Physical and Mechanical Properties of Polypropylene-Wood-Carbon Fiber Hybrid Composites

Djamila Kada,[a,b,c] Sébastien Migneault,[c] Ghezalla Tabak,[b] and Ahmed Koubaa [c,*]

Effects of the addition of short carbon fibers (CFs) on the mechanical, physical, and morphological properties of polypropylene (PP) and wood-polypropylene composites (WPCs) were investigated. Hybrid composites (mix of wood and CFs) were manufactured in a two-stage process, pellet extrusion and samples mold injection with varying amounts of poplar wood fiber (0%, 20%, 30%, and 40%) and CFs (0%, 3%, 6%, and 9%), with and without maleic anhydride grafted PP (MAPP) as a coupling agent. The composites were prepared with extrusion blending followed by injection molding. The samples where then tested for mechanical and physical properties, and fractured surfaces where observed with scanning electron microscopy. The results indicated that the addition of CFs to WPCs improved the tensile and flexural strength and the modulus of elasticity but had only a small influence on elongation at break and impact strength. The density of hybrid composites slightly increased with CFs proportion but their water absorption was not affected. Scanning electron micrographs of the tensile fractured specimens showed improved adhesion of CFs and poplar with the PP matrix in the presence of a coupling agent.

Keywords: Polypropylene; Short carbon fiber; Poplar wood; Hybrid composite; Mechanical properties

*Contact information : a: École Préparatoire Sciences et Techniques d'Alger, BP 474 Place des Martyrs, Alger, Algeria, 16000; b: Université des Sciences et de la Technologie Houari Boumediene, BP 32 El-Alia, 16111, Alger, Algeria; c: Université du Québec en Abitibi-Témiscamingue, 445 boul. de l'Université, Rouyn-Noranda, Québec, Canada, J9X5E4;*Corresponding author: ahmed.koubaa@uqat.ca*

INTRODUCTION

Natural fiber-thermoplastic composites such as wood-plastic composites (WPCs) are a new alternative to traditional materials. These commodities are growing in popularity because of their low cost, dimensional stability, fungal and termite resistance, and low maintenance. Furthermore, adding wood fibers to plastic products is an efficient use of waste wood. Poplar wood (*Populus tremuloides*) is among the most abundant hardwood species in Canada. This species is underutilized in some areas and it is therefore available for the production of WPCs. A previous study has shown the potential of poplar wood for WPC applications (Migneault *et al.* 2014). Currently, WPCs are widely used for decking, outdoor spacing such as landscaping timbers, fencing, playground equipment, building louver facades, industrial pallets, bridges, and automotive interior parts (Kim and Pal 2011). Despite their advantages, WPCs suffer from disadvantages such as hydrophilicity, poor mechanical properties, and poor thermal properties. As a consequence, these products are only used for non-structural applications.

Hybrid composites can be described as systems with two or more reinforcing materials in a single matrix. Hybridization of natural fibers with stronger and more corrosion-resistant synthetic fibers, such as glass fibers or carbon fibers (CFs), can improve

the stiffness, strength, and moisture resistance of natural fiber-reinforced thermoplastics. Given the reinforcement of CFs in a PP matrix (Tamura *et al.* 2003; Karsli and Aytac 2011; Enoki *et al.* 2014), CFs could be used for the hybrid reinforcement of WPCs.

Few studies have been conducted on the hybridization of WPCs. Anuar *et al.* (2008) studied the hybridization of thermoplastic natural rubber reinforced with CFs treated with sulfuric acid and kenaf fiber. Composites were formed with a melt-blending method in an internal mixer. The results revealed that the flexural strength and the modulus of elasticity increased with increasing fiber proportion. However, the flexural properties for the composite with a single type of reinforcement were better than those of the hybrid composite. The impact strength also increased with increasing fiber proportion. The use of a coupling agent reduced the strength of the hybrid composites. Lin *et al.* (2013) combined impact-resistant PP, coir, and electrically conductive CFs to form hybrid composites. The prepared hybrid composites were electromagnetically shielding and recyclable and thus broaden the range of applications of natural fiber-reinforced thermoplastic composites. Zolfaghari *et al.* (2012, 2013) investigated the potential of glass fibers for reinforcing HDPE composites. They demonstrated the feasibility of processing hybrid composites using an extrusion process (Zolfaghari *et al.* 2012), and clearly reported substantial improvements in tensile and impact strengths (Zolfaghari *et al.* 2013) in comparison to wood-polymer composites. Turku and Karki (2014) investigated the effect of micro-sized glass fibers and CFs and nano-sized montmorillonite organoclay addition on the mechanical and physical properties of wood flour/PP composites. The addition of CFs did not affect the composite tensile strength. The tensile modulus of elasticity and the impact strength increased by 18% and 7%, respectively, while the impact strength decreased by 19%. Zhou *et al.* (2014) studied the effect of maleic anhydride grafted polyethylene (MAPE) on the properties of hybrid composites. The flexural and tensile strengths of hybrid composites were very close to that of structural timber. The mechanical (tensile strength, flexural strength, and impact strength) and electrical (volume electrical resistivity) properties were considerably improved with the addition of MAPE.

Hybridization of WPCs represents a good potential for increasing the performance of the material for structural applications. However, the lack of interaction between the synthetic fibers, wood fibers, and matrix limits the effective reinforcement (Lu *et al.* 2007; Parka *et al.* 2003). Given the reinforcement of CFs in a PP matrix (Tamura *et al.* 2003; Karsli and Aytac 2011; Enoki *et al.* 2014), CFs could be used for the hybrid reinforcement of WPCs.

A better understanding of the interaction between CFs, wood fibers, and PP has both scientific and practical interest. Therefore, the objective of the present study was to investigate the effect of CFs and MAPP on the mechanical, morphological, and physical properties of PP/wood poplar/short CFs hybrid composites.

EXPERIMENTAL

Materials

Virgin polypropylene (PP 4150H, Pinnacle Polymers, USA) was used as a matrix. Its melt flow index is 55 g/10 min at 230 °C, the density at room temperature is 0.90 g/cm^3, the tensile strength at yield is 20.7 MPa, and the flexural modulus is 1000 MPa. Anhydride modified homo polymer PP (MAPP) (Admer AT2305A, Mitsui Chemicals America, USA)

was used as coupling agent. Its melt flow index is 1000 g/10 min at 230 °C, and its density at room temperature is 0.90 g/cm³.

Chopped Panex CFs with 2.75% epoxy-based sizing (type-65) was supplied by Zoltek (Bridgeton, Missouri, USA). Its unpacked bulk density is 0.425 g/cm^3, and the fibers are 6 mm in length.

Poplar wood fibers (*Populus tremuloides*) from eastern Canada were used as reinforcement. Poplar is an underused species in the northern part of the province of Quebec, Canada. Poplar wood was ground with a Thomas model 4 Wiley mill (Thomas Scientific, Swedesboro, New Jersey, USA), mounted with a 2-mm opening sieve. Poplar fibers were then sieved with a Ro-Tap test sieve shaker RX-29 model (W.S. Tyler Industrial Group, Mentor, Ohio, USA). The 150 to 710 μm opening fraction (100 to 25 US mesh) was used in composite formulations. Finally, the fibers were oven-dried at 80 °C to approximately 3% dry basis moisture content.

Composite Preparation

The composite formulations were prepared by extrusion blending with a conical twin screw extruder (Thermo Scientific, HAAKE PolyLab OS Rheodrive 7 with Rheomex OS extruding module, USA) at a temperature of 185 °C and a mixing speed of 30 rpm. The extruded material was cooled in a water bath at room temperature and then cut into 3-mm-long pellets.

After being oven-dried at 80 °C overnight to a dry basis moisture content of about 3%, the pellets were mold-injected for preparation of tensile, flexural, impact, and water uptake specimens with an Arburg (Loßburg, Germany) 370 A 600 kN injection molding machine. Injection molding parameters were 25 °C mold temperature, 140 MPa injection pressure, 1.7 s injection time, 40 MPa holding pressure, 9.65 s holding time, barrel temperature profile 195 to 171 °C, and 17 s cooling time. Processing conditions were selected for optimal mixing and sample quality according to the parameters obtained in previous studies (Migneault *et al.* 2014). All samples were conditioned until mass stabilization at 20 °C and 50% relative humidity prior to testing.

The composite formulations, presented in Table 1, can be classified into different categories: PP, carbon fiber-reinforced polypropylene (CFRP), wood-polypropylene composites (WPCs), and hybrid wood fiber-carbon fiber-polypropylene composites (hybrid composites).

WPC Sample Characterization

Composites apparent density, water uptake, and water absorption of soaked samples were measured according to the ASTM D1037 (1999) standard. Specimens were tested in triplicate. Three-point bending properties were measured according to ASTM D790 (2003) with a span-to-depth ratio of 16:1 and at a speed of 1.4 mm/min. Unnotched cantilever beam impact resistance was measured according to ASTM D4812 (1999) with a 2.75-J pendulum. Tensile properties were measured according to the ASTM D638 (2003) standard using specimen Type I and a speed of testing of 4 mm/min. Tensile fractured surfaces were observed with scanning electron microscope (SEM) Hitachi (Tokyo, Japan) S-3500 using secondary electron mode. The operating conditions were 20 keV, approximately 100 μA, and 5 mm working distance.

Table 1. Composite Formulations (Percent by Weight)

Type	Code	Poly-propylene	Wood	Carbon Fiber	Coupling agent
PP[a]	PP	100	0	0	0
CFRP[b]	PP91/CF9	91	0	9	0
CFRP	PP94/CF3/MAPP3[c]	94	0	3	3
CFRP	PP91/CF6/MAPP3	91	0	6	3
CFRP	PP88/CF9/MAPP3	88	0	9	3
WPC[d]	POP20/PP80[e]	80	20	0	0
WPC	POP30/PP70	70	30	0	0
WPC	POP40/PP60	60	40	0	0
WPC	POP20/PP77/MAPP3	77	20	0	3
WPC	POP30/PP67/MAPP3	76	30	0	3
WPC	POP40/PP57/MAPP3	57	40	0	3
Hybrid Composite	POP20/PP74/CF3/MAPP3	74	20	3	3
Hybrid Composite	POP20/PP71/CF6/MAPP3	71	20	6	3
Hybrid Composite	POP20/PP68/CF9/MAPP3	68	20	9	3
Hybrid Composite	POP30/PP64/CF3/MAPP3	64	30	3	3
Hybrid Composite	POP30/PP61/CF6/MAPP3	61	30	6	3
Hybrid Composite	POP30/PP58/CF9/MAPP3	58	30	9	3
Hybrid Composite	POP40/PP54/CF3/MAPP3	54	40	3	3
Hybrid Composite	POP40/PP51/CF6/MAPP3	51	40	6	3
Hybrid Composite	POP40/PP48/CF9/MAPP3	48	40	9	3
Hybrid Composite	POP40/PP51/CF9/MAPP0	51	40	9	0

[a]Polypropylene. [b]Carbon fiber reinforced polypropylene. [c]Maleic anhydride grafted polypropylene. [d]Wood-polypropylene composite. [e] Poplar wood

RESULTS AND DISCUSSION

Physical Properties

Adding 9% CFs increased the density of PP by 4% due to the higher density of the CFs compared to that of PP. Density also increased with increasing wood content (Fig. 1). This result indicates that wood fiber lumens are filled with PP or collapsed, as the apparent density of solid poplar wood is approximately 350 to 400 kg/m³ and the density of the fiber cell wall is approximately 1450 kg/m³ (Rowell *et al.* 1999). The addition of CFs in WPCs also resulted in a slight increase in density. For example, the bulk density of WPC-POP40/PP57/MAPP3 is 1067 kg/m³, and it is 1110 kg/m³ for the hybrid composite POP40/PP48/CF9/MAPP, containing 9% CFs.

As expected, the water absorption rate of the neat PP was lower than that of the composites (Fig. 2). CFRP showed a low absorption rate, very close to that of neat PP. This is due to the hydrophobic nature of CFs (Turku and Karki 2014). The coupled CFRP-PP88/CF9/MAPP3 showed a lower water absorption rate than that of the uncoupled CFRP-

PP91/CF9. This is because MAPP improved adhesion between the CFs and the PP matrix by reducing the potential of microvoids, thus reducing the access of water molecules. Figure 2 clearly indicates that water absorption increased with increasing wood fiber proportion. This result can be explained by the hydrophilic nature of wood combined with the diffusivity of water into the composites *via* voids and flaws at the interfaces between fiber and matrix (Ghasemi and Kord 2009; Gwon *et al.* 2011). Additionally, fiber lumens are porous, tubular structures that allow the penetration of water by capillary action (Kiani *et al.* 2011). The coupling agent reduces water absorption because of the formation of covalent bonds of anhydride groups in the MAPP with hydroxyl groups of cellulose. Thus, the polar functions of wood are no longer available for water molecules. The high density of WPCs (Fig. 1) also suggests that there are very few voids in the material.

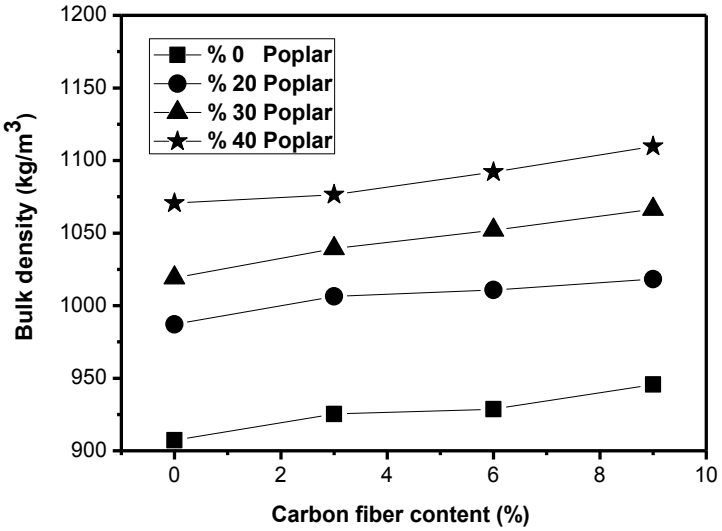

Fig. 1. Effect of carbon fiber content on the bulk density of PP and composites

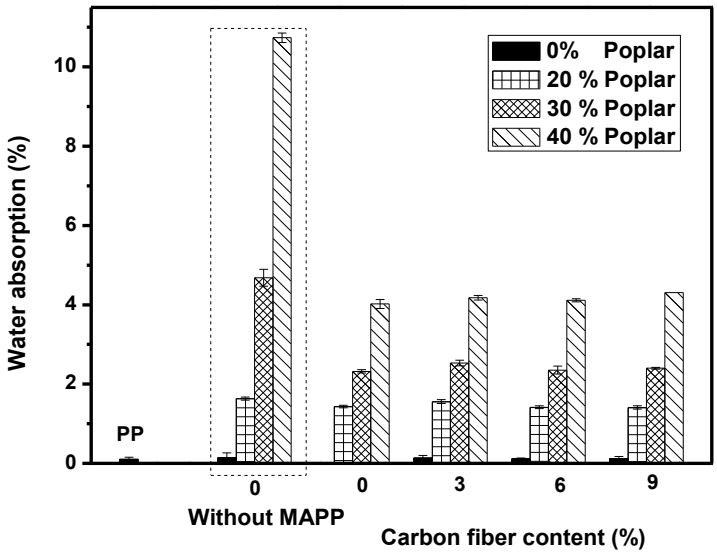

Fig. 2. Effect of carbon fiber content and MAPP on water absorption of PP and composites after 125 days of water immersion

Hybridization of poplar fibers with CFs had very little effect on water absorption (Fig. 2). The substitution of PP with CFs reduced the amount of polymer coating around the poplar wood fibers; thus, it was expected to increase the water absorption. However, the presence of impermeable CFs filler particles might increase the tortuosity factor in the material, leading to a longer water diffusion pathway throughout the composite (Turku and Karki 2014).

Tensile Properties

The effect of CFs proportion on the tensile properties of the composites with and without MAPP is shown in Figs. 3, 4, and 5. The tensile strength of PP increased with the addition of CFs accompanied by MAPP (Fig. 3). However, without MAPP, CFs did not increase the tensile strength. This result indicates that MAPP increased the fiber-matrix interfacial adhesion.

The tensile modulus of elasticity of PP also increased with the addition of CFs (Fig. 4). The gain was observed with and without the coupling agent, but it was more important with the presence of the coupling agent. Similarly, the tensile strength and modulus of elasticity of the CFRP increased with increasing CFs proportion (Figs. 3 and 4). The improvement of the tensile properties with increasing proportion of CFs was expected because of the high mechanical properties of the CFs compared to those of PP. This reinforcement was only possible in the presence of the coupling agent because it increased the fiber-matrix adhesion and thus fiber-matrix stress-transfer. This may be attributed to the reaction between MAPP and the sizing agent on the surface of carbon fiber. As reported, sizing agents are chemical coatings applied on the surface of CFs to increase fiber-matrix interfacial adhesion (Nakamura *et al.* 2010).

Without the use of MAPP, very little or no increase of WPC tensile strength was observed with increasing wood content (Figs. 3 and 4). This result was due to the absence of chemical bonding between the polar wood and the non-polar PP matrix. Also, the interfacial area increases as wood content increases, resulting in a decreased tensile strength (Bengtsson *et al.* 2007).

However, with the use of MAPP, the tensile strength of the composites increased with increasing wood content because of the formation of ester bonds between the carbonyl groups of the MAPP and the hydroxyl groups of the wood fiber (Ndiaye and Tijani 2012; Zhou *et al.* 2013). The modulus of elasticity of the WPCs increased with increasing wood content, with or without MAPP (Fig. 4). However, the gain was higher in the presence of a coupling agent.

The hybrid composites showed an increase in the tensile strength and the modulus of elasticity with increasing CFs proportion (Figs. 3 and 4). The modulus of elasticity and the tensile strength of hybrid composites were higher than those of the WPCs with the same wood content.

The positive effect of hybridization also increased with increasing CFs content. This result suggests that MAPP is effective at bonding both fibers to PP. The hybrid composite POP40/CF9/PP48/MAPP3 is one interesting case because it had a very similar tensile strength to that of CFRP-PP88/CF9/MAPP3. Thus, using up to 40% of bio-sourced material did not reduce the tensile strength.

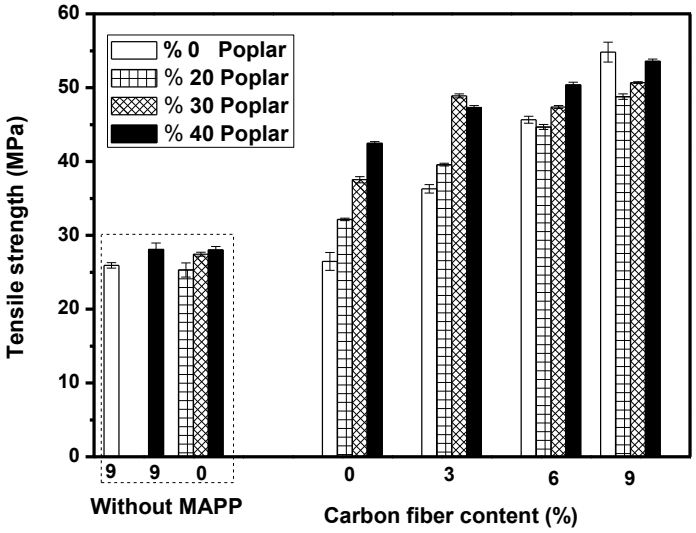

Fig. 3. Effect of carbon fiber content on tensile strength of PP and composites

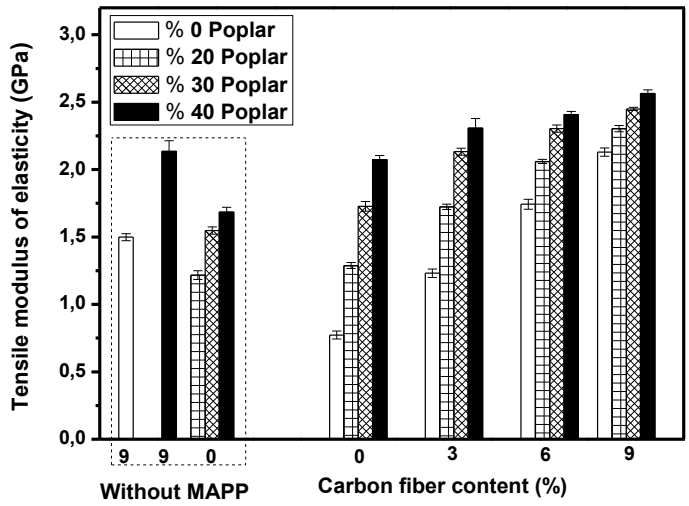

Fig. 4. Effect of carbon fiber content on tensile modulus of elasticity of PP and composites

Figure 5 shows the elongation at break of PP, CFRP, WPCs, and hybrid composites. The neat PP had much higher elongation at break than all composites (the elongation at break of neat PP is not shown in Fig. 5 because it is out of scale, 534%). It can be seen that the addition of both fibers, individually and as a hybrid, resulted in a substantial decrease in elongation at break. The addition of poplar fibers to PP resulted in deep modification of the mechanical behavior, from highly ductile to almost brittle. For example, the elongation at break of PP was 534% and decreased to 4.3% with 40% wood content.

The elongation at break of PP was reduced with the addition of CFs. The elongation of the CFRP with 9% CFs was 5.4% in the presence of MAPP. However, the elongation at break for the same formulation without MAPP was 370% (not shown in Fig. 5). This result is explained by the good adhesion between the stiff CFs and the matrix in the presence of

MAPP, resulting in stiffer composites. Accordingly, the plasticity of the system was reduced. For the same reason, the elongation at break decreased as the poplar content increased. The elongation at break of the CFRP decreased with increasing CFs proportion for the 20% poplar level only (Fig. 5). With higher poplar proportions, CFs proportion had little impact on elongation. The embrittlement effect of CFs on elongation is due to the limited elongation of CFs. This result is in contradiction with reports from Hariharan and AbdulKhalil (2005) and Sheshmani *et al.* (2013).

Flexural Properties

The flexural modulus of elasticity (Fig. 6) and strength (Fig. 7) of CFRP increased with increasing CFs content. This variation was attributed to the favorable interfacial properties of the CFs combined with the effect of the coupling agent (MAPP), as discussed for the tensile properties.

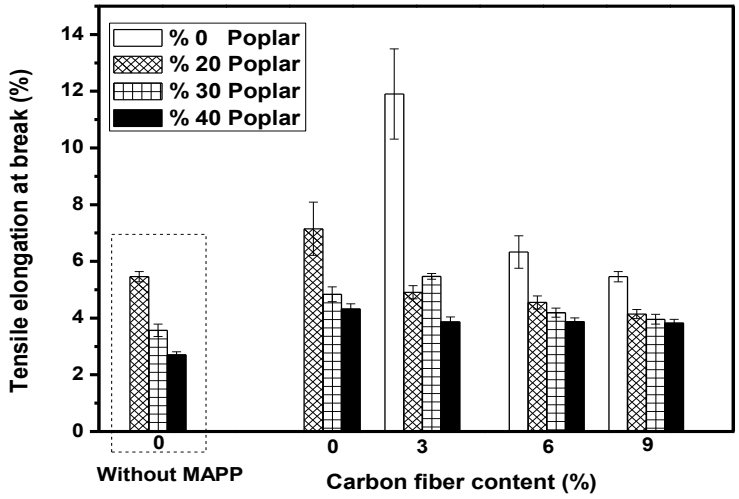

Fig. 5. Effect of carbon fiber content on tensile elongation at break of PP and composites

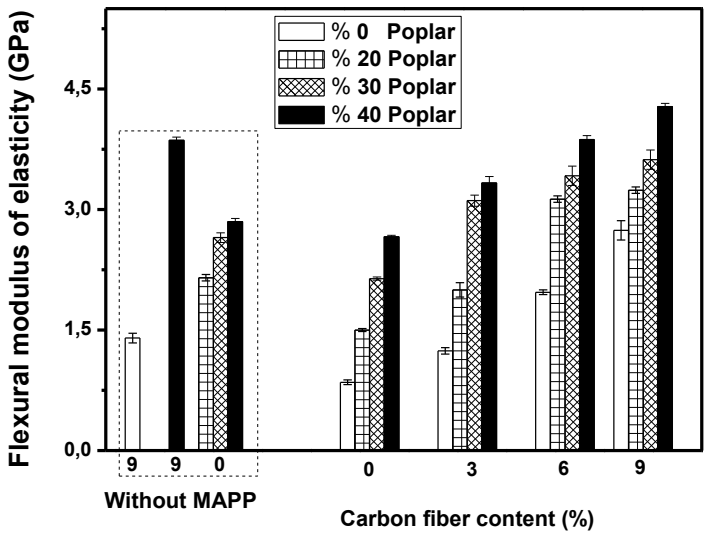

Fig. 6. Effect of carbon fiber content on flexural modulus of elasticity of PP and composites

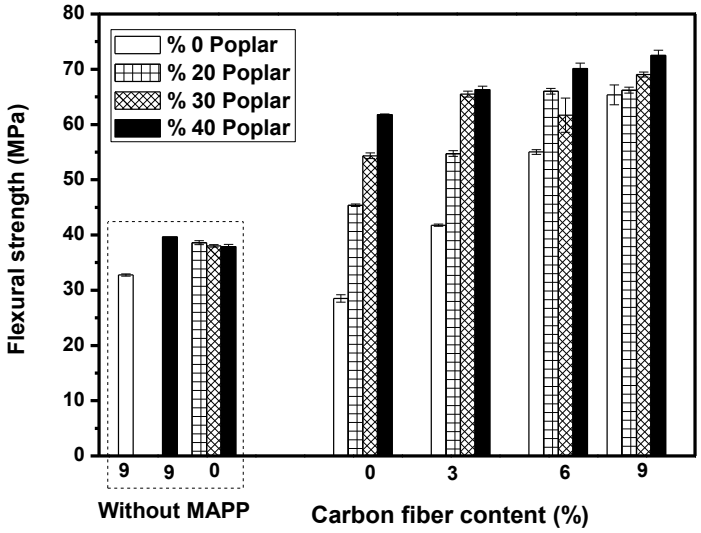

Fig. 7. Effect of carbon fiber content on flexural strength of PP and composites

In the absence of MAPP, the reinforcement of CFs was much lower. For example, the flexural strength of CFRP-PP91/CF9 was 32.8 MPa, compared to 65.4 MPa for the CFRP-PP88/CF9/MAPP3.

Figure 7 shows the variation of the flexural strength and modulus of elasticity of WPCs with and without coupling agent. The modulus of elasticity increased with increasing poplar fiber content with and without MAPP. This increase is attributable to the stiffness and uniform dispersion of the wood fibers. The WPCs with coupling agent showed a higher flexural strength than those without coupling agent because of the bonding effect of MAPP.

The variations in the flexural modulus of elasticity (Fig. 6) and strength (Fig. 7) of the hybrid composites increased with increasing CFs content. This increase could also be explained by the desirable properties of CFs and the effect of MAPP, as discussed for the tensile properties. An increase of up to 42% was observed with the addition of only 9% CFs with 20% wood content. The highest flexural strength (72.5 MPa) was obtained with the hybrid composite POP40/CF9/MAPP3, an improvement of 17% compared to the WPC-POP40/PP57/MAPP3 and 10% compared to the CFRP-PP88/CF9/MAPP3 (Fig. 7). In the absence of MAPP, the flexural strength of the hybrid composite dropped to 39.6 MPa. Therefore, the coupling agent improved the strength by 83%. Overall, the highest flexural strength and stiffness were reached with hybrid reinforcement.

Impact Strength

The effect of fiber content on composite impact strength is shown in Fig. 8. The impact energy of the neat PP is by far the highest, as the sample did not break; its energy was higher than the pendulum capacity (Fig. 8). Thus, the addition of fibers, individually or mixed, negatively impacted the toughness of PP. Without MAPP, the CFRP had the next highest impact strength. The impact strength of the CFRP-PP88/CF9/MAPP3 was lower than that of the CFRP-PP91/CF9. This is ascribed to a good fiber-matrix adhesion, which does not allow slippage at the fiber-matrix interface, thus decreasing polymer chain mobility. It therefore increased the rigidity and decreased the ductility of the composites (Enoki *et al.* 2014).

The impact strength of WPCs decreased as wood fiber proportion increased. Increasing the proportion of wood particles led to an increase in the stress concentration areas because of poor bonding between the polymer and wood (Ndiaye and Tijani 2012).

The effect of CFs proportion on the impact strength of WPCs is shown in Fig. 8. The impact strength of hybrid composites was similar to that of WPCs, increasing slightly as CFs content increased. Within hybrid composites, optimal impact strength was obtained at 6% CFs. This result suggests a combined synergistic effect of the two fibers, or that CFs helped the dispersion of poplar fibers in the PP matrix.

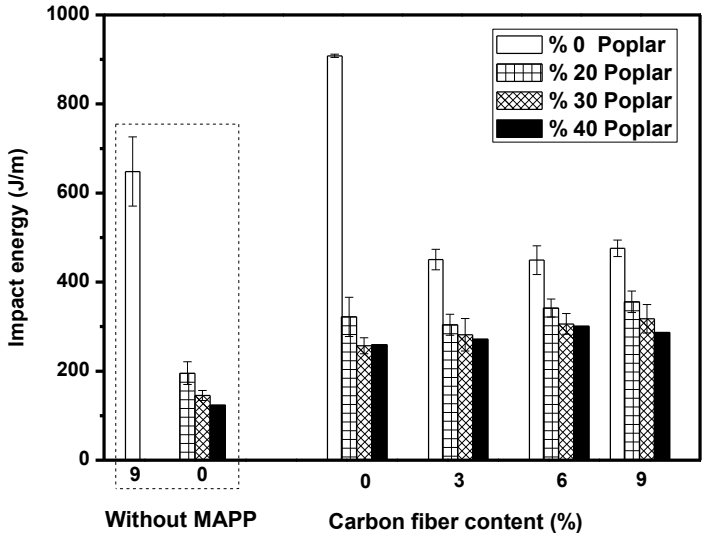

Fig. 8. Effect of carbon fiber content on impact energy of PP and composites

Scanning Electron Microscopy Analysis

SEM was carried out to evaluate the state of dispersion and adhesion of poplar and CFs into the PP matrix. Micrographs of the fractured surfaces of the tensile specimens are presented in Fig. 9. Images of CFRP fractured surfaces showed uncoated CFs in the absence of a coupling agent (Fig. 9a). In addition, there were many cavities surrounding the fiber, detached fibers, and circular holes from fiber withdrawal. With MAPP, however, there were few detached CFs and evidence of the polymer on CFs surfaces (CFs are coated with the polymer) (Fig. 9b). These observations support the results obtained from the mechanical tests.

In the case of the uncoupled WPC-POP40/PP60 (Fig. 9c), the fiber recovery in the matrix was low, indicating a weak bonding. The matrix/fiber affinity was low, and the primary mechanism of rupture was fiber pull-out. However, in the presence of MAPP (POP40/PP57/MAPP3, Fig. 9d), SEM images showed the polymer bonded on the wood fiber surface, suggesting a good fiber-matrix adhesion. Once again, these observations support the results obtained from the mechanical tests.

Figures 9e and 9f show the ruptured surfaces of two of the hybrid composites, POP40/CF9/PP51 and POP40/CF9/PP48/MAPP3, respectively. In the absence of MAPP, there were several detached fibers and voids. Interestingly, in the presence of MAPP, CFs were bonded to poplar fibers. This result shows that MAPP adhered to both fibers and explained the increase in mechanical properties of the hybrid composites with increasing CFs content.

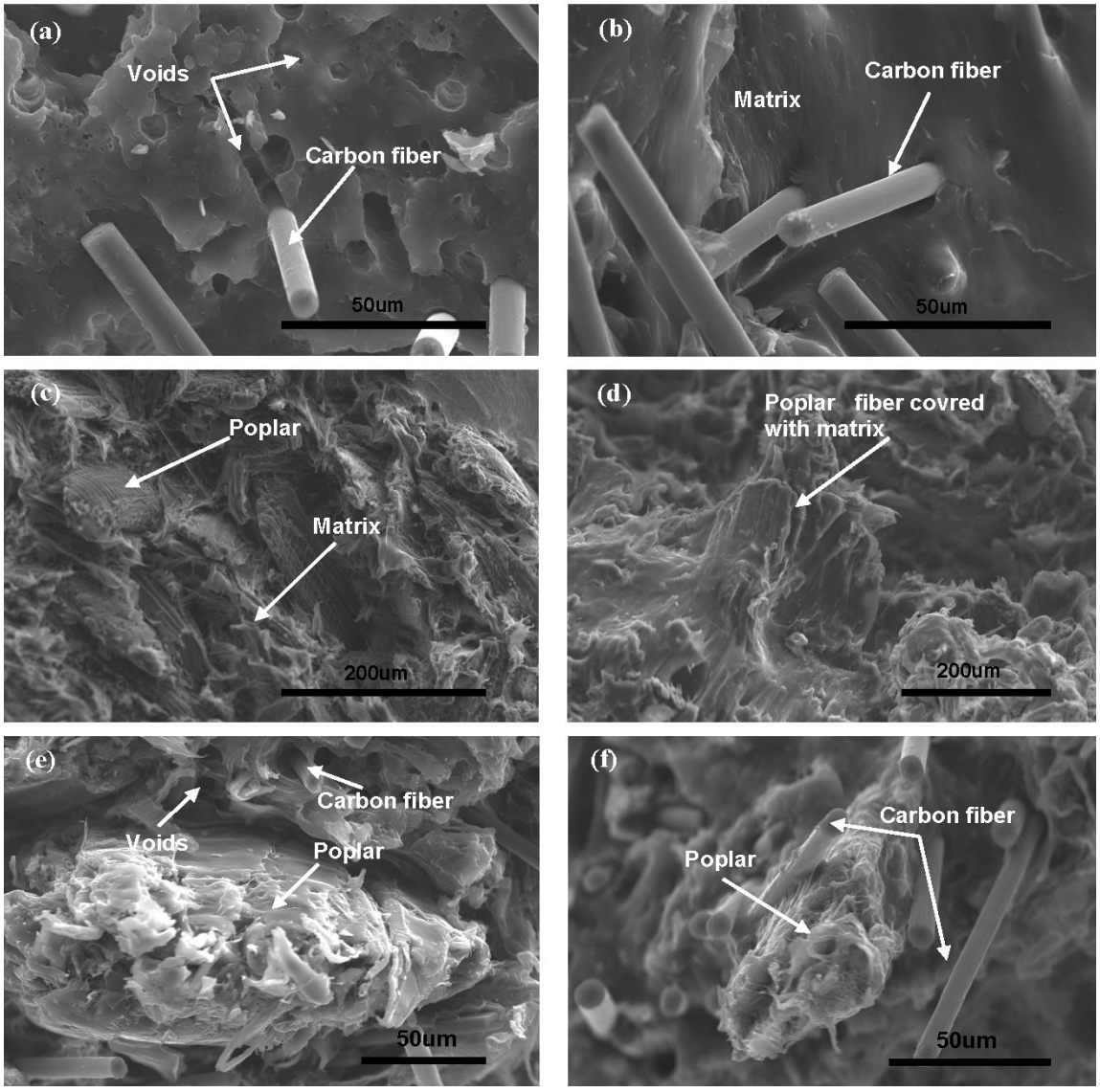

Fig. 9. SEM micrographs of tensile fractured surfaces of composites: (a) PP91/CF9, (b) PP88/CF9/MAPP3, (c) POP40/PP60, (d) POP40/PP57/MAPP3, (e) POP40/PP51/CF9, and (f) POP40/PP48/CF9/MAPP3. Note the scale indication (black) at the lower right of each image.

CONCLUSIONS

The main goal of this work was to study the effect of carbon fiber (CFs) reinforcement in wood-plastic composites (WPCs). Three types of composites were prepared by injection molding: carbon fiber-reinforced PP (CFRP), WPCs, and hybrid CF-wood-plastic composites. From the results obtained, the following conclusions can be drawn:

1. The addition of CFs to WPCs resulted in substantial improvement in the tensile strength, tensile modulus of elasticity, flexural strength, and flexural modulus of elasticity. In contrast, elongation at break was reduced as CFs content increased. Few variations were observed for impact energy.

2. The composite formulations without the use of the compatibility agent (MAPP) showed a lower stiffness, lower strength, and higher water absorption than the coupled ones. The greatest difference was observed for strength.
3. The flexural strength and modulus of elasticity of hybrid composites were higher than those of the CFRP.
4. The apparent density of WPCs increased with the addition of CFs.
5. Hybridization of the reinforcement (mix of wood and CFs) had a small effect on water uptake.
6. SEM micrographs confirmed the adhesion of both CFs and wood fibers with the PP matrix in the presence of a coupling agent.

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