

Impact Strength Improvement of Wood Flour–Recycled Polypropylene Composites

Saman Ghahri,¹ Saeed Kazemi Najafi,¹ Behbood Mohebbi,¹ Mehdi Tajvidi²

¹Department of Wood and Paper Science and Technology, Faculty of Natural Resources and Marine Sciences, Tarbiat Modares University, Iran

²Department of Wood and Paper Science and Technology, Faculty of Natural Resources, University of Tehran, Iran

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ABSTRACT: In this research, the improvement of the impact strength of wood flour–recycled polypropylene (PP) composites through impact modification was studied. For this purpose, a virgin polypropylene (VPP) was thermomechanically degraded by five extrusions under controlled conditions in a twin-screw extruder at a rotor speed of 100 rpm and a temperature of 190°C. PP (VPP and recycled PP at the second and fifth stages) and wood flour were compounded at 50 wt % wood flour loading in a counterrotating twin-screw extruder in the presence of different contents of ethylene vinyl acetate (EVA) to produce

the wood flour–PP composites. From the results, the composites containing recycled PP exhibited significantly lower impact strengths. The addition of EVA up to 9 wt % increased the impact strengths of the composites made with PP recycled two and five times by about 63 and 41%, respectively. The composites containing VPP exhibited higher impact strengths than those containing recycled PP and EVA. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 1074–1080, 2012

Key words: extrusion; mechanical properties; recycling

INTRODUCTION

The utilization of recycled plastics for manufacturing wood–plastic composites (WPCs) has been investigated by a number of researchers.^{1–3} As for virgin plastics, any recycled plastic that melts and can be processed below the degradation temperature of wood (lingocellulosic fillers, 200°C) is usually suitable for producing WPCs.³

The results of previous researches show that the recycling of plastic materials changes several properties, such as the melt viscosity, molecular weight, crystallinity, and melting point.^{4–10} These changes affect the mechanical properties of plastics and the resultant composites.

Different results have been reported for the flexural and tensile properties of composites made from recycled plastics in comparison to those made from virgin plastics. Some of these researches indicated similar properties for composites made from virgin and recycled plastics,^{3,11,12} some reported superior properties^{2,13,14} and others showed lower properties.^{1,2} In contrast to the flexural and tensile properties, the results of all researches have shown lower impact strengths in composites made from recycled

plastics and lingocellulosic materials in comparison to composites made from virgin plastics.^{1,2,14} The lower impact strength of the recycled plastic is responsible for the lower impact strength of the WPCs containing them. Plastic degradation frequently occurs when a polymer is submitted to a process or service. Degradation usually decreases the impact strength of plastics. The decrease in the impact strength depends on type and level of degradation. Kazemi et al.¹⁴ showed that the thermomechanical degradation during two extrusions greatly decreased the impact strength of polypropylene (PP) and the resulting WPCs.

To improve impact strength of WPCs made from recycled plastics, several methods, such as the use of blending recycled plastic with virgin plastic,¹¹ adding nonlignocellulosic fibers (e.g., glass fibers),^{3,14} and preparing hybrid WPCs,¹⁵ have also been considered. However, it is not possible to increase the impact strength of WPCs containing recycled plastic as much as the impact strength of WPCs made from virgin plastics with such methods, especially if the plastics have undergone a multitude of degradation processes.

One of the methods used to improve the impact strength of plastics and resultant composites, such as WPCs, is the use of impact modifiers (IMs) and the addition of elastomeric materials to the plastic. Many elastomeric materials have been used as IMs for PP and filled/reinforced PP systems, such as WPCs.^{16–21} These materials are ethylene–propylene copolymers or ethylene–propylene–diene terpolymer, styrene–

Correspondence to: S. Kazemi Najafi (skazemi@modares.ac.ir)

butadiene styrene rubber, styrene-ethylene-butylene-styrene (SEBS), maleic anhydride grafted SEBS, acrylonitrile-butadiene rubber, ethylene vinyl acetate (EVA), chlorinated polyethylene, polyisobutylene, ethylene-propylene rubber, styrene-butadiene-rubber, maleic anhydride grafted ethylene-propylene-diene terpolymer, and maleic anhydride grafted SEBS. Although, the IMs have a positive effect on increasing the impact strength, their negative effects on other mechanical properties must also be considered.

Because of the lower impact strength of WPCs made from recycled plastics (especially those having undergone multiple reprocessing), the aim of this study was to investigate the improvement in the impact strength of wood flour-recycled PP composites with EVA as an effective IM. Although EVA has been successfully used to improve impact properties in virgin plastic composites, no information on its effectiveness for recycled plastics is currently available.

EXPERIMENTAL

Materials

The homopolymer PP was grade SI080 and was obtained from Polynar Co. (Tabriz, Iran). The density and melt flow index (MFI; temperature = 230°C, load = 2.16 kg) of PP were 0.91 g/cm³ and 5.8 g/10 min, respectively. Maleated polypropylene (MAPP) produced by Kimia Javid Factory (Isfahan, Iran), with an MFI of 100 g/10 min, and 1.1% coupled maleic anhydride was used as a compatibilizer. EVA copolymer (as an IM) with 18% vinyl acetate content was supplied by LG, Lotte Daesan Petrochemical Co. Ltd (Daesan South Korea). Wood flour was obtained by the screening of industrial sawdust of Iranian beech (*Fagus orientalis*) collected from local mills to +60/-40 mesh particle size. The wood flour was dried in an oven for 24 h at 80°C.

Preparation and properties of recycled PPs

The virgin polypropylene (VPP) supplied by the company was thermomechanically degraded under controlled conditions in a twin-screw extruder at a screw speed of 100 rpm and a temperature of 190°C. The VPP was designated VPP, and R2PP and R5PP represent PP extruded two and five times, respectively.

The MFI of VPP and the degraded PPs were determined with MFI testing Santam Machine (Tehran, Iran) according to ASTM D 1238-98.²² The tests were performed at 190°C under a load of 2.16 kg, and the results are reported in terms of grams per 10 min.

Differential scanning calorimetry (DSC) was carried out with a Rigaku Thermoplus DSC-8230 analyzer (Tokyo, Japan). The tests were performed from room temperature to 200°C at a heating rate of 20°C/min in a nitrogen atmosphere flowing at 100 mL/min. Around 10 mg of the materials were heated in an aluminum sample pan. An empty pan was used as the reference. The fractional crystallinity (X_c) of the samples was calculated according to the following equation:²³

$$X_c(\%) = \frac{\Delta H_f}{\Delta H_f^0 w} \times 100 \quad (1)$$

where ΔH_f is the heat of fusion of the VPP and recycled PPs and composites, ΔH_f^0 is the heat of fusion of 100% crystalline PP (209 J/g),^{24,25} and w is the mass fraction of PP in the composites.

Thermogravimetry analysis (TGA) was carried out with a Rigaku Thermo plus TG 8129 TGA-differential thermal analysis (DTA) analyzer. The tests were performed from room temperature to 500°C at a heating rate of 20°C/min in an air atmosphere, which flowed at 100 mL/min.

Thin films of VPP and recycled PP were prepared, and Fourier transform infrared (FTIR) spectra were obtained with a Nicolet MAGNA-IR 860 spectrometer at a spectral resolution of 4 cm⁻¹ over the spectral range 4000-400 cm⁻¹.

Composite manufacturing and properties

Oven-dried wood flour with a moisture content of less than 3%, PP (VPP or degraded), the coupling agent (MAPP), and IM (EVA) were weighed for each formulation according to Table I. The composites were manufactured according to the method described by Kazemi Najafi et al.¹⁴ As controls, VPP and recycled PP samples were also prepared. All specimens were conditioned at 23 ± 2°C and 65 ± 3% relative humidity for at least 2 weeks before the tests were performed.

The flexural modulus and strength of the wood flour-recycled PP composites were determined according to ASTM D 7031-04 specifications²⁶ with a computer-controlled DARTEC machine. The speed of the crosshead was set at 5 mm/min. Unnotched impact tests were carried out according to ASTM D 256-90 specifications²⁷ with an Izod testing machine (Santam). At least five replicates of each formulation were tested for each test.

RESULTS AND DISCUSSION

The MFI of the VPP was 5.8 g/10 min and with two extrusions and five extrusions of VPP, the MFI

TABLE I
Composition of Evaluated Formulations (wt %)

Formulation	Wood flour	MAPP	EVA	VPP	R2PP	R5PP
WVPP	50	2	0	48	0	0
WR2PPE0	50	2	0	0	48	0
WR2PPE3	50	2	3	0	45	0
WR2PPE6	50	2	6	0	42	0
WR2PPE9	50	2	9	0	39	0
WR5PPE0	50	2	0	0	0	48
WR5PPE3	50	2	3	0	0	45
WR5PPE6	50	2	6	0	0	42
WR5PPE9	50	2	9	0	0	39

W, wood flour; V, virgin; PP, polypropylene; R2, two-time recycled; R5, five-time recycled; M, MAPP; E, EVA.

increased around three and five times for R2PP (15.5 g/10 min) and R5PP (31.4 g/10 min), respectively. This indicates that multiple-extrusion caused a significant change in the MFI values. An increase in MFI during the extrusion process has been previously reported for PP by several authors.^{4,5,8,28} It suggests a decrease in the molecular weight of PP and, correspondingly, its melt viscosity.^{6,29,30} This behavior was expected as a result of polymer chain degradation because of severe thermal and stress cycles exerted during the extrusion process, which led to a molecular weight reduction.^{6,31}

Figure 1 indicates the DSC thermograms of PPs. The effect of multiple extrusion processes on the thermal properties of PP is also shown in Table II. The heat of fusion of VPP was 81.2 J/g, whereas for R2PP and R5PP, it was 61.7 and 65.6 J/g, respectively. The results indicate a decrease in the fractional crystallinity of (two times extruded PP) R2PP and (five times extruded PP) R5PP. From the DSC results, Baquero et al.³² also reported a decrease in the fractional crystallinity and an increase in MFI of recycled PP. These results show a change in the structure of recycled PP.

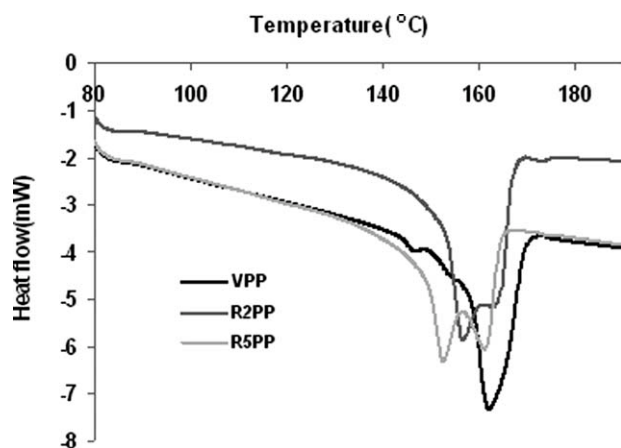


Figure 1 DSC heating curves of VPP and recycled PPs.

TABLE II
Properties of the VPP and Extruded PPs

PP	MFI (g/10 min)	Melting temperature (°C)	Heat of fusion (J/g)	Fractional crystallinity (%)
VPP	5.8	162.0	81.3	38.9
R2PP	15.5	156.8, 162.9	61.8	29.6
R5PP	31.4	152.7, 161.7	61.8	31.4

V, virgin; R2, two-time recycled; R5, five-time recycled.

In general, because of the presence of hydrogen linked to a tertiary carbon in the backbone chain, PP degraded preferentially by chain scission, which reduced the molecular weight.⁶ With the continuation of the degradation, the amount of short chains increased; this led to an increase in the chain ends, which acted as imperfections to increase the crystallinity in recycled PP. A decrease in crystallinity was also reported for high-density polyethylene by Baquero et al.³² and Shojaei et al.²⁸ with DSC study.

The DSC curves (Fig. 1) also showed that VPP had a melting temperature (T_m) of about 162°C, whereas R1PP and R2PP had two main melting peaks. The melting temperature corresponding to the first peak was found to decrease with the extrusion rounds as the melting temperature of VPP was higher than the melting temperature corresponding to first peak of R1PP and R2PP, respectively. However, the changes in the melting temperature corresponding to second peak were small. Multiple melting peaks have been reported for several types of PP,^{33–35} and some reasons, such as secondary crystallization, a difference in the degree of crystalline perfection, a different lattice structures, partial melting followed by recrystallization, a different extent of stereoirregularity, and inhomogeneities in molecular weight, have been suggested as possible causes of multiple melting peaks.

The thermal stability of PP is a very important parameter for the processing and use of this material in WPC manufacturing. Figures 2 and 3 illustrate the mass loss (TGA curve) and its derivative as a function of temperature (Differential thermal gravimetry (DTG) curve) of VPP, R2PP, and R5PP. It was noticed that R5PP exhibited a slightly higher thermal stability than VPP and R2PP.

The FTIR spectra of VPP, R2PP, and R5PP in the range 4000–400 cm^{-1} are presented in Figure 4. The carbonyl group peak, which might have formed due to oxidation during extrusion, should have been seen around 1900–1600 cm^{-1} .³⁶ Negligible carbonyl peaks were observed for R2PP and R5PP at 1725 cm^{-1} . Some researchers have reported no oxidative reactions during the extrusion process of PP.^{5,37} Therefore, it could be concluded that the multiextrusion process only reduced the molecular weight due

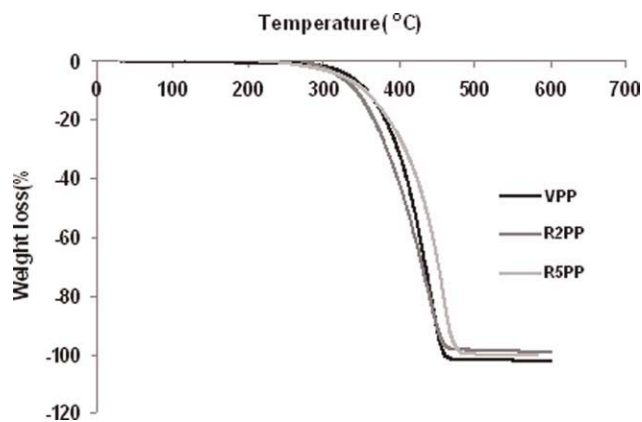


Figure 2 TGA curves of VPP and recycled PPs.

to chain scission. The slight oxidation of PP during thermomechanical degradation can be a positive event to produce WPC because of the polarization of PP, which may improve the compatibility between PP and wood.¹⁴

Table III shows that the thermomechanical degradation significantly decreased the impact strength of PP. The impact strengths of R2PP and R5PP obtained were 3683 and 2557.5 J/m², respectively, whereas the VPP samples did not break. Duncan's multiple-range test results showed that the impact strength of R5PP was significantly lower than that of R2PP. A decrease in the molecular weight of degraded PP led to a decrease in the impact strength.³⁸

Also, the flexural properties of the VPP and extruded PPs are summarized in Table III. As can be clearly seen, the flexural properties of PP decreased with the increase in the number of extrusion cycles. From Duncan's multiple-range test results, the flexural modulus and strength of VPP and recycled PPs were found to be significantly different. It can be seen that VPP and R5PP exhibited the highest and lowest flexural modulus and strength, respectively.

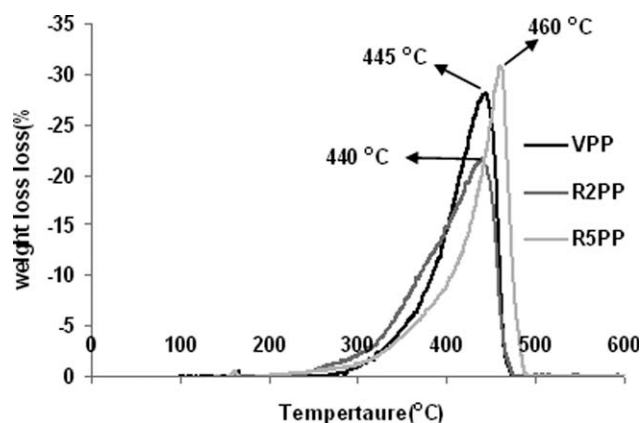


Figure 3 Differential thermal gravimetry (DTG) curves of VPP and recycled PPs.

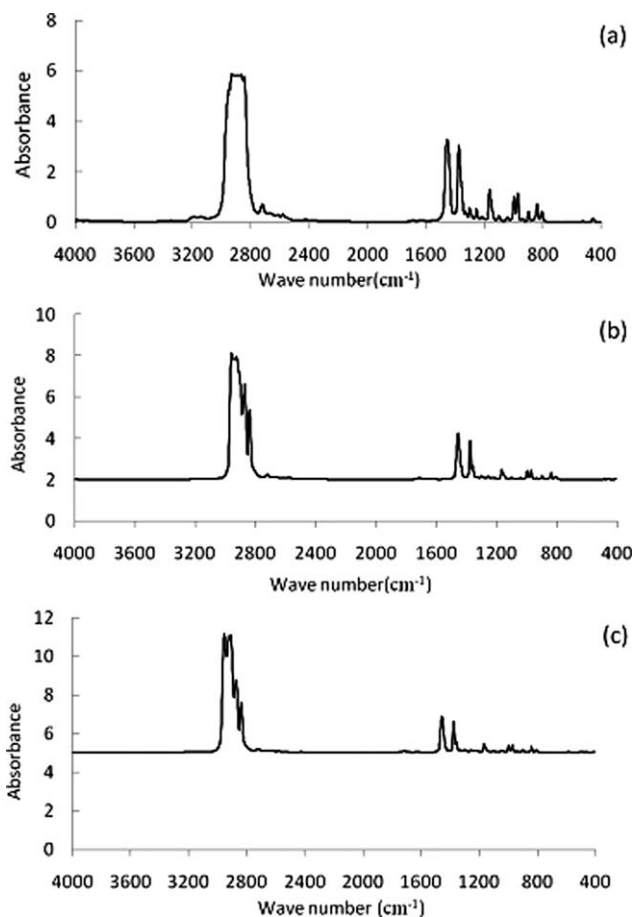


Figure 4 Transmission FTIR spectra of VPP and recycled PPs: (a) VPP, (b) R2PP, and (c) R5PP.

Figure 5 illustrates the flexural modulus of the wood flour-PP composites. It can be seen that the flexural modulus of the wood flour-PP composites made from R2PP was significantly higher than the composites containing VPP. The higher flexural modulus of composites containing R2PP could be attributed to the lower melt viscosity of R2PP and, therefore, the improvement of wood flour encapsulation by the resin.^{14,39} Increasing the recycling times up to five times caused a considerable increase in the MFI values of R5PP. The higher MFI might have adversely affected the system by interfering with the mixing process. Hence, the flexural modulus of WPCs containing R5PP drastically decreased and

TABLE III
Mechanical Properties of the VPP and Extruded PPs

Mechanical property	VPP	R2PP	R5PP
Flexural modulus (MPa)	1999 ± 225 ^a	1112 ± 52 ^b	1071 ± 35 ^b
Flexural strength (MPa)	30.6 ± 0.5 ^a	13.7 ± 0.5 ^b	10.7 ± 0.6 ^c
Impact strength (J/m ²)	Not broken	3683 ± 99 ^a	2558 ± 144 ^b

Mean ± Standard error values in the same row followed by the same letter(s) are not significantly different ($p = 0.05$) on the basis of Duncan's multiple-range test.

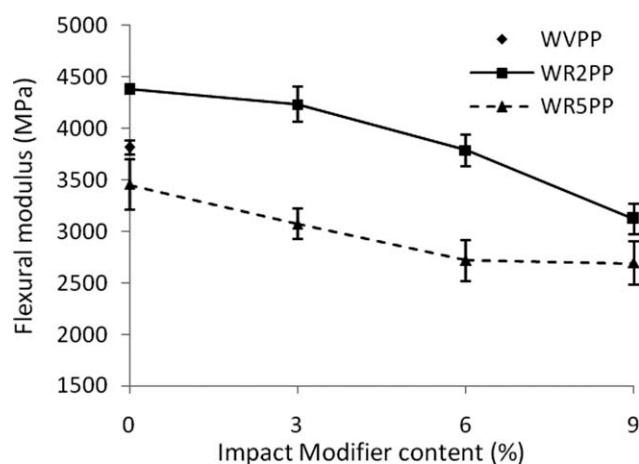


Figure 5 Flexural modulus of the wood-PP composites.

reached values less than the flexure modulus of the composites containing VPP.

The effect of IM content on the flexural modulus of the composites is shown in Figure 5. The flexural modulus decreased as the IM content increased. The flexural moduli of the composites containing R2PP and R5PP modified with 9 wt % EVA were about 29 and 22% lower, respectively, than composites without modifier. In general, IMs reduce the flexural modulus of composites.⁴⁰ Oksman and Clemons¹⁶ also reported that the IMs improved the PP impact strength and elongation at break but decreased the elastic modulus of the composites. This phenomenon was caused by structural characteristics of IM, that is, low modulus of elasticity and weak adhesion between EVA and the PP matrix.⁴¹ Dikobe and Luyt⁴² also confirmed the immiscibility of EVA and PP due to a lack of interaction between PP and EVA.

Figure 6 illustrates the flexural strength of the wood flour-PP composites. The results show that with increasing number of extrusion cycles of PP, the flexural strengths of the composites containing R2PP and R5PP significantly decreased by about 15 and 39%, respectively. Kazemi Najafi et al.³ also reported a lower flexural strength of WPCs containing recycled plastic. A similar result, indicating a reduction of the flexural strength of WPCs, has also been reported.^{2,43} Figure 6 also shows the effect of IM content on the flexural strength of the composites. In general, the flexural strength decreased as the IM content increased. A significant decrease in the flexural strength of composites could be observed with 9 wt % EVA. The flexural strengths of the composites containing R2PP and R5PP modified by a high percentage of EVA (9 wt %) were about 45 and 34% lower, respectively, than the flexural strength of composites without EVA. The reason for the decrease in the flexural strength of the composites was related to the structure of the IM, such as the low flexural strength.^{41,44}

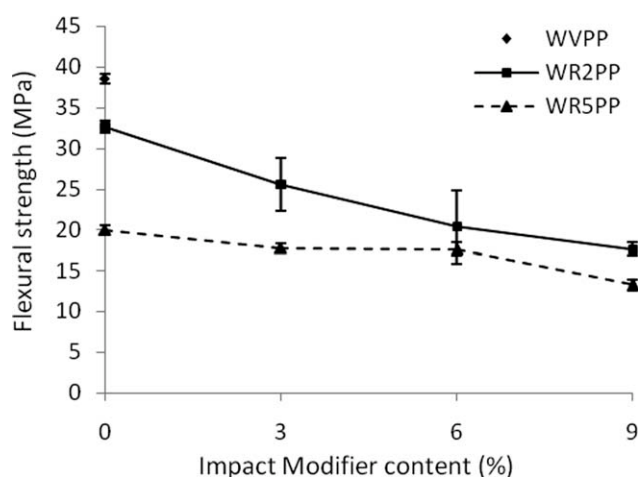


Figure 6 Flexural strength of the wood-PP composites.

The impact strength of the wood flour-PP composites is shown in Figure 7. The impact strength of the composites containing R2PP and R5PP was significantly lower than composites containing VPP. With the increase in the number of extrusion cycles, the molecular weight of recycled PP decreased; this could have led to a decreased stress transfer via covalent linkages and an increased crack initiation and propagation in the polymer matrix (recycled PP).¹⁴ Figure 7 also illustrates the effect of IM content on the impact strength of wood flour-PP composites. With the addition of IM, the impact strength significantly increased. The impact strength of the composites containing R2PP and R5PP modified with 9 wt % EVA were about 63 and 41% more, respectively, than composites without EVA.

Cracks usually originate from points where interfacial bonding between the polymer and filler phases is too weak.¹⁴ IM particles disperse in the plastic matrix and absorb created energy by deformation stress with microcrack formation. Under impact loading, microcracks form near the interfacial surfaces between IM and PP. IMs absorb impact energy and control crack propagation by the formation of many

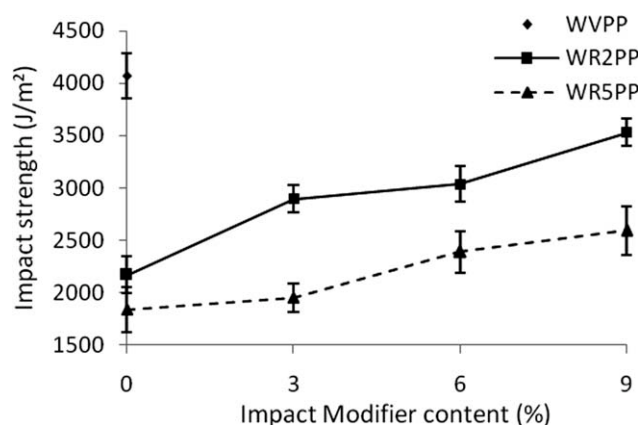


Figure 7 Impact strength of the wood-PP composites.

energy-absorbent microcracks and by the termination of crack propagation before irrecoverable defect levels are reached.⁴⁵

Moreover, EVA is a thermoplastic elastomer and is both nonpolar and polar in structure. It is then believed that EVA can further improve the compatibility between the fibers and matrix. EVA is a copolymer of ethylene, which permits the adhesion to the PP matrix, and vinyl acetate, which could bond, because of its acetate groups, to the hydroxyl groups on the fibers.⁴⁶ EVA, with an affinity for the particles, partially encapsulated the wood flour. This phenomenon caused good stress transfer (similar to MAPP) and could increase the impact strength in modified WPCs.

It can be concluded from Figure 7 that although the addition of EVA increased the impact strength of composites containing recycled PP, the impact strength of these composites were still significantly lower than the impact strength of composites containing VPP (not modified with IM). However, the addition of higher contents (>9%) of IM may have increased the impact strength of the modified wood flour-recycled PP composites, but its negative effects on other mechanical properties should be considered. Therefore, it is necessary to use other ways, such as the use of more effective IMs and/or the addition of VPP to wood flour-recycled plastic composites.

CONCLUSIONS

The improvement of the impact strength of composites made of wood flour and recycled PP with IMs was studied in this research. The following conclusions could be drawn from the results and discussions presented previously:

- Multiple extrusion of VPP decreased the heat of fusion, melting point, and crystallinity and increased MFI.
- The flexural modulus, flexural strength, and impact strength of recycled PP decreased significantly with multiple extrusion of VPP.
- Composites containing degraded PP exhibited significantly lower impact strengths than composites containing VPP.
- The impact strength of the composites containing recycled PP was improved with EVA.
- With increasing EVA content up to 9%, the impact strength of the composites containing recycled PP increased but was still lower than the impact strength of composites containing VPP.
- EVA decreased the bending properties (flexural strength and modulus) of wood flour-recycled PP composites.

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