INVESTIGATION OF DECAY RESISTANCE OF POPLAR WOOD IMPREGNATED WITH ALKALINE COPPER, UREA–FORMALDEHYDE, AND PHENOL–FORMALDEHYDE RESINS

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Abstract. The decay resistance of modified fast-growing poplar lumber with ion-based preservative and thermosetting resins were determined in laboratory and field stake tests. Commercial alkaline copper quat-type D (ACQ-D), thermosetting phenol–formaldehyde (PF) resin with varying concentrations, and urea–formaldehyde (UF) resin were used to improve decay resistance of low-grade fast-growing poplar. The results indicated that the concentrations of PF and weight gains of resin-modified specimens were highly correlated, and target resin retains can be achieved from resin concentration. The retention rates of ACQ of 1.1% concentration, UF of 41.5% concentration and PF of 10% concentration were 6.72, 18.43, and 7.31 kg/m^3, respectively. For PF, the retention rate is linearly related to concentration. The mass losses (ML) for the untreated, ACQ-treated, and UF-treated specimens were 26.34%, 8.91%, and 11.66% after 12 wk of incubation in laboratory, respectively. The ML for the treated specimens were 9.74%, 7.32%, 3.14%, 2.38%, and 2.41% for PF impregnated wood at concentrations of 10%, 15%, 20%, 25%, and 40%, respectively, after 12 wk of incubation in the laboratory.

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After 24-mo field tests, the loss and damage rates of the treated specimens were 83%, 67%, 50%, and 100% for the ACQ-D, UF, PF, and untreated specimens, respectively. The specimens modified with thermosetting resin have very good decay resistance.

Keywords: Decay, durability, mass loss, poplar, resin.

INTRODUCTION

Wood is the building material with relatively low processing cost and high strength. However, wood will decay, resulting in a loss of mechanical properties (Ryu et al 1991; Deka and Saikia 2000). Wood is exposed to environmental factors, and fungi and other pathogens feed on wood if used without any treatments (Yang et al 2012). The physical and chemical modifications have been used to improve its resistance to the attacks of fungi and mold. Copper-based preservatives have been extensively used to improve the decay resistance because of their high effectiveness and low toxicity to humans and animals (Humar et al 2001). However, research has also shown that water-soluble copper-based preservatives are less effective in protecting hardwoods than softwoods (Butcher 1979; Smith et al 1996; Lebow et al 2010). The ammonium copper quaternary compound has been widely accepted because of its excellent resistance to the decay fungi (Goodell et al 2007; Gaspar et al 2010). ACQ-B and ACQ-D in the ACQ series are widely used. The quaternary ammonium was didecyldimethylammonium chloride (DDAC) with the ratio of CuO to quat of 2:1 in ACQ-B according to American Wood Protection Association. For ACQ-D, the increases in concentration of ethanolamine ligand resulted in increased coordination of copper with ethanolamine ligand and decreased free copper ions in the solution (Pankras et al 2009). The effect of CCA and ACQ on the mechanical properties of wood was negligible (Yildiz et al 2004; Barnes and Lindsey 2009). The copper in waterborne preservatives is vulnerable to leaching during service (Zhang and Kamdem 2000). It is hard to completely inhibit the leaching (Ung and Cooper 2005; Humar et al 2006; Humar and Žlindra 2007; Tascioglu et al 2008; Lin et al 2009; Yu et al 2009).

As a nonchemical modification to increase the decay resistance of wood, heat treatment has been proved to be efficient. During heat treatment at 180-260°C, hemicelluloses and lignin degraded and new compounds emerged in the wood (Jämsä and Viitaniemi 2001; Tjeerdsma and Militz 2005). Thermal modification cause mass loss of up to 47% for southern pine and reduce MOR by up to 42.28% (Kandem et al 2002; Icel et al 2015). The mold and termite resistance of thermally treated wood was not effective (Kocaefe et al 2007; Surini et al 2012). The heat treatment is not suitable for wood used as structural materials (Wang et al 2013).

Phenol–formaldehyde (PF) and urea–formaldehyde (UF) resins are thermosetting resins and have been in the market for a long time. PF is majorly used in exterior applications, whereas UF used in indoor products (Forest Products Laboratory 1999). The fungi and termite resistance of both resins have been proved to be excellent (Mallari et al 1990; Kajita and Imamura 1991; Ryu et al 1993). The literature indicated that PF can be an efficient preservative because of the lower mass loss of the treated wood than the untreated specimens (Yue et al 2008). Kajita and Imamura (1991) found that the biological properties of PF-based particleboards was much better than that of the control using burial in moist and unsterile soil test. The reference reported that the average life of PF-modified specimens was longer than that of the untreated specimen (Woodward et al 2011). Bakar et al (2013) found that the mass losses (ML) of wood impregnated with phenolic resin were lower than those of the untreated wood on 28-d exposure to termites and a white-rot fungus. Low molecular weight oligomeric PF and UF are composed of small molecules that can impregnate cell lumens and pits easily (Ryu et al 1993; Kamke and Lee 2007). The small molecules filled the cell lumen and cell walls and were polymerized under thermal treatment to form a cross-linked complex (Laborie 2002; Furuno et al 2004). A small amount of resin components reacted with the ingredients of the cell wall by chemical bonding (Gindl 2001; Gindl and Gupta 2002).

The objectives of this study was to investigate the decay resistance of fast-growing poplar and improve it using low molecular weight thermosetting resins. To study the effect of concentration of PF on the decay resistance of treated wood, the untreated, ACQ-treated, and UF-treated specimens were used for comparison, and a combination of incubation in the laboratory and field tests was employed.

MATERIALS AND METHODS

Materials

A 11-year-old fast-growing poplar tree with the diameter of 38.3 cm at breast height was felled in Jiangsu Province, Southeast China. Poplar wood is a low density hardwood, and it has poor decay resistance. The defect-free specimens were cut from lumber having annual ring widths between 28.5 and 31.5 mm. The specimens were then dried to a MC of 11-13%. The specimens with dimensions 25 mm × 55 mm × 950 mm were divided into eight groups with each group having six specimens. The eight treating conditions are as follows: unimpregnated (control), ACQ-D treated (TA), 41.5% UF treated (TU), 10% PF treated (T10P), 15% PF treated (T15P), 20% PF treated (T20P), 25% PF treated (T25P), and 40% PF treated (T40P). Specimens with dimensions 20 mm^3 × 20 mm^3 × 10 mm^3 for laboratory tests and 20 mm^3 × 20 mm^3 × 300 mm^3 for field tests were prepared.

The formula and synthesis of UF and PF were followed according to Yue et al (2016). Their viscosities were 17.5 and 15.8 s at 20°C. The two resins were stored at 0-5°C to prevent curing before use. The ACQ-D concentrate (66.7% CuO and 33.3% DDAC, w/w) had a concentration of 15%, which was supplied by a wood preservatives company in Guangdong Province, China. Before each test, the resins and preservative were diluted and homogenized for 3 min using a mixer. UF had a concentration of 41.5%; ACQ-D had a concentration of 1.1% (Ung and Cooper 2005); and PF had five concentrations of 10%, 15%, 20%, 25%, and 40%.

The wood decay fungus used in the test was *Gloeophyllum trabeum (Pers.) Murrill* CFCC 86617 supplied by China Forestry Culture Collection Center (CFCC).

Impregnation Treatment

The specimens were exposed to a 15-kPa vacuum for 30 min in a stainless steel reactor and then impregnated at 450 kPa and 45-50°C for 4 h. The procedure was followed with the removal of excess chemicals under a 15 kPa vacuum for another 30 min. The impregnated specimens were air-dried at room temperature (15-25°C) for 1 wk, and then put in an oven at 60°C for 12 h to obtain the MC of 14-20%. The ACQ-treated specimens were treated for 21 d at room temperature to obtain optimal fixation of preservative (Jiang and Wang 2002). The UF-treated specimens were treated for 2 h at 80-105°C. The PF-modified specimens were treated for 5 h at temperatures between 80°C and 140°C until full curing was reached. Then, the specimens were finally conditioned at 20 ± 2°C and 65 ± 5% RH for 7 d until a constant weight was achieved.

Weight gains of treated specimens with resin were used as an assessment of the treating levels, and are defined by Eq 1 as follows:

$$WG = \frac{m_{1t} - m_0}{m_0} \times 100, \quad (1)$$

where WG is the weight percentage gain of the modified specimen (%), m_0 is the initial oven-dried weight of the specimen before treatment with resin (g), and m_{1t} is the oven-dried weight of the specimen after impregnation (g).

Decay Resistance

A combination of laboratory and field stake methods were used to evaluate the effect of wood impregnation on decay resistance. The tests were conducted by subjecting the specimens to brown rot fungus *Gloeophyllum trabeum (Pers.) Murrill* CFCC 86617 according to GB/T 13942.1-2009 (2009). Petri dishes with a diameter of 90 mm were filled with sterile medium (20 mL) prepared from malt (40 g) and agar (20 g) in distilled water (1 L) and inoculated with a piece of mycelium of a freshly grown brown rot fungus. Petri dishes were incubated at 28 ± 2°C and more than 70% RH for 7-10 d until full colonization of the surface by the mycelium. Wood specimens were sterilized at 121°C and 0.1 MPa in an autoclave for 30 min to achieve a MC ranging from 40% to 60% after measuring the initial dry weights. Then, the specimens were cooled and inoculated with fungal mycelia. Three specimens were placed in each petri dish and exposed to fungi. Sixteen Petri dishes were used for each group. After 3, 6, 9, and 12 wk of incubation at 28 ± 2°C and 80 ± 5% RH, four petri dishes were randomly taken out from each group to measure the ML of specimens. The surface fungus mycelium and impurities on the specimens were cleared out and the specimens were dried at 103°C until weight stabilization and measurement. The ML due to fungal attacks after decay testing were determined as percentages of the initial dry weights of the wood specimen.

For the field stake testing, the field is located in Nanjing, China (32°04″N, 118°48″E). The annual average temperature, RH, and rainfall precipitation of test plot are 15.4°C, 76%, and 1106 mm, respectively. The test site has a warm humid climate in the summer that is considered to be favorable for decay and termite activities. The field test site was flat and had sandy loam soil. 2/3 of the specimens' length was buried in soil (Fig 1). The row spacing *Dr* between test pieces ranged from 15 cm to 20 cm and the column spacing *Dc* was set from 30 cm to 40 cm according to GB/T 13942.2-2009 (2009). Twelve specimens with dimensions of 20 mm × 20 mm × 300 mm for each group were planted. After a 12-mo field exposure, half of the specimens were taken out from each group, and their biological properties were determined. The other half of the specimens were kept on site for another 12 mo and then retrieved. A blunt knife was used to remove the mycelium, soil, and other loose surface debris. The degree of deterioration of specimens was calculated.

Mass difference between the initial and final weights were used to assess the levels of decay resistance according to Chinese standard. The percentage ML was defined by Eq 2 in standard laboratory method as follows:

$$\mathrm{ML} = \frac{m_{2t} - m_i}{m_i} \times 100, \qquad (2)$$

where ML is the percentage ML of the decayed specimen (%), m_i is the oven-dried weight of the specimen after modification with modifiers or preservatives (g), and m_t is the oven-dried weight of the specimen after decay (g).

RESULTS AND DISCUSSION

Weight Percent Gain of Modified Wood Specimens

According to Eq 1, chemical retention in modified wood specimens with 1.1% ACQ and 41.5%

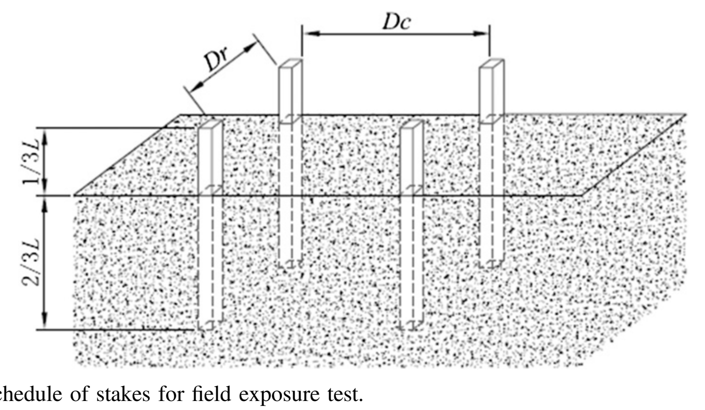


Figure 1. Detailed schedule of stakes for field exposure test.

UF was 6.72 kg/m^3 and 18.43 kg/m^3, respectively. The retention of ACQ specimens was more than 6.4 kg/m^3 and met the demands of the normal ground contact retention regulated in AWPA standards. Weight gains of treated poplar with PF were plotted in Fig 2. The relationship between weight gains and resin concentration was liner, so it can be fitted by a linear function as determined by Eq 3 as follows:

$$y_\eta = a + b \times x_\eta, \tag{3}$$

where y_η is the weight percent gain of resin specimens (%); x_η is the concentration of PF (%); and a and b are the two constants.

The retention of PF specimens achieved a range from 7.31 kg/m^3 to 31.07 kg/m^3 after PF with concentrations ranging from 10% to 40% was employed (see Fig 2).

The initial weight gains of specimens before impregnation can be determined by Eq 4 as follows:

$$y_0 = a + b \times x_n = a + b \times 0 = a = 0. \tag{4}$$

Therefore, Eq 3 can be simplified into Eq 5 as follows:

$$y_0 = b \times x_n. \tag{5}$$

According to the obtained weight gains data of impregnated specimens with different concentrations of PF resins, a fitted linear function model can be drawn as shown as Eq 6 using Origin software.

$$y_\eta = 0.7518 \times x_\eta \tag{6}$$

As shown in Fig 2, the required weight percent gain of resin-poplar wood can be achieved using the adjusting concentration of resin.

Laboratory Method

Figure 3 represents the average ML and standard deviations. Chemical modification can improve the decay resistance significantly according to the conventional laboratory method. Figure 4 shows the infested state of four kinds of poplar specimens tested.

The ML were measured on the untreated and treated specimens. However, the difference of decay resistance between untreated and treated poplar wood was significant. The untreated specimens had the largest mass loss of 26.34%

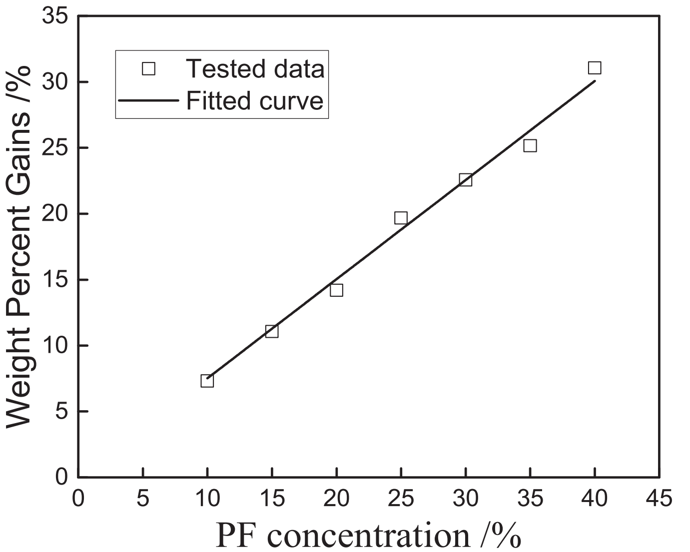


Figure 2. Curves of weight percent gains in phenol–formaldehyde (PF) specimens with resin concentration. The goodness of fit values was 0.9907.

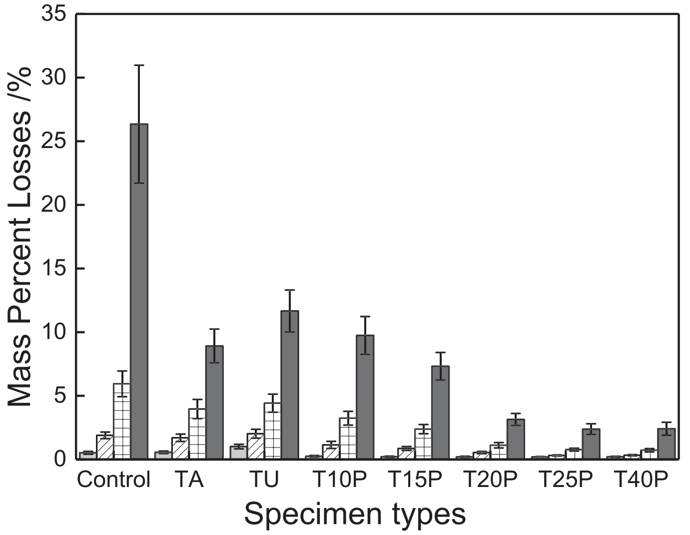
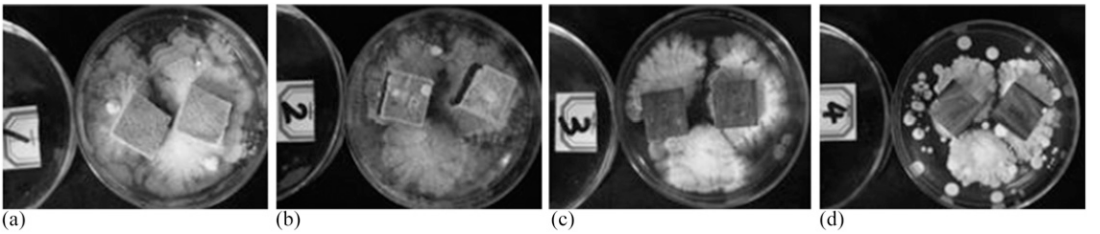


Figure 3. Mass loss caused by brown-rot fungus *Gloeophyllum trabeum (Pers.) Murrill* CFCC 86617 at 3-wk intervals during laboratory tests (Note: The light gray, diagonal, gridded, and black columns expressed mass losses after 3-, 6-, 9-, and 12-wk tests, respectively). CFCC, China Forestry Culture Collection Center.

after a 12-wk incubation, whereas the ML for ACQ-D and UF-modified specimens after a 12-wk incubation were 8.91% and 11.66%, respectively. The ML for PF-modified specimens were 9.74%, 7.32%, 3.14%, 2.38%, and 2.41% at 10%, 15%, 20%, 25%, and 40% concentrations, respectively. The higher the PF concentration the specimens were treated with, the less the mass losses. The mass loss for the untreated specimens reached 0.52% at the end of the first 3 wk, then increased to 1.89%, 5.94%, and 26.34% after 6, 9 and 12-wk incubations. For the UF specimens, the average mass loss was 10%. It was larger than that of the ACQ specimens. The PF specimen displayed the best decay resistance, followed by the ACQ- and UF-modified specimens.

After a 12-wk incubation, the control specimens were covered with mycelium, and seriously infested (see Fig 4). For the ACQ-modified specimen, mycelium grew on some surfaces. Large growth of the mycelium on the wood surfaces of the PF specimens after 12-wk incubation was hardly seen (Fig 4).

ACQ contains the copper ions and DDAC, which are toxic to wood decay fungi. The copper ions can bond with functional groups such as carboxyl, carbonyl, and phenolic hydroxyl (Jin and Archer 1992), and DDAC can be attached to wood cells in the cation exchange process (Xie et al 1995; Ren 2001), where vanillin served as a lignin model compound. The UF or PF can react



(a) (b) (c) (d)

Figure 4. Typical specimen after 12-wk incubation in laboratory test. (a) Control group, (b) ACQ specimen, (c) urea–formaldehyde specimens, and (d) 20% phenol–formaldehyde specimens.

with wood chemical components and creates a strong chemical bond (Liu et al 2000). Because thermosetting resin reacts with wood fiber and generates three-dimensional network polymers, the fungi cannot easily penetrate into the substrate because of resin and wood cross-linking. The results indicated that the resin retains the effect of decay resistance on poplar wood. Also, there was some residual formaldehyde in the cured formaldehyde-based resins, and formaldehyde was an effective insecticide (Li et al 2000). When wood was treated with PF and UF resins that penetrated into the cell wall and set, bulked composites with insoluble polymers were generated which will not be leached out. The susceptibility to biodegradative organisms was reduced significantly because of bulked polymer formation (Rowell and Banks 1985). The earlier findings also indicated that PF resins are also an effective wood preservative in above-ground applications.

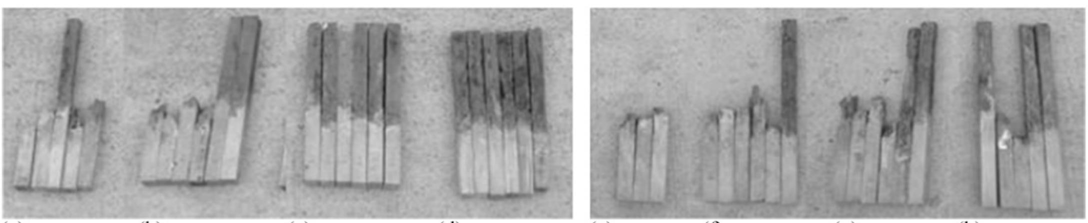
Field Stake Method

Figure 5 presents the damaged states of four groups in the field stake test. All specimens showed varying degrees of damage after exposure.

After the 12- and 24-mo field tests, the loss and damage rate of the control reached 83% and 100%, respectively. The percentage of broken specimens were 83%, 67%, and 50%, respectively, after treatment with ACQ-D, UF, or 20% at the end of the 24-mo field exposure. The laboratory and field stake tests indicated that the decay resistance of poplar wood and the modified wood was as follows: PF specimen remained the best, ACQ and UF specimens were better, and the untreated specimens were the worst. The specimens decayed more in field test than in laboratory test, and the reasons may be that the fruiting bodies of brown rot can only attack wood, the duration was longer, the test was more severe, other types of fungi may be present in soil, and the soil provided nutrients that the fungi need in attacking the treated wood in field exposure.

In both laboratory and field stake tests, the untreated wood exhibited the least decay resistance. The untreated specimens were damaged severely after exposure. The specimens were fully destroyed after the 24-mo field test. Similar findings presented that most of aspen solid wood controls were totally destroyed by termites at the end of the 27-mo test period (Schirp 2008). The decay resistance of ACQ specimens was not satisfied because the efficiency of copper-based waterborne preservatives on durability in protecting hardwoods was worse than that in protecting softwoods (Smith et al 1996; Lebow et al 2010). Higher retentions of waterborne copper naphthenate are necessary for the protection of red maple from white rot because the efficiency of waterborne copper naphthenate was lower than the oil-borne formulation against white-rot fungi (Smith et al 1996).

The experimental assessment of decay resistance of modified poplar wood showed that the mass loss of modified wood specimens with PF represented 19% of the average value of the untreated using laboratory soil-block test (Yue et al 2008). The durability of particleboards with PF adhesive was tested using burial in moist and unsterile soil test, and the experiment exhibited



(a) (b) (c) (d) (e) (f) (g) (h)

Figure 5. Decayed specimen after field stake test. (a-d) Decayed specimens after the 12-mo test and (e-h) decayed specimens after the 24-mo test (Note: (a) and (e) Control group; (b) and (f) ACQ specimen; (c) and (g) urea–formaldehyde specimens; and (d) and (h) 20% phenol–formaldehyde specimens).

that the control was softened severely, whereas the PF-modified specimens with 10-15% resin uptake were virtually unchanged in color and texture (Kajita and Imamura 1991). The reference reported that the average life of untreated southern pinewood specimens was 3.4 yr and that of PF-modified specimens was extended to 5.0 yr as PF retention achieved 48.21 kg/m^3 after stake tested (Woodward et al 2011). Bakar et al (2013) found that the weight losses of untreated oil palm wood were 27.94% and 16.9% after 28-d exposure to termites and a white-rot fungus, respectively, whereas the weight losses decreased to 9.58% and 8.99% after phenolic resin impregnation was used. The previous findings about the decay resistance of PF-modified wood basically correlated with the experimental results in this study (Kajita and Imamura 1991; Yue et al 2008; Woodward et al 2011; Bakar et al 2013).

ML of UF-modified specimen may be resulting from the weight of decomposition of UF. UF-based composites were not suitable for exterior application because of the bad resistance of UF against varying environmental conditions (Pizzi 1993; Uysal 2005). Compared with the untreated, ACQ-modified, and UF-modified specimens, the mass loss for PF-modified specimens was the least in the laboratory test, and its deterioration was the slightest in the field stake test. PF can be used as a wood preservative. These results were in good agreement with the conclusions from other researches (Ryu et al 1991). There was mass loss for PF-modified specimens during incubation in laboratory and field tests. These may have resulted from PF and unprotected part in wood. It was reported that the decay resistance of cured PF was strong, but was biodegradable, and Gusse et al (2006) proved it with the white-rot fungus *Phanerochaete chrysosporium*. The decay resistance of PF specimens was improved significantly and exhibited best decay resistance. The slight change in color and texture can be observed on the surface of the PF specimen.

CONCLUSIONS

The fast-growing poplar wood has been impregnated with PF, UF, and ACQ to improve its durability. The retention rates of ACQ of 1.1% concentration, UF of 41.5% concentration, and PF of 10% concentration can be controlled to 6.72, 18.43, and 7.31 kg/m^3, respectively. For PF, the retention rate is linearly related to concentration. The ML for the untreated, ACQ-modified, UF-modified, and PF-modified poplar wood were 26.34%, 8.91%, 11.66%, and 9.74% after 12 wk of incubation in laboratory, respectively. After 24-mo field tests, the ML were 83%, 67%, 50%, and 100% for ACQ, UF, PF, and untreated specimens. The poplar wood modified with thermosetting resin has very good decay resistance, with the PF impregnating the highest decay resistance.

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