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Effect of Materials Content on Dimensional Stability, Nano Roughness and Interfaced Morphology for Virgin or Recycled Polypropylene Based Wood Composites

Shamsul Haq

Abstract

The compositions of mango wood-polypropylene composites (WPCs) are formulated, with different compositions of virgin polypropylene (PP) or recycled PP, mango wood waste and a coupling agent. The compositions are fabricated via melt extrusion compounding pursued by injection hot molding. The tests of the prepared compositions are carried out for, water absorption, thickness swelling, surface properties at a nano-scale and interfaced morphology. Comparative study of WPCs composition has done on respective properties. All processing variable conditions are constant for different compositions. The recycled PP based wood composites with or without the coupling agent possessed superior properties in comparison to virgin PP based composites. FESEM images show that coupled composite is having the better bonding strength and smoothness along with a higher dimensional stability in comparison to none coupled composite. Future endeavor should be focused on optimizing the composition of reinforcement wood and recycled plastics matrix according to intended application. The quality of WPCs can also be improved with the co-ordination of latest development in technology and processing technique relevant to them. WPCs study supports "turning waste into something useful". This provides the mileage in price performance ratio and also the product's environmental footprints to be adjusted to suit the products application.

Keywords: polymer wood composites, interface morphology, nanoscale roughness, water absorption, sustainability, wood-based products

1. Introduction

Wooden materials are one of the firstly natural construction materials, as utilized by the human being. It is used in most of the cases. But it is applied in maximum cases, according to the traditional approach methodologies. This is showed by past experience and utilization tactics of wood. As a view of the above, in the field of engineering perspective and application, wood and the wood-based material

is one of the low tech or second tier materials. But the fact is that the exploitation of wooden based material uninterrupted to steadily expand in construction and structural field. To reduce the consumption of neat wood or tree plant, many of the approaches are developed to create wood based material for sustained in different engineering applications. One of the approaches is based on the combination wood material with other material. Wood composites are based on a component of wood and thermoplastic are gaining an importance in the current century. Wood plastic composites are one of the newly developed materials for the different field of application. It is still a very new material in comparisons to the long history of natural lumber as a construction material and plastics.

The wooden article is readily biodegradable and renewable. On the other hand, thermoplastic has an option for the recyclability. For wood plastic composites (WPCs), thermoplastic and wood combinations have exerted enough pressure recently to develop the WPC products, due to their favorable properties, processing characteristics and eco-friendly advantages [1, 2]. Polypropylene (PP) belongs to the polyolefin family and most widely used plastics today. It is also light in weight. In WPCs, thermoplastics and wood flours are acting as a matrix and reinforcement materials respectively [3–5]. The filler wood flour has bearded the load across its interface with the matrix and procures the WPCs against break-down under the applied loads [6, 7]. Most of the physical and mechanical properties are broadly investigated and listed in the previous research works in this field [8–14]. Hygroscopic thickness swelling of composites was estimated for 50:50 wood plastic ratios [14]. Influence of artificial UV conditioning on the roughness of the composite was estimated for one composition of wood and PP [15]. Roughness characteristics of such like WPCs should be investigated for finding out its bonding and finishing properties [16, 17]. Also, WPCs are used in many interior and exterior applications. WPCs are gaining increasing potential in many construction applications including decking, fencing, (**Figure 1**) siding, paneling, furniture industries, windows, doors, siding, sign board, barrier, cabinet etc. In general, WPCs may be used in almost all application where wood or plastic already are utilized. Also, improving the performance of WPCs materials, additives may be used for the different function such like as Lubricant, pigments, colorant, an anti-microbial agent, antioxidants, UV stabilizer, fire retardants agent, coupling agent, stabilizers etc. Surface quality and dimensional stability of wood-based panels are one of the most

Figure 1.
Fencing and deck board made from WPCs.

important characteristics. It is stimulated by the manufacturing processes or their adhesive strength and also performs an important preface in the determination of the attributed product for an ultimate application. Therefore minute observation of dimensional stability, surface roughness and interfaced morphology of these WPCs materials is desirable. The objective of this work is to study various compositions of WPCs, fabricated by mango (scientific name-Mangifera indica) wood waste, virgin (v) PP, recycled (r) PP and maleic anhydride polypropylene (MAPP). Fabricated compositions of WPCs are compared and correlated in terms of water absorption, thickness swelling, nano-roughness and interface morphology. Also, estimated the effect of mango wood flour, vPP and rPP quantities are on the abovementioned properties. Effect of coupling agent on mango wood-PP composites is also studied in rPP matrix based compositions. Surface roughness parameters, roughness profile and 3D surface variations have analyzed the roughness of compositions.

The developments in the quality life of peoples are provided through better uses and exercises on available unutilized or utilized resources. From ancient times humans have fabricated wood and wood-based products as the most available materials in the surrounding. However, led to environmental consciousnesses are in exclusively on recycling of utilized materials, degeneration in forest resources and warming in global condition. These factors have forced the peoples to make efforts to form bio-composites from waste wood resources or non-wood residues [11]. Similarly, the plastic waste which is a major part of municipal solid waste (MSW) should also be utilized. In past research work, efforts have been made to utilize these waste thermoplastics and wood in sequence to decrease the eco-system imbalance and production of the neat plastics and wood. The important factor for promoting the recycling of a waste plastic product is to achieve an appropriate composition with the intended application. Worldwide environmental degradation motivated the researchers to focus on fabrication of the materials using several recycled and waste stream [2, 18]. Also, WPCs product can be re-molded under the application of heat many times after the useful service [19]. Correlation between nano-roughness and dimensional stability of above like mango-PP composites are not admitted as much as other engineered materials. So this study concerned for the surface, stability and interface between mango wood flour and polypropylene.

2. Materials, fabrication, and experimentation

Wood flour is biodegradable and cheap in cost, with low density and abrasion in comparison to synthetic reinforcement [20]. Mango wood sawdust waste is procured from the local Kanpur (U.P. India) saw mill. It is heated at 100°C for 2 hours in order to remove the moisture content up to less than 3%. The Sieve analysis is performed with 50 and 30 mesh sieve and the corresponding size of the wood particles are found between 297 μm < D <595 μm. Thermal degradation temperature of the wood flour is 200° C [21, 22], hence there is no mass loss at the processing and molding temperature in this study. Lubricant Zinc stearate ($C_{36}H_{70}O_4Zn$) helps to improve the flow of melt in mold and also worked as a mold releasing agent. Polypropylene is obtained from the RIL, India. Recycled and virgin PP is used in this study. PP granules are heated in an oven at 50°C for 8 hours before melt compounding with reinforcing flours. It is a homopolymer, h10 ma grades with a density of 0.9 g/cm³. MFI of PP is 11.9 ± 0.29 g/10 minute according to ASTM D1238. MAPP worked as a compatibilizer or coupling agent to WPCs. It is procured from Defense Material & Stores Research and Development Establishment Kanpur. MAPP (with Mw – 9, 100 by GPC, Mn – 3, 800 by GPC and maleated anhydride

1.5 wt.%) is used in this study. Twin screw micro conical co-rotating extruder is used for the melt compounding of the flours of wood with v or r PP and MAPP. Haake mini CTW5 lab performed the melt compounding and produced WPCs blend. Composites are formulated by the weight ratio in percentage. The different compositions and their codes are given in **Table 1** [23]. The twin screw with speed of 65 revolutions per minutes in the co-rotating extruder, compounding in the house is for two minutes and temperature range (start to end) in the chamber is 175 to 180°C. Same machine parameters are applied for the compounding of both wood flour with rPP or vPP and MAPP. The conical co-rotating twin screw, intercepted the agglomeration from start to end at the various stages of compounding due to its design characteristic. Haake mini jet micro injection molding is used to fabricate WPC samples as per ASTM standard by the molding of WPC blend or pellets. The temperature of the cylinder is about 188 to 190°C and of a mold is 55°C. The pressure of 450 bars is maintained for 7 sec. at the cylinder formfull filling the cavity of the mold.

Dimensional-stability (water absorption and thickness swelling) test is followed the ASTMD570–98 [24]. In this test, samples are immersed in a container of distilled water for 2 hours and then for 24 hours, at a temperature about 23 ± 1°C. All surface waters are wiped off with a dry cloth before all measurements. Surface topography measured by the attractive forces between the probe and the sample in a non-contact mode by AFM (Agilent Technologies, USA) at nano-level. AFM provides three-dimensional image and highresolution information. AFM cantilevers are fabricated with an integrated sharp tip of silicon nitride. The Agilent AFM, raster scanning in the X and Y axes, tip in the Z axis and produces the 2D profile and 3D surface structure of the composites. Surface roughness parameters data followed by ISO 4287 standards. It scans about 2 × 2 μm surface area of the sample. AFM produces the result with high accuracy and calibration but used with a limitation of little region scanning. AFM has collected a roughness parameter (Ra, Rz, and Rmax), 3D surface structure and roughness profile at a nanoscale [25]. The fracture interface of the samples arem characterized by FESEM, DMSRDE Kanpur. The scanning data between the interfaces of cracked composites are analyzed at 500× magnifications.

S. No.	Code for samples	WPCs composition	Polypropylene form	Polypropylene content	Mango flour content	MAPP (CA) content
Mango wood flour – polypropylene composites						
1.	I	vPP100	virgin	100	0	0
2.	II	rPP100	recycle	100	0	0
3.	III	vPP70W30	virgin	70	30	0
4.	IV	rPP70W30	recycle	70	30	0
5.	V	vPP60W40	virgin	60	40	0
6.	VI	rPP60W40	recycle	60	40	0
7.	VII	vPP50W50	virgin	50	50	0
8.	VIII	rPP50W50	recycle	50	50	0
9.	IX	rPP47W50CA3	recycle	47	50	3
10.	X	rPP45W50CA5	recycle	45	50	5

Table 1.
WPCs formulations of sample given below in table (percentage by weight).

3. Results and observation

3.1 Dimensional stability

Water absorption and thickness swelling of the compositions is examined for both rPP and vPP based WPCs. The dimensional stability test is also done in coupled composites. Data from the test is presented in **Tables 2** and **3**. It is found that the water absorption, increased with the higher quantity of mango flour in the WPCs composition. This trend is exactly right for both 2 and 24 hours water immersion for all samples. It is also calculated that the water absorption for 2 hours immersion increases from 0.02 to 6.51%, and after 24 hours water immersion, the water absorption rises from 0.056 to 9.08% dependent on the wood-plastic composite compositions. Sample V has a value of 3.72% and 7.46% and VI has a value of 3.38% and 7.04% for 2 hours and 24 hours respectively. It is established that rPP have lesser water absorption in comparisons to those fabricate of vPP for same plastic to a wood composition. The same behavior is pursued by sample III, IV, VII and VIII. Dispersion of wood flour and interface bonding, due to the existence of oxidized impurities are improved for rPP based composites [26]. The stability is increased by the presence of oxidized impurities in rPP by the development of ester bond with wood flour. So that micro gaps or crack at across the interface between the reinforcing flour and rPP matrix are reduced. It has provided a lower surface wetting for the same. That is why the dimensional stability of rPP based WPCs are higher than vPP based. It is pointed that the MAPP can significantly reduce the water absorption. This trend is followed by the samples IX and X for rPP based composites. In addition, the sample VIII exhibited more water absorption than sample VI. This shows that the composites made from higher plastic content have less water absorption.

It is also true for vPP based composites. This is due to the hydrophobic properties of polypropylene because it is covered the functional polar hydroxyls groups of wood flours and made chemically in active.

The composition of WPCs with high water absorption displayed higher thickness swelling for all sample compositions. The result of thickness swelling test is

Composite sample code	Water absorption (%)	
	(2 hours)	(24 hours)
I	0.024 (0.026)	0.078 (0.039)
II	0.02 (0.0245)	0.056 (0.0167)
III	2.88 (0.335)	5.94 (0.152)
IV	2.48 (0.192)	5.46 (0.114)
V	3.72 (0.165)	7.46 (0.207)
VI	3.38 (0.134)	7.04 (0.294)
VII	6.51 (0.222)	9.08 (0.228)
VIII	6.13 (0.12)	8.52 (0.13)
IX	3.38 (0.148)	6.6 (0.424)
X	2.7 (0.158)	5.63 (0.403)

The numbers in parenthesis are standard deviation value.

Table 2.
Result from water absorption test of the WPCs.

Composite sample code	Thickness swelling (%)	
	(2 hours)	**(24 hours)**
I	0.0125 (0.017)	0.0625 (0.022)
II	0.0063 (0.014)	0.05 (0.028)
III	2.07 (0.198)	5.16 (0.247)
IV	1.69 (0.175)	4.7 (0.214)
V	2.85 (0.164)	6.68 (0.376)
VI	2.57 (0.194)	6.26 (0.408)
VII	5.46 (0.199)	8.44 (0.247)
VIII	4.78 (0.219)	8.01 (0.215)
IX	2.49 (0.218)	6.1 (0.313)
X	2.08 (0.186)	5.19 (0.204)

The numbers in parenthesis are standard deviation value.

Table 3.
Result from thickness swelling test of the WPCs.

presented in **Table 3**. The existence of other polar groups and hydroxyl in several constituent elements of wood produces the poor compatibility between hydrophobic polypropylene and hydrophilic wood. So that, these wood-plastic based composites have the ability to take up water under humid environment due to the availability of various hydroxyl groups present for interaction via hydrogen bonding with water molecules. MAPP treated composition shows, lower thickness swell than the composition with the absence of MAPP at the same wood content. The anhydride moieties of MAPP enters into with hydrophilic groups of mango wood through an esterification reaction [27]. It forms monoester and diester bond between a wood particle and thermoplastic, consequential improve the compatibility. This is reduced water absorption sites in MAPP coupled compositions. Above properties of rPP based composites are improved by adding 3 to 5% coupling agent (MAPP).

3.2 Surface characterization

The roughness properties of composition at a nano-scale are estimated by the Average roughness (Ra), maximum roughness (Rmax) and mean peak-to-valley height (Rz) using AFM. The roughness parameters of mean values of are given in **Table 4**. The sample V and VII have reinforcing flour quantity 40 and 50 percentage respectively. The Ra values of sample V and VII are 1.47 nm and 2.38 nm respectively. It is clear from the above Ra values that with the decrease of plastic quantity from 60–50%, Ra value increases for the WPCs samples. A similar trend is also followed by rPP based wood composition VI and VIII. Roughnesses of the WPCs composition are significantly higher by the increase in the wood flour quantity for all composition of WPCs. On the other side, the Ra value of the sample VII and VIII are found to be 2.38 nm and 1.63 nm respectively. From these Ra values of sample VII and VIII, it can be concluded that vPP based composite has a more roughness in comparison to rPP based WPCs. The same trend is followed by the sample V and VI. It is because of the well dispersed wood flour in the rPP matrix [28]. Resultantly, rPP based WPCs obtained a good interfacial bonding between wood reinforcement and rPP matrices. Due to which, Ra value is lower for rPP

Composites code	Roughness parameters (mean value) (nm)		
	Ra	**Rz**	**Rmax**
V	1.47	4.97	9.95
VI	0.952	2.42	4.15
VII	2.38	9.12	16.4
VIII	1.63	3.92	8.18
X	0.74	2.59	3.64

Table 4.
Mean value of roughness parameters.

based composition. The roughness parameters (Rz and Rmax) also pursued the same trend of Ra result for samples.

The influence of coupling agent upon the roughness characteristics of WPCs composition VIII is also analyzed. With the addition of 5% MAPP, a roughness of the surface is decreased. Sample X has surface parameters, Ra, Rz, and Rmax values 0.74 nm, 2.59 nm, and 3.64 nm while VIII has values of 1.63 nm, 3.92 nm, and 8.18 nm. This proves that sample X has a smoother surface than sample VIII. This result shows the effect of MAPP and confirms the improvement of the interface bonding between the flours of wood and the rPP matrices for the sample [27, 29].

Particular roughness profile and the 3D surface variation of WPCs sample V, VI, VII, VIII and X also supported the result for the roughness parameters. 3D roughness variation is shown in **Figure 2**. Also, it must be noted that profile of a tip probe upon the surface does evaluate the variation in the roughness profile. 3D roughness image shows an actual 3D morphology of the zigzag structure for the respective composition at a nano-scale.

3.3 FESEM analysis

Mango wood flour of a weight ratio of 50% with vPP and rPP matrix is shown by FESEM morphology in **Figure 3** (VII) and (VIII) respectively. Outcomes of this morphology that interface bonding between vPP mango wood flour and rPP mango wood flour are showing a distinct lumen, cavity and gaps for sample VII and VIII. The pattern of **Figure 3** (VII) and (VIII) shows that the wood flours are so inadequately connected to the PP matrix. So it is recommended that the interface across the reinforcing flour and PP matrices are poorer due to the bad compatibility and dispersion. Mango wood flour shows a better and uniform dispersion with rPP matrix (sample VIII) in comparison to vPP matrix (sample VII). Because the mango reinforcement is in filling the small cavity and micro-gaps in the rPP matrix. Mechanical interlocking is improved due to better-dispersed flours filler in the rPP matrices [28]. So it possesses good entanglement in connections at the interface for composition VIII.

Image of coupled composite sample X in **Figure 3** (X) displayed no apparent cavity between reinforcement and matrix materials which indicates, the strong bond at the interface. It is observed from the morphology of sample X that there is a much little magnitude of ragged matrices, informing that the composite X is stable than those VII and VIII composites which have no coupling agent. The meshes of MAPP (coupling agent) are developed at the interface and fill the gaps. There is a lowest aggregation in the MAPP coupled composites. While in non-coupled composite, wood flour set out the aggregates for sample VIII. The presence of the -OH groups

Figure 2.
3D surface structures for sample V, VI, VII, VIII, and X respectively.

(VII)   (VIII)

(X)
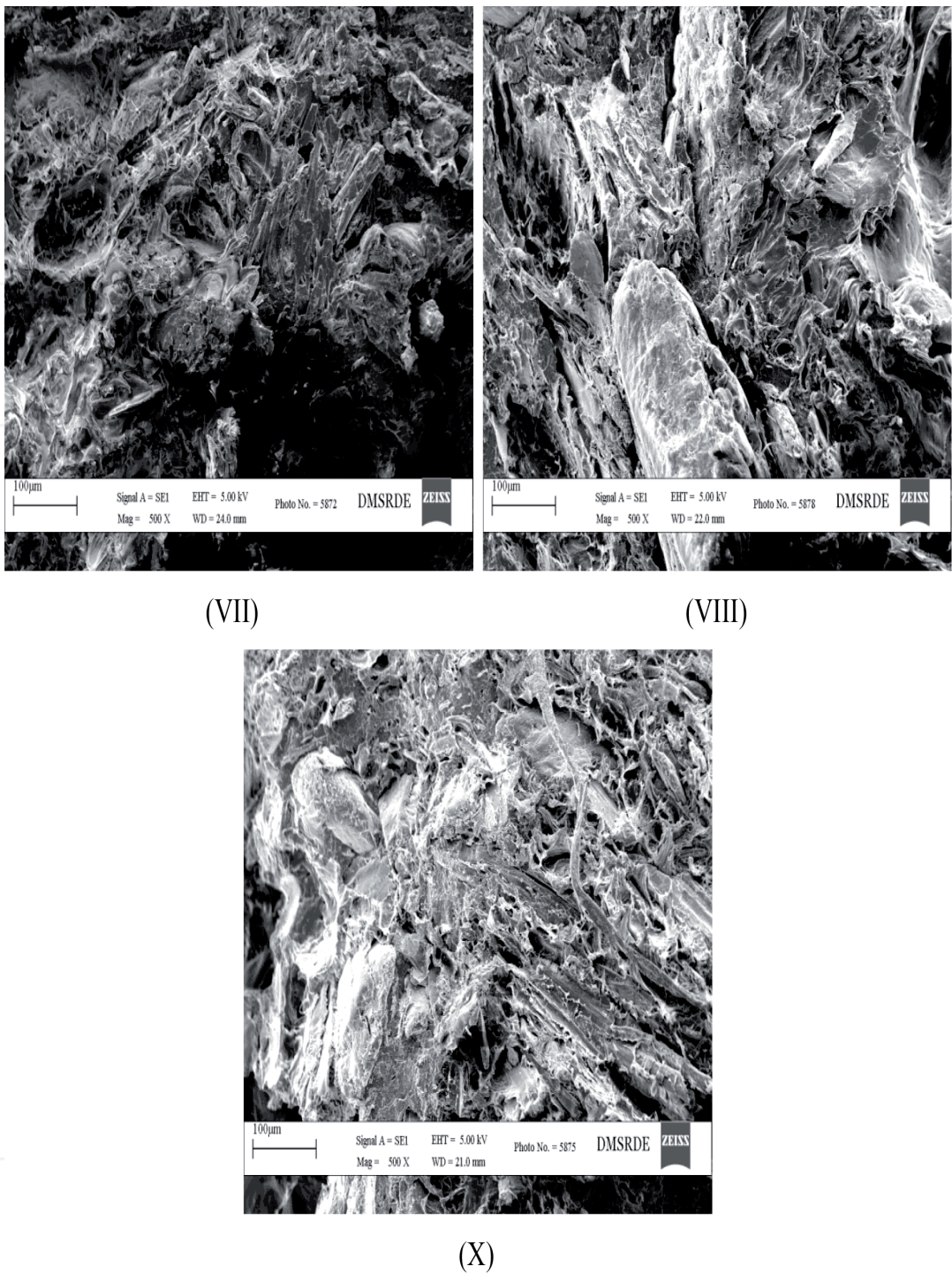
Figure 3.
FESEM morphology of interface for sample VII, VIII and X respectively.

in the reinforcement flour, create hydrogen bonds and tempted to each other [19]. The addition of MAPP has certainly altered the chemical character of the wood flour surface [27] and improved crystallinity of the structure [23]. It is favorable for densification and reduces the roughness of WPCs [16, 26]. This enhances in turned the dimensional stability as well as surface smoothness.

4. Conclusions

Water absorption of the composites based on rPP is lower for those based on vPP in all set. Also, rPP based compositions show better smoothness in comparison to vPP based compositions. The coupling agent is improving the surface

characteristics, highest in the same class and decreasing the water absorption in rPP based composites. This is reduced water absorption [(6.13% to 3.38% and 8.52% to 6.6%) & (6.13% to 2.7% and 8.53% to 5.63%)] for 2 h and 24 h and with addition of 3 and 5% MAPP respectively. The performance of surface roughness and water absorption of composites improved with reducing plastic content. The rPP based compositions displayed a better outcome in the same class either with MAPP or without MAPP, observed from the experimentations. FESEM interface images morphology is also validating the results. The Present research work demonstrates that all WPCs compositions which have lower water absorption or better dimensional stability shows higher smoothness and dense interface morphology and vice versa. The intended application of WPCs is suitable, where dimensional stability and smoothness condition merits are important especially in humid condition. Formulation contents and raw materials characteristics directly affect the quality of above properties. The present study will be further helpful for Wood based products development and construction applications.

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