WOOD-RUBBER COMPOSITES FABRICATED FROM RUBBER MIXING AND VULCANIZATION MOLDING PROCESS

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Abstract. Wood-rubber composites (WRC) has been successfully manufactured through the rubber mixing and vulcanization molding process. The morphological properties of the composite panels were characterized using scanning electron microscope (SEM). It showed that the wood powders were well embedded in the tire rubber matrix. The functional groups of the composites were observed by Fourier transform infrared spectroscopy. The curing characteristics were analyzed on the WRC fabricated with different wood powder loadings. The mechanical properties of the WRCs including tensile strength, elongation at break, hardness, and rebound resilience, were examined. It was determined that the wood powder loading should not be more than 50 wt.%. Incorporation of wood powder increased the hardness of the WRC with about 53% improvement when the wood powder loading increased from 0% to 50% (from 60° to 92°). The water absorption of the WRC was lower than that of traditional wood-based composites.

Keywords: Wood powder, tire rubber, wood-rubber composites (WRC), rubber mixing and vulcanization molding process.

INTRODUCTION

With a limited resource of large diameter trees, plantation species, such as Poplar (*Popupus genus* group), became popular in China. Poplar is a fast-growing and high-yielding species, but low density, low strength, and easy to warp, which have limited its utilization lower end applications, such as packaging, matches, and disposable chopsticks.

Mixing two or more types of raw materials can be beneficial to producing products with superior properties (Gardiner 1968; Woods and Davidson

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1976; Shershnev 1982) manufacturing wood-rubber composites (WRC). As a hygroscopic material, wood absorbs or losses moisture causing dimensional instability, and is attacked by fungi and insects (Nami and Nadir 2005). Rubber is almost hydrophobic, only minimally affected by atmospheric humidity. Tire rubber would be an ideal raw material for the functional composites because it possesses some unique properties, such as excellent energy absorption, better sound insulation, durability and abrasion resistance, and causticity and rot resistances (Blackley 1997). The combination of wood and rubber may improve performance of the composite products, as it couples the advantages of the both wood and rubber, which enables them for some special applications. In the processing of WRC, waste wood materials, such as branches, wood chips, can be used. Compared with the wood-based composites, WRC have a potential to improve the performance in decay resistant, water resistant, acoustic properties, antistatic, damping, and thermal insulation. The WRC could be used as indoor insulation and sound-absorbing materials, decoration materials, and different industrial rubber sheets used in cars, manufacturing facilities, such as machine seat cushion and printing rubber sheet.

A number of studies have been conducted on WRC fabricated from waste tire rubber and wood. These methods used adhesive as a bonding agent, and a compression molding process was employed (Wang et al 2000; Maria and Joekes 2002; Frantzis 2003; Martins and Mattoso 2004; Stefani et al 2005; Zhou et al 2007). Song and Hwang (1997, 2001), Zhao et al (2008), and Evren et al (2009) used urea formaldehyde (UF) and polymeric methylene-4,4'-diphenyl diisocyanate resins as bonding agent to fabricate WRC. In the process, waste tire rubber, wood, and adhesive were incorporated separately, which would have issues of uneven mixing causing poor mechanical properties of the composite. Because a bonding agent was used, a high production cost would incur, with a concern of volatility organic compound emissions, such as free formaldehyde. There is also a limitation in the product applications due to the use of adhesives. For example, the UF resin bonded composites can only be used for indoor application. Comparing to the traditional compression molding process, the wood rubber composites fabricated from the rubber mixing and vulcanization molding process uses tire rubber as binder which has no concern on formaldehyde emission, and is environmental friendly.

Tire rubber is noncross-linked and has good fluidity and viscidity. Usually, tire rubber consists of natural rubber (NR), butadiene rubber (BR) and butadiene styrene rubber (SBR), and other ingredients. In the processing of tire rubber, unvulcanized raw rubber (such as NR or BR) and other ingredients (such as carbon black and sulfur) are mixed at a certain ratio using a mixer. The raw rubber is in its original uncured form, which has low strength. The raw rubber softens at an elevated temperature and brittle at a low temperature, which has limited its usage (Hoffman 1967). Other ingredients may be mixed to improve the performance of the raw rubber, such as hardness and elasticity properties (Yang et al 2004). The tire rubber can be used in harsh ambient conditions, due to its ability to withstand high and low temperatures, and their high causticity and rot resistant properties (Nadie et al 2009a).

The vulcanization molding process for rubber mixing has mainly three steps: mixing, shaping, and vulcanizing. Vulcanization, is a key step for the process, is a chemical process where long chains of rubber molecules are cross-linked, transforming the plastic material into a strong elastic product with high and reversible deformability and good mechanical properties owing to the strain-induced crystallization, low hysteresis, excellent dynamic properties, and fatigue resistance (Aprem et al 2005). After the vulcanization, the rubber loses its tackiness to be insoluble in solvent with more resistant to heat, light, and aging (Zhao et al 2011). Rubber, carbon black, and sulfur are the major ingredients for the tire improved initial elasticity modulus and durability (Zhang et al 2006).

The objective of this study was to investigate the feasibility to manufacture composites using

poplar wood powder and tire rubber through the vulcanization molding process, and to determine the maximum wood powder loading for the composites. The morphological properties, curing characteristics, and mechanical properties of the WRC were evaluated.

<div style="text-align:center">MATERIALS AND METHODS</div>

Materials

Poplar wood powders were provided by Wooden Forest Woods products co., LTD with a particle size of 80-100 meshes and a MC of 1% (Fig 1). The tire rubber was provided by Xingda Rubber Factory in Harbin, China, containing ingredients (parts per hundreds of rubber, phr): NR, 30; BR, 24; SBR, 6; carbon black (N330), 30; stearic acid, 2; zinc oxide, 2.5; sulfur, 1; spindle oil, 3; and antioxidant 1.5.

Sample Preparations

Based on our initial experiments, when the wood powder loading was higher than 50%, mixing torque during the rubber mixing process decreased with no recovery, indicating that the compound could not be vulcanized. Therefore, it could be concluded that the maximum wood powder loading was determined as 50 wt.%. Wood-rubber composite panels of $260 \times 260 \times 2$ mm were manufactured with wood powder contents of 0%, 10%, 20%, 30%, 40%, and 50% by weight (referred as W0, W1, W2, W3, W4, and W5, respectively). Four replicates were used for each formulation.

The tire rubber was premixed at 60°C for 2 min in a twin rotor mixer (XH-409, Zhuosheng mechanical equipment Co., Ltd, Dongguan, China). The main rotor speed was 25 r/m and the ratio was 1.3. The wood powders were gradually added into the mixer and continued to blend for 5 min. Mixing was carried out using a laboratory two roll mill (XH-401A, Zhuosheng mechanical equipment Co., Ltd) for 3 min at 60°C. The two roll mill consisted of two parallel rolls with different rotation speed and produces a homogeneous sheet mixture. The speed ratio between the two

rolls was 1.2 and the gap was 2 mm. Mixing time and the sequence of the ingredients incorporation were the same for all samples. The sheets from the double roller open mill were conditioned at a temperature of 23 ± 2°C for 24 h in a closed container before vulcanization. Vulcanization was carried out in a plate vulcanizing machine (XH-406B, Zhuosheng Mechanical Equipment Co., Ltd) at 160°C under a pressure of 15 MPa for the optimum curing time (t_c90). The optimum cure time (t_c90) was determined when the material to construct the highest cross-linking density through the No Rotor Rheometer. The molded sheets were conditioned in desiccators for 24 h prior to further testing.

Characterization

The surface morphology of the samples was characterized by a scanning electron microscope (SEM) system, Quanta 200 microscope (FEI Company, Hillsborough, OR) at 10 KV. The specimens were frozen under liquid nitrogen, fractured, coated with a gold layer of about 15 nm to avoid charging. Cryogenically fractured surface of the samples were examined. Fourier transform infrared spectroscopy (FTIR) technique was used to exam the functional groups of the WRC. The attenuated total reflection spectra was obtained using the Nicolet 6700 spectrometer (Thermo Fisher Scientific, Waltham, MA) in a range of 550 to 4000 cm^{-1} at a resolution of 4 cm^{-1}. The curing characteristics, such as minimum torque (ML), maximum torque (MH), scorch time ($ts2$), and cure time (t_c90), were examined by a Jingzhuo No Rotor Rheometer according to ISO 3417, at −160°C. The tensile and elongation properties of the composites were measured according to ISO 37 standard using dumb-bell test pieces by the Instron 4505 Universal Testing Machine with a crosshead speed of 500 mm/min. The hardness of the composites was measured by the shore type A Durometer according to the procedure described in ISO 7619. The rebound resilience was tested based on the procedure described in ISO 4662 using the Elastic impact tester (Yangzhou Jingzhuo Instruments Factory, China). The water absorption of the composites (sample sizes: $50 \times 50 \times 2$ mm)

was measured using 24-h water soaking method at room temperature. The water absorption was calculated according to the following formula:

$$TS = \frac{h_1 - h_0}{h_0} \times 100\%$$

where TS is the thickness swelling, h_0 is the thickness of the sample before testing, and h_1 is the thickness of the sample after water absorption.

RESULTS AND DISCUSSION

Phase Morphology

Figure 1 shows a comparison of the images at different stages of the WRCs processed with 50 wt.% wood powder content. The image of pure tire rubber (W_0) is shown in Fig 1a. Figure 1b shows the mixture wood-rubber after mixing, from which a homogeneous mixture of wood powders and rubber matrix is observed. The wood powder was well encapsulated in the rubber matrix due to adhesion between wood powder and rubber matrix. Figure 1c shows a sheet of the WRC before the vulcanization. The white dots shown in the picture are wood powders. It is shown that the wood powder is uniformly distributed in the rubber matrix, indicating that the mixing process in the experiment was efficient. After the vulcanization, the wood powder was not -visible in the composite panel (Fig 1d), and the board showed black color. For all WRC samples fabricated from the rubber mixing and vulcanization process, the wood powder was covered by the cured rubber matrix. The woody materials could not be seen from the surface, and the appearance of the composite material is regular black color. However, for the WRC samples fabricated from traditional compression molding process (Garcia et al 2007), the wood powder
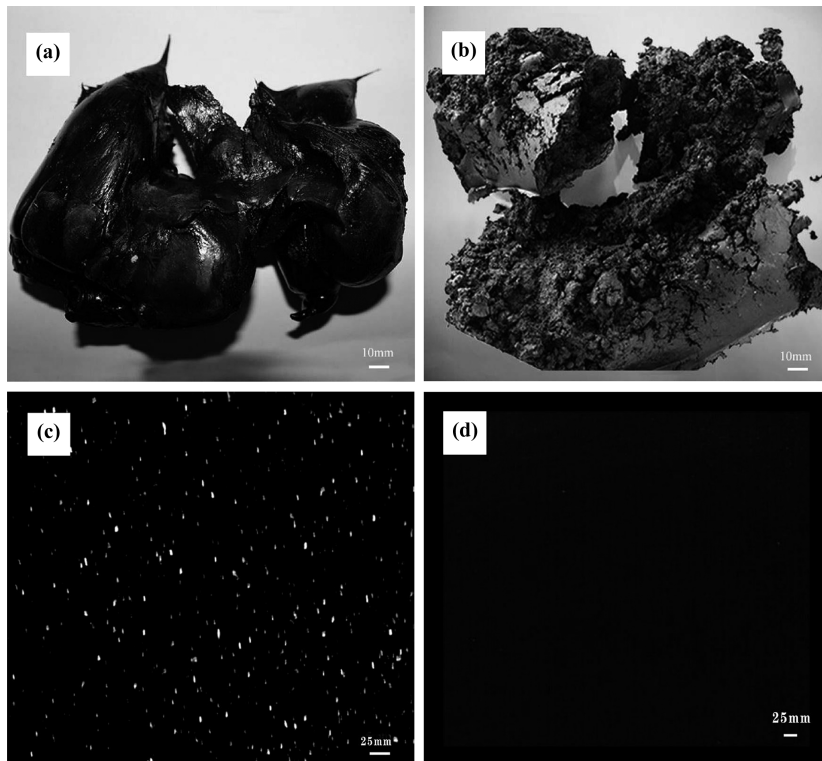

Figure 1. The material in the process (a) pure rubber matrix after mixing; (b) 50 wt.% wood powders after mixing; (c) 50 wt.% wood powders in a sheet before vulcanization; (d) the composites panel with 50 wt.% wood powders after vulcanization.
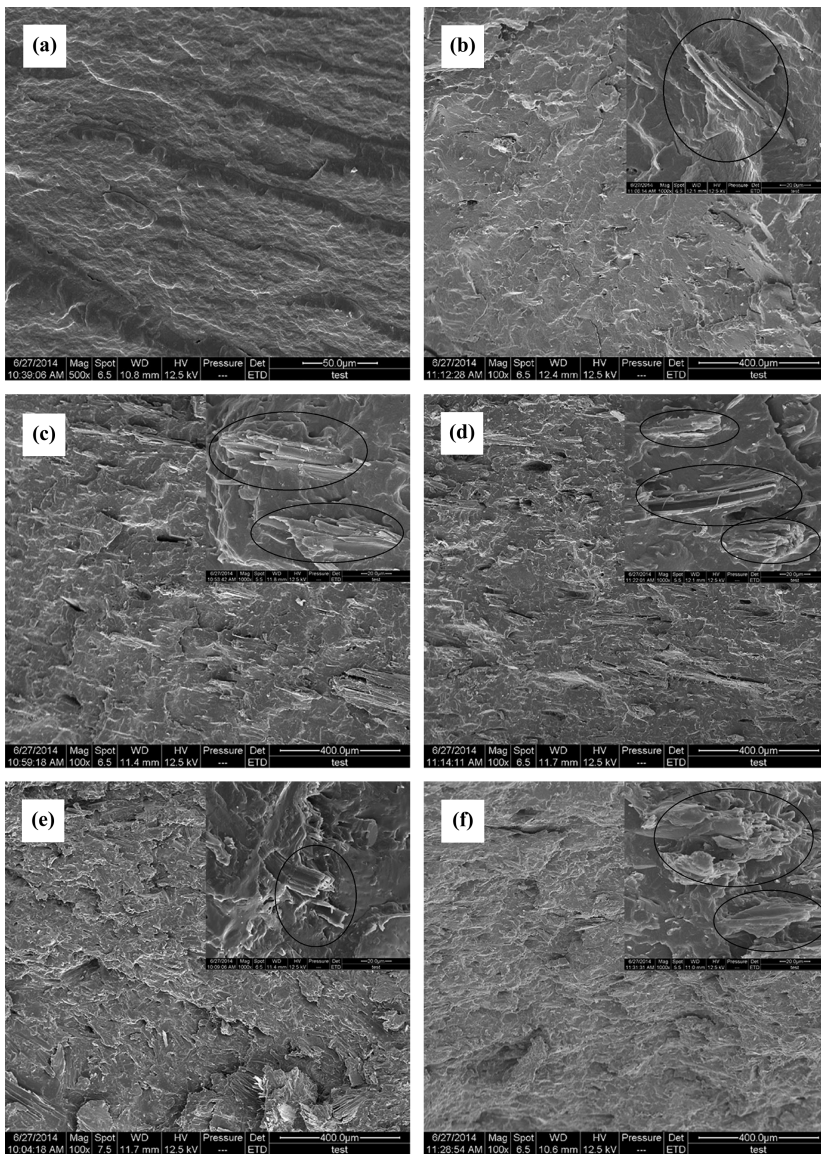
Figure 2. The scanning electron microscope photograph of (a) pure rubber matrix; (b) 10 wt.% wood powder loading; (c) 20 wt.% wood powder loading; (d) 30 wt.% wood powder loading; (e) 40 wt.% wood powder loading; (f) 50 wt.% wood powder loading.

was covered with small black spots from the images, which could not be used directly and needed surface preparation.

Figure 2 shows the SEM images of the cryogenically fractured surface of the WRCs with different wood powder contents from 0 wt.% to 50 wt.%. Compared with the micrograph of the control sample in Fig 2a, the wood powder in the rubber matrix can easily be identified in Fig 2b-d. It was found that the amount of wood powder on the fracture surface of the composites increased as the wood powder loading increased. The wood powder was well surrounded by the rubber matrix with a smooth fracture surface indicating a strong bonding at the wood powder and rubber interface. When wood powder content was greater than 40 wt.%, the panel surface became

rougher (Fig 2e-f). However, no obvious separation was observed between the wood powder and the rubber on the fracture surface. Under a high magnification for the image, it was shown that on the fracture surface, wood powder was embedded in the rubber matrix. The above phenomenon suggested that the composites had gone through simultaneous curing and formed a continuous segment at different wood powder contents. From prior work (Zhao et al 2010), the SEM images of the WRC fabricated from traditional compression molding process showed that there were some spaces between wood and rubber. From the SEM images shown in Fig 2 on the samples fabricated in this study using the rubber mixing and vulcanization process, tight bonding between wood and rubber in the composites was clearly seen, indicating that the bonding between the rubber and wood was much closer.

FTIR analysis of WRC

Figure 3 shows the FTIR spectra of WRCs with different wood powder contents. The characteristic vibration bands for the tire rubber (W0) have been noted where the existence of -CH_2- at 2920 cm^{-1}, -CH_3 at 2850 cm^{-1}, C=C at 1594 cm^{-1}, C-CH_3 at 1408 cm^{-1}, and C-H at 870 cm^{-1}. These bands of each wood-rubber composite can be observed. After adding the wood powder, the bands reduced, suggesting that the characteristic peaks in wood are weak. It was also seen from Fig 3 that the intensity of these bands of rubber was decreased as the wood powder loading increased. The strong bond at 3332 cm^{-1} was characterized as the O-H, at 1660 cm^{-1} as C=O (in lignin) and at 1020 cm^{-1} as C-O (in cellulose and hemicellulose), which are all characteristic bands of wood powders. The intensity of these bands of wood was increased as the wood powder loading increased. The FTIR spectra of WRC was similar to the IR spectrum of tire rubber, and the composites did not generate new functional groups. Therefore, it was confirmed that the rubber had played a leading role to form the composites, and the addition of wood powder did not change the chemical property of the rubber.
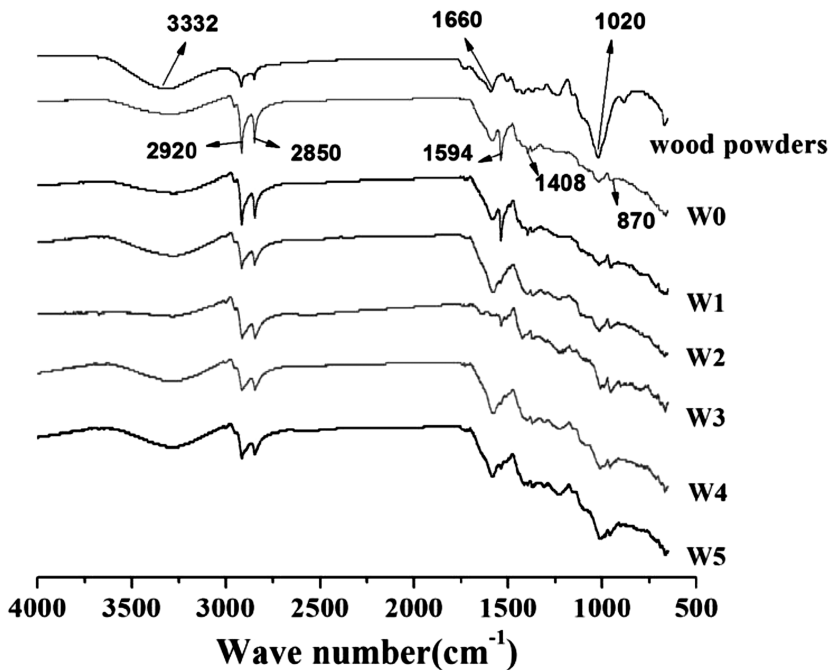

Figure 3.   Fourier transform IR spectroscopy spectra of wood-rubber composites with different wood powder loadings.

Curing Characteristics

The curing characteristics reveal the vulcanizing process of the composites. The minimum torque (ML) in the rheograph indicates the material flow characteristics, such as viscosity. The maximum torque (MH) is a measure of cross-link density and stiffness in the rubber (Jacob et al 2004a, b). In general, for all the mixtures used in our experiments, the torque decreased initially, then increased, and leveled off eventually. The initial decrease in torque to a minimum value was due to the softening of the rubber matrix. The increase in torque was due to the cross-linking of rubber. The leveling off indicated the completion of the curing. The scorch time (t_s2) indicates when the rubber starts to vulcanize. The optimum curing time (t_c90) is the material to construct the highest cross-linking density with the different wood powder contents - the longer the optimum curing time, the slower the curing rate, and the lower the yield. Figure 4 shows that the presence of wood powders generates an increase in viscosity of the mixes. The torque decreases initially, then increases and levels off eventually as the wood powder content increased from 0 wt.% to 50 wt.%.

Table 1 shows that both ML, MH, and t_s2 and t_c90 increased as the wood powder content increased from 0 wt.% to 40 wt.%, then decreased when the wood powder content continued to increase to 50 wt.%. The increase of the minimum torque indicated that the increase of the viscosity of the mixture as the wood powder content increased. The increase in maximum torque indicated that the stiffness of the composites increased when the wood powder content increased from 0 wt.% to 40 wt.%. The increment in torque values with increasing wood powders loadings indicated that more and more wood powders get into the rubber matrix, the mobility of the macromolecular chains of the rubber reduced, resulting in more rigid vulcanizates (Stefani et al 2005). The decrease in maximum torque could be explained by the lack of sulfur. Sulfur content may affect the cross-linking density, as the bonding of rubber is mainly through sulfur-sulfur bonds and carbon-sulfur bonds, which are achieved under the high temperature and pressure in vulcanization (Garcia et al 2007). The increase in the scorch time and optimum cure time of the blends indicated that the cross-linking reactions started later and the
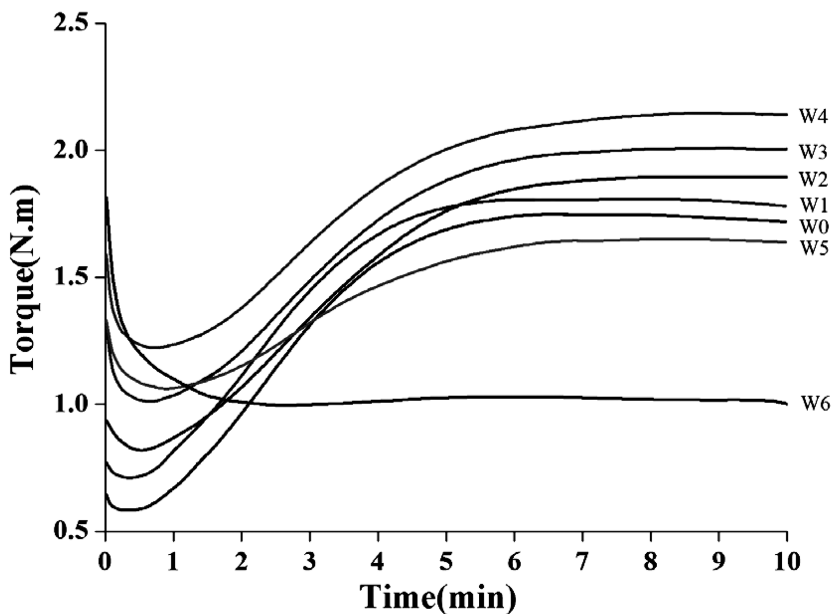

Figure 4.   Curing curves of wood-rubber composites with different wood powder loadings at a curing temperature of 160°C.

Table 1.   Curing characteristics of wood-rubber composites.

Sample code	ML (N.m)	MH (N.m)	t_c2 (min)	t_c90 (min)
W0	0.60 ± 0.05	1.78 ± 0.12	1.33 ± 0.11	4.33 ± 0.44
W1	0.72 ± 0.10	1.81 ± 0.10	1.35 ± 0.08	4.57 ± 0.21
W2	0.80 ± 0.08	1.83 ± 0.21	1.35 ± 0.10	4.88 ± 0.52
W3	1.00 ± 0.05	2.01 ± 0.11	1.65 ± 0.12	5.15 ± 0.36
W4	1.19 ± 0.11	2.15 ± 0.20	1.82 ± 0.10	5.42 ± 0.34
W5	1.04 ± 0.06	1.65 ± 0.13	1.77 ± 0.14	5.37 ± 0.40

vulcanization process was slow. Wood powder contains hydroxyl groups (-OH), which absorbs the curative agents, reduces the active sulfuring agent (Hanafi et al 2002). That was why the cure rate of the mixture was slowed down when wood powder was incorporated.

A good vulcanization process should have: 1) enough scorch time to fit the machining process; 2) fast curing rate to improve the production efficiency; 3) long flat vulcanizing period to ensure the safety of sulfide in the operation; and 4) all the above requirements met at the same time, higher performance, namely high peak. In conclusion, the incorporation of wood powder into the tire rubber reduced the processing, the cure characteristics changed little as the wood powder loading increased, and can meet the requirements of good vulcanization process. It was concluded that the process ability of the composites did not change remarkably and was still smooth.

Mechanical Properties

The mechanical properties of the WRCs with different wood powder contents are listed in Table 2. It is seen from Table 2 that as the wood powder content increased, the hardness of the WRC increased, whereas the tensile strength, the elongation at the break, and rebound resilience decreased. The decreases in tensile strength and

elongation at the break of the composites decreased would be due to the inability of the filler to support stresses transferred from the polymer matrix (Hanafi et al 2002). The hardness of the wood-rubber composite panels was higher than that of the pure rubber panels. These results indicated that the incorporation of wood powders into the rubber matrix enhanced the stiffness of the composites. When wood powder loading increased from 0 wt.% to 50 wt.%, the hardness increased by about 53% (from 60 to 92 IRHD) demonstrating a good reinforcement of rubber matrix by well dispersed wood powders. The decrease in rebound resilience was evidently due to the reduction of the volume fraction of the elastomer. However, when the loading of wood powder was increased up to 50 wt.%, the rebound resilience remained as 18, suggesting that the incorporation of wood powder did not affect the resilience properties.

The WRCs could be used as industrial rubber sheet. Industrial rubber sheet is a common industrial material, that can be used as a machine seat cushion, car inside apply to plastic board, printing rubber sheet, different acid, alkali, oil protection boards, rubber mat, cushion, etc. Rubber sheet is that rubber has gone through vulcanization process with a certain thickness and relatively large area of sheet products. Products with high hardness and low mechanical properties

Table 2.   Mechanical properties of wood-rubber composites.

Sample code	Tensile strength (MPa)	Elongation at break (%)	Hardness (Shore A)	Rebound resilience
W0	16.4 ± 1.1	634.1 ± 19.7	60 ± 0.00	22 ± 0.00
W1	10.9 ± 0.7	485.0 ± 21.7	68 ± 0.58	22 ± 1.53
W2	6.6 ± 0.4	398.1 ± 9.2	78 ± 0.58	21 ± 0.00
W3	6.3 ± 0.5	298.8 ± 10.5	82 ± 0.58	20 ± 0.58
W4	4.4 ± 0.6	109.5 ± 15.5	89 ± 1.15	19 ± 1.32
W5	5.0 ± 0.2	34.6 ± 3.5	92 ± 0.58	18 ± 0.58

can be obtained. These products could be used in somewhere with low pressure resistant with an ambient temperature of 0-60°C. As the wood powder content increased, the hardness of the WRCs was increased, whereas the tensile strength was decreased. High hardness and low strength of the WRC conforms to the industrial rubber standard GB5574-85 on the high hardness rubber sheet 1704 and 1804 (rubber sheet specifications) requirements, and it could be used as a rubber mat for table and floor.

Water Absorption

The 24-h water absorption of W0, W1, W2, W3, W4, and W5 were obtained as 0%, 0.19%, 0.81%, 0.99%, 2.90%, and 4.13%, respectively, which are much lower than those wood-based composites, which are usually more than 20% (Shi and Wu 2009; Shi and Wang 1997; Shi et al 1999). The thickness swelling of 24-h soaking of the WRC increased as wood powder content increased. The increased thickness swelling of the WRC is also due to the hygroscopic nature of wood powder (Shi and Gardner 2006a, 2006b). However, rubber is almost hydrophobic and only minimally affected by atmospheric humidity. For all the wood-rubber composite samples, the thickness swelling were lower than the material made by waste rubber, wood, and adhesive with compression molding process (Xu and Li 2012; Xu et al 2014) indicating that most of the wood powders were encapsulated in the polymer matrix, as the tire rubber is almost hydrophobic (Nadir et al 2009b). Comparing with the traditional WRC, directly using rubber wrapping the wood powder exhibited less sensitivity to the MC of wood and a good water resistance.

CONCLUSIONS

Wood powder and tire rubber can be successfully combined to fabricate WRC using the rubber mixing and vulcanization process. Compared with the compression molding process, using the rubber mixing and vulcanization process to fabricate wood rubber composites presented a better interfacial bonding at the wood and rubber interface, and uniform distribution of the wood powders. The addition of wood powder did not change the chemical property of the tire rubber. The curing characteristics, such as minimum or maximum torques of the WRC, increased as the wood powder loading increased, whereas the scorch time and optimum curing time decreased. The wood powder loading cannot be more than 50 wt.%. The incorporation of wood powders increased the hardness of the WRC significantly with a marginal decrease in tensile strength, the elongation at the break, and rebound resilience. The water absorption of the wood rubber composites was much reduced compared with the wood composites, low, the composites had a good water resistance.

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