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Evaluation of properties of propylene-pine wood Plastic composite

J. Bhaskar [1], S. Haq [1], A.K.Pandey [2], N. Srivastava [3]

[1]*Department Of Mechanical Engineering Harcourt Butler Technological Institute, Kanpur-208002 India*
[2]*Head, Polymer Processing Lab, DMSEDRE, Kanpur*
[3]*HOD, Mech Engg Deptt. PSIT, Kanpur, India*

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Corresponding Author, E-mail: briseddik@gmail.com,

Abstract
The low maintenance cost of wood plastic composite (WPC) is generating a boom in the market of wood composites products. An attempt is made to fabricate WPCs with saw dust of pine wood flour which is wastages of wood/furniture industry. These composites (WPCs) are made using matrices of recycled polypropylene (rPP) with sawdust (Pine Wood flour) as filler. Corresponding WPCs are also made using virgin plastics (vPP) for comparison with the recycled plastic based composites. All varieties of these WPCs are made through melt compounding and injection moulding with varying formulations based on the plastic type (PP), plastic form (recycled and virgin), wood four content and addition of coupling agent(MAPP). The water absorption and thickness swelling evaluated. To understand the changes in WPCs stability and durability performance, microstructure of the composites are examined, Incorporation of maleated polypropylene (MAPP) coupling agent in composite formulation improved the stability. Scanning electron microscopy (SEM) images of the fractured surfaces of WPCs confirmed that the MAPP coupling improved the interfacial bonding between the plastic and the wood filler for the series of composites.

Keywords: rPP, vPP, MAPP, wood flour, water absorption, thickness swelling, microstructure characterization

1. Introduction
The plastic based composites, the polymers, either thermoplastics, act as a matrix and flour of wood or other natural flour are reinforcement. The reinforcing flour are the main load-carrying component in the composites. It provides high strength and stiffness as well as resistance to bending and breaking under the applied stress. Interface bonding between the fillers and the matrix is the key to transfer the stress from the matrix into the fillers across the interface. The interface adhesion between the polymer matrix and wood fillers can be improved using coupling agents. The coupling agents will form a bond between the wood flour (reinforcement) and the thermo-plastic (matrix) through the improved compatibility and developing a mechanical or chemical bonding. The performance of wood plastic composite (WPC), made using either recycled polypropylene (rPP) or virgin PP together with wood sawdust (Pine flour), is evaluated and compared in terms of water absorption and thickness swelling and SEM . It is found that composites made

form rPP through injection moulding exhibited excellent dimensional stability, which is comparable to those made from virgin PP (vPP). Effects of maleated polypropylene (MAPP) as a coupling agent is also investigated in the rPP based composites. Incorporation of 3-5 wt. % MAPP significantly improved the dimensional stability of the rPP based composites at all wood flour contents. MAPP modified composites confirmed improved interfacial adhesion due to enhanced flours dispersion and stability resulting from the chemical reaction between maleic anhydride in the MAPP and the hydroxyl groups of wood flour. It is clearly observed in SEM test.

2. Materials

2.1. Wood Filler

Pine wood flour is used as a reinforcing material in this study which is collected from the local Kanpur saw mill G.T.Road The fresh sawdust of pine wood received is dried at 103°C for 24 h to a moisture content of about 2-3% and then grounded to finer flour by using a sieve analysis. The sieve analysis found that most of the wood particles remained in the 35-45 mesh sizes with corresponding particle diameter ranging between 0.2 and 0.5 mm (200um<D<500um).

2.2. Thermoplastic Polymer

Both virgin and recycled post-consumer thermoplastics of PP are used in this study. Polypropylene purchased from the reliance industries limited (RIL) which is manufacturing at Hazier manufacturing division Surat, village Mora. It is homo polymer and it is full recyclable and reusable. The grade is H10 MA. After arriving at the laboratory, the plastic granules are dried at 65°C for 12 hr before mixing and compounding with wood flour in a twin-screw extruder.

2.3. Coupling Agent

The coupling agent used is maleated polypropylene (MAPP). Which collected from Defence Material & Stores Research & Development Establishment Kanpur.

3. Composite preparation

3.1. Mixing and Compounding

The wood flour is compounded, respectively, with the recycled and the virgin plastic granules (PP) in the co-rotating twin-screw extruder. Operating condition, speed of the screw 80 rpm, cycle time 3 min temperature 186 to 190 °C.

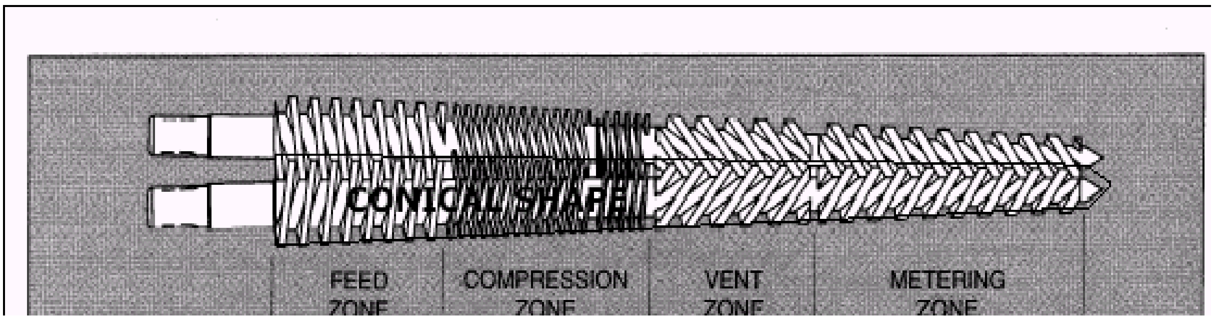

Fig. 1. Conical twin screw extruder

The operation conditions of the co-rotating twin-screw extruder compounding including extruder barrel temperature at different extruding zones, melt pressure, and screw speed employed for the compounding of both wood flour and plastics (PP). The wood flour and the plastic are fed through feeders at the extruder. The plastic pellets are firstly fed from the main feeding hopper at the end of the extruder, and then the wood flour is fed through a feeder. The extruded strand coming out from the die head is then passed through a water bath and subsequently palletized. The composite formulations are designed as per the mass proportion in percentage. The plastic composition was varied from 50-100 wt. % while the wood flour varied from 0- 50 wt. % in the composites. In some formulations, coupling agent (MAPP) is added at the proportion of 3 or 5 wt. %. In the text of this paper v, r, W and CA will be used to represent virgin, recycled, wood flour and coupling agent and the composition is given by the percentage values (% wt.) in the formulations. In the formulations where the MAPP is added, the plastics mass is reduced correspondingly thus, the total proportion of the plastics and the agent is either 50% (rPP47W50CA3 and rPP45W50CA5).

3.2. Injection Moulding of Pellets

Injection molding (British English: **moulding**) is a manufacturing process for producing parts from both thermoplastic and thermo set plastic materials. Wood plastic pellets material is fed into a heated barrel, mixed, and forced into a mold cavity where it cools and hardens to the configuration of the mold cavity. Operating condition of the injection moulding temperature of the cylinder is about 200 ^{0}C, temperature of the mould 50 ^{0}C and holding pressure up to 10 sec is 500 bar.

Table 1 WPCs formulations for PP series composites (percent by weight)

Composite sample code	Plastic type	Plastic content	Wood flour content	Coupling agent content type
Wood flour –PP series composite				
vPP100	Virgin	100	0	0
rPP100	Recycle	100	0	0
vPP60W40	Virgin	60	40	0
rPP60W40	Recycle	60	40	0
vPP50W50	Virgin	50	50	0
rPP50W50	Recycle	50	50	0
rPP47W50CA3	Recycle	47	50	3
rPP45W50CA5	Recycle	45	50	5

4. Experiemnts

4.1. Dimensional Stability Tests

Water absorption and thickness swelling tests are conducted in accordance with ASTMD570-98[15], in which the specimens are immersed in water for 2h and 24h, respectively, at a temperature 23±1°C. The weight gain and thickness increase are measured after 2h and 24h the samples are removed from the water. After 2h and 24 h water immersion tests, all of the specimens are weighted before and after the water immersion test.

In the water immersion tests, thickness of each composite sample is also measured for determination of the thickness swelling (TS).

4.2. Scanning Electron Microscopy (SEM)

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity. The fracture surfaces of the flexural test specimens were characterized with high resolution field emission scanning electron microscopy (FESEM).The scanning data are analyzed at magnifications of 500 X , 1000 X and 1500 X. Approximately 12 SEM images are taken and analyzed for each composite formulation.

5. Results and discussion

5.1 Water Absorption properties

Dimensional stability of the composites is investigated for both vPP and rPP with and without addition of the MAPP coupling agent. From the experimental results illustrated in table 2. It is found that the water absorption is increased with increasing wood content in the composites that is true both for 2 h and for 24 h water immersion. It is also found that the water absorption for 2h immersion varied from 0.02 to 1.3%, and after 24 h water immersion, the water absorption increased from 0.04 to 4.1% depending on the composite formulations. The rPP50W50 (50 wt. % rPP and 50 wt. % wood flour) composite exhibited more water absorption than the rPP60W40 composite (60 wt. % rPP and 40 wt. % wood flour). With the increase in wood content, there are more water-residence sites thus more water is absorbed. On the other hand, the composites made with higher plastic content have less water-residence sites and thus lower water absorption. The water absorption of the entirely rPP or entirely vPP was only 0.02-0.03% after 2 h and 0.04-0.05% after 24 h water immersion. The composites made of rPP have lower water absorption compared to those made of vPP given the same wood to PP ratio. It is also noted that the coupling agent (MAPP) can significantly reduced the water absorption. As the rPP may have experienced chain scission beforehand a part of OH- of polymer already consumed by the wood flour, thus having lesser water resident sites. Additionally the wood flour dispersion is poor in case of vPP matrix composites. When coupling agent is added, the influence of the plastic to wood ratio is no longer as important as in the composite without the coupling agent. With 3-5 wt. % MAPP, the water absorption is reduced at 2h and 24h immersions is in the rPP50W50 composite formulation.

Table 2 Experimental results for the water absorption.

Composite sample code	Water absorption (%)	
	2h	24h
vPP100	0.03	0.05
rPP100	0.02	0.04
vPP60W40	1.28	2.67
rPP60W40	0.94	2.15
vPP50W50	2.1	4.1
rPP50W50	1.3	3.6
rPP47W50CA3	0.58	1.3
rPP45W50CA5	0.49	1.11

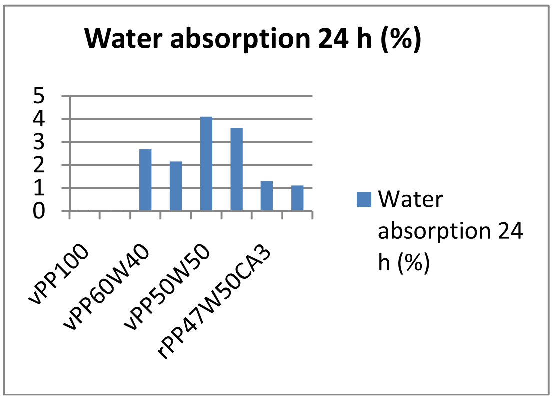

Fig 2 Water absorption

5.2 Thickness Swelling properties

Thickness swelling of the wood-PP composites has a similar trend as the water absorption and composites with high water absorption also showed higher thickness swelling.

The thickness swelling values for the 2 h immersion varied from 0.01 to 0.32%, and these values are increased after 24 h immersion, varying from 0.02 to 1.21% depending on the composite formulation. Samples made with lower content of wood flour have the lowest thickness swelling as for the water absorption. However, MAPP coupled composites showed less thickness swell than composite samples without the coupling agent at the same wood content. In general, the composite made of virgin and recycled PP has similar dimensional stability properties without adding the coupling agent. However, the stability properties of these composites are improved by adding 3-5 wt. % MAPP coupling agent. The impact of wood to plastic ratio on the water absorption and thickness swelling can be explained by water absorption behaviors of wood and plastic. As cellulose fibre is the main component in the wood flour, the absorbed water mostly resides in the regions such as the flour lumens, the cell wall, and the gaps at the interface between the wood flours and the polymer matrix.

Table 3 Experimental result for the thickness swelling.

Composite sample code	Thickness swelling(%)	
	2h	24h
vPP100	0.01	0.03
rPP100	0.01	0.02
vPP60W40	0.21	0.71
rPP60W40	0.18	0.59
vPP50W50	0.32	1.21
rPP50W50	0.28	1.09
rPP47W50CA3	0.16	0.55
rPP45W50CA5	0.11	0.43

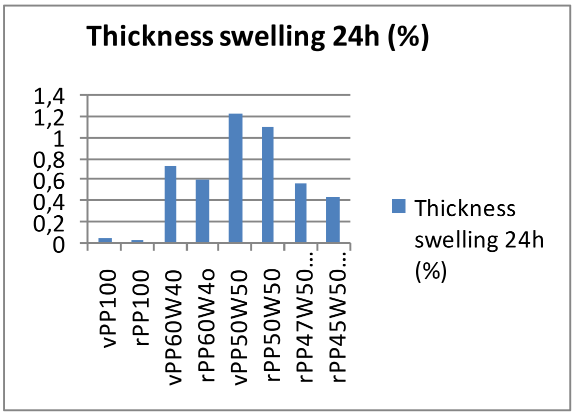

Fig. 3. Thickness swelling properties

The presence of hydroxyl and other polar groups in various constituents of the wood flour resulted in poor compatibility between hydrophilic wood flour and hydrophobic plastic, which increased the water absorption. With the increase in wood content, there are more water residence sites thus more water was absorbed. With the addition of MAPP (3-5%) the compatibility between wood flour and PP is improved because the anhydride moieties in MAPP entered into an esterification reaction with the surface hydroxyl groups of wood flour. This lowered the water absorption sites and reduced the water absorption in MAPP coupled composites. The composites based on rPP based composites absorbed less water compared with vPP based composites for same wood and plastic content. The possible reason could be the enhanced dispersion and interfacial bonding due to the presence of chemical impurities through better surface wetting during processing. Water absorption in composites is mainly due to the presence of lumens, fine pores and hydrogen bonding sites in the wood flour, the gaps and flaws at the interfaces, and the micro-cracks in the matrix formed during the compounding process.

5.3. Microstructure Characterization

Microstructure of the fractured surface of specimens tested in tensile is examined using SEM. SEM images of the wood flour-PP composites at filler loading of 50 wt. % for vPP and rPP matrices are shown in Fig.4(a) and (b), in 1000× magnification. From these images, it is clearly observed that there are distinct cluster and gaps between polymer matrix and wood. The patterns from wood flours that are so weakly bonded to the matrix have been released from the matrix during fracture. The failure surface is undulated with clear wood flour surfaces with visible trachaids and lumen, indicating the path of weaker part through the wood-wood interface and weakest polymer matrix. This suggests that the interface between the wood and PP matrix is weaker due to the poor dispersion and compatibility.

The dispersion of the wood flours in the rPP matrix (Fig. 5(b)) is uniform as compared to vPP matrix (Fig. 4(a)). This may be due to the different grade of plastic and other impurities in the rPP. In some cases, the part of the wood lumen is filled with plastic that could increase the strength of the composites because of mechanical interlocking. When wood content is increased, the polymer matrix is no longer continuously distributed and many wood flours are in direct contact with one another, resulting in poor bonding at adhesion at the interface.

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Fig. 5(a) and (b) show SEM images of fracture surface of the 3 and 5 wt. % MAPP incorporated composites filled with 50 wt. % wood flour. SEM image showed that there are no clear gap between wood flour and PP matrix, indicating the good interface bonding. The fracture surface of the composite showed a very limited amount of torn matrix, suggesting that the matrix is more brittle than those composites without MAPP.

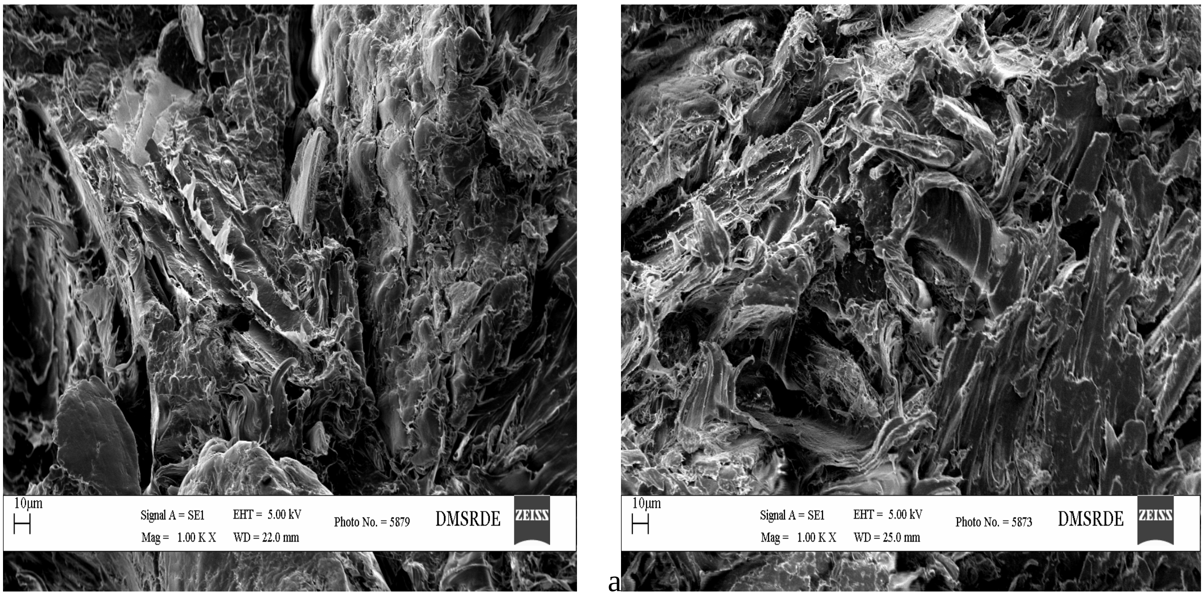

Fig. 4. SEM images (×1000) of fractured surface of (a) vPP50W50, (b) rPP50W50

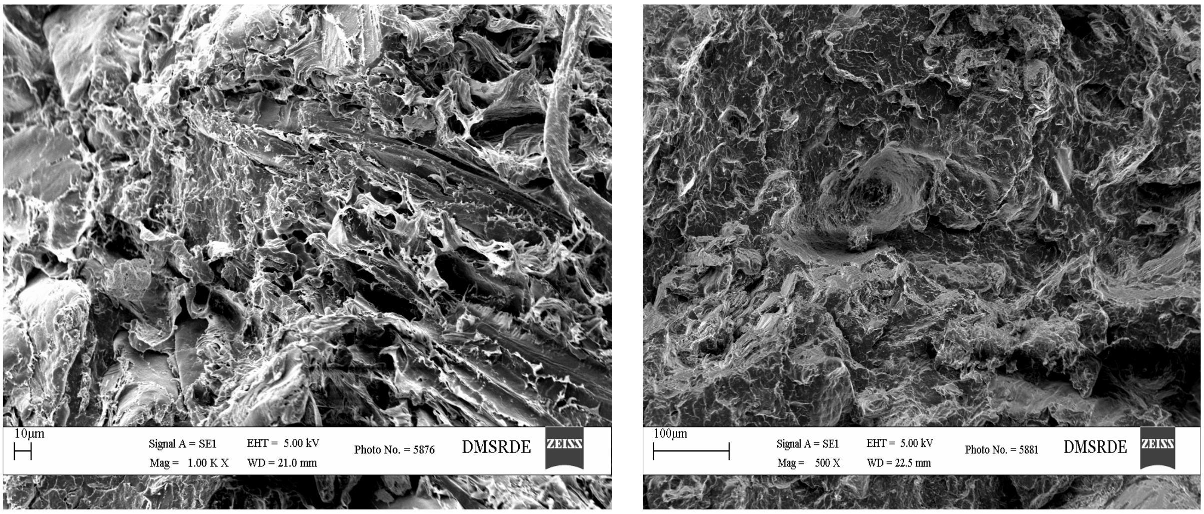

Fig.5. SEM images (×1000) of fractured surface of (a) r PP47W50CA3, (b) rPP45W50CA5

It is also seen that a crack running through the wood flours, and this could be an indication of stress-transfer from the matrix to the wood flours. The interfacial bonding between the filler and the PP matrix is improved due to the esterification mechanism and the fracture occurred at the filler itself. This means that the stress is well propagated between the filler and the matrix polymer, resulting in enhanced flexural strength and modulus in response to stress. In addition, the fracture surface showed a very limited amount of torn matrix, suggesting that the composite is more brittle. In general, coupling agent is randomly distributed in composites and randomly reacted with wood flours and the matrix to form graft polymerization. Hence, grafting sites are randomly distributed on wood, and a network of coupling agent is formed at the interface. However, there is a limit for chemical coupling reaction and only part of coupling agent was grafted onto wood surface and even cross-linked at the interface. Further, the fracture surface of the composite containing 5 wt % MAPP showed a very limited amount of torn matrix, suggesting that the matrix is more brittle than those in composites containing 3 wt % MAPP. This phenomenon is mainly due to the excessive modification of the base polymer.

Comparison between Fig. 4 and 5 it is observed that non-coupled composite samples had a weak interfacial region and damage mainly occurred along the loose and weak interface between the wood flour and PP matrix under loading. However, with the MAPP coupled composites, the wood flour is combined with the PP matrix through the covalent bonding or strong interfacial bonding, and interfacial fracture usually accompanied with a cross section damage of the wood flour. Hence, after the failure, the flour surface in the untreated composites is smooth; whereas the wood flour in the MAPP treated composites has a rough surface. It is embedded in the matrix with a chemical link.

Conclusions

SEM images of the fractured surfaces of composites confirmed that an addition of the MAPP coupling agent improved the interfacial bonding between the polymer and the wood filler for the rPP based composites. Stability and mechanical properties of the WPCs can be achieved by addition of coupling agents. SEM images of the fractured surfaces of composites confirmed that bonding strength between recycle PP and wood flour stronger then the bonding strength between virgin PP and wood flour. Dimensional stability propertied of the WPCs are improved with the addition of 3-5 wt. % MAPP coupling agent in the same composite formulations. Wood plastic composites (WPCs) are made using recycled polypropylene (rPP) with wood flour (Pine radiate) as filler. Post-consumer plastics and waste sawdust are used as raw materials. Corresponding WPCs are also made for some composite formulations using virgin plastics (vPP) for comparative studies. WPCs sample are made through melt compounding and injection moulding based on plastic type (PP), plastic form (virgin, recycled), wood flour content and adding of MAPP coupling agent.

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