

The Effects of Binder on the Physical and Mechanical Properties of Chemically Treated Sawdust-Reinforced Polypropylene Composites

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ABSTRACT: This study investigates the effects of Polyvinyl Alcohol (PVA) as a binder on the mechanical and physical properties of wood polymer composites (WPCs). The WPCs were manufactured from tropical sawdust and polypropylene using a hot press molding method at five levels of sawdust loading (10–30 wt). For the manufacturing of the composites, the sawdust was chemically treated with 2-ethylhexyl methacrylate before 10 wt % PVA was added. The flexural strength, flexural modulus, Young's Modulus, and Rockwell hardness were found to improve with the treatments. However, the binder treated wood polymer composite (BTWPC) samples exhibited better mechanical properties than the raw wood polymer composite and the treated wood polymer composite (TWPC) samples. Moreover, water absorption decreased significantly for the TWPC and the BTWPC samples, indicating that their water resistance is higher than that of the raw samples. The SEM micrographs revealed that the interfacial bonding of the BTWPC had significantly improved, suggesting that the chemical treatment led to better dispersion of the filler into the matrix, and this improved further after the addition of the binder. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 129: 1534–1540, 2013

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INTRODUCTION

Due to the increasing awareness of environmental pollution and the scarcity of resources, non-toxic and environmentally friendly materials containing specific characteristics are becoming more sought after. Wood sawdust polymer composites (WPC) have been receiving considerable attention due to their renewability, availability, and improved physical and mechanical performance.^{1–3} WPC have the largest market share out of all natural fiber/plastic composites in building materials, furniture, and internal car parts.⁴ The addition of sawdust as a filler has also been found to improve the stiffness of polypropylene (PP) composites.^{5–7} Nevertheless, previous research has indicated that even though WPC possess acceptable mechanical properties, surface incompatibility between the sawdust and PP still poses a major challenge.^{8,9} This incompatibility is caused by the hydrophilic characteristics of the hydroxyl groups that exist in the

sawdust fiber, which reduce the mechanical properties and cause degradation. These composites are incompatible due to the fact that PP is a non-polar and hydrophobic material, whereas lignocellulose is a polar and hydrophilic material that contains large amounts of hydroxyl groups ([oxygen bond]OH).^{10,11} These problems can be minimized or fixed with the modification of the fiber by chemical treatment and the application of a coupling agent or binder to the composites.^{12,13}

The selection of a binder usually depends on the polymer used. For instance, Maleated Polypropylene is one of the most popular additives or binders used by researchers to improve the adhesion and compatibility between sawdust and PP.⁸ In general, a binder or coupling agent is typically added in small percentages to the WPC to reduce the interfacial tension between the non-polar polyolefin matrix and the wood filler, thereby enhancing the mechanical properties of the polymer composites.

This additive also enhances the morphology of the composites by evenly dispersing the discontinuous filler throughout the dominant polymer matrix.

Previous studies have shown that Polyvinyl Alcohol (PVA) is mainly used as a de-characterizing agent of thermosetting resin. It has also been used as a binding agent such as carbamide, formaldehyde resin, and melamine, for plywood, artificial board, and timber processing. When mixed with melamine-formaldehyde resin, PVA helps improve the viscosity and shortens the curing and cooling time due to its stronger initial cohesion. Moreover, PVA is also used as an adhesive agent for profiles, e.g., prefabricated plasterboard and sound absorption boards that are made of organic fibers (cane, residue, and wood-shavings) or non-organic materials that are highly cohesive and water proof. PVA can also be used to make profiles through compression and cohesion. A large number of research studies have been conducted on sawdust-reinforced WPC and their chemical modifications. Very little or no work, however, has been devoted to 2-ethylhexyl methacrylate chemical-treated tropical sawdust as a reinforcing filler in combination with PVA binder.

Motivated by our earlier study, the current investigation was carried out to determine the effects of binder on the physical and mechanical properties of chemically treated sawdust-reinforced PP composites.¹⁴ Tropical sawdust was chosen because it is produced on a large scale in sarawak's timber industry. Moreover, it has a minimal effect on the environment due to its biodegradability.¹⁴ The sawdust was treated with 2-ethylhexyl methacrylate, and 10% PVA binder was added in order to increase the adhesion and compatibility of the fiber to the polymer matrix. Thus, this study aims to determine the effect of PVA binder in the presence of 2-ethylhexyl methacrylate monomer on the physical and mechanical properties of sawdust-reinforcement pp composites (WPC) at different sawdust loading levels (wt %).

EXPERIMENTAL

Materials

The sawdust was oven-dried at 70–80°C to achieve a moisture content of 3–5%, then stored in a polyethylene bag until needed. PP, which was used as the polymer matrix, has a melt index of 0.28 g/10 min with a density of 0.938 g/cm³. The molecular weight of PP and PVA was 42.08 g/mol and 44.00 g/mol. The PP was supplied by Petrochemical Ind. Co. (Seoul, Korea). The particle sizes of the sawdust were in the range of 0.5 mm. The chemicals used to treat the sawdust were 2-ethylhexyl methacrylate (density- 0.885 g/mL and specific gravity- 0.87) and PVA, which were supplied by Merck (Darmstadt, Germany).

Methods

Chemical Treatment of Tropical Sawdust. Prior to treatment, the sawdust was dried at 105°C for about 24 hours until a constant weight was reached in order to obtain 1–2% moisture content. The dried sawdust was then kept in a sealed container. 200 mL of 2-ethylhexyl methacrylate (C₁₂H₂₂O₂) solution was measured into a 500 mL beaker. About 500 g of sawdust was submerged in the solution for about 1 hour at about 70°C in an oven. After about 1 hour, the sawdust was taken out of the beaker, washed with water, and finally dried in open air.

Preparation of WPC, TWPC, and BTWPC. The raw wood polymer composite (RWPC) and treated wood polymer composite (TWPC) were prepared from raw and treated sawdust and PP. Both raw and treated tropical sawdust (10, 15, 20, 25, and 30 wt %) was mixed thoroughly with PP in a beaker. For all binder treated wood polymer composite (BTWPC), 10 wt % PVA was added as a binder at room temperature. The mixtures were stirred continuously without any external heating until they were uniformly mixed, after which they were pre-heated in an oven for 24 hours at 80°C to ensure that the mixing was homogeneous. The mixture was introduced into a mold measuring 270 mm × 270 mm × 5 mm and then hot-compressed at the temperatures of 200 ± 5°C. The molding board was then cut to the test specimen sizes that were appropriate for each test. The molding conditions were as follows: pressure, 6.8 MPa; preheating time, 15 min; heating time, 45 min; and cooling under a slight pressure to ambient temperature.

FTIR Spectroscopy Analysis. The infrared spectra of the RWPC, TWPC, and BTWPC specimens were recorded on a Shimadzu Fourier Transform Infrared Spectroscopy (FTIR) 81001 for the purpose of characterizing the chemical change of the sawdust and the composites upon chemical treatment and use of the binder. The transmittance range of the scan was 4000–400/cm. Samples were dried, ground, and mixed with potassium bromide (KBr) in a ratio of 1:100 and pressed with hydraulic pressure to form pellets. A KBr pellet was used as a reference.

Scanning Electron Microscopy (SEM). The surface morphology of the manufactured composite was examined using a SEM (JSM-5510) supplied by JEOL Company (Tokyo, Japan). The samples were sputter-coated with platinum and observed under the SEM. The micrographs were taken at a magnification of 300×.

Mechanical Properties. In order to examine the mechanical properties of the samples, specific tests for tensile, flexural, and hardness were conducted. For each test, 10 specimens were utilized and the average values were reported.

Tensile Test. The tensile tests were carried out in accordance with ASTM D 638-01¹⁵ using a Shimadzu Universal Testing Machine with a loading capacity of 300kN. Each test was performed at a crosshead speed of 10 mm/min. The dimension of the specimen was 148 mm × 10 mm × 4 mm.

Flexural Test. Flexural tests were conducted in accordance with ASTM D 790-00¹⁶ using the same aforementioned testing machine at the same crosshead speed. The dimension of the specimen was 79 mm × 10 mm × 4.1 mm. The modulus of elasticity and flexural strength were calculated using the following equations,

$$\text{Flexural strength, } \sigma = 3PL/2bd^2 \quad (1)$$

$$\text{Flexural modulus, } E = L^3m/4bd^3 \quad (2)$$

$$\text{Water absorption \%} = (W_2 - W_1/W_1) \times 100\% \quad (3)$$

where P is the maximum applied load, L is the length of support span, m is the slope of the tangent, and b and d are the width and thickness, respectively, of the specimen.

Hardness Test. The hardness of the composites was measured using a Rockwell Hardness Testing Machine in accordance with ASTM D785-98.¹⁷ Results are shown in the following section.

Water Absorption Test. RWPC, TWPC, and BTWPC samples with the dimensions of 40 mm × 10 mm × 4.1 mm were prepared for the water absorption test. Prior to immersion in a static de-ionized water bath, the samples were air-dried at 80°C until a constant weight was reached. The specimens were periodically taken out of the water, wiped with tissue paper to remove water on the surface, reweighed, re-measured, and immediately put back into the water. Water absorption was calculated according to the given formula,

$$\text{Water absorption (\%)} = (W_2 - W_1/W_1) \times 100\% \quad (3)$$

where W_2 is the specimen weight after soaking and W_1 is the specimen weight before soaking.

RESULTS AND DISCUSSION

FTIR Spectroscopy Analysis

The expected chemical reaction of the sawdust with 2-ethylhexyl methacrylate and the formation of composites were reflected in the FTIR spectroscopic analysis as shown in Figure 1. From Figure 1, it can be seen that the absorption peaks of the RWPC (3408, 2961, 2918, 2839, 1459, 1377, 1256, 1166, 1056, 995, and 973 cm^{-1}) shifted towards higher or lower wave numbers (i.e., 3415, 2960, 2921, 2838, 1459, 1377, 1166, 1058, and 973 cm^{-1}) for the TWPC sample. The characteristic absorption bands of cellulose and hemicelluloses are indicated by the peaks at 1650 and 3200 to 3600 cm^{-1} in the TWPC samples.^{18,19} These absorption peaks are due to the C[dbond]O, H-bond and [oxygen bond]OH stretching vibration in the sample. Meanwhile, C[sbond]H stretching and bending can be observed at 2890–2940 cm^{-1} . A few new peaks at 2723 and 1034 cm^{-1} , which may

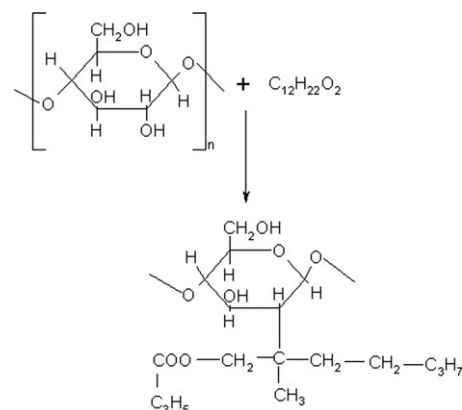


Figure 2. The chemical reaction scheme of cellulose in sawdust with 2-ethylhexyl methacrylate.

correspond to a new chemical reaction between the sawdust and 2-ethylhexyl methacrylate, were found in the TWPC samples. These results suggested that the chemical reaction was successful and that it yielded the cellulose-ethylhexyl methacrylate compound (Figure 2) in the composite as observed by other researchers.^{20,21}

Additionally, the more prominent peaks of the [oxygen bond]OH groups shifted to higher wave numbers with narrow band intensity, which provided further evidence of the above reaction (Figure 1). However, in the BTWPC, a few new peaks were also found at 2723, 1509, and 1034 cm^{-1} , suggesting a new interaction that occurred due to the addition of the PVA. It is also observed that OH peak of BTWPC is higher compared to other two samples. The reason may be due the additional OH groups in PVA. All the characterized absorption bands clearly confirmed that the composites consisted of sawdust and PP, and that a few changes had occurred due to the chemical treatment and binder effect.

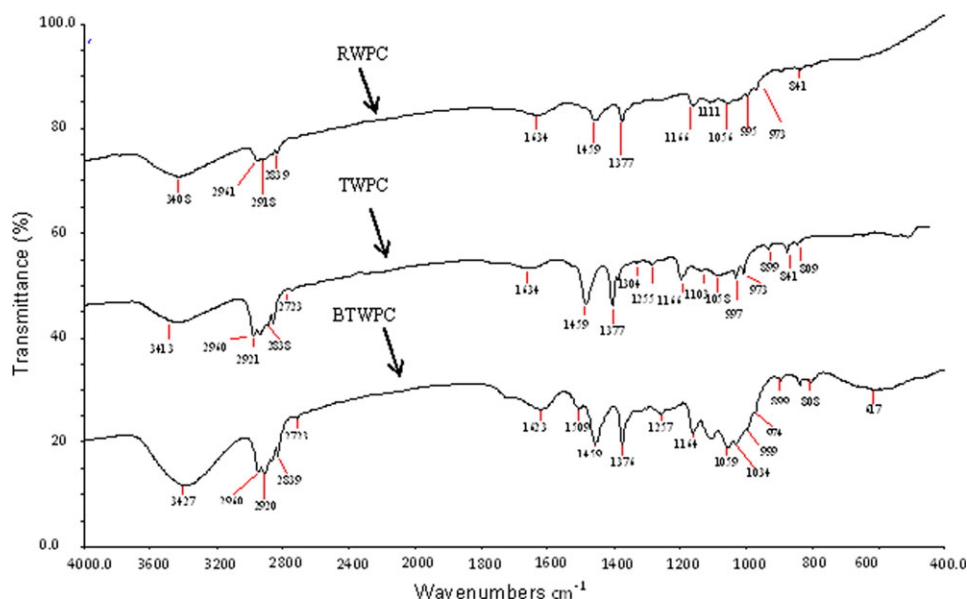


Figure 1. IR spectrum of RWPC, TWPC, and BTWPC composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

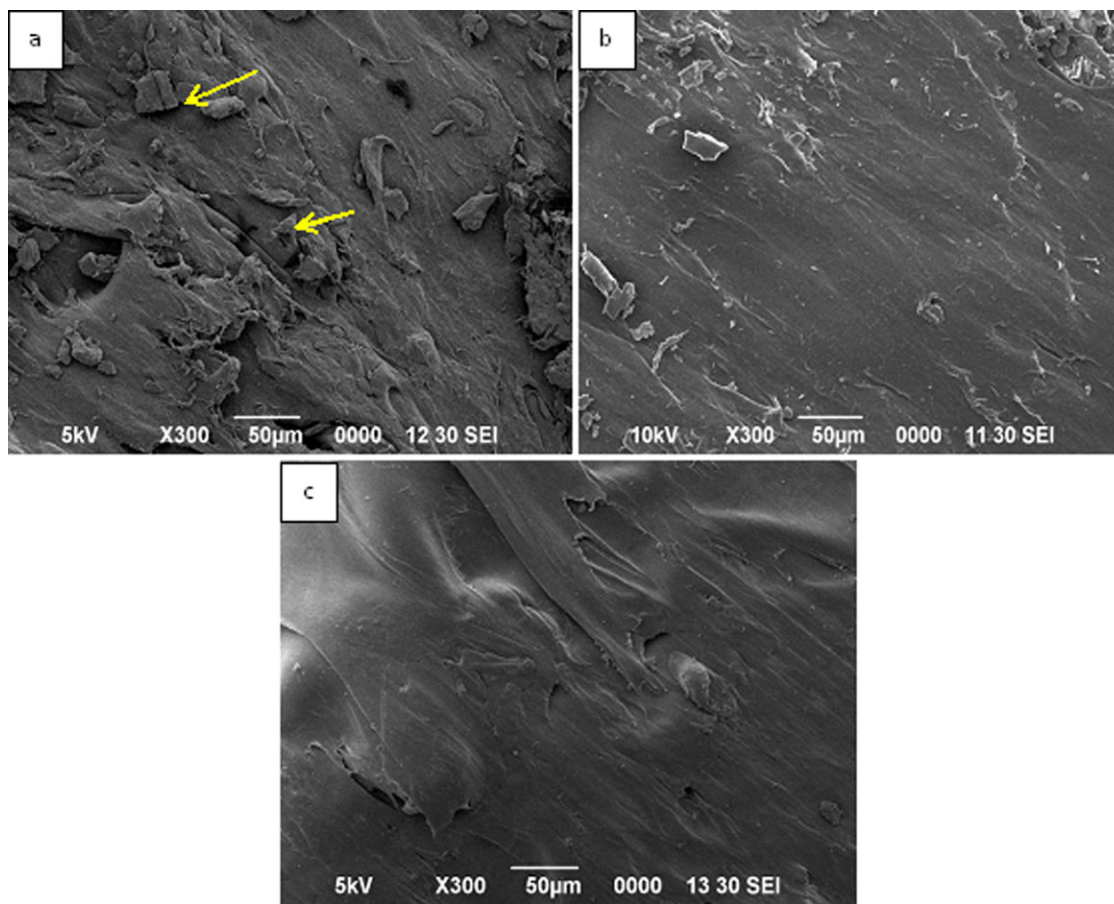


Figure 3. Typical SEM morphology of the PP reinforced with 30% sawdust (a) RWPC, (b) TWPC, and (c) BTWPC. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Scanning Electron Microscopy (SEM)

The typical SEM micrographs of the RWPC, TWPC, and BTWPC at 30% tropical sawdust loadings are shown in Figure 3. The SEM image of the WPC shows an agglomeration of sawdust that clearly demonstrates the existing roughness and void between PP and the sawdust reinforcement in the composite matrix [Figure 3(a)]. This feature indicated that there was poor dispersion and weak interfacial bonding between the matrix and the sawdust, which confirmed that the interfacial bonding between the filler and the matrix polymer was poor and weak.²²

In contrast, the SEM image for the TWPC shows a better dispersion of sawdust throughout the matrix [Figure 3(b)]. This resulted in better interfacial bonding between the sawdust and the matrix, as is reflected in the FTIR results. However, the best dispersion and interfacial bonding was observed in the BTWPC sample in Figure 3(c). As is clearly depicted in the micrograph, the agglomeration of sawdust was substantially reduced in comparison to the composites. The incorporation of PVA binder strongly reduced the surface roughness and void spaces in the composites as illustrated. Consequently, the BTWPC had a smoother surface texture than the others. It can be deduced from the SEM micrographs that the PVA binder enhanced the phase compatibility and adhesion between the sawdust and the PP matrix, which significantly increased the surface morphology

of the composite. It can also be observed that the dispersion of sawdust in the BTWPC sample was more uniform than that of TWPC and RWPC, a characteristic that would greatly affect the mechanical behavior of the composites.²³

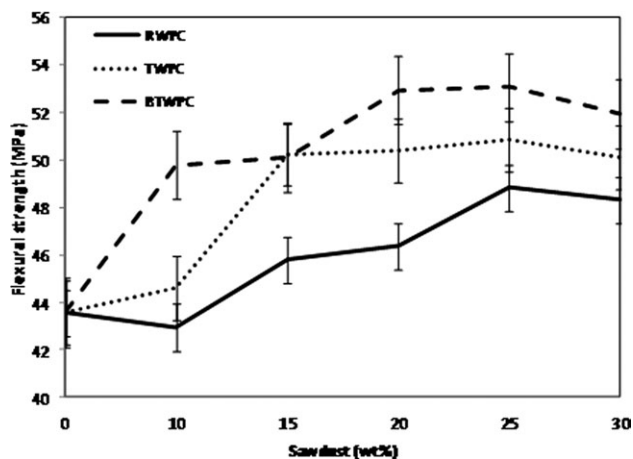


Figure 4. (A). Typical stress–strain curve obtained during flexural test of the PP reinforced with 30% sawdust (a) RWPC, (b) TWPC, and (c) BTWPC. (B) Variations of flexural strength of composites at different filler loadings.

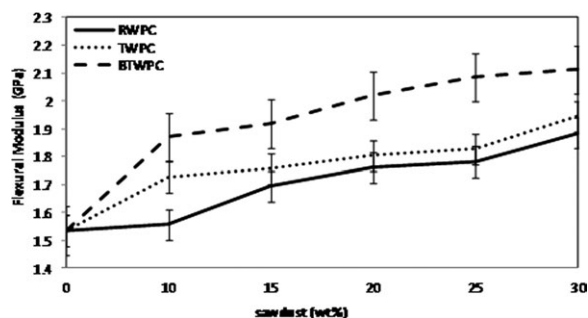


Figure 5. Variations of flexural modulus at different filler loadings showing possible data trend.

Flexural Properties

The flexural strength and modulus of the RWPC, TWPC, and BTWPC at different levels of sawdust loading are shown in Figures 4 and 5 respectively. From Figure 4, it is apparent that the BTWPC yielded higher flexural strength than did the TWPC and RWPC for all loading levels. The ranges of flexural strength for BTWPC, TWPC, and RWPC were 49.78–51.92, 44.6–50.10, and 42.94–48.31 MPa, respectively. It was also found that the flexural strength increased approximately 7.5–15.9% for the BTWPC and 3.7–9.7% for the TWPC with respect to the RWPC. These positive results were due to the combined technique (i.e. chemical treatment and binder use), which increased the inter-phase interaction and compatibility between the sawdust and PP in the composites, thus significantly enhancing flexural strength. Similar results have also been reported by other researchers.⁴ It can also be seen from Figure 4 that the flexural strength increased with the increase in sawdust loading; however, there was a slight decrease at the 30 wt % sawdust loading level for all the manufactured composites.²⁴

Figure 5 illustrates the variations in the flexural modulus at different filler loadings. It can be seen that the flexural modulus of the BTWPC composites was higher than those of the TWPC and RWPC. The average values of the flexural modulus were 1.87–1.97, 1.73–1.94, and 1.56–1.88 GPa for BTWPC, TWPC, and RWPC, respectively. The increase in the flexural modulus was 7.47–15% for BTWPC and 3.7–9.74% for TWPC compared to their corresponding RWPC. The improvement in this flexural modulus can be explained by the strong interaction and adhesion between the reinforced filler and the PP in the composites, as mentioned. Furthermore, the higher modulus of the BTWPC in comparison to the TWPC and RWPC provided evidence of a homogeneous distribution of the sawdust particles within the matrix, which led to better matrix-sawdust interaction. The flexural modulus of composites depended on the morphology of the WPC as well.

Figure 5 also shows that the flexural modulus increased as the filler loadings increased up to 30 wt %. The highest value was observed at 30 wt % sawdust loading in the BTWPC sample (2.1 GPa), followed by the TWPC (1.94 GPa) and RWPC (1.88 GPa) samples. These results revealed that the higher content of sawdust in the composites demanded a higher stress for the same deformation. Furthermore, the increase in sawdust content in the PP matrix provided for additional stress transfer between them, which significantly increased the flexural modulus of the composite.

Tensile Strength

The tensile property of the RWPC, TWPC, and BTWPC against different filler loadings is presented in Figure 6. Based on Figure 6, it is clear that the BTWPC exhibited a higher tensile strength than the TWPC and RWPC at all sawdust loadings. The value of tensile strength for BTWPC, TWPC, and RWPC were in the range of 26–27, 25–20, and 25–19 MPa, respectively. It can also be observed from Figure 6 that the tensile strength for RWPC was higher than that of TWPC, but that between 10 and 30 wt % sawdust loadings it decreased gradually by approximately 3.92 to 9% (RWPC) and 2.3 to 6.88% (TWPC). However, the tensile strength for the BTWPC increased by about 7.98–42.49% between 10 and 25 wt % sawdust loadings. At 30 wt %, the BTWPC tensile strength decreased slightly, but nonetheless was 47.17% higher than that of RWPC. This may be due to the incorporation of PVA into the PP matrix, which increased the compatibility and adhesion of the sawdust fiber with the PP, thereby resulting in higher tensile strength for BTWPC. These results also suggested that stress is expected to transfer from the sawdust fiber to the matrix, indicating a better interfacial bonding with a consequent improvement in the tensile properties.

On the other hand, the tensile strength for the RWPC and TWPC decreased gradually with the increase in sawdust loading. Similar results have also been reported by other researchers.^{1,6,24,25} This happens because as the sawdust loading increases, the weak interfacial area between the sawdust and the matrix increases as well, which consequently decreases the tensile strength.^{26–28} This low improvement of properties is attributed to the low chemical compatibility between the components of the composite at the interfacial area, as mentioned previously.²⁹ The compatibilization of composite surfaces with no binder agents is related to a physics mechanism involving mechanical anchoring due to surfaces pores of the sawdust fibers. However, when the material is modified with a binder, the improvement in tensile strength is better than that of the raw materials.³⁰ For the BTWPC at a low sawdust composition up to 10 wt % created phase discontinuity and heterogeneity inside the composite specimens, resulting in a drop in tensile strength (Figure 6). However, when the sawdust loading increased from 10 wt % to 25 wt %, the tensile strength gradually showed a significant increase after the addition of PVA binder.

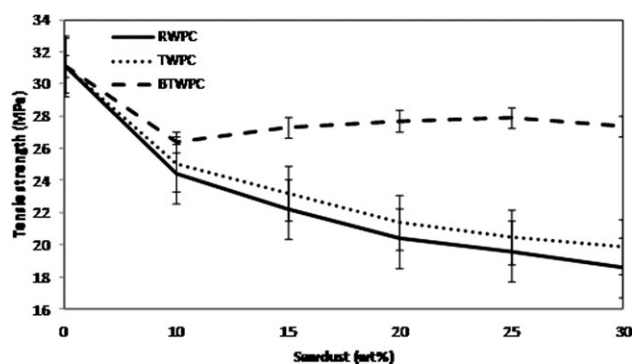


Figure 6. Variations of tensile strength of WPC at different filler loadings

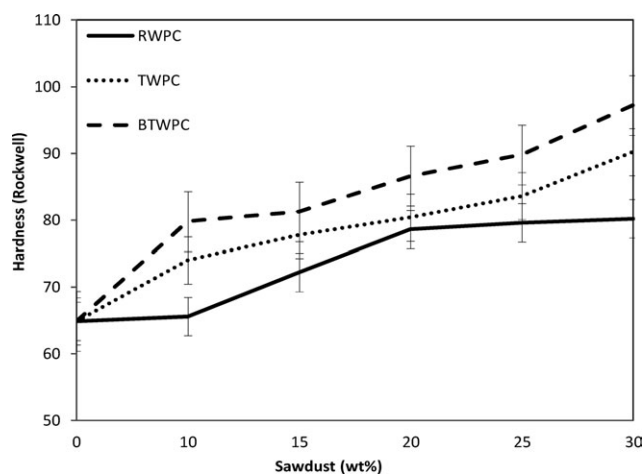


Figure 7. Variations of hardness at different filler loadings for RWPC, TWPC, and BTWPC.

Hardness Test

Figure 7 shows the hardness values of various manufactured composites at different sawdust reinforcement loadings. The results showed that both the TWPC and the BTWPC exhibited significantly higher hardness values than the raw materials at all sawdust loadings. It is generally accepted that the hardness of a composite depends on the distribution of the filler into the matrix.^{31,32} The results of this study showed that the average hardness values increased upon chemical treatment and sawdust loading.

As is shown in Figure 7, the incorporation of PVA into the treated sawdust polymer composites increased the surface hardness. On the other hand, the average hardness of the TWPC and the post-BTWPC increased with the increase of the sawdust reinforcing fillers. This was expected because lignocellulosic fillers display considerably higher hardness than the soft polymer matrix. This may be due to the increased stiffness of the respective composites. The BTWPC, meanwhile, showed better hardness properties than did the raw and treated samples, which could be attributed to better filler-matrix interfacial wetting and surface crystallization upon chemical treatment of the fiber.^{26,33}

Water Absorption Behavior

Figure 8 shows the water absorption results of the composites against the filler loadings. The water absorption of TWPC at 10, 15, 20, 25, and 30 wt % sawdust loading levels were 1.701, 2.104, 2.704, 2.983, and 3.638% respectively. Meanwhile, the water absorptions of BTWPC at 10, 15, 20, 25, and 30 wt % were 1, 1.143, 1.2, 1.35, and 1.741% respectively, which showed a significant decreasing trend. As is displayed in Figure 5, the RWPC exhibited higher water absorption than the TWPC and PTWPC. It was also found that the 30 wt % sawdust loading sample for all composites (i.e., RWPC, TWPC, and BTWPC) absorbed more water than the lower loading samples. These results are explained by the fact that the increase in sawdust loading in the composites gave rise to a higher number of hydroxyl groups, which consequently increased the water absorption.^{31,32} It has been established that cellulose and hemicelluloses in sawdust are the major factors involved in the high

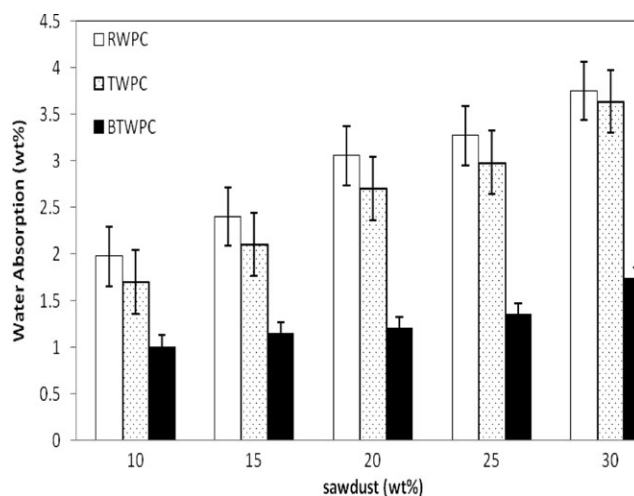


Figure 8. Variations of water absorption of RWPC, TWPC, and BTWPC at different filler loadings after 16 days of 24-hour soaking in water.

water absorption of natural fibers, since both contain hydrophilic hydroxyl groups that attract water molecules from the surrounding environment to form hydrogen bonding.^{18,19}

However, the TWPC and BTWPC samples showed significantly decreased water absorption compared to the RWPC sample at the same sawdust content level, as seen in Figure 8. This is due to the chemical reaction between the cellulose in sawdust and 2-ethylhexyl methacrylate, which significantly decreased the amount of [oxygen bond]OH groups in the sawdust. As a result, the sawdust became less hydrophilic, thus leading to less water absorption. It should also be noted that the BTWPC sample showed a greater decrease in water absorption than both the RWPC and TWPC samples did. This improvement in water absorption for the BTWPC was expected; the PVA binder increased the interaction and adhesion between the sawdust and the PP, which in turn blocked the void spaces and sorption sites of the water molecules from the composites. This is clearly illustrated in the SEM micrographs. Consequently, fewer water absorption sites remained, which also contributed to the reduction in water uptake.²⁴ More precisely, it also indicated that the favorable interaction between the sawdust and matrix managed to reduce the voids in the composites, demonstrating the lower water absorption tendency of the composites.

CONCLUSIONS

The following conclusions can be drawn from the present study:

1. The tensile strengths of the composites decreased slightly as the sawdust loading increased; however, the composites retained an acceptable strength. Nevertheless, there was an increase in the tensile strength of the BTWPC between 10 and 25 wt % sawdust loadings.
2. The Young's modulus, flexural modulus, flexural strength, and hardness of the composites increased after the application of PVA as the sawdust loading increased. These results were better than those of the raw materials. The stiffness of the TWPC increased significantly in comparison to RWPC. These results established that even at 30 wt % sawdust

loading, the imbued materials had higher tensile strength and achieved better results in Young's modulus, flexural modulus, and hardness tests, although their flexural strength was lower than that of 25 wt % sawdust loading.

3. The tensile strength, Young's modulus, flexural strength, flexural modulus, and hardness of the BTWPC were higher than those of raw and treated WPC.
4. Physical properties such as water absorption were gradually reduced when the PVA was introduced into the composites. Better results were achieved with the BTWPC. With the addition of PVA, the interfacial bonding between the sawdust filler and the matrix polymer improved greatly, resulting in enhanced water absorption behavior.
5. The authors proposed that the 25 wt % sawdust-reinforced composites had the optimum set of mechanical and physical properties in comparison to other manufactured composites. In order to have better mechanical properties at higher sawdust contents, the bonding between the sawdust and the PP matrix had to be improved. Chemical treatment and application of a binder can help achieve this by improving the adhesion between hydrophilic and hydrophobic materials.
6. The current results and findings of this work provide evidence that the addition of the binder agent PVA can be used successfully to improve the performance of WPC in terms of its physical and mechanical properties. This is, nonetheless, only acceptable at the research level, and so far is less suitable for commercial exploitation.

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