

Long term hygroscopic characteristics of polypropylene based hybrid composites with and without organo-modified clay

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Abstract Recycled PP contains various impurities and has poor and variable mechanical properties compared to virgin PP. This, in general, rules out the use of recycled PP in the original applications and in other high-value applications. Hence, this study investigated the effects of polymer matrix type, weight fraction of wood flour and organoclay on the thickness swelling and water absorption behavior of PP based hybrid composites. WPCs based on polypropylene (virgin and recycled), wood flour, organoclay and maleated polypropylene (MAPP) were made using melt compounding and subsequent injection molding. Composites made from recycled polypropylene (rPP) exhibited better dimensional stability compared to the virgin (vPP) based ones. Besides, wood flour did not completely encapsulate in the polymer matrix at 50 % weight fraction. Incorporation of nanoclay exhibited a beneficial effect on both the water absorption and thickness swelling by creating a tortuous path as a result of its characteristic barrier property. The improvements in hygroscopic characteristics of hybrid composites using rPP and nanoclay were further supported by scanning electron microscopy and X-ray diffraction methods. Conclusively, PP recycled from post-consumer applications can be used

in value-added composites without accepting the expense of separating out impurities from the polymer.

Hygroskopische Langzeiteigenschaften von Polypropylen-basierten Verbundwerkstoffen mit und ohne organisch modifiziertem Ton

Zusammenfassung Rezykliertes Polypropylen (PP) enthält starke Verunreinigungen und hat verglichen mit frischem Polypropylen schlechte und unterschiedliche mechanische Eigenschaften. Dadurch ist die Verwendung von rezykliertem PP in üblichen Anwendungen sowie solchen mit höherer Wertschöpfung ausgeschlossen. In dieser Studie wird der Einfluss des Polymermatrixtyps, des Masseanteils von Holzmehl und von organischem Ton auf die Dickenquellung und die Wasseraufnahme von Hybridwerkstoffen auf Polypropylenbasis untersucht. WPCs auf Basis von Polypropylen (rezykliert oder frisch), Holzmehl, organischem Ton und Maleinsäure-Anhydrid-Polypropylen (MAPP) wurden durch Schmelz-Compoundierung und anschließendem Spritzguss hergestellt. Verbundwerkstoffe aus rezykliertem Polypropylen (rPP) wiesen eine bessere Dimensionsstabilität als Verbundwerkstoffe aus frischem Polypropylen (vPP) auf. Bei 50 % Masseanteil wurde das Holzmehl nicht vollständig in die Polymermatrix eingekapselt. Die Zugabe von Nanoclay wirkte sich aufgrund seiner charakteristischen Barriereigenschaften positiv auf die Wasseraufnahme und die Dickenquellung aus. Die Verbesserung der hygroskopischen Eigenschaften der Verbundwerkstoffe auf Basis von rPP und Nanoclay wurden auch mittels Rasterelektronenmikroskopie (REM) und Röntgenstrahlbeugung (XRD) bestätigt. Abschließend ist zu sagen, dass Polypropylen aus rezyklierten Produkten für hochwertige Verbundwerkstoffe ohne kostenintensive Beseitigung von Verunreinigungen aus dem Polymer verwendet werden können.

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1 Introduction

Polypropylene (C_3H_6) (PP), a semi-crystalline polymer, has been used in a wide assortment of applications. PP has excellent resistance to stress and can readily be recycled mechanically several times using conventional equipment. Recycled PP contains various impurities and has poor and variable mechanical properties compared to virgin one that restricts the use of recycled PP in high-value applications (Khanjanzadeh et al. 2012). Attempts have been made to reuse waste plastics in order to reduce the environmental impacts and consumption of virgin plastics (Hannequart 2004). The worldwide production of plastics is about 245 million tons per annum resulting in a significant amount of municipal solid waste (Plastics Europe 2009). Since most plastics take a very long time to break down in nature, the disposal of polymers after use is an important issue (Hirunpraditkoona and Garcia 2009). In the wood industry, a large amount of wood waste is generated at different stages of the wood processing which mainly ends up in landfills (dump sites in a few areas) or is burned (Poletto et al. 2011). The volume of wood flour waste generated in the wood industry is very high and estimated at around 16 % of its total production (Ares et al. 2010).

Wood plastic composites (WPCs) have experienced significant market expansion in recent years as a replacement for solid wood, mainly in outdoor applications (Afrifah et al. 2010). Manufacturing wood-plastic composites using waste wood and recycled plastic out of the municipal and industrial solid wastes produced daily in vast quantities can have an important meaning both in the stabilized supply of raw materials and solution of solid waste problems. Using recycled PP to formulate WPCs is ideal from the point of view that adding wood particles has the potential to mask and even over-power the composition-driven as well as molecular weight-driven variations in the properties of the base polymer.

In polymers of low polarity such as polypropylene (PP), low compatibility between the polymer and the clay is observed during mixing that can lead to poor dispersion (Ferreira et al. 2011). The use of maleated polypropylene as a compatibilizer enhances the dimensional stability of the polypropylene, wood flour and nanoclay composite via improving adhesion between polypropylene-wood flour as well as interaction between polypropylene-organoclay (Pettarin et al. 2008; Deka and Maji 2010; Chan et al. 2011).

Natural fillers such as wood flour have a number of advantages over synthetic fillers; they are abundant resources, environmental friendly and cheap. Despite these advantages, there are some problems associated with using lignocellulosic materials in natural fiber/thermoplastic composites including moisture absorption, bio-deterioration, and thickness swelling. Moisture absorption is one of the most important

problems of WPCs exposed to environmental conditions which will determine their end use applications. Water absorption figures for wood-plastic composite materials typically are 0.7–2 % after 24 h, 1–5 % after 1 week, and up to 18–22 % after several months (Klyosov 2007). During long term exposure of wood plastic composites to water, changes in microstructure can occur and a great number of large openings could be created in the composites resulting in decreased bonding between the fiber and matrix, hence, loss of mechanical properties is unavoidable (Butylina et al. 2011). Therefore, moisture absorption has to be taken into account in the design of WPCs for final applications (Xu et al. 2010). It has undesirable effects on the mechanical properties and the dimensional stability of fibers and in long-term results in their embrittlement, which is linked to the degradation of the macromolecular skeleton by hydrolysis (Espert et al. 2004; Anuar et al. 2008). Water molecules easily bond to these hydroxyl groups via hydrogen bonding and push apart the fibrils causing cell wall to swell (Steckel et al. 2007). Water absorption of lignocellulosic fibers place stress on the surrounding matrix and lead to micro-cracking, which would eventually cause the composite to fail catastrophically (Abdul Khalil et al. 2011). This gives rise to other problems such as microbiological decay. The rate at which water is absorbed by a composite depends on many variables including type of fiber and matrix, temperature, difference in water distribution within the composite, and reactions between water and the matrix (Butylina et al. 2010).

One of the new ways to enhance water resistance as well as mechanical properties of both thermoplastic and thermoset based wood composites that has gained great attention in academic and industrial sectors is addition of small amounts of nanoclays (Arafa et al. 2004; Lei et al. 2008; Adhikary et al. 2008; Chan et al. 2011; Valente et al. 2011; Ferreira et al. 2011). Montmorillonite (MMT) is a member of the smectite mineral group, a naturally occurring layered material having high aspect ratio and specific surface area (Moucka et al. 2011). Clays are organophobic in nature, but they can be changed into organophilic by replacing the Na^+ or Ca^{2+} cation originally present in the galleries by one organic cation such as alkyl ammonium ions via an ion-exchange reaction (Pettarin et al. 2008). MMT is composed of 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 (Moucka et al. 2011). An optimum or synergistic usage of nanoclay in the resins is essential to achieve the highest physical property as well as other tailored features that they offer (Haq et al. 2008).

Ashori and Sheshmani (2010) evaluated the moisture absorption and thickness swelling behavior of hybrid composites made from recycled materials. They found that composites containing more fractions of recycled

newspaper fiber exhibited maximum water absorption during the entire duration of immersion and stated that the effect can be contributed to the presence of a high amount of holocellulose present in the recycled newspaper fiber, compared to the poplar wood flour.

Adhikary et al. (2008) studied long-term moisture absorption and thickness swelling behavior of HDPE based wood plastic composites. They found that water absorption and thickness swelling increase with wood content and water immersion time before equilibrium condition was reached and reported that hygroscopic properties can be improved significantly with incorporation of a coupling agent (maleated polypropylene) into the composite formulation. The composites made from the recycled plastics showed comparable results to the composites made of virgin plastics.

Therefore, it is important to study in detail long term hygroscopic characteristics not only in order to estimate the consequences that the absorbed water may have, but also how this water absorption can be minimized in some ways. The main objective of this work was to investigate recycled polypropylene (as well as virgin one as a benchmark) and waste wood flour in manufacturing hybrid composites, and to study the hygroscopic characteristics of these composites in terms of long-term water absorption and thickness swelling. The effect of nanoclay incorporation was also investigated.

2 Materials and methods

2.1 Materials

Polymers used for making composites were post-consumer recycled polypropylene (rPP) and virgin polypropylene (vPP) in the form of pellets. The melt flow index (MFI) of rPP and vPP were 8.5–9.5 and 5–7 g/10 min (at 230 °C/2.16 kg), respectively and their density was 0.9 g/cm³. Underutilized wood flour (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 (1–2 %) before compounding.

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

The clay sample, Cloisite 15A, an organically modified montmorillonite (OMMT), was purchased from Southern Clay Products Inc. The properties of nanoclay are given in Table 1.

2.2 Compounding and injection molding

Prior to compounding, the wood flour was oven-dried at 100 °C for 24-h for removal of any additional moisture.

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

Compounding was performed in Haake internal mixer (HIB, sys 90, USA). Mixing was carried out at 175 °C with a rotation speed of 60 rpm. After melt blending in the Haake mixer, the mixed materials were cooled for about an hour at room temperature and grounded 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

Table 1 Specification of the Nanoclay

Tab. 1 Eigenschaften von Nanoclay

Typical properties		
Organic modifiers	Modifiers concentrations (meq/100 g clay)	Moisture (%)
2M2HT ^a	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 Å°		

^a Dimethyl, dehydrogenated tallow, quaternary ammonium

Table 2 Wood plastic composite formulations (percent by weight)

Tab. 2 Zusammensetzung der Holz-Kunststoff-Verbundwerkstoffe (Masseanteile)

Composite sample code	Wood flour content	vPP content	rPP content	MAPP content	Nanoclay content
(A) W40/vPP/M5	40	55	–	5	–
(B) W40/vPP/M5/N3	40	52	–	5	3
(C) W40/vPP/M5/N5	40	50	–	5	5
(D) W50/vPP/M5	50	45	–	5	–
(E) W50/vPP/M5/N3	50	42	–	5	3
(F) W50/vPP/M5/N5	50	40	–	5	5
(G) W40/rPP/M5	40	–	55	5	–
(H) W40/rPP/M5/N3	40	–	52	5	3
(I) W40/rPP/M5/N5	40	–	50	5	5
(J) W50/rPP/M5	50	–	45	5	–
(K) W50/rPP/M5/N3	50	–	42	5	3
(L) W50/rPP/M5/N5	50	–	40	5	5

vPP, rPP, W, M and N codes are related to virgin polypropylene, recycled polypropylene, wood flour, maleated polypropylene and nanoclay, respectively

180 °C, a molding temperature of 40 °C, an injection pressure of 10 MPa and cooling time of 20 s. Specimens for water absorption and thickness swelling testing were made according to ASTM specifications.

3 Characterizations

3.1 Moisture content and thickness swelling

For moisture content and thickness swelling tests, five specimens of each formulation were selected and dried in an oven for 24 h at 100 °C. The weight and thickness of dried specimens was measured to a precision of 0.001 g and 0.01 mm, respectively. The specimens were then placed in distilled water and kept at 23 ± 1 °C. For each measurement, specimens were removed from the water, and the surface water was wiped off using absorbent paper. Weight and thickness of the specimens were measured at different times during the prolonged immersion. These tests were conducted in accordance with ASTM D570 (2005).

3.2 Scanning electron microscopy

After the water absorption test, the specimens were broken under tensile strength and fracture surfaces were investigated using a scanning electron microscope (SEM) (Model VEGA-II TESCAN). All specimens were sputter coated with gold prior to examination.

3.3 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 K α radiation ($\lambda = 1.54$ nm). The samples were scanned in 2θ ranges $2\text{--}10^\circ$ 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: $2d \sin\theta = \lambda$.

4 Results and discussion

4.1 Water uptake

Figures 1 and 2 show the water absorption percentages for the composites at different periods of immersion, which vary depending upon the type of polymer matrix, fiber and nanoclay loadings. In all the cases, the water uptake was found to increase with increasing time of immersion. Generally, the water absorption curves of the hybrid

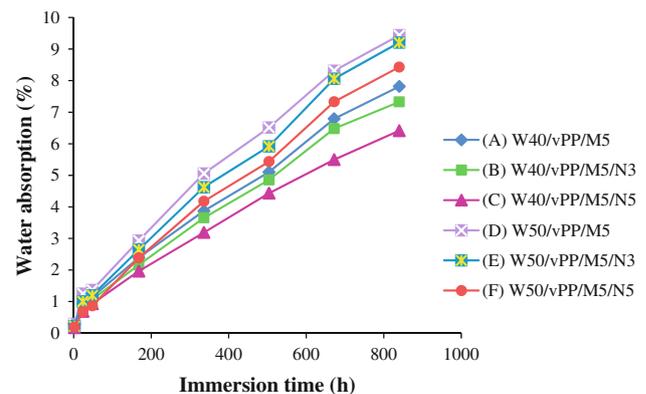


Fig. 1 Water absorption behaviour of wood flour-vPP composites
Abb. 1 Wasseraufnahme von Holz-vPP-Verbundwerkstoffen

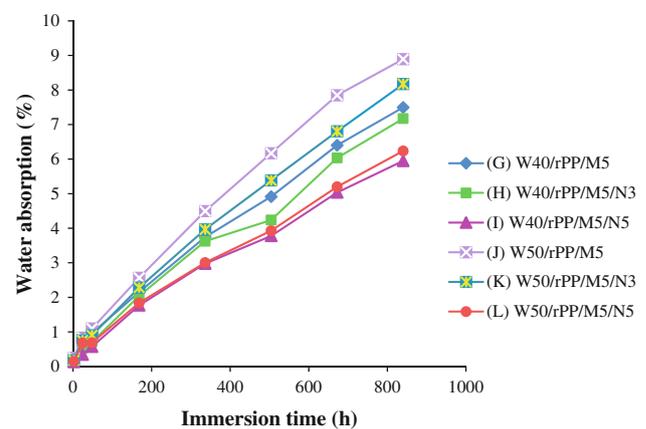


Fig. 2 Water absorption behaviour of wood flour-rPP composites
Abb. 2 Wasseraufnahme von Holz-rPP-Verbundwerkstoffen

composites increase with an increase in soaking time until equilibrium conditions are reached.

It is interesting to note that composites based on the recycled polypropylene absorbed less water compared with the virgin polypropylene matrix. One possible reason is the enhanced dispersion and interfacial adhesion due to the presence of chemical impurities, different molecular and compositional differences (melt flow index and crystallinity) between the virgin and the recycled plastics (Adhikary et al. 2008). Hygroscopic characteristic of wood based composites is affected by bond quality and resin property (Abdolzadeh et al. 2011). Post-consumer polypropylene is partially aged and contains carbonylic groups, as a consequence of its thermal or photo-chemical oxidative degradation which these hydrophilic groups may improve the interaction between the hydrophilic wood fibers and the polymer matrix (Spinace et al. 2009). Since water absorption by WPCs generally depends on their porosity, amount of cellulose fiber, and their availability for incoming water (Klyosov 2007), lower water absorption of rPP based WPCs in comparison to the virgin ones can also

be due to lower availability of wood flours to water as a result of better interfacial adhesion and encapsulation of fibers in the former composites.

The water absorption increases with increasing wood flour content in the composites. For instance, water absorption in sample type D with 50 % wood flour content increased to 9.44 % as compared to 7.82 % water absorption of the corresponding composite with 40 % wood flour content (sample type A) (Fig. 1). Wood flour is assumed to be encapsulated in the polymer matrix but results of this study imply that it is not completely valid, especially when the filler content is 50 %. Wood flour is a lignocellulosic material that is a three-dimensional composite, substantially composed of macromolecules of cellulose, hemicelluloses and lignin (Ndazi et al. 2006). The water uptake of lignocellulosics can be mainly ascribed to hydrogen bonding of water molecules to the free hydroxyl groups presented in cellulose and hemicelluloses (Gwon et al. 2010; Nourbakhsh et al. 2011). Cellulose is a natural polymer containing many hydroxyl groups, and these groups and their ability to form hydrogen bonds govern the physical properties of cellulose; in addition, non-cellulosic carbohydrates or hemicelluloses have an amorphous structure and hydrophilic characteristic, so water can be absorbed in hemicelluloses either (Gwon et al. 2010). However, lignin is totally amorphous and hydrophobic, therefore, water absorption cannot occur in lignin (Pirayesh et al. 2012). Additionally, large numbers of porous tubular structures present in fiber accelerate the penetration of water by the capillary action (Abdul Khalil et al. 2011). Therefore, with increasing wood flour content, there are more water residence sites (OH groups) resulting in more absorbed water. Under the combined influence of pressure and heat, cell lumens in hardwoods collapse and some fractures in cell walls develop which are responsible for the dimensional instability of the fibers in the form of hygroscopic swelling (Buyuksari et al. 2012).

The effect of clay on water absorption was investigated by using organoclay at 0, 3 and 5 wt %. The organoclay, Cloisite 15A, is hydrophobic in nature and does not disperse well in water, hence, the water absorption of wood flour/PP composites decreased with the incorporation of organoclay. The higher the amount of clay the lower was the water uptake. Organically modified clay increases the tortuous path for water transport and as a result water diffusivity decreases (Alexandre et al. 2006; Zahedsheijani et al. 2011). Decreasing of the available space for water absorption due to occupation of void spaces in the wood flour by the polymer and nanoclay can also be a mechanism for the lower water uptake of nanocomposites (Deka and Maji 2010). Another reason can be obstruction of capillaries in wood by nanoparticles so that the water lost its main channel (Shi et al. 2007). The presence of organoclay

in the composite hinders the permeation of water through the composite (Deka and Maji 2011). The surface of modified clay has a tendency to immobilize some of the moisture (Lee and Kim 2009). The result of water absorption is consistent with other researchers (Arafa et al. 2004; Lei et al. 2008; Kord et al. 2010; Doosthoseini and Zarea-Hosseini 2010; Zahedsheijani et al. 2011; Khanjanzadeh et al. 2012).

4.2 Thickness swelling

The thickness swelling of hybrid composites follows a trend similar to the water absorption behavior, increasing with immersion time until equilibrium condition is attained. As time goes on, water can penetrate the interphase and also the loose cellular cellulose network structure of wood flour, resulting in high water uptake. The effects of matrix type and nanoclay loading on the thickness swelling of composites are presented in Figs. 3 and 4. Thickness swelling of pure PP (both virgin and recycled) is almost negligible (e.g., 0.076 and 0.158 % after 2 and 24 h of water immersion for the virgin PP, respectively). Thickness swelling of rPP based composites is significantly lower than vPP-based ones. The results indicate that rPP-based composites have a better interfacial adhesion compared to vPP-based ones. Besides, thickness swelling (after 840 h) decreased with incorporation of nanoclay significantly. The higher the nanoclay content the lower is the thickness swelling. For specimens containing 40 % wood flour the thickness swelling decreased from 4.72 % for specimens without nanoclay to 4.32 % for specimens containing 3 % nanoclay and 3.85 % for specimens containing 5 % nanoclay. Dimensional instability leads to a moisture build-up in the fiber cell wall (fiber swelling) and also in the fiber–matrix interface and it is responsible for

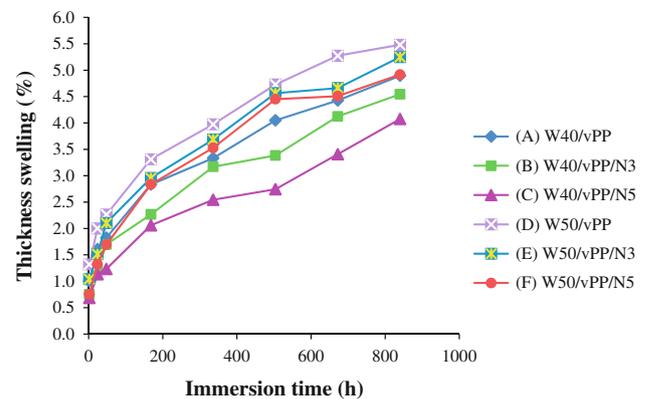


Fig. 3 Thickness swelling versus water immersion of vPP-based composites

Abb. 3 Dickenquellung in Abhängigkeit der Wasserlagerung der vPP-basierten Verbundwerkstoffe

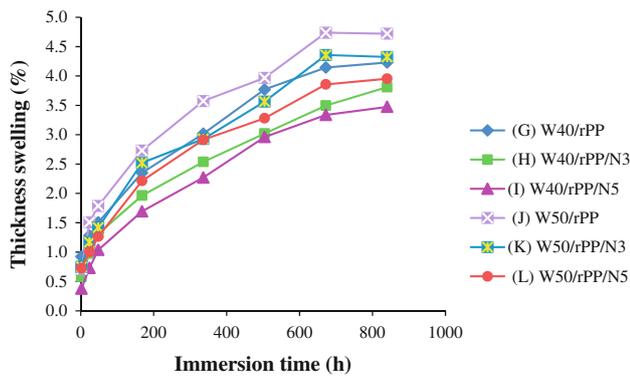


Fig. 4 Thickness swelling versus water immersion of rPP-based composites

Abb. 4 Dickenquellung in Abhängigkeit der Wasserlagerung der rPP-basierten Verbundwerkstoffe

the changes in the dimension of cellulose-based composites, particularly in the thickness due to reversible and irreversible swelling of the composites (Abdul Khalil et al. 2007; Nourbakhsh and Ashori 2010). As a consequence, the fiber–matrix adhesion is weak and outdoor applications of the composite will be greatly restricted (Wichman et al. 1993; Butylina et al. 2011) and this can give rise to other problems such as microbiological decay. Reasons for improvement of thickness swelling with substitution of vPP with rPP as well as nanoclay loading are the same as water absorption discussed earlier. These results of thickness swelling are consistent with other researchers (Arafa et al. 2004; Kord et al. 2010; Doosthoseini and Zarea-Hosseini-abadi 2010; Zahedsheijani et al. 2011; Valente et al. 2011; Khanjanzadeh et al. 2012; Ashori and Nourbakhsh 2011).

4.3 SEM study

SEM micrographs (Fig. 5) taken from the surface of broken specimens presents partial implication of failure mode and interfacial adhesion. Figure 5a shows a good interface between matrix and wood flour so that the fracture mode is debonding instead of fiber pull out for rPP based composite containing 40 % wood flour and 3 % nanoclay. At higher wood flour and clay contents (50 and 5 %, respectively) some localized agglomerations of fillers occurred (Fig. 5b), and from the micrographs it is evident that pull-out along with high number of holes dominate the fracture surface of the nano-composite. High amount of the fillers has caused the matrix to be discontinued and there are a number of gaps as a result of fiber pull out indicating poor interfacial bonding as proposed in the discussion on the physical properties. Figure 5c shows a WPC without nanoclay (50 % wood flour) and it is evident that the mixture is not homogenous and consistent and due to the high amount of wood flour flocculation is seen and wood flour is not

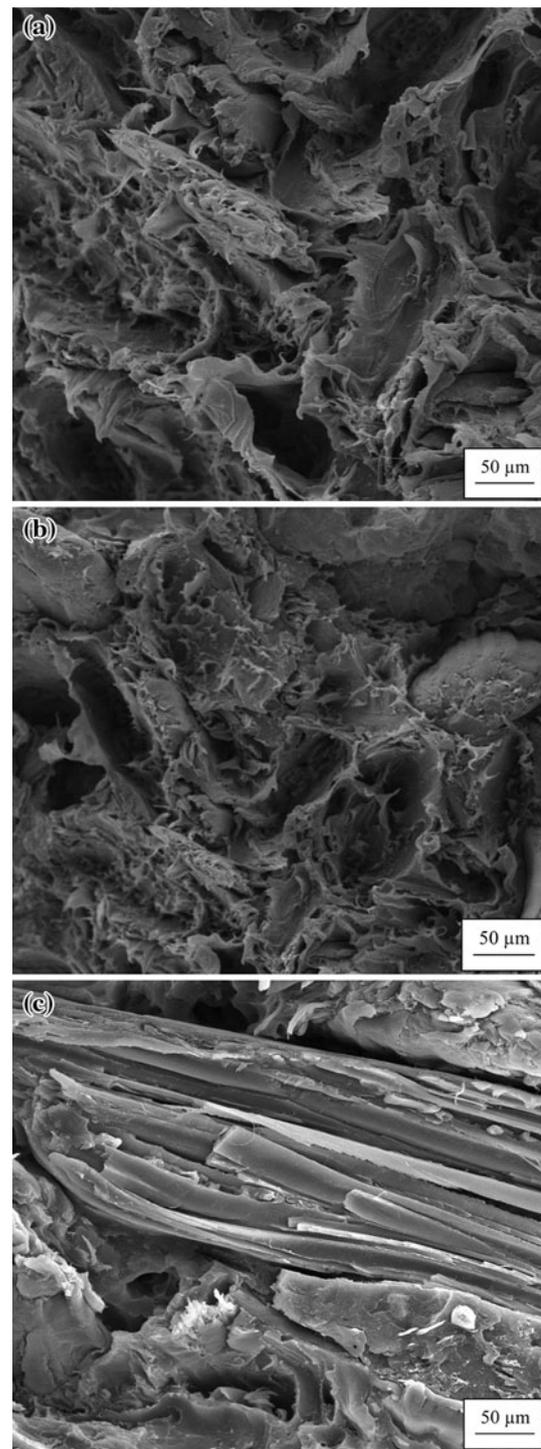


Fig. 5 SEM micrographs of fracture surfaces for: **a** W₄₀/rPP/M₅/N₃, **b** W₅₀/vPP/M₅/N₅, **c** W₅₀/vPP/M₅
Abb. 5 REM-Aufnahmen der Bruchfläche von: **a** W₄₀/rPP/M₅/N₃, **b** W₅₀/vPP/M₅/N₅, **c** W₅₀/vPP/M₅

completely encapsulated by the polymer. The roughness of a fracture surface is generally attributed to the fracture properties and critical strain energy release rates (Haq et al. 2008); smooth featureless fracture surfaces are attributed to

brittle nanocomposites and rougher fracture properties relate to tough nanocomposites (Wang et al. 2006). The roughness of the fracture surfaces increased relatively with increasing nanoclay content up to 3 %. These features provide an explanation of the increase in water absorption as well as thickness swelling.

4.4 XRD results

The XRD patterns of pure OMMT (cloisite 15A) and selected composite samples containing nanoclay are shown in Fig. 6. 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 (2θ) than the pure cloisite 15A, indicating an increase in interlayer spacing of silicate layers and intercalation of polymer chains between clay layers. The reduced peak intensity is attributed to the low concentration of clay in the samples. Shifting of the peak from higher diffraction angle to lower diffraction angle has been stated by different researchers (Lee and Kim 2009; Ravindra Reddy et al. 2010).

5 Conclusion

Matrix type, wood flour and nanoclay loading have significant effect on hygroscopic characteristics of PP based WPCs. Composites based on rPP had significantly lower water absorption and thickness swelling compared to vPP based ones. Wood flour is not completely encapsulated in the polymer matrix especially at 50 % wood weight

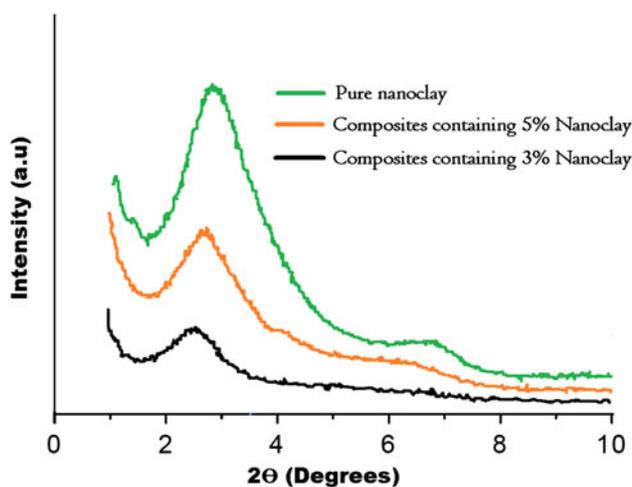


Fig. 6 X-ray diffraction patterns of clay (cloisite 15A) and its polypropylene composites with wood flour

Abb. 6 Röntgenstrahlenbeugungskurven von Ton (Cloisite 15A) und der Holz-Polypropylen-Verbundwerkstoffe mit unterschiedlichem Tonanteil

fraction. Water absorption and thickness swelling of the composites decreased with incorporation of nanoclay to the composite. The SEM micrographs confirmed the poor interfacial bonding for specimens containing higher amount of fillers (50 % wood flour and 5 % nanoclay). XRD data showed that the order of intercalation and relative intercalation in the case of composites containing 3 % nanoclay is higher than for 5 % nanoclay. Consequently, PP recycled from post-consumer applications can be used in value added composites without going the expense of separating out impurities from the polymer.

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