Structure and Properties of Polymer-Impregnated Wood Prepared by *in-situ* Polymerization of Reactive Monomers

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This study demonstrates the preparation, structure, and properties of polymer-impregnated wood (PIW) based on novel *Muchelia macclurei* wood through the *in-situ* polymerization of vinyl and functional monomers. Mixed monomers of glycidyl methacrylate (GMA) and ethylene glycol dimethacrylate (EGDMA) (2:1 molar ratio) were effectively vacuum/pressure impregnated into the cellular structure of the fast-growing wood, and then they underwent a catalytic-thermal polymerization process to form a polymer-impregnated wood composite. Scanning electron microscope (SEM) observations showed that the polymer formed from the monomers occupying the cell lumens and formed tight bonds with the wood substrate. Fourier transform infrared (FTIR) microscopy analysis indicated that the *in-situ* generated polymer probably chemical bonded to the wood cell wall. The resultant PIW exhibited remarkably improved mechanical properties and durability relative to untreated wood. This was attributed to the possible grafting of the polymer onto the wood cell wall and the resultant reinforcement of the wood by the polymer.

Keywords: Polymer-impregnated wood composite; In-situ polymerization; Properties; Glycidyl methacrylate; Ethylene glycol dimethacrylate

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INTRODUCTION

Wood has long been an important material because of its excellent structural properties, abundant reserves, and sustainable nature. Now, it is widely used in areas such as construction, transportation, home furnishing, and sports equipment. Wood exhibits a complex cellular structure that is composed of the biopolymers cellulose, hemicelluloses, and lignin.

The porous structure of wood imparts favorable mechanical properties to the wood for its potential use in structural applications. However, wood is quite sensitive to microorganisms and is easily damaged by pests. It is also susceptible to changes in dimension by moisture because of the unique porous structure and components containing abundant hydroxyl groups, which limit it, especially as a high-quality material, to be used in wider applications (Li *et al*. 2010).

In addition, wood consumption has been rapidly increasing every year. However, high-quality wood production has been sharply decreasing (Ding *et al*. 2008). Such a significant contradiction has impelled scientists to seek substitutional inferior materials for additional value-added commodities. In order to realize these objectives, appropriate technique is required in order to enhance the inferior wood qualities for practical uses (Li 2011a).

Because of wood's abundant hydroxyl groups and porous structure, wrapping the reaction points or filling in the voids may provide enhancement to the water resistance properties and improve the physical structure and durability (Li *et al*. 2009; Li 2011a; Li *et al*. 2011b,c,d, 2012, 2013).

Several popular treatments to change the structure and improve the physical properties of wood include chemical modification, chemical impregnation, compression during heating, and heating at a high temperature (Li 2011a; Xie *et al*. 2012). Among these, the formation of polymer-impregnated wood composites (PIW) *via in-situ* polymerization of functional monomers with carbon-carbon double bonds is one of the most popular methods to improve the physical properties of wood (Li *et al*. 2010; Xie *et al*. 2012).

The resultant macromolecules are proposed to improve the wood's physical properties and dimensional stability, as well as provide decay resistance (Hakan *et al*. 2008; Locs *et al*. 2009; Wang *et al*. 2011). This versatile process may assist in the avoidance of preservatives that may be leached from the modified wood and into the surroundings, strengthen the wood, avoid dimensional deformation of compressed wood, and resist a color change when heat-treated (Li *et al*. 2011d).

Vinyl monomers in wood are typically formed into polymers through a mechanism of free-radical polymerization. This process is superior to condensation polymerization reactions because the free radical initiator requires neither acidic nor basic conditions, which would cause the degradation of cellulose chains, leading to wood brittleness. Also, the reaction does not produce any byproducts. The vinyl-type polymers possess properties ranging from soft rubber to hard, brittle solids depending on their molecular backbones, such as styrene (St), and various acrylates, especially methyl methacrylate (MMA), glycidyl methacrylate (GMA), and acrylonitrile (Ding *et al*. 2008; Devi *et al.* 2008; Li *et al*. 2009; He *et al*. 2011a,b; Koubaa *et al*. 2012; Trey *et al*. 2012; Ding *et al*. 2013; Li *et al*. 2013b).

This study employed a new group of reactive monomers containing glycidyl methacrylate (GMA) and ethyl glycol dimethyl methacrylate (EGDMA), combined with maleic anhydride (MAN) as the reactive catalyst, to treat *Muchelia macclurei* wood. In theory, this group of monomers should be able to polymerize under a catalyst-thermal conditions, and concurrently graft onto wood substrates via the reaction between hydroxyl groups on the components and the epoxy group and/or cyclic anhydride group on the monomers. The reaction mechanism is presented as shown in Fig. 1.

The goal of this work was to develop a novel treatment using a new group of functional monomers for the improvement of wood's comprehensive properties. The microstructure of the prepared WPC was characterized, and the reaction between the wood and the resultant polymers was analyzed.

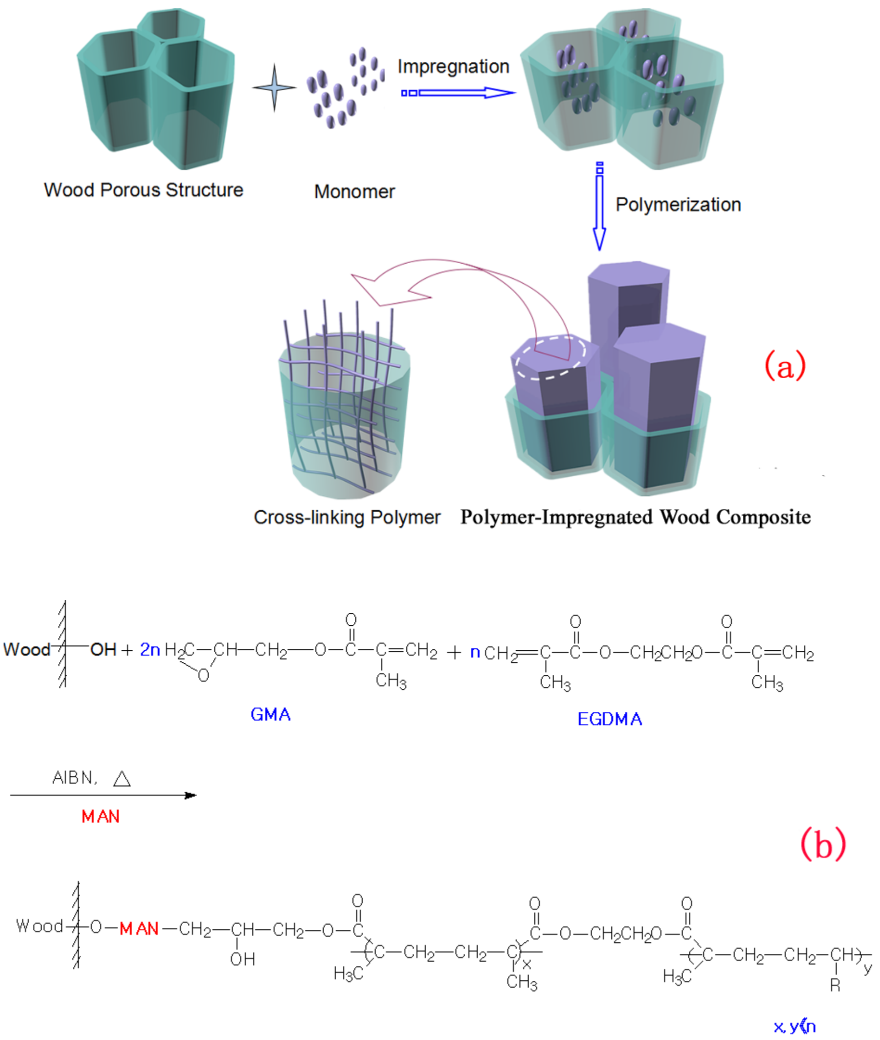

Fig. 1. Schematic of (a) the preparation of the polymer-impregnated wood composites (PIW) and (b) the reaction mechanism of the wood and the functional monomers

EXPERIMENTAL

Materials

Both the MAN and the initiator, 2,2'- azobisisobutyronitrile (AIBN) (Shanghai Chemical Reagent Factory, Shanghai) were recrystallized before use. Analytical-grade GMA purchased from Nanjing Jiulong Chemical Industry Co., Ltd. (Nanjing, China) and EGDMA obtained from the Yantai Yunkai Chemical Industry (China) were directly used.

The wood samples of *Muchelia macclurei* were obtained from the original plantation in Guangxi Yulin, located in southern China. Test samples were cut from the wood samples and then oven-dried at 105 °C to constant weight. The samples were stored at room temperature for testing.

Methods

Preparation of WPC

The MAN was dissolved in acetone to form a mixed solution of 10 wt% concentration. Then, AIBN, as an initiator, was dissolved in GMA and EGDMA (2:1 molar ratio) to obtain a 1.0 wt% concentration. Thereafter, the MAN solution was combined with the mixed GMA and EGDMA monomers solution containing AIBN to form a mixture, in which MAN accounted for 6.0 wt% of the mixed monomers of GMA and EGDMA. Then, the wood samples were vacuum/pressure impregnated with the solution (0.08 MPa for 20 min, followed by 0.8 MPa for 20 min) by a self-made instrument. First, the samples into the instrument, and then the device was sealed. The pressure in the equipment was then set to 0.08 MPa and maintained for 20 min. After that, the pressure was released to atmospheric pressure and then quickly set to 0.8 MPa and maintained for 20 min. Then, the treated wood samples were wrapped in aluminum foil and oven-dried at 80 °C for 8 h, followed by 110 °C for 8 h. Finally, the samples were labeled as P(GMA-co-EGDMA)-impregnated wood composite. The conversion rate is represented by Eq. 1,

$$\text{Conversion rate (\%)} = 100 \times (w_p - w_d) / (w_i - w_d) \qquad (1)$$

where w_p and w_d are the weights of the dry wood after and before the final treatment, respectively, and w_i is the wet weight of the wood after impregnation.

The resultant WPC obtained a 72.4 ± 3.7 percent increase in weight. For comparison purposes, PEGDMA-impregnated wood and PGMA-impregnated wood were prepared with a 73.1 ± 2.2% and 68.4 ± 4.3 % increase in weight, respectively.

Characterization and analysis

The structural morphology of wood was evaluated using an environmental scanning electron microscopy (ESEM) instrument (QUANTA 200, FEI Inc., USA). The surface was cut using a surgical blade, and then mounted on sample holders with double-sided adhesive tape, followed by sputter-coating with gold. The Fourier transform infrared (FTIR) test, using the KBr technique, was conducted with a Magna-IR560 E.S.P (Thermo Nicolet Inc., USA). The resolution ratio was 4 cm to 1 cm, and 40 spectra were accumulated. Before testing, the samples were discretely ground into powder, with a particle size below 100 mesh screen using a disintegrator (FW-200, Beijing Kewei Yongxing Corp., China). The sample was extracted using Soxhlet equipment for 24 h, and then subsequent drying to constant weight.

Mechanical properties, decay resistance, and dimensional stability

The physical and mechanical properties of the samples were tested *via* the "General Requirements for Physical and Mechanical Tests of Wood" (GB/T 1928-2009). Samples with the dimensions of 20 × 20 × 300 mm^3 (radial × tangential × longitudinal), 20 × 20 × 30 mm^3, and 20 × 20 × 50 mm^3 for the compression strength, hardness test, and modulus of rupture test were obtained from a pair of control and treated samples, respectively. Each experiment required five samples. All of the mechanical property tests were conducted using a Universal Testing Machine (AG-10TA, Shimadzu, Kyoto, Japan).

The decay test was conducted according to the Chinese Forest Industry Standard, “Laboratory Methods for the Toxicity Test of Wood Preservatives on Decay Fungi,” (1998), which corresponds to the International Fungal Decay Test (Li *et al*. 2011b).

Samples with the dimensions of 20 × 20 × 10 mm^3 were obtained from control and modified wood. Each experiment required five samples. Test samples were autoclaved for 30 min and set on wood chips with sizes of 22 × 22 × 2 mm^3 in the incubator. Each incubator involved three wood samples, which were separately set on three wood chips. The relative humidity and the temperature were 80% and 28 °C, respectively. Each sample weight loss for the decay resistance was calculated after exposing the sample to decay fungi for 12 weeks. The brown decay fungus, *Gloeophyllum trabeum* (Pers. ex Fr.) Murr was used in this experiment.

The dimensional stability of the samples was obtained according to the GB/T1928-2009 testing standard (2009). Samples with the dimensions of 20 × 20 × 20 mm^3 were cut from the control and WPC, and were further submerged in distilled water for 10 days. The average data of the five specimens for each measurement were recorded.

The polymer loading rate (R_m) (%) was calculated using Eq. 2,

$$R_m = (G_i - G_0) / G_0 \times 100\% \tag{2}$$

where G_i (g) represents the mass of the WPC and G_0 (g) represents the mass of wood before the treatment.

The volume swelling efficiency (V_m) (%) was calculated using Eq. 3,

$$V_m = (V_1 - V_0) / V_0 \times 100\% \tag{3}$$

where V_1 (cm^3) represents the volume of the samples after immersion in water and V_0 (cm^3) represents the volume of the samples before immersion.

RESULTS AND DISCUSSION

SEM Observations

Figure 2a presents the wood cellular structure of different pore sizes, which are micrometers in diameter. Figure 2b shows the polymer being polymerized and effectively blocking most pores, indicating that the conditions employed for the composite were reasonable. However, it shows visible gaps between wood cell wall (the pore substrate) and the resulted polymer, PEGDMA, indicating their poor interfacial interaction. Figure 2c shows that the formed polymer bonds with the wood substrates without any obvious gaps; thus providing evidence of a strong interaction between the respective phases, which is either physical, chemical, or a combination of both. Figure 2d presents the impact section of the P(GMA-co-EGDMA)-impregnated wood composites, which clearly indicates the resulted polymer insertion into wood pores and reveals necking fracture morphologies that are characteristic of ductile materials. Also, the polymer tightly contacts the wood substrates, similar to that of Figure 2c.

Considering the structure and the reaction mechanism as shown in Fig. 1, it could be concluded that the chemical reaction contributed to the intense interaction between the wood cell wall structure and the resultant polymer, and the polymer contributes toughness to the resulted polymer-impregnated wood composite. This was further evidenced by the following FTIR characterization and the mechanical properties.

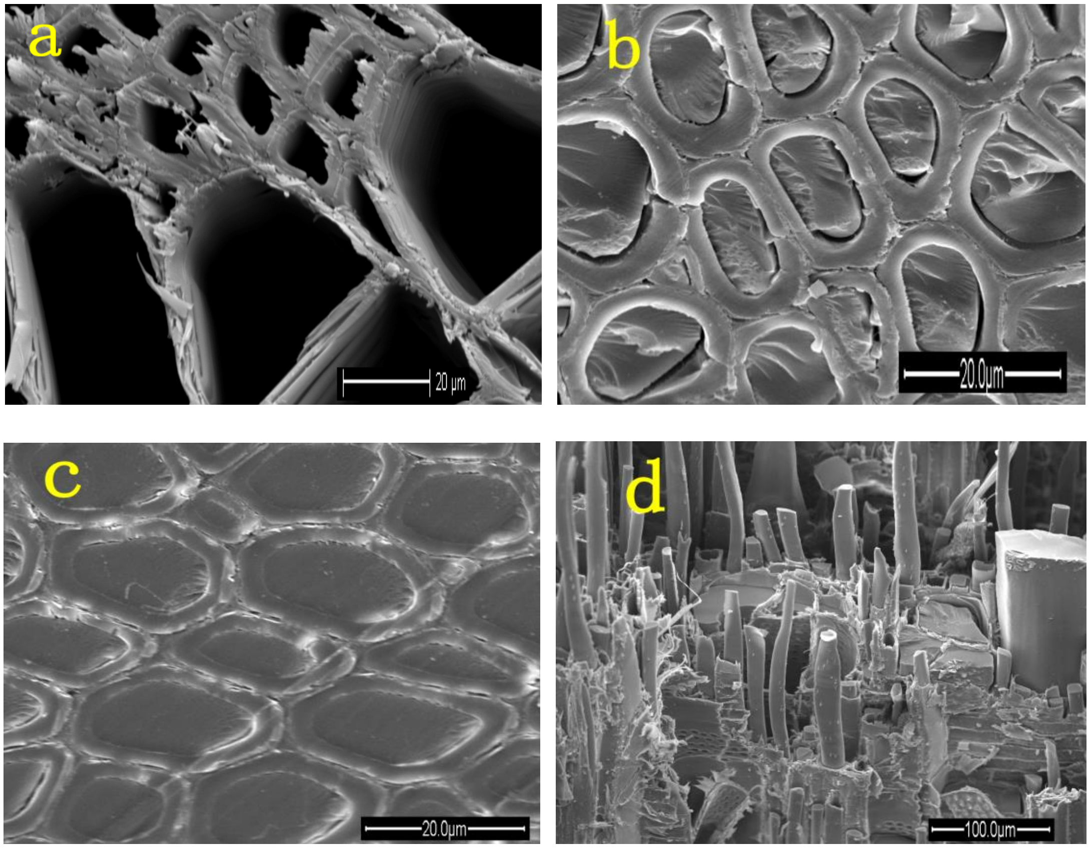

Fig. 2. The SEM morphologies of wood (a), PEGDMA-impregnated wood composites (b), and P(GMA-co-EGDMA)-impregnated wood composites with cut section (c) and with impact section (d)

FTIR Analysis

Figure 3 shows that there was an increased intensity in an absorbance peak at 1735 cm^{-1} corresponding to the carbonyl stretching vibration of the MAN-impregnated wood composites, PGMA-impregnated wood composites, and the P(GMA-co-EGDMA)-impregnated wood composites, respectively. This feature demonstrated the presence of MAN, GMA, and EGDMA (Li *et al*. 2011b,c,d). The hydroxyl groups on the wood and the three polymer-impregnated wood composites peaked at 3394 cm^{-1}, representing the stretching vibration. The peak of the later three samples, especially MAN-impregnated wood composite, was weakened in comparison to the untreated wood, indicating reactions occurring between the chemicals and wood substrates. The decrease of hydroxyl groups and the increase of carbonyl groups indicated the grafting of the monomer/polymer onto the wood matrix through the reaction of the cyclic anhydride of MAN/epoxy groups of GMA with the hydroxyl groups on wood, as shown in Fig. 1b.

The C-O-C asymmetrical stretching vibrations peaking at 1164 cm^{-1} showed a visible enhancement as evidence of the reaction between GMA/EGDMA copolymers with the hydroxyl groups on wood, resulting in polyethers (Li *et al*. 2011b,c,d; Li *et al*. 2012). Consequently, it can be concluded that the resultant polymers exhibited a chemical interaction with the wood cell walls, and thus contributed toughness and strength to wood matrix, which was further evidenced by the following mechanical properties.

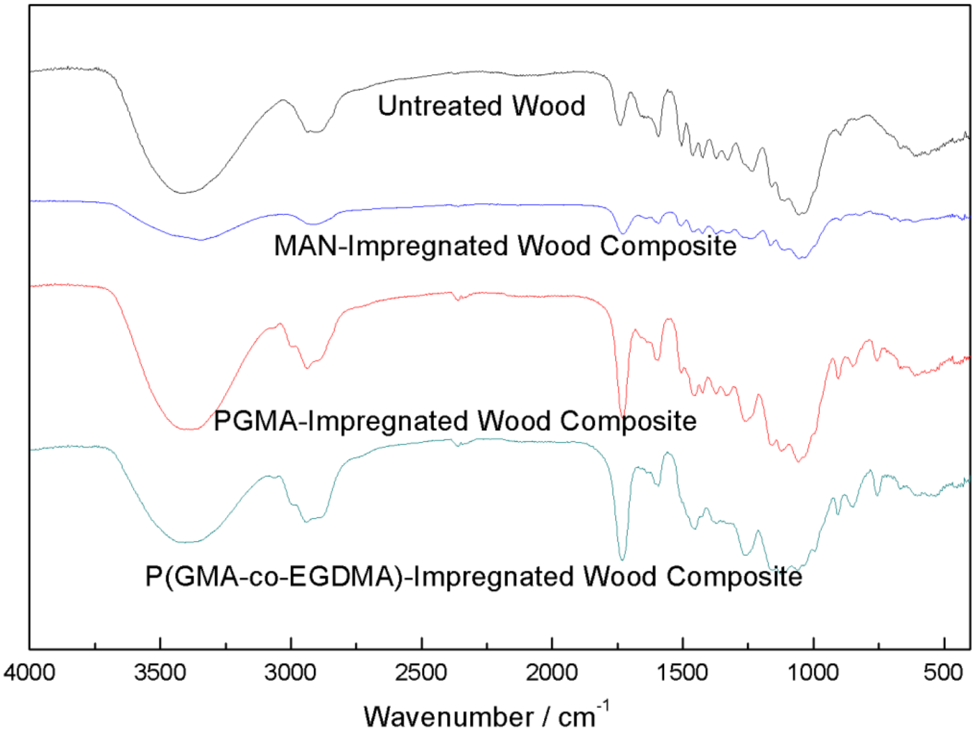

Fig. 3. The FTIR spectra of wood, MAN-impregnated wood composites, PGMA-impregnated wood composites, and P(GMA-co-EGDMA)-impregnated wood composites

Integrated Properties

Table 1 shows the integrated properties of the untreated wood, PGMA-impregnated wood composites, PEGDMA-impregnated wood composites, and P (GMA-co-EGDMA)-impregnated wood composites. In comparison with the untreated wood, the modulus of rupture, compressive strength, impact toughness, and hardness were improved by 54%, 60%, 88%, and 151%, respectively, for P(GMA-co-EGDMA)-impregnated wood composites; the PEGDMA-impregnated wood composites was improved by 42%, 39%, 135%, and 119%, respectively; and the PGMA-impregnated wood composite was improved by 34%, 59%, -13%, and 151%, respectively. The improvement of toughness corresponded to the SEM observation results in Fig. 2d. Therefore, it can be concluded that the addition of MAN and GMA/EGDMA monomers remarkably improved the mechanical properties of wood, which is consistent with the FTIR analysis. In addition, most of the mechanical properties of the P(GMA-co-EGDMA)-impregnated wood composites exceeded those of untreated wood, as well as other treated wood composites.

Table 1 shows the resistance of the P(GMA-co-EGDMA)-impregnated wood composites, PGMA-impregnated wood composites, and PEGDMA-impregnated wood composites to microorganisms in terms of weight loss against the brown decay fungus was improved by 95.10%, 90.62%, and 86.10% over that of untreated wood, respectively. This indicated that the wood after treatment by *in-situ* formation of polymer was remarkably improved regarding decay resistance. The resulted polymer resisted the addition of moisture from accessing the wood cell wall, and the hydroxyl groups were partially blocked because of the grafting reaction. Thus, it was more difficult for microorganisms to gain access to the wood's internal structure (Li *et al*. 2013a). The reaction of the polymers with the cell wall partly changed the structure of the wood components, which were not fully recognized by the microorganisms, and also contributed to the enhanced resistance against the fungi (Li *et al*. 2013a). Consequently, the chemical modification of wood by the polymers improved the decay resistance of the wood against the brown fungus. However, as the chemical modification degree differs among the different types of monomers; therefore, the value of decay resistance of the P(GMA-co-EGDMA)-impregnated wood composites against the brown decay fungus was highest among the polymers in chemically modified wood materials. This suggested that the reaction was complete between the wood and the resultant polymers from GMA/EGDMA monomers.

Table 1. Integrated Properties of the Treated and Untreated Wood Materials

Properties / Samples	Modulus of rupture (Tangential section)		Compressive strength (Crossing section)		Impact toughness (Tangential section)	
	Value (MPa)	Times versus untreated wood	Value (MPa)	Times versus untreated wood	Value (KJ/m^2)	Times versus untreated wood
Untreated Wood	103.69 (2.01)	—	79.48 (1.03)	—	41.66 (1.34)	—
PGMA-impregnated wood composites	138.67 (3.97)	1.34	126.83 (2.14)	1.59	36.41 (1.60)	0.87
PEGDMA-impregnated wood composites	147.72 (3.55)	1.42	110.77 (1.95)	1.39	97.87 (2.28)	2.35
P(GMA-co-EGDMA)-impregnated wood composites	159.51 (2.47)	1.54	127.06 (2.36)	1.60	78.13 (2.02)	1.88

Properties / Samples	Hardness (Tangential section)		Decay Resistance		Dimensional Stability	
	Value (N)	Times versus untreated wood	Weight-loss Ratio (%)	Improved times versus untreated wood (%)	Volume swelling efficiency (%)	Improved times versus untreated wood (%)
Untreated Wood	4817.08 (12.97)	—	70.05 (1.76)	—	11.14 (0.26)	—
PGMA-impregnated wood composites	13150 (20.46)	2.73	6.57 (0.42)	90.62	4.93 (0.40)	55.75
PEGDMA-impregnated wood composites	11060 (16.77)	2.29	9.74 (0.71)	86.10	6.85 (0.38)	38.51
P(GMA-co-EGDMA)-impregnated wood composites	12090 (17.35)	2.51	3.43 (0.25)	95.10	3.30 (0.19)	70.38
Note: The data in parentheses are standard deviations						

Table 1 shows that the volume swelling efficiency of untreated wood achieved 11.14% after being immersed in water for 240 h, while that of P(GMA-co-EGDMA)-impregnated wood composites, PGMA-impregnated wood composites, and PEGDMA-impregnated wood composites reached 3.30%, 4.93%, and 6.85%, respectively. In other words, P(GMA-co-EGDMA)-impregnated wood composites obtained the highest dimensional stability, with an improvement of 70.4% over untreated wood. Based on the SEM observations and FTIR analysis, a greater dimensional stability was attributed to the reaction bonding between the polymer and wood substrates and the physical blocking of wood's porous structure by the polymers, which effectively wrapped hydroxyl groups in the wood cell walls, and thus stopped moisture from passing into the wood substrates.

CONCLUSIONS

1. The SEM observations and FTIR analysis indicated that the polymers in the P(GMA-co-EGDMA)-impregnated wood composites resulted from the polymerization of reactive monomers, which occupied wood pores and may have reacted with the main components of wood substrates.
2. In comparison with untreated wood, the mechanical properties of the *in-situ* polymer-impregnated wood composites including hardness, impact toughness, modulus of rupture, and compressive strength were notably improved.
3. The durability, including dimensional stability and decay resistance, were also improved in comparison to untreated wood. Consequently, the integrated performance of the fast-growing and middle-grade *Muchelia macclurei* wood was remarkably improved by the newly introduced combination of monomers. This treatment could be employed as a promising way to add valuable applications to wood.

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