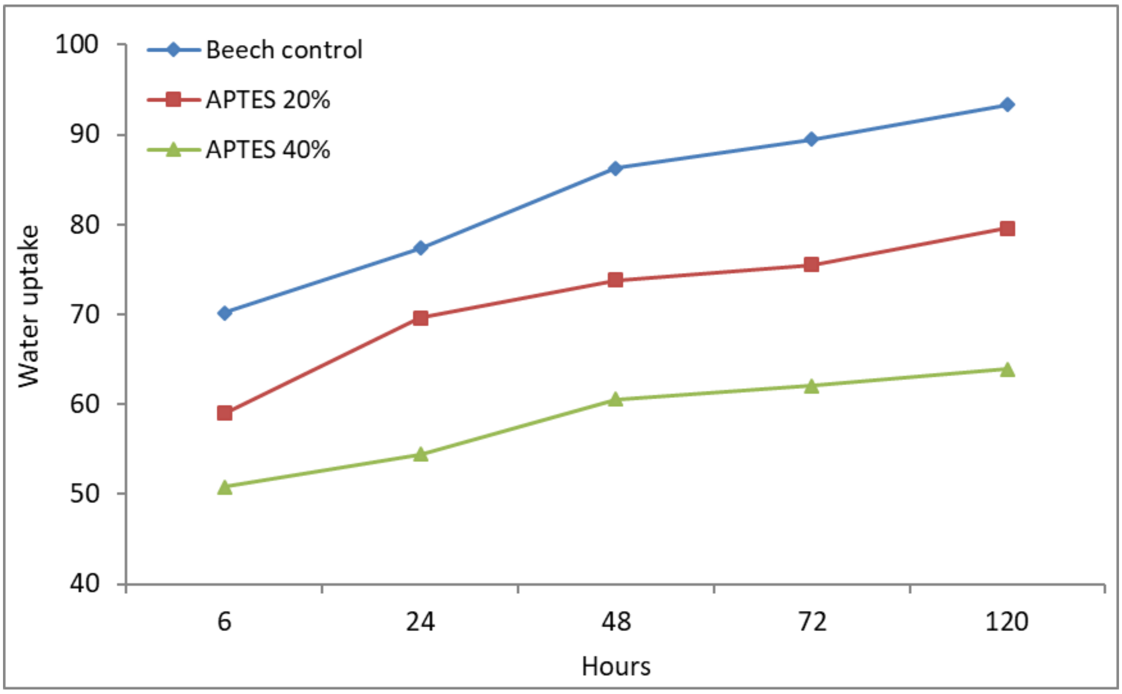

Figure 1. Effect of APTES treatment on water uptake of beech over a 5-day immersion period

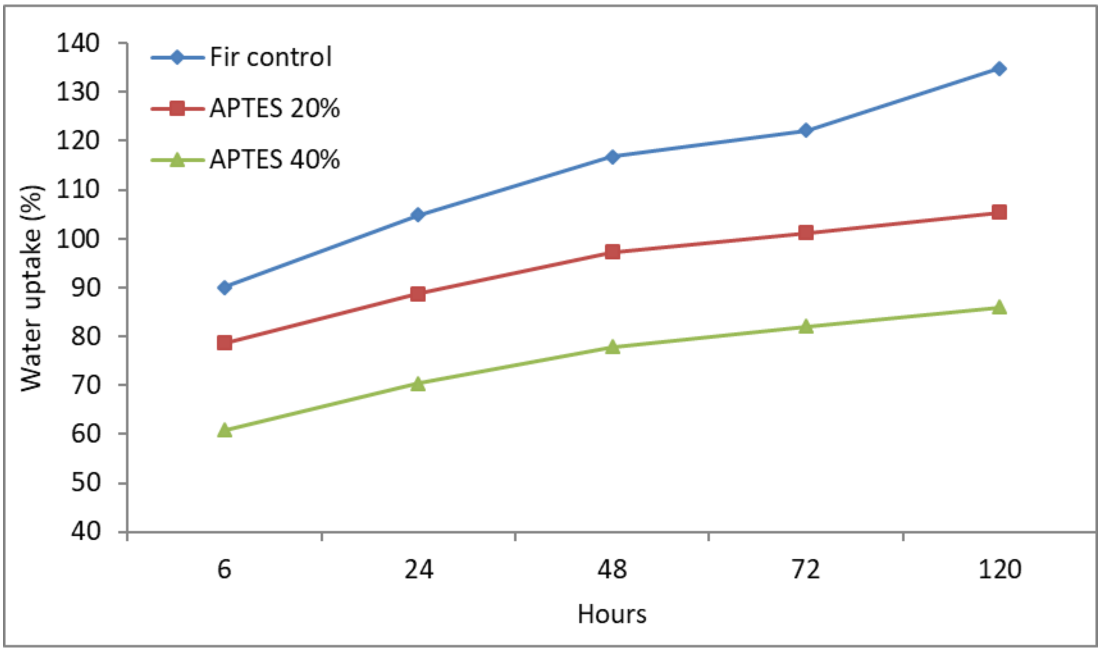

Figure 2. Effect of APTES treatment on water uptake of fir over a 5-day immersion period

3.2 Thermal Analysis of Wood Samples Modified with APTES

TGA results for untreated and APTES treated beech and fir were similar early in the heating phase up to 250 °C, but then losses slowed for APTES treated samples of both species (Figure 3). APTES concentration had little effect on weight loss for either species, but both levels produced a noticeable difference in final weight loss suggesting that APTES treatment rendered the substrate more resistant to heating.

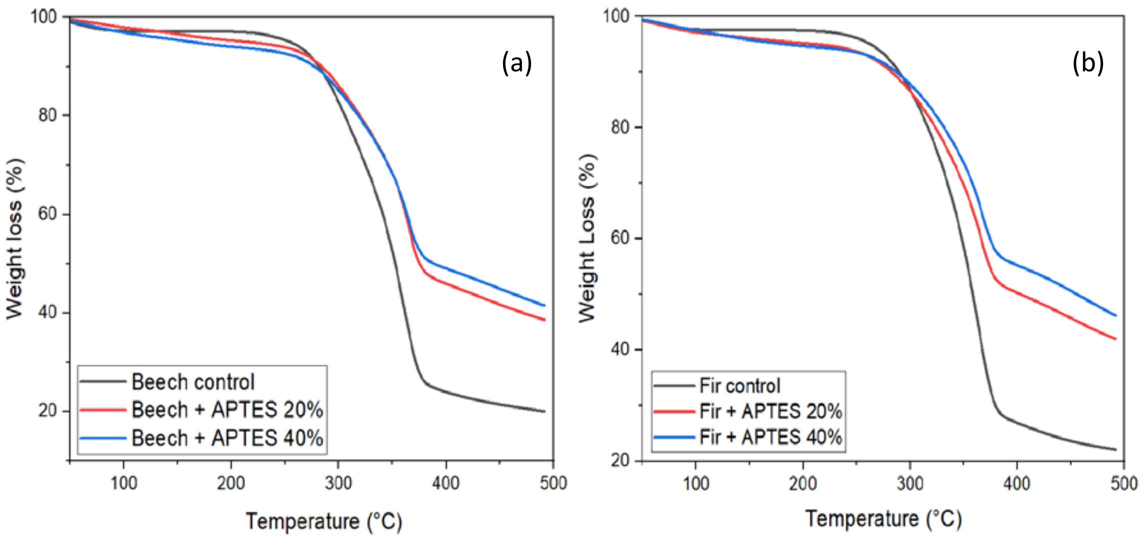

Figure 3. TGA curves of untreated beech (a) and fir (b) as well as samples of the same species treated with 20 or 40 % APTES

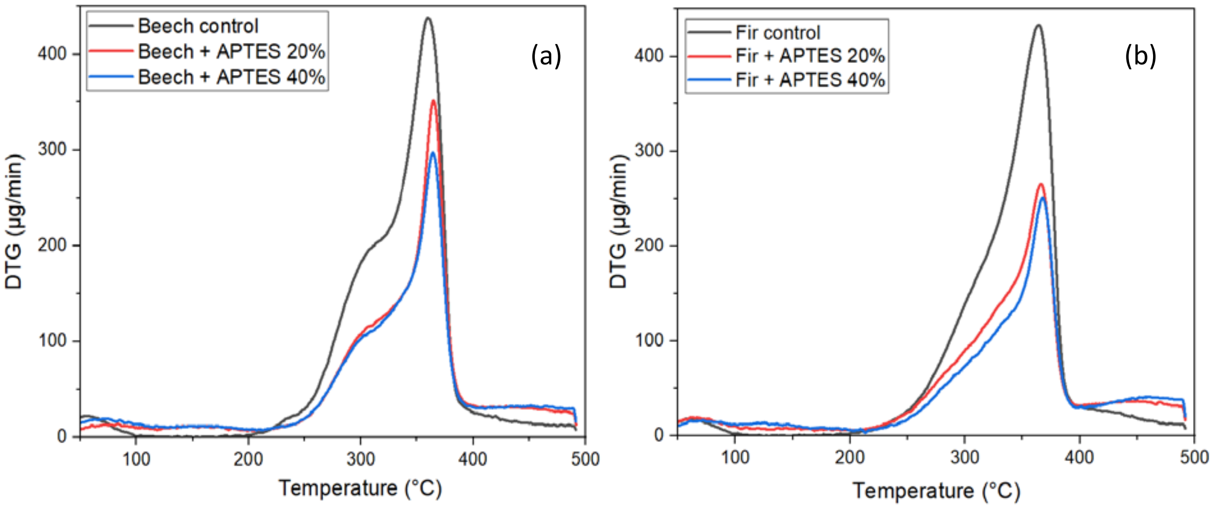

Figure 4. DTG curves of untreated beech (a) and fir (b) as well as APTES treated samples of the same species.

DTG curves were similar for the beech and fir for the untreated as well as APTES treated samples up to around 200°C, but then the untreated samples of each species experienced a sharp spike compared to either of the treatments (Figure 4). The peaks for both the 20 and 40 % APTES treated samples were far lower than those for the untreated controls, but there was little difference between the two concentrations. The results suggest that APTES modification had a limited effect on thermal behaviour.

Silane chemicals have been studied with composite materials by several authors. For example, γ-aminopropyl triethoxy silane showed improved thermal stability with the wood fiber component, which was attributed to the homogeneous distribution of the silane (Coutinho et al. 1998). Kim et al. (2011) investigated the effect of silane treatments on some properties of wood fiber-reinforced polypropylene composites and did not observe any effect of silane treatments on the thermal properties.

3.3 Mechanical Properties

Table 2

Flexural properties of untreated and APTES treated beech and fir

Species	APTES Conc (%)	MOR (N/mm^2)	MOE (N/mm^2)
Beech	None (control)	$98,67^b \pm 4,75$	$13165,52^a \pm 806,67$
	20	$106,01^b \pm 8,13$	$13990,31^a \pm 1046,22$
	40	$103,95^b \pm 6,74$	$13855,40^a \pm 667,43$
Fir	None (control)	$67,19^a \pm 8,24$	$11119,62^a \pm 2074,42$
	20	$72,06^a \pm 13,01$	$12006,14^a \pm 2148,63$
	40	$67,76^a \pm 6,74$	$11365,14^a \pm 1883,83$

Average MOR values for beech were greater than those for fir, which is consistent with previous reports for these two species. MOR's of APTES treated beech appeared to increase slightly compared to the controls, but there was considerable variation among the treatments and the differences were not significant (Table 2). Similar results were observed with APTES treatment of fir and suggest that the treatment had no significant negative effects on bending strength. These results also support the premise the APTES did not react with the cellulose or otherwise negatively affect the lignocellulosic matrix.

MOE values followed similar trends with slightly higher values for beech, and no real differences between untreated and the two APTES treatments (Table 2). One concern with compounds that react with the wood cell wall is that crosslinking or disruption of the lignocellulose matrix will embrittle the wood. This does not appear to be the case with either treatment level, and again supports the premise that APTES is not reacting with the cell wall polymers but is more likely bulking the cells.

3.4 Decay Resistance

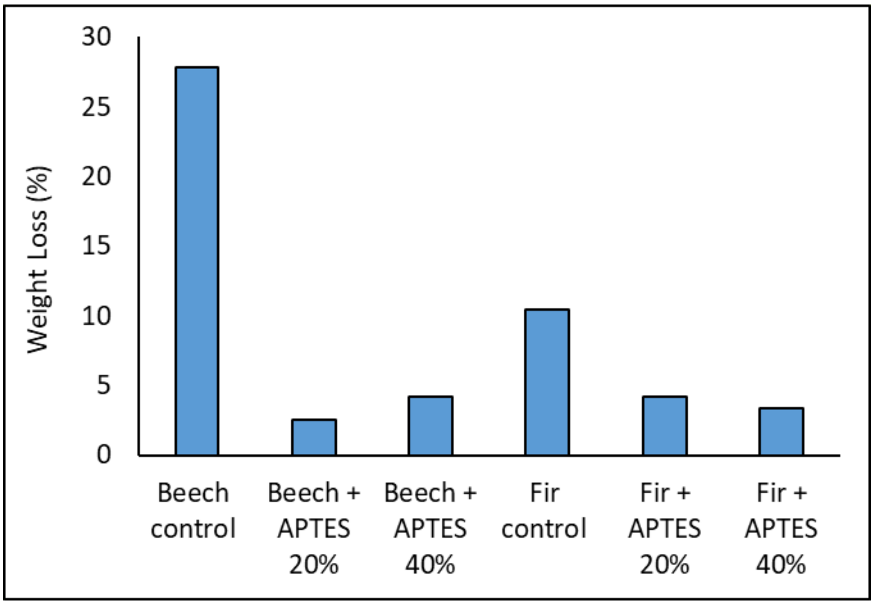

Figure 5. Effect of APTES treatment on mass losses of beech blocks exposed to *T. versicolor* or fir blocks exposed to *C. puteana* in an agar block decay test

Mass losses averaged 27.8 % for the untreated beech blocks exposed to *T. versicolor* and 10.5 % for the fir blocks exposed to *C. puteana*. The mass losses associated with beech indicated that conditions were suitable for aggressive fungal attack while those for fir were less vigorous, but still sufficient to begin to discern treatment differences. Increased decay resistance was also obtained with pine wood modified with a silicone-oligomer system containing amino groups (Mai et al. 2005).

Mass losses for beech blocks treated with 20 or 40 % APTES were both below 5 %, indicating that impregnation markedly improved decay resistance. While mass losses were slightly higher for the 40 %, the differences were slight and given the mass losses found in the water uptake test, could reflect leaching losses rather than fungal attack. Mass losses on APTES treated fir exposed to *C. puteana* were also lower than those for the controls but were less dramatic because of the low weight losses obtained with the untreated controls. In both cases, however, APTES treatment appeared to improve decay resistance. Alkoxysilanes treatments of *Araucaria angustifolia* including aminopropyl methyldiethoxysilane, aminopropyl triethoxysilane and an aminopropyl methyldiethoxysilane/aminopropyl triethoxysilane mixture were studied against brown rot (*Polyporus meliae*) and white rot (*Coriolus versicolor*) fungi. The best result was obtained with aminopropyl triethoxysilane, followed by the mixture of both alkoxides and finally aminopropyl methyldiethoxysilane (Giudice et al. 2013).

Earlier studies reported that organo-silanes methyltrimethoxysilane (MTMS), vinyltrimethoxysilane (VTMS), and propyltrimethoxysilane (PTMS) the impregnated into Scots pine sapwood were slightly effective against the brown-rot fungus *C. puteana* and the white-rot fungus *T. versicolor*, while3-aminopropyltrimethoxysilane (APTMS) with an amino group (NH_2) showed higher decay resistance (Reinprecht and Grznarik 2015).

According to Reinprecht et al. (2017), Scots pine sapwood modified with methytrimethoxysilane (MTMS) alone did not provide sufficient decay resistance against C. *puteana* or *T. versicolor*. However, they obtained more effective results when they two-step treatment incorporated a fungicide.

4. Conclusions

The APTES (Amino Propyl Triethoxy Silane) treatment has demonstrated significant improvements in the performance of wood samples. This treatment has notably reduced water uptake, slowed down thermal decomposition, and enhanced resistance to fungal attacks, particularly standing out for these benefits. However, it should be noted that when applied to fir wood samples, there seemed to be a slight increase in swelling.

Importantly, these treatments did not have any adverse effects on the flexural properties of the wood. The absence of any negative impact suggests that, at the tested levels, APTES primarily functions as a bulking agent with minimal interactions with the lignocellulosic matrix.

Author Contributions

Hüseyin Sivrikaya, designed the research, participated in the analysis process, and wrote the article.

Ahmet Can, analyzed the data and conducted a literature review.

Mesut Yalçın, collected data and conducted a literature review.

Jeffrey Morrell, commented the test results and made final edits of the article.

Conflict of Interest

The authors declared no conflict of interest.

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