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Research Article

WATER ABSORPTION, ANTI-SHRINK EFFICIENCY AND DECAY RESISTANCE OF TREATED WOOD BY SILICA BASED SOLUTIONS

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ABSTRACT

In this study, the effect of two different silica (SiO_2) based solutions on water absorption, anti-shrink efficiency and decay resistance of Scots pine wood was studied. Sol-gel process was used in order to prepare SiO_2 based solutions. One of the SiO_2 based solutions (Sol-gel 1) was prepared by using tetraethoxysilane (TEOS) and de-ionized water (TEOS:H_2O=1:1/2). The same precursors was used for preparing the other solution (Sol-gel 2) but with a different molar ratio of TEOS:H_2O=1:4. Scots pine wood specimens were first vacuum impregnated with the solutions and then cured. The level of water absorption and anti-shrink efficiency were determined with cyclical wetting tests, total of 14 days. Specimens were exposed to brown rot fungus, *Coniophora puteana* attack according to modified EN 113 standard to determine the best SiO_2 based solution for sufficient decay resistance. Leached specimens were also suspected to decay test in order to evaluate any loss in effectiveness in decay resistance due to possibility of silica leaching. Both solutions had similar weight percent gains in wood, around 25%. SiO_2 treated specimens decreased water absorption of wood as 20% in comparison with un-treated controls. Anti-shrink efficiency of wood was found as 26% for Sol-gel 1 solution and 35% for Sol-gel 2 solution at the end of the test. Decay resistance of treated specimens was in the range of 63-91% in comparison with controls. Sol-gel 2 solution were found efficacious in suppressing *Coniophora puteana* attack when no leaching prior the decay test was used, however, Sol-gel 1 solution seemed to be ineffective against fungus attack that exhibited more than 3% weight loss. Leached specimens had higher weight loss than un-leached specimens. The silica in leached wood supposed to be not sufficient to prevent brown rot fungus attack on wood. Results clearly showed that Sol-gel 2 solution had better water absorption and anti-shrink efficiency rates, and decay resistance than Sol-gel 1 solution.
Keywords: Scots pine, sol-gel, decay resistance, anti-shrink efficiency, water absorption.

1. INTRODUCTION

Wood has been used as an environmentally friendly material in indoor and outdoor applications. However, it has disadvantages such as biodegradability, dimensional instability, flammability and photo-degradation. To overcome those disadvantages, appropriate preservation methods are needed. Due to increasing environmental concerns, new methods and chemicals are still being investigated to be an alternative to traditional ones [1-6].

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One of the strategies of the recent studies is to protect wood with using inorganic silicon compounds. Actually, silanes are known as modifying agents in the plastic, textile, construction and paper industries. They are used for hydrophobization of ceramics, scratch resistant surfaces, soil proofing and anti-graffiti coatings, or as an adhesion promoter between organic and inorganic materials [7]. Wood treated with tetraalkoxysilanes showed improved dimensional stability, durability and fire resistance to a certain degree [8]. In addition, wood treatment with tetraethoxysilane and alkyltriethoxysilanes provide strong hydrophobicity and partial fungal resistance [7-9]. Donath et al. [7] compared performance properties of wood specimens impregnated with three different sol-gel precursors, namely: TEOS, methyl triethoxysilane, and propyl triethoxysilane. They observed that wood properties – such as cell wall bulking, anti-swelling efficiency, moisture uptake, and durability – improved significantly in wood specimens treated with the monomeric sol-gel precursors compared to those specimens that were treated with oligomeric sol-gel particles. Sebe et al. [10] showed that n-propyl-trimethoxysilane reacted by alcoholysis with hydroxyl groups present in the wood substrate. This study showed that despite the sensitivity of the C-O-Si bond to hydrolysis, a significant amount of silane remained in the wood even after 14 days in water. Tanno et al. [11] investigated the application of sol-gel formulations to wood to increase its dimensional stability and resistance to termites and fire, and also developed a sol-gel system that increases the fungal resistance of wood. In that study, the sol-gel system consists of tetraethoxysilane (TEOS) precursor mixed with small amounts of 2-heptadecafluorooctyl trimethoxysilane and 3- (trimethoxysilyl) -propyl- (carboxymethyl) -desylmethyl ammonium hydroxide. In the study of Saka and Tanno [12], TEOS as a silane component was compared with methyltrimethoxysilane (MTMOS). The weight percent gain (WPG) of wood treated with MTMOS and TEOS was similar, but MTMOS resulted in a higher volume although the concentration in the impregnation solution was lower.

The sol-gels in the study were developed for a coating of glass to prevent migration of alkalis from the surfaces in a previous study by Sam et al. [13]. The researchers found that glass coatings that have a SiO_2 barrier layer exhibited better performance by giving higher photoactivity constants. This study was conducted to determine whether a similar barrier layer could be formed in the wood with the same sol-gels. For this purpose, wood was impregnated with two different SiO_2 based solutions, and then water absorption, anti-shrink efficiency and decay resistance against *C. puteana* attacks were investigated.

2. MATERIALS AND METHODS

2.1. MATERIALS

Wood specimens from sapwood of Scots pine (*Pinus sylvestris* L.) with dimensions of 15 mm (radial) x 5 mm (tangential) x 30 mm (longitudinal) were prepared for the study. Specimens without any visible defects such as cracks, strain and knots were selected for the experiments, and then oven-dried prior the treatments. Tetraethoxysilane (TEOS) was sourced from Sigma Aldrich (St. Louis, MO, USA). Two SiO_2 based solutions were prepared. One of the SiO_2 based solutions (Sol-gel 1) was prepared by using TEOS and de-ionized water (TEOS:H_2O=1:1/2). The same precursors was used for preparing the other solution (Sol-gel 2) but with a different molar ratio of TEOS:H_2O=1:4.

2.2. METHOD

The specimens were first vacuum impregnated with solutions at 700 mmHg for 45 min, and were then immersed in the solutions for 60 min at atmospheric pressure. The specimens were cured at 100°C for 24h. Weight percent gain of the specimens (WPG, %) was calculated on the basis of the oven dry weight of specimens before and after impregnation. Then, specimens were conditioned at 20°C and 65% RH for 2 weeks.

Six replicates of treated and untreated wood specimens were placed into beakers filled with deionised water. After defined times (24h, 48h, 72h, 96h, 120h, 144h, 168h and 336h), the specimens were removed from the water, dabbed of with tissue and weighed. The dimensions of the specimens were measured. This procedure continued for a total of 14 days. Relative water uptake (WA, %) (Eq. 1) and anti-shrink efficiency (ASE, %) (Eq. 2) were determined.

$$WA = [(W2- W1) / W1] \times 100 \qquad (1)$$

$$ASE = [(Su- S) / Su] \times 100 \qquad (2)$$

Where,

W2 = wet weight of wood specimens after wetting with water
W1 = initial dry weight
Su = volumetric swelling of untreated wood
S = volumetric swelling of treated wood

Decay test was performed according to principles of EN 113 [14] with some modifications both for leached (L) and unleached (UL) specimens with 6 replicates for each group. Water immersed specimens were used as leached specimens. Brown rot fungus, *Coniophora puteana* (Schumach.) P. Karst. (Mad-515) was used in the test. Malt extract agar of 4.8% concentration and specimens were sterilized in an autoclave at pressure of about 0.1 MPa at 120°C for 25 minutes. Fungi cultures were inoculated to sterile malt extract agar medium in the petri dishes. After incubation period of inoculated petri dishes, one impregnated and one control specimens were placed on the growing mycelium in each petri dish. The petri dishes were then incubated at 20ºC and 70% RH for 8 weeks. After the test, all wood specimens were removed from the petri dishes and cleaned from the surface mycelium. Then, they were dried at a temperature of 103±2°C, weighed, and the weight loss (WL, %) was calculated on the basis of oven dry weight before the test. Decay resistance of impregnated specimens defined as a percentage change was calculated based on the weight loss of control specimens.

3. RESULTS AND DISCUSSION

3.1. WEIGHT GAIN (WPG), WATER ABSORPTION RATE (WA) AND ANTI-SHRINK EFFICACY (ASE)

The weight percentage gain (calculation based on the weight difference between untreated and treated wood specimens) is a good indicator of an efficient impregnation (i.e., chemical bonding or interaction between the introduced chemical and wood cell wall polymers) [15]. Weight percent gain of specimens was found to be 25.02 (±1.99) and 26.29 (±2.53)% for sol-gel 1 and sol-gel 2, respectively. Both solutions had similar weight percent gains in wood. The WPGs were consistent with the previous studies by Donath et al. [7, 16]. Donath et al. [16] found WPG value of approximately 22.3% after impregnation with three monomeric silanes in pine sapwood, and Donath et al. [7] found WPG value between 25-30% after impregnation with alkoxysilanes in beech.

The average values of water absorption and anti-shrink efficiency of specimens are shown in Figs. 1 and 2. Water absorption of the control specimens increased from 69.48% to 155.04% during immersion in water. All treated specimens had lower water absorption than that of the controls after 14 days. Impregnated specimens had water absorption rate from 93.11 to 124.65%. At the end of the water immersion test, treated specimens decreased water absorption of wood as 20% in comparison with controls. In the beginning periods of the test, up to 120h, treated specimens absorbed more water than controls. A continuous increase was observed on absorbed water by controls while a slight increase on WA values was observed with treated specimens along with water immersion periods. A remarkable difference on WA was not observed between two solutions. Donath et al. [16] found that water repellence of specimens treated with three types of silanes strongly diminished after a longer submersion time (24 h). It was reported that the reduction in hydrophobicity after longer submersion time was not caused by removal of silanes during submersion. This was explained by continued condensation of unreacted silanol groups in the aqueous functional silanes during the wetting-drying cycles.

Anti-shrink efficiency of Sol-gel 1 was found to be 32.12% for 24h and 25.90% for 336h. In the case of Sol-gel 2, it was found as 40.56 and 35.04% for 24h and 336h, respectively. The ASE values decreased during 72h but then they tend to increase slightly until the end of the test. The reduction of ASE from beginning to end of the test was 19% for Sol-gel 1 and 14% for Sol-gel 2. This could be due to Si–O–C-bonds are susceptible to hydrolysis [17]. Sol-gel 2 exhibited better dimensional stability than Sol-gel 1. Sol-gel 2 might penetrate and polymerize in the wood better than Sol-gel 1 since it was prepared with less molar ratio of TEOS to H_2O (1:4). High rate of un-reacted silanol groups might presence on wood impregnated with Sol- gel 1.

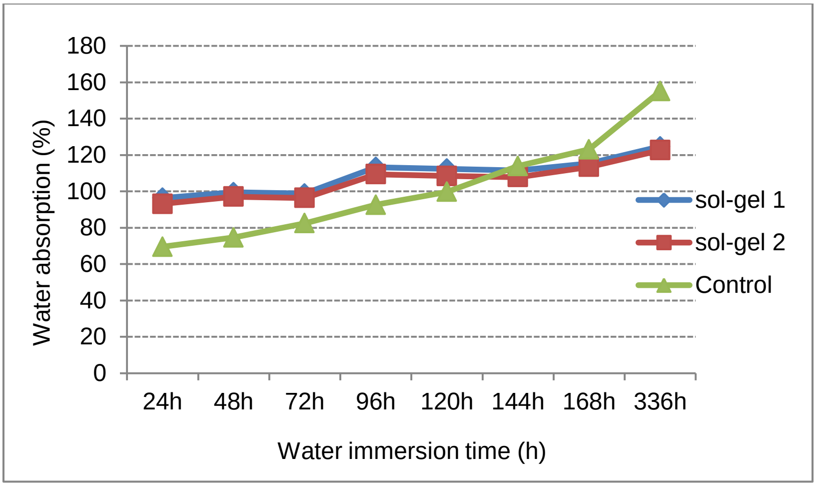

Figure 1. Water absorption of specimens during 14 days of water immersion

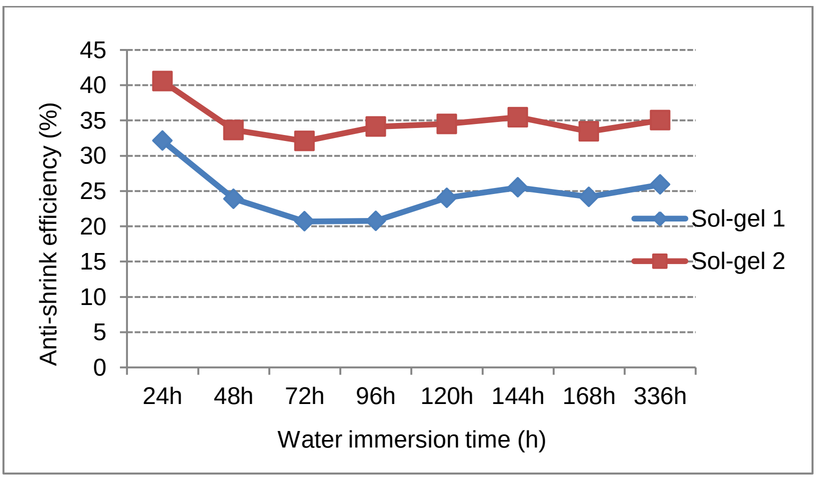

Figure 2. Anti-shrink efficiency of specimens during 14 days of water immersion

3.2. DECAY RESISTANCE OF SPECIMENS

Fig. 3 summarizes the average values and standard deviations of the weight loss caused by *C. puteana* attack. The control specimens were severely degraded by the fungus. The weight loss of the control specimens was found to be greater than 25%. All treated specimens exhibited better decay resistance than controls. Weight loss of specimens treated with Sol-gel 1 was higher than required a maximum weight loss (3%) for a candidate wood preservative according to EN 113 [14]. Anti-fungal effect could not be proven by Sol-gel 1. Only the slightly improved decay resistance was probably achieved by water repellency. Sol-gel 2 was found efficacious in suppressing *C. puteana* attacks when no leaching prior the decay test was used, however, it seemed to be ineffective after leaching. Goethals and Stevens [18] reported that poor activity was observed with a silylation reaction for pine and beech against *C. versicolor* and *C. puteana* attacks. The poor decay resistance of the sol-gels may also probably caused by the fact that not all of the silanol groups present react with the hydroxyl groups of wood [18]. Hydrolysis and condensation reaction of silanes was also reported [16, 17]. This finding is also compatible with ASE results.

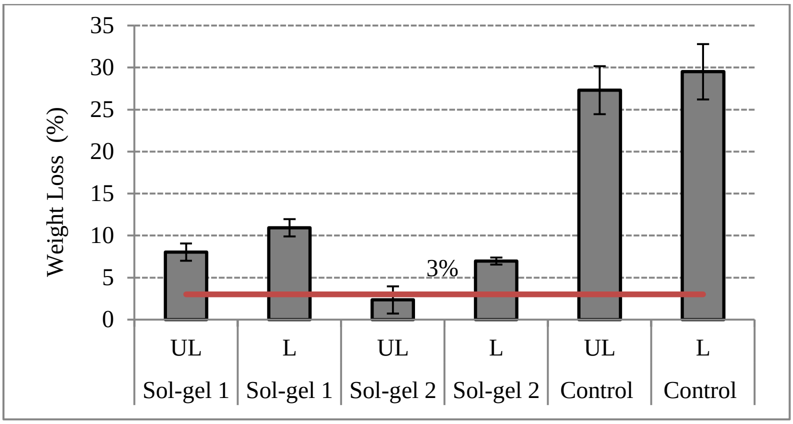

Figure 3. Weight loss of decayed specimens by *C. puteana*

4. CONCLUSIONS

In this study, two different silica based solutions were evaluated in order to investigate their water absorption and anti-shrink efficiency rate, and decay resistance against *C. puteana* attacks. Results showed that solutions increased water repellency and dimensional stability of wood. However the efficiency tended to decrease after longer water immersion periods, probably related with hydrolysis of silanes. Sol-gel 2 gives a promising result on preventing attack of *C. puteana*. However after leaching, sufficient decay resistance was not obtained with Sol-gel 2. As a consequently, the performance of the solutions were far poorer than that of the commercially available formulations in outdoors. However, impregnated wood can be used for interior purposes (Hazard class 1) and for internal humid conditions and protected external use (Hazard class 2). SEM images can help to understand the protection mechanism of Sol-gel 2 and are needed for further studies.

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