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BRIEF ORIGINAL

Wood treatment by inorganic alumino-silicate polymers

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Abstract
This work assesses the effectiveness of dilute inorganic alumino-silicate polymer suspensions in enhancing wood's durability, fire resistance, and mechanical properties. Alumino-silicate polymer-treated wood exposed to *Coniophora puteana* lost less than 3% mass on average, compared to 20% for controls. The moduli of elasticity and rupture of treated specimens were nearly twice as high as those of the controls. Coating with the inorganic polymer was also effective as a fire retardant. Results indicate that inorganic alumino-silicate polymers are a very promising wood treatment.

1 Introduction

Inorganic alumino-silicate polymers refer to aluminosilicate solids that are produced by reacting an aluminosilicate precursor, with alkali activator which results in an amorphous solid (Sá Ribeiro et al. 2016). Alkali-aluminosilicate-based coatings (Krivenko et al. 2013) and geopolymer-based paints (Guzii et al. 2020) have previously been applied to increase the fire resistance of wood. Wood particles are commonly employed as filler material within alumino-silicate polymers but, inversely, the use of alumino-silicate polymers as a wood impregnant has not been tested. The aim of this paper is to scope the potential of using dilute inorganic alumino-silicate polymer precursor as a sole ingredient in wood preservation. We show that its dilute suspensions will enhance the sapwood specimens' biological resistance, mechanical properties and fire retardancy.

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2 Materials and methods

2.1 Inorganic alumino-silicate polymer and specimen preparation

Metakaolin (Metaver® M, Newchem GmbH, Austria) was added in an ice bath to activation solution of sodium hydroxide, sodium silicate and water stirred in a closed plastic container for 24 h with a mass ratio between Si: Al: Na: H_2O of 4:1:1:17, making a total dilution of 26.1%. To be used as a wood impregnant, the resulting mixture was diluted to 5, 10 and 20% using MilliQ water (Merck KGaA, Darmstadt, Germany), resulting in a total solid content in water of 1.3%, 2.6% and 5.2% respectively.

Scots pine (*Pinus sylvestris*) sapwood specimens of dimensions $5 \times 10 \times 40$ mm^3 (radial × longitudinal × tangential) were oven dried at 50 °C and weighted. Each inorganic polymer suspension was used to impregnate 20 sapwood specimens at room temperature: The sapwood specimens were submerged in the dilute inorganic polymer suspension (either 5%, 10% and 20%) and then vacuumed at 20,000 Pa. After 30 min, pressure was subsequently increased to 1,000,000 Pa. After one hour, the pressure was released and the specimens were taken out of the dilutions, sealed into Ziplock bags and kept 24 h at 40 °C and 14 days at room temperature for curing. After final oven drying at 50 °C their dry mass was again recorded. Untreated wood specimens were used as controls. The samples for the fire-retardancy test (cone calorimetry) were prepared by coating plywood specimens ($10 \times 10 \times 2$ cm^3) with 5%, 10%, 20% and undiluted inorganic polymer suspension, sealing them

into Ziplock bags at 40 °C and kept 14 days at room temperature. Untreated plywood specimens were used as controls.

2.2 Mini-block decay test

The decay performance of the inorganic polymer-treated specimens ($5 \times 10 \times 40$ mm^3) was assessed exposing the wood specimens to the wood-decaying fungus *Coniophora puteana* (strain BAM 112), following a modified version of EN 113 (2021). All the wood specimens were autoclaved (120 °C, 15 min). 10 replicates from each treatment (5, 10 and 20%) and control were placed in each Petri dish (Ø 90 mm and 20 mm height) with a colony of *C. puteana* previously grown in malt agar media. A plastic mesh was placed between the fungus and the wood specimens to avoid direct contact of the wood, and the Petri dish was then sealed with parafilm and kept in a growth chamber at 20 ± 2 °C and 65 ± 5% RH. After 16 weeks the wood specimens were taken out from the dish and the hyphae that grew over them were gently removed. The specimens were weighed after drying at 50 °C when specimens reached constant mass to calculate decayed mass loss (%). The wood's durability class was assessed according to EN 350 (2016).

2.3 Mechanical test (3-point bending test)

10 replicates from each treatment and controls were conditioned to constant weight at 20 °C and 65% RH. A three-point bending test was performed using a Zwick Roell 050 materials testing setup, to calculate the modulus of elasticity (E_{mod}) and modulus of rupture (MOR).

2.4 Cone calorimetric analysis

The fire resistance of the inorganic polymer-treated plywood specimens ($10 \times 10 \times 2$ cm^3) was analyzed following the ISO 5660-2 Standard Test Method, with a cone calorimeter (ConeTool 1; SGS Govmark Ltd., Farmingdale, IL, USA) at a radiant flux of 50 kW/m^2. The measurement of the heat release rate was conducted by analysing the oxygen consumption and flow rate within the combustion product stream. Two replicates per treatment concentration were analysed.

2.5 Statistical analysis

The statistical analyses of the decay test of alumino-silicate polymer treated wood specimens compared to controls were performed using IBM SPSS Statistics 29.0, using Tukey's range test as post-hoc for ANOVA.

3 Results and discussion

The dry mass gain was 18.6%, 15.2% and 15.4% for specimens treated with 5%, 10% and 20% solutions respectively. The visual appearance of treated wood specimens was like the untreated ones, even if the colour was slightly brighter and a thin whiteish layer was visible in the longitudinal direction of the specimens. The lowest alumino-silicate inorganic polymer concentration with the lowest viscosity yielded the best-performing specimen highlighting the need for further recipe optimization. The effects of the alkalinity of the inorganic polymer in the wood specimens has not been studied.

All inorganic polymer concentrations protected wood from the decay caused by *C. puteana*, as the avg. mass loss of the specimens of all the treatments was less than 3% (Table 1) and was significantly lower than that of control specimens based on the statistical analyses. Based on EN 350 (2016), the controls were slightly durable (Durability class 4) while all the inorganic polymer treated specimens exhibited a very durable durability class (Durability class 1). These first results show high potential of alumino-silicate inorganic polymers against decay, but several long-term durability analyses are still required to verify this. Based on the statistical analysis, the moduli of elasticity and rupture of treated specimens increased significantly, being about 100% higher than the mean of the control specimens. The mechanism behind the durability improvement has not been tested but could be related to filling of the pores of the wood and the alkalinity of the inorganic polymer on wood, which have not been studied and may impact the durability and mechanical properties of wood, requiring further investigation.

The time to ignition increased when the wood was treated with any of the inorganic polymer solutions (Table 2) what agrees with previous findings (Krivenko et al. 2013). The

Table 1 Mean mass loss after 16 weeks of exposure to *Coniophora Puteana*, durability class based on EN 350 (2016), moduli of elasticity (E_{mod}) and rupture (MOR) of controls and treated wood specimens. Treatments that do not share the same letter are significantly different from each other based on Tukey's range test

Treatment	Mass loss (%)	Durability class	E_{mod} (GPa)	MOR (MPa)
5%	1.3 ± 0.3^{a}	1 (Very durable)	3.8 ± 0.2^{b}	84.1 ± 2.6^{b}
10%	1.7 ± 0.3^{a}	1 (Very durable)	4.5 ± 0.5^{bc}	90.6 ± 4.4^{bc}
20%	2.6 ± 0.3^{a}	1 (Very durable)	4.4 ± 0.5^{bc}	87.0 ± 4.1^{bc}
Control	20.5 ± 3.8^{b}	4 (Slightly durable)	2.2 ± 0.1^{a}	48.1 ± 1.2^{a}

Table 2 Fire test results of Scots pine sapwood specimens treated with alumino-silicate polymer (5%, 10%, 20% and undiluted) and untreated specimens. Two replicates were used for all the treatments, apart from 100% alumino-silicate polymer which had only 1 replicate

Sample	Replicate	Time to ignition (s)	Mean heat release rate (kW/m²)	Peak heat release (kW/m²)	Mass loss (%)	Total smoke (m²/m²)
Control	1	13.0	90.7	273.0	40.6	95.5
	2	13.0	103.3	261.0	46.3	148.6
5%	1	16.0	69.4	189.4	48.4	25.3
	2	13.0	71.4	143.8	50.2	18.6
10%	1	25.0	80.4	141.4	41.7	34.7
	2	17.0	88.5	127.6	45.4	43.6
20%	1	19.0	68.8	123.4	48.1	13.3
	2	17.0	76.3	136.6	52.1	29.2
100%	1	44.0	46.8	153.4	63.8	394. 5

total smoke, mean and peak heat release decreased, while the mass loss was similar for all. The wood specimen coated with undiluted inorganic polymer showed very high time to ignition and low mean and peak heat release compared to controls, but also a higher amount of total smoke. However, in most of the undiluted polymer-treated samples the coating cracked, having only one remaining specimen to test.

4 Conclusion

It can be concluded that the use of alumino-silicate inorganic polymers as a treatment for wood is a very promising method which improves the wood properties. Even at the lowest concentration tested, inorganic polymers provided an adequate wood protection based on EN 113 and the modulus of elasticity and rupture were improved by almost 100% from that of the untreated wood specimens. Alumino-silicate inorganic polymers seem to perform very well also as a fire-retardant wood coating.

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Data availability The data will be made available on request.

Declarations

Competing interests The authors declare no competing interests.

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