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# Morphology, dimensional stability and mechanical properties of polypropylene–wood flour composites with and without nanoclay

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**Abstract**

Several composites based on recycled—virgin polypropylene, wood flour and organically modified montmorillonite (commonly called 'nanoclay') were prepared by melt compounding. This paper aims to evaluate the potential for the use of recycled polypropylene and underutilized wood flour as material for the development of wood–plastic composites, as well as reinforcement effect of organically modified montmorillonite on them. In order to improve the poor interfacial interaction between the hydrophilic wood flour and hydrophobic polypropylene matrix, as well as polypropylene–organically modified montmorillonite, maleic anhydride-grafted polypropylene was used as a compatibilizer. Some mechanical and physical properties were evaluated. Findings of this work show that both recycled and virgin polypropylene can be used in manufacture of wood–plastic composites and there is no significant difference in the properties of resulting nanocomposites. It was found that mechanical properties of polypropylene containing 40 wt% wood flour reinforcement remain essentially unchanged when the virgin polypropylene in the matrix is replaced be recycled polypropylene. Morphologies of the nanocomposites were analyzed by scanning electron microscopy and X-ray diffraction, and the results showed increased d-spacing of clay layers indicating enhanced compatibility between polypropylene and clay and wood flour. Consequently, polypropylene recycled from postconsumer applications can be used in high-value nanocomposites without going the expense of separating out impurities from the polymer.

**Keywords**

melt compounding, mechanical properties, dimensional stability, scanning electron micrograph, wood–plastic composites

## Introduction

Lately, the consumption of plastic materials has increased enormously due to their various advantages. The worldwide production of plastics is about 245 million tons per annum, resulting in a significant amount of municipal solid waste.[1] Since most plastics take a very long time to decompose in nature, the disposal of polymers after use is an important problem.[2] Recycling and reusing are the processes to reduce the environmental pollution caused by postconsumer plastic materials.[3] Besides, owing to the significant increase in global oil prices, utilization of recycled plastics for manufacturing wood–plastic composites (WPCs) has recently gained considerable attention and is now a great task for researchers and industry. A large body of published literature in the area of virgin fiber-reinforced thermoplastic composites exists.[4–6] Using recycled polypropylene (rPP) to formulate WPCs is ideal from the point of view that adding wood particles have the potential to mask and even overpower the composition-driven as well as molecular weight-driven variations in the properties of the base polymer. It should be noted that recycled material can

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often have an unpleasant odor or color and that these potential problems can be overcome by using a thin layer of virgin polymer, by, say, the process of coextrusion.[7]

Studies had shown that waste wood in the form of wood flour (WF), fibers or pulp is suitable as a filler for polyolefin matrix composites.[8] In the wood industry, a large amount of wood waste is generated at different stages of the wood processing, this waste mainly ends up in landfills (dump sites in a few areas) or burned.[9] The volume of WF waste generated in the wood industry is very high, estimated at around 16% of its total production.[10] The addition of WF waste to plastics renders the resulting composites viable from both the mechanical properties and the environmental points of view.[11]

Using organically modified montmorillonite (OMMT; commonly called 'nanoclay') to reinforce polymer-based composites have raised great attention to academic and industrial sectors, since the addition of small amount of nanoclay could substantially enhance the mechanical properties of pristine polymers.[12] A number of researchers have reported improvement of properties by employing low amount of clay in production composites with different matrices.[13–15] OMMT is a member of the smectite mineral group, naturally occurring layered materials having high aspect ratio and specific surface area. OMMT is composed from aluminium silicate layers which were organized in a parallel fashion to form stacks with a regular van der Waal gap in between them called interlayer spacing or gallery.[16] Clays are in nature organophobic, but they can be changed into organophilic by replacing the $Na^+$ or $Ca^{2+}$ cation originally present in the galleries with one organic cation such as alkylammonium ions via an ion-exchange reaction.[17]

In polymers of low polarity, such as polypropylene (PP), low compatibility between the polymer and the clay is observed during the mixing leading to poor dispersion.[14] The use of maleated polypropylene (MAPP) as a compatibilizer enhances the dimensional stability and mechanical properties of the PP, WF and nanoclay composite via improving adhesion between PP–WF as well as interaction between PP–organoclay.[12,13,17]

The goal of this work was to formulate WPCs containing 40 wt% of WF and nanoclay using virgin/recycled PP and to compare both the structure and properties of the resulting WPCs.

## Materials and sample fabrication

### Materials

Polymers used for making composites were postconsumer rPP and virgin polypropylene (vPP) in the form of pellets. The melt flow index (MFI) of rPP and vPP were 8.5–9.5 g/10 min and 5–7 g/10 min (at 230°C/2.16 kg), respectively, and their density was 0.9 g/cm$^3$. Underutilized WF of poplar was kindly supplied by a local mill and screened to obtain 40–60 mesh particle sizes. Flours were oven-dried at 103 ± 2°C for 24 h to reach target moisture content (1–3%) before compounding.

MAPP was obtained from Kimya Baspar Asia Co. (Iran) in the form of pellets, which had a MFI of 64 g/10 min and 2% (by weight) maleic anhydride (MAH) content.

The clay sample, Cloisite 15A, which is an OMMT, was purchased from Southern Clay Products Inc. The properties of nanoclay are given in Table 1.

### Compounding and injection molding

Formulations of the composites and their mass ratios used for the respective blends are given in Table 2.

Compounding was performed in Haake internal mixer (HIB, sys 90, United States). The mixing was carried out at 175°C with a rotation speed of 60 r/min.

After melt blending, in Haake, the mixed materials were cooled for about an hour at room temperature and ground to prepare the granules using a pilot scale grinder (model WIESER, WGLS 200/200). The granulated samples were then dried for 12 h at 100°C in an oven prior to injection molding.

Finally, the composite samples were produced using an injection molding machine (Imen Machine Company, Iran) at a melting temperature of 180°C, a molding temperature of 40°C, an injection pressure of

**Table 1.** The specification of the nanoclay.

| Trade name | | |
|---|---|---|
| Cloisite 15A[a] | | |
| Typical properties | | |
| Organic modifiers | Modifiers concentrations (meq/100 g clay) | Moisture (%) |
| 2M2HT[b] | 125 | <2% |
| Typical dry particle sizes (microns, by volume) | | |
| 10% less than 2 μm | 50% less than 6 μm | 90% less than 13 μm |
| X-ray results | | |
| $d_{001}$ = 31.5 A° | | |
| Color | | |
| Off white | | |

[a]Cloisite 15A is a natural montmorillonite modified with a quaternary ammonium salt.
[b]Dimethyl, dehydrogenated tallow, quaternary ammonium.

**Table 2.** Wood–plastic composite formulations (percentage by weight).[a]

| Composite sample code | vPP content | rPP content | Wood flour content | MAPP content | Organoclay content |
|---|---|---|---|---|---|
| vPP | 100 | — | — | — | — |
| rPP | — | 100 | — | — | — |
| vPP/W40/M5 | 55 | — | 40 | 5 | — |
| rPP/W40/M5 | — | 55 | 40 | 5 | — |
| vPP/W40/M5/O3 | 52 | — | 40 | 5 | 3 |
| rPP/W40/M5/O3 | — | 52 | 40 | 5 | 3 |
| vPP/W40/M5/O5 | 50 | — | 40 | 5 | 5 |
| rPP/W40/M5/O5 | — | 50 | 40 | 5 | 5 |

[a]vPP, rPP, *W*, *M* and *O* codes relate to virgin polypropylene, recycled polypropylene, wood flour, maleated polypropylene and organoclay, respectively.

10 MPa and cooling time of 20 s. Specimens for flexural, tensile and impact properties testing were made
1 according to ASTM specifications.

## Characterizations

### Flexural testing

The composite flexural strength and modulus were determined using the three-point bending test method following the ASTM D-790 test method. A 63 mm span was used in a 5 kN load cell. The load was placed midway between the supports. Bending test was conducted using an Instron Universal Testing Machine (model 4486) at a speed of 5 mm/min. Flexural properties were determined for five samples of each composition.

### Tensile testing

The composite tensile strength and modulus measurement were performed according to the ASTM D-638 test method. The samples were tested for tensile properties in an Instron Universal Testing Machine (model 4486) at a constant crosshead speed of 5 mm/min. Tensile properties were determined for five samples of each composition.

### Izod impact testing

The notched Izod impact strength tests were performed according to ASTM D-256 at room temperature in a Santam testing machine (model SIT 20D). The values obtained are averaged measurements of five samples.

### Dimensional stability tests

The thickness swelling (TS) and water absorption tests were conducted in accordance with ASTM D-570. Before testing, the weight and dimensions, that is, length, width and thickness, of each specimen were measured. The conditioned specimens were immersed in distilled water for 2 and 24 h at a temperature of 23°C. At the end of the preset time, the specimens were removed from the water; all surfaces were wiped off with a cloth and then weighed. After 2 h immersion, the specimens were replaced in water and weighed again after 24 h. Water absorption was calculated according to the following equation.

$$WA(\%) = (M_2 - M_1)/M_1 \times 100 \quad (1)$$

where, $WA$ is the water absorption in percentage and $M_1$ and $M_2$ are the sample weights before and after immersion (g). The values of the TS in percentage were calculated using the following equation.

$$TS(\%) = (T_2 - T_1)/T_1 \times 100 \quad (2)$$

where, $T_1$ is the initial thickness of the sample and $T_2$ is the thickness of the wetted sample.

### Scanning electron microscopy

Fracture surfaces of the tensile test specimens were investigated using a scanning electron microscope (SEM; Model VEGA-II TESCAN). All specimens were sputter coated with gold prior to examination.

### X-ray diffraction

An X-ray diffractometer was used to measure the basal spacing between silicate layers in the nanocomposites. The X-ray diffraction (XRD) was performed in a Bruker X-ray diffractometer (D8 Advanced, Germany) using Cu-Kα radiation ($\lambda = 1.54$ nm). The samples were scanned in 2θ ranges 2–10° at a rate of 0.3 deg/min. The generator was operated at 40 kV and 30 mA. The interlayer spacing ($d_{001}$) of clay was calculated in accordance with Bragg equation: $2d \sin\theta = \lambda$.

### *Statistical analysis*

Composite formulations (Table 2) were produced according to a factorial design. Factors were kind of
2 polymer (two levels; recycled and virgin PP) and OMMT loading (three levels; 0, 3 and 5%). This experimental design allows an evaluation of the effects of polymer kind and OMMT content on the physical and mechanical properties of WPC. Analysis of variance (ANOVA) and Duncan multiple comparison tests (5% significance level) were conducted using SPSS software.

## Results and discussion

### *Mechanical properties study*

*Flexural properties.* Figure 1 shows the flexural properties of composites made with both kinds of PP resin at different nanoclay loadings. The flexural strength and modulus of composites with matrix type slightly but with nanoclay content significantly different. While the flexural strength and modulus of vPP and rPP vary significantly, the flexural strength and modulus of the composites made from rPP was almost similar to composites made with vPP. The samples containing no WF showed flexural strength of 26.2 MPa and 23.1 MPa for vPP and rPP, respectively. The addition of 40 wt% WF increased the flexural strength of vPP and rPP-based composites to 29.4 MPa and 30.3 MPa, respectively (with 5% MAPP), that is 12.2% and 31% higher compared to the neat polymer (rPP).

Besides, with incorporation of nanoclay up to 3 wt% into the formulations, flexural strength and modulus increased significantly and slightly, respectively. The reinforcement effect of nanoclay for vPP and rPP-based composites was similar. At higher nanoclay content (5 wt%), the values decreased. The increased strength and modulus of the clay-containing samples up to 3 wt% nanoclay content may be attributed to the high aspect ratio, as well as surface area of stiff silicate layers in the polymer matrix that result in a higher extent of interaction with the polymer chains[18] and good interfacial adhesion between the nanoscale clay particles and the PP matrix, so that the mobility of polymer chains is restricted under loading.[19,20] The mechanical properties of the composites are determined by several factors, such as intrinsic strength of individual fibers, fiber aspect ratio, fiber–matrix interfacial adhesion and also the fiber orientation in the composites.[18,21] Nevertheless, with further increase in nanoclay loading from 3 to 5 wt%, a decrease in the strength and modulus of the clay-containing samples was observed. This indicates that the nanoclay was dispersed more uniformly through the polymer matrix at low concentrations (3 wt%) to increase the surface attraction between the clay and the polymer matrix. One possible reason for this kind of behavior may be attributed to

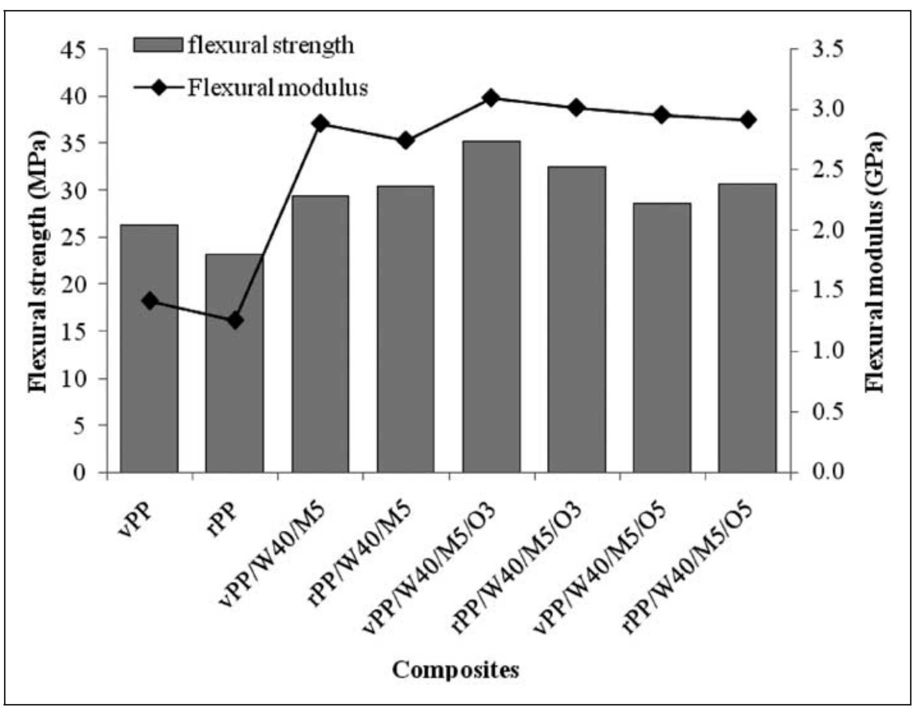


**Figure 1.** Flexural properties of the investigated formulations.

the agglomeration/clogging of the nanofillers or the filler–filler interaction, resulting in induced local stress concentration in the nanocomposites and reduction in the clay aspect ratio, thereby reducing the contact surface area between the organoclay and the polymer matrix and formation of agglomerated clay tactoids.[19,22,23]

Postconsumer PP is partially aged and contains carbonyl groups, originating from its photochemical and thermal oxidation that leads to better interaction between hydrophilic wood fiber and polymer matrix and consequently better water resistance property.[24]
3 Another possible reason ~~in~~ the enhanced dispersion and interfacial adhesion due to some percentage of chemical impurities, different molecular and compositional differences (MFI and degree of crystallinity) between the virgin and recycled plastics.[8] These results are consistent with the results of Butylina et al.,[24] Faruk and Matuana[25] and Najafi et al.[26]

*Tensile properties.* The trend of tensile properties of the WPCs as a function of matrix type and nanoclay content was similar to that of flexural properties discussed earlier. From Figure 2, it is evident that tensile strength of composites in which rPP was substituted with vPP was similar to each other and in some cases even slightly higher. The tensile strength and modulus of vPP was significantly higher than the rPP. The samples containing no WF showed tensile strength of 15.2 MPa and 13.7 MPa for vPP and rPP, respectively. The addition of 40 wt% WF increased the tensile strength of vPP- and rPP-based composites to 16.1 MPa and 16.2 MPa, respectively (with 5% MAPP), that is 5.8% and 17.6% higher compared to the neat polymers. The improvement in tensile strength at the presence of cellulose materials is expected as they are too much stiffer than polymer matrix and hence add stiffness to the composites.[18]

Similar to the flexural strength, with increasing nanoclay content up to 3 wt% in the formulations, the tensile strength significantly and modulus slightly increased. At higher nanoclay content (5 wt%), the values decreased. There was no principal difference in the reinforcement effect of nanoclay for vPP and rPP-based composites. When rPP was employed, the tensile strength varied from 16.2 to 18.8 MPa as the amount of nanoclay increased from 0 to 3 wt% based on the amount of the polymer. Since the recycled polymers contain impurities, their tensile and impact strength could be lower than that of virgin polymers.[7]

*Impact strength.* The notched Izod impact test was conducted at room temperature. Figure 3 illustrates the impact strength of the composites. The addition of 40% WF decreased the impact strength of rPP and vPP-based composites to 21.4 J/m and 22.5 J/m, respectively, that is 23.5% and 18.7% lower compared to pure PP (rPP). Impact strength of PP is higher than its WPC. WF is a kind of stiff organic agent, so adding fiber could decrease the impact strength of

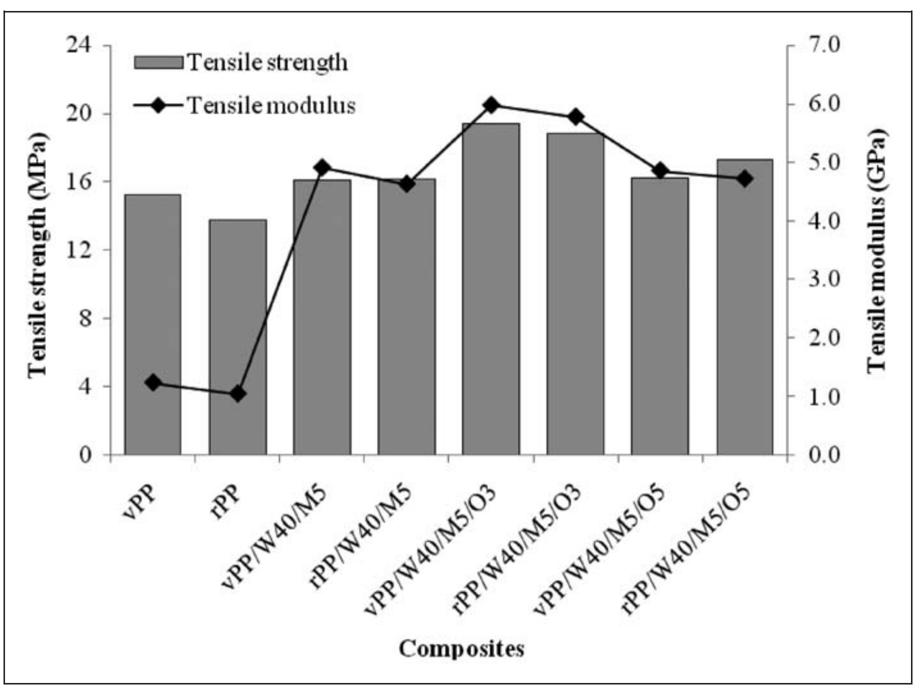


**Figure 2.** Tensile properties of the investigated formulations.

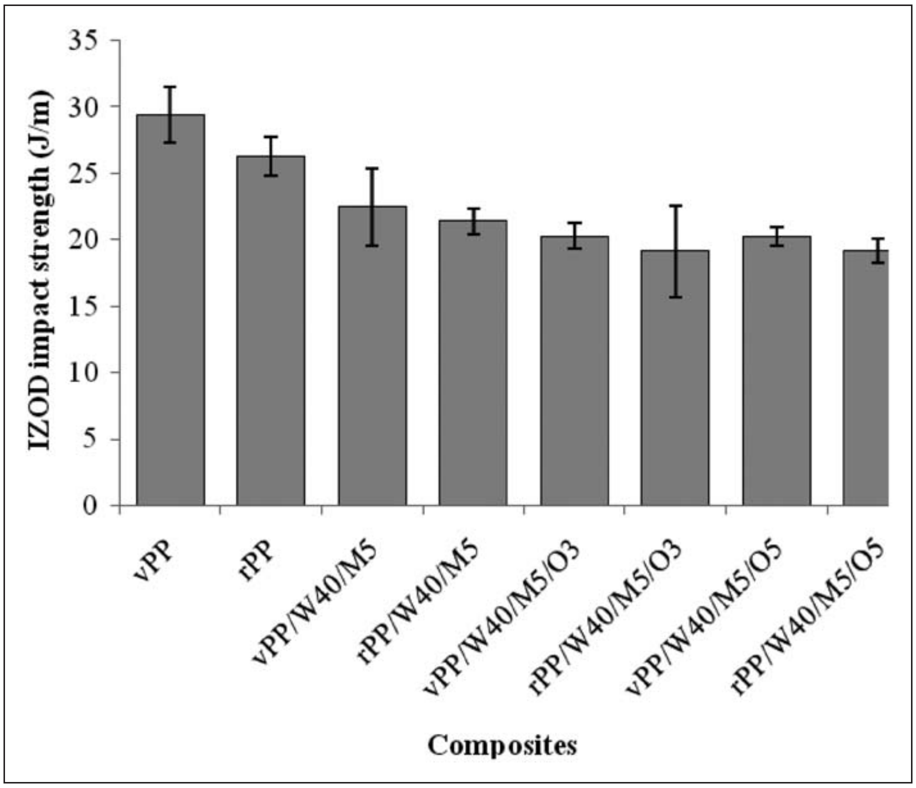


**Figure 3.** Izod impact properties of the investigated formulations.

composite. Typically, a polymer matrix with high loading of fillers has less ability to absorb impact energy. Fillers disturb matrix continuity and individual filler is a site of stress concentration, which can act as a microcrack initiator.[27] As flexural and tensile properties, Izod impact strength of composites decreased with substitution of rPP with the vPP, however, differences were not significant. Large difference in brittleness between the virgin and recycled polymers is not apparent in wood-reinforced PP (Figure 4). In light of nanoclay effect, contrary to flexural and tensile properties, impact strength slightly decreased (not significant) with incorporation of nanoclay content. The higher the nanoclay content, the lower was the impact strength. With increasing nanoclay content in the mixture, the impact strength decreased.[14,28] Regarding ductility, large impact strength differences between the vPP and rPP show up as small differences in the corresponding mechanical property of wood-reinforced PP. These results are in consistent with Nourbakhsh et al.[21]

## *Physical properties study*

*Water absorption.* Table 3 shows the percentage of water uptake of the nanocomposites with different matrix and nanoclay contents after 2 and 24 h water immersion times. Water absorption increased with increasing time of immersion. Water uptake is one of the key parameters in quality assessment of WPCs. Water uptake varies depending on the matrix type and nanoclay contents. The water absorption of pure PP, however, was so low (0.04 and 0.06% after 2 and 24 h water immersion, respectively) because PP is hydrophobic in nature, but with addition of WF to the formulations, water absorption increased. The hydrophilic nature of WF (high contents of hydroxyl groups in cellulose and hemicelluloses) was responsible for this.[21] Besides, PP–WF composites based on the rPP absorbed less water compared with the vPP matrix. The possible reason is the enhanced dispersion and interfacial adhesion due to the presence of chemical impurities, different molecular and compositional differences (MFI and crystallinity) between the virgin and the recycled plastics.[8] On the other hand, postconsumer PP is partially aged and contains carbonyl groups, originating from its thermal or photochemical oxidative degradation which these hydrophilic groups may improve the interaction between the hydrophilic wood fibers and the polymer matrix.[29] 4

The water absorption of WF/PP composites decreased with the incorporation of nanoclay. The higher the amount of clays, the lower was the water uptake. Organically modified clay increases the tortuous path for water transport and, as a result, water diffusivity decreases.[30] Decreasing available space for water absorption due to occupation of voice spaces in

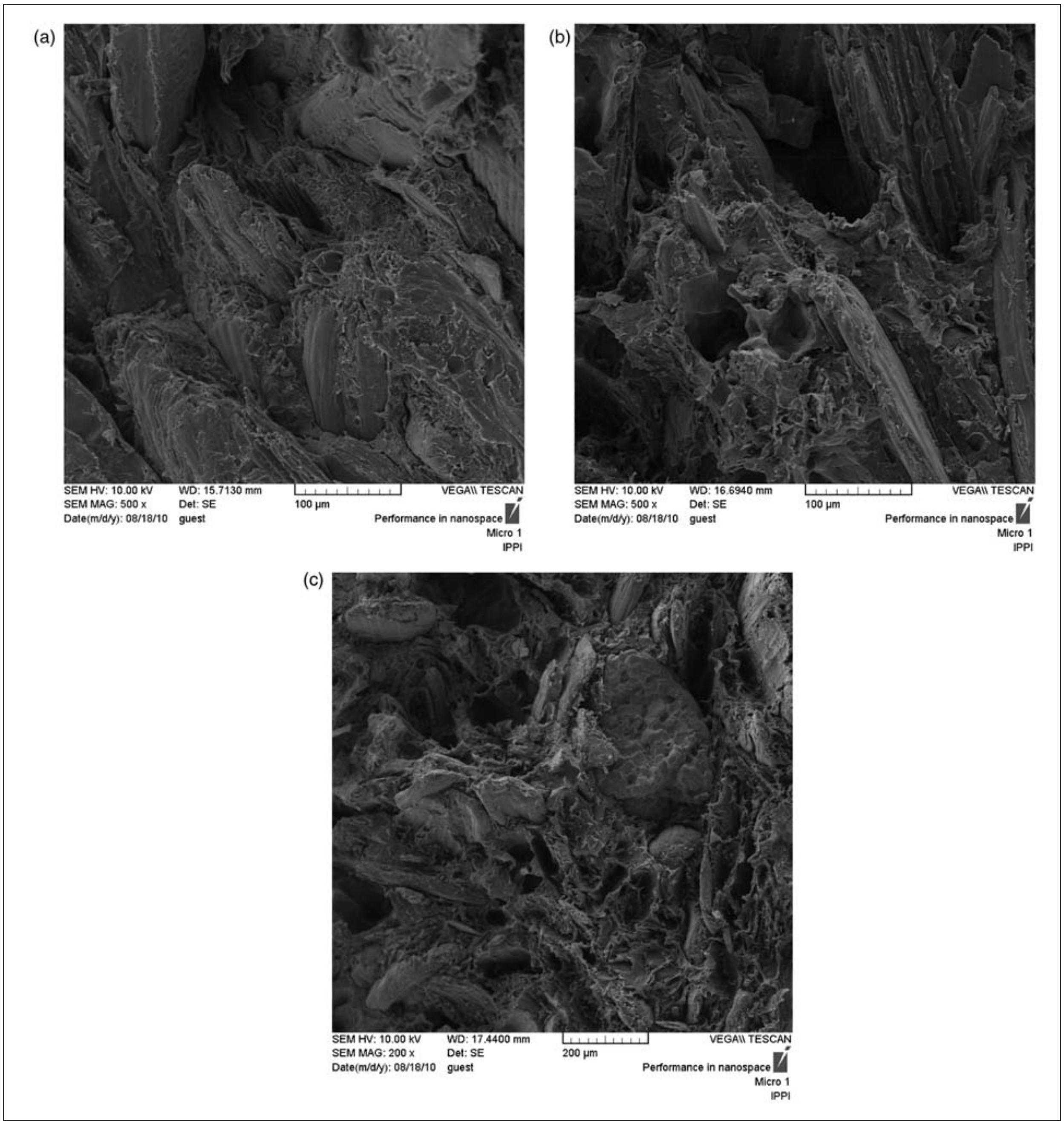


**Figure 4.** Scanning electron micrographs of tensile fracture surfaces of (a) polypropylene (PP)/wood flour with 3% nanoclay, (b) PP/wood flour without nanoclay and (c) PP/wood flour with 5% nanoclay.

the WF by the polymer and nanoclay can be another mechanism for the lower water uptake of nanocomposites.[13] The presence of nanoclay in the composite hinders the permeation of water through the composite.[3] The surface of modified clay had a tendency to immobilize some of the moisture.[31]

*Thickness swelling.* The effect of matrix type and nanoclay loading on the TS of composites is presented in Table 3. As it can be seen, TS of rPP-based composites is significantly lower than vPP-based ones. Besides, TS decreases with incorporation of nanoclay meaningfully. The higher the nanoclay content, the lower was the TS. TS of pure PP (both virgin and recycled) is almost negligible; for instance, 0.08% and 0.16% after 2 and 24 h water immersions for the virgin one. Reasons of improvement of TS with substitution of vPP with rPP as well as nanoclay loading are the same as water absorptions that was discussed earlier. These results are consistent with Valente et al.[32]

**Table 3.** Water absorption and thickness swelling of studied formulations.[a]

| | Water absorption (%) | | Thickness swelling (%) | |
|---|---|---|---|---|
| Composite sample code | 2 h | 24 h | 2 h | 24 h |
| vPP | 0.041 (0.01) | 0.057 (0.01) | 0.076 (0.01) | 0.158 (0.05) |
| rPP | 0.023 (0.01) | 0.052 (0.02) | 0.064 (0.03) | 0.135 (0.03) |
| vPP/W40/M5 | 0.297 (0.06) | 0.927 (0.10) | 1.321 (0.18) | 1.918 (0.33) |
| rPP/W40/M5 | 0.233 (0.04) | 0.760 (0.09) | 1.040 (0.14) | 1.524 (0.08) |
| vPP/W40/M5/O3 | 0.197 (0.02) | 0.860 (0.05) | 0.734 (0.09) | 1.043 (0.04) |
| rPP/W40/M5/O3 | 0.170 (0.03) | 0.533 (0.16) | 0.349 (0.22) | 0.821 (0.19) |
| vPP/W40/M5/O5 | 0.170 (0.03) | 0.690 (0.01) | 0.629 (0.24) | 1.046 (0.12) |
| rPP/W40/M5/O5 | 0.127 (0.04) | 0.343 (0.00) | 0.199 (0.14) | 0.502 (0.22) |

[a]vPP, rPP, *W*, *M* and *O* codes relate to virgin polypropylene, recycled polypropylene, wood flour, maleated polypropylene and organoclay, respectively.

*SEM study.* SEM micrographs taken from the fracture surface of specimens broken during the tensile test are shown in Figure 4.

SEM micrographs taken from the surface of broken specimens present partial implication of failure mode and interfacial adhesion. Figure 4(a) presents the fracture surface of specimens (PP/WF) prepared with 3% nanoclay, the coverage of the wood with the polymer and the relatively small number of holes related to debonding or fiber pullout indicate good adhesion between PP and WF and effectiveness of nanoclay in improvement of the interaction. The high number of debonding particles and holes indicates poor adhesion of PP/WF specimens without nanoclay (Figure 4(b)). The addition of nanoclay to the formulations altered the fracture modes. The roughness of a fracture surface is generally attributed to the fracture properties and critical strain energy release rates,[33] smooth featureless fracture surfaces are attributed to brittle nanocomposites and rougher fracture properties relate to tough nanocomposites.[34] SEM micrographs for specimens with no nanoclay revealed fracture surfaces that were relatively smooth and featureless. The roughness of the fracture surfaces relatively increased with increasing nanoclay content up to 3%. At high nanoclay loading (5%) SEM micrographs showed agglomeration and, as a result, ineffectiveness of them in providing good interaction between PP and WF (Figure 4(c)) and this is why reinforcement effect of nanoclay decreased at higher content.

*XRD results.* The XRD patterns of pure OMMT (Cloisite 15A) and selected composite samples containing nanoclay are shown in Table 4. The organically modified nanoclay showed its characteristic intense peak at $2\theta = 2.8^\circ$. The composite specimens with 3 and 5 wt% nanoclay loading showed a peak shift to lower diffraction angle ($2\theta$) than the pure Cloisite 15A, indicating an increase in interlayer spacing of silicate layers and intercalation of polymer chains between clay layers (Figure 5). The reduced peak intensity is attributed to the low concentration of clay in the samples. Similar observations have been recorded by many authers.[15,31]

**Table 4.** XRD results of the OMMT and selected composites.

| Formulation | 2θ (°) | d-spacing (nm) |
|---|---|---|
| OMMT | 2.8 | 31.35 |
| vPP/40WF/5MAPP/5OMMT | 2.57 | 34.1 |
| vPP/40WF/5MAPP/3OMMT | 2.51 | 34.49 |

XRD: X-ray diffraction; OMMT: organically modified montmorillonite; vPP: virgin polypropylene; MAPP: maleated polypropylene.

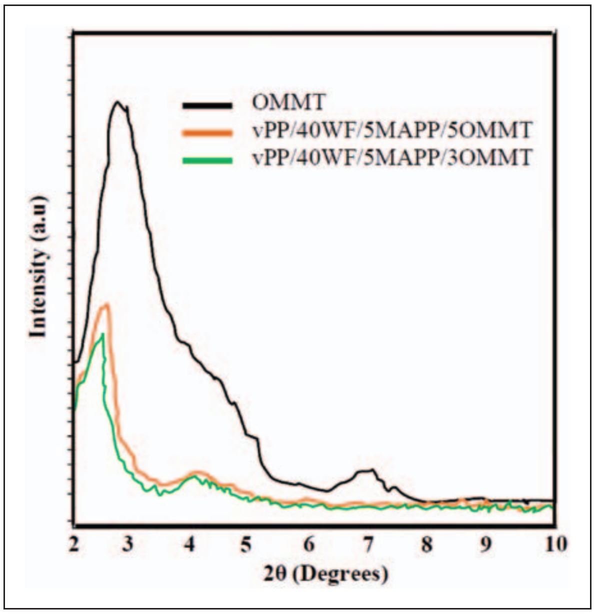


**Figure 5.** X-ray diffraction (XRD) patterns of clay (organically modified montmorillonite [OMMT]) and its polypropylene composites with wood flour.

## Conclusions

PP (recycled–virgin)/WF/OMMT nanocomposites were made via melt-blending method using HIB. The primary goal of this work was to establish the beneficial effect of WF addition on the mechanical properties of rPP from postconsumer applications and comparing it with the virgin one. The rPP contains various impurities and it has poor and variable mechanical properties compared to vPP. This, in general, roles out the use
5 of rPP in the original applications and in other high-value applications. Upon addition of sufficient amount of WF (40 wt%) to the PP, though, it is found that the mixture's mechanical and physical properties depend primarily on the wood content as well as nanoclay content and not on the mechanical properties of the matrix polymer. Thus, the mechanical properties of PP containing 40 wt% WF reinforcement remain essentially unchanged when the vPP in the matrix is replaced by rPP. It was found that, while impact and flexural strengths of the virgin and recycled polymers differ significantly, the composite properties vary only slightly from each other. Consequently, rPP from postconsumer applications can be used in high-value nanocomposites without going the expense of separating out impurities from the polymer.

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7 [redacted].

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