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Janis Kajaks*, Karlis Kalnins, Sandris Uzulis, and Juris Matvejs

Some Exploitation Properties of Wood Plastic Hybrid Composites Based on Polypropylene and Plywood Production Waste

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Abstract: During the last 20–30 years many researchers have paid attention to the studies of properties of the wood polymer composites (WPC). A lot of works are closely related to investigations of exploitation properties of wood fibres or wood flour containing polyolefine composites [1, 2]. The most useful from wide selection of polyolefines are polypropylenes, but timber industry waste materials comprising lignocellulose fibres are often used as reinforcement of WPC [3–12]. Plywood industry is not an exception – part of waste materials (by-products) are used for heat energy, *i.e.* burned. In this work we have approbated reinforcing of polypropylene (PP) with one of the plywood industry by-products, such as birch plywood sawdust (PSWD), which contains wood fibre fractions with different length [13]. The main fraction (50%) includes fibres with length l = 0.5 – 1 mm. Our previous study [13] has confirmed that PSWD is a promising filler for PP reinforcing. Addition of PSWD up to 40–50 wt.% has increased WPC tensile and flexural modulus, but decreased deformation ability of PP matrix, impact strength, water resistance and fluidity of composite melts. It was shown [13] that modification of the composites with interfacial modifier – coupling agent maleated polypropylene (MAPP content up to 5–7 wt.%) considerably improved all the abovementioned properties. SEM investigations also confirmed positive action of coupling agent on strengthening of adhesion interaction between components wood and PP matrix. Another way how to make better properties of the WPC is to form hybridcomposites [1, 14–24]. Very popular WPC modifiers are nanoparticle additions like organonanoclays, which increase WPC physical-mechanical properties - microhardness, water resistance and diminish barrier properties and combustibility [1, 2, 14–17, 19, 20]. The goal of this study was to investigate organonanoclays influence on plywood production industry by-product birch plywood sawdust (PSWD) containing polypropylene wood hybrid composites (WPHC) physical-mechanical and other exploitation properties.

Keywords: Birch plywood industry by-product; polypropylene; nanoclays; hybridcomposites; physical-mechanical properties; modification; water resistance

***Corresponding Author: Janis Kajaks:** Department of Polymer Materials Technology, Riga Technical University, P. Valdena street 3, LV-1048, Riga, Latvia; Email: kajaks@ktf.rtu.lv
Karlis Kalnins: Department of Polymer Materials Technology, Riga Technical University, P. Valdena street 3, LV-1048, Riga, Latvia
Sandris Uzulis: MNKC SIA, Dzerbenes street 27, LV-1006, Riga, Latvia; Email: sandris.uzulis@finieris.lv
Juris Matvejs: MNKC SIA, Dzerbenes street 27, LV-1006, Riga, Latvia

1 Introduction

During the last 20–30 years a lot of research papers have paid attention to the studies of thermoplastic polymer composite materials based on natural fibres, wood flour, wood fibres and polyolefine matrices [1, 2]. Natural lignocellulose fibres and their agriculture and textile industry waste containing polymer materials have ecological and technological advantages as opposed to traditional polymer composites reinforced with high modulus fibres, for example glass fibres [1]. Fibres of natural origin are environmentally friendly materials and their utilization in innovative polymer composites is defined by low cost and density, as well as practically unlimited resources in nature with very fast ability to recover [1, 2]. Polymer composites containing natural lignocellulose fibres have acceptable technological properties because polymer composite melts maintain suitable fluidity [3] that allow to use for processing these compositions by traditional polymer processing methods like injection molding and extrusion. Moreover, natural fibres do not break during processing and they do not have abrasive influence on processing equipment. The most popular thermoplastic polymer matrices for this purpose are polyolefines like polypropylene [4–12]. From all polyolefines polypropylene (PP) matrix has the highest modulus of elasticity and at the same time rather low melting temperature (below 175°C), that is assumed for systems containing natural fibres. Timber industry, *e.g.* plywood production, also generates different types of waste (by-products) which actually are lignocellulose, containing materials like natural vegetable fibres. Our previous studies [13] showed that birch plywood sawdust (PSWD) is promising filler for PP reinforcing. Additions of PSWD up to 40–50 wt.% increased WPC tensile and flexural modulus, but decreased deformation ability of PP matrix, impact strength, water resistance and fluidity of composite melts. Modification of the composites with interfacial modifier maleated polypropylene (MAPP up to 5–7 wt.%) considerably improved all the above-mentioned properties. Another way how to make better properties of the WPC is to form hybridcomposites [1, 2]. Quite popular modifiers for strengthening WPC and another natural fibre containing composite materials are nanoparticle additions like organically modified nanoclays which increase WPC physical-mechanical properties, microhardness, water resistance and diminish combustibility [2, 14–17, 19, 20]. Due to nanoclay particles great ratio of length (l) and diameter (d) nanoclay and hybrid wood plastic composites have excellent barrier properties [14–16, 19, 20]. High values of the l/d and great specific surface of nanoparticles causes and effect of nanoclay containing composites significantly higher exploitation properties. Nanoclay materials are also widely available and these fillers are relatively cheap. To reach better distribution of nanoclay particles in polymer matrix and intensification of adhesive interaction between fillers and polymer, interfacial modifiers like maleated polyolefines (MAPE, MAPP) are often used [2, 10, 12, 15–17, 19, 20, 22]. Scanning electron microscopy investigations also show positive influence of MAPE and MAPP modifiers on WPC hybridcomposites properties [12, 14, 19, 20, 24]. Furthermore, it is possible to make wood fibre and nanoclay hybridcomposites by mixing fillers in polymer melts [2, 16, 18, 20, 21]. The best method is to mix components in twin-screw extruder [14]. The goal of the present work was to approbate the influence of organo nanoclay particles additions on plywood industry by-product: birch wood plywood sawdust (PSWD) containing polypropylene composites exploitation properties.

2 Materials and investigation methods

For preparing polymer composites the following materials were used. As polymer matrix was chosen polypropylene (PP) type Mosten MA-712 with melt flow index MFI = 12 g/10 min. (at temperature 230°C) and melting temperature 153°C. To improve interfacial interaction between components, maleated polypropylene (MAPP) granules type Licocene PP MA-7452 (acid number 41 mg KOH/g, T_m = 159°C) were used. As polypropylene thermal stabilizer served Hostanox 03P powder (from previous studies 1.5 wt.% from PP weight was chosen), but for PP reinforcing one type of the by-product was approbated during plywood production technology: birch plywood sawdust (PSWD). This by-product type contains more fractions with different particle and fibre sizes. PSWD contains the following fractions: fibres with length > 1 mm (5.32%), 0.5–1 mm (45.48%), 0.25–0.5 mm (25.92%), 0.1–0.25 mm (14.28%), 0.05–0.1 mm (8.52%), and dust fraction < 0.05 mm (0.48%). In PSWD fibres with length 0.5–1 mm (45.48%) prevails. PSWD also contains a small amount (2.0–2.4 wt.%) of cured phenol-formaldehyde resin dusts. For WPC composites modification was used montmorillonite modified with quarternary ammonium salt (modifier concentration 125 meq/100 g clay) organonanoclay type Cloisite 15 A (average size of the particles 2–13 microns).

Prior to mixing by-product was dried for 12–24 h at 105°C in thermostat. Samples of the composites (filler content from 20 up to 60 wt.%) were manufactured by mixing components on two-rolls mill (T = 170–175°C, t = 10–12 min.), then cooled, granulated (average size of the chips was 2–4 mm) and pressed (T = 180°C, t = 5 min., P = 2 MPa) in 0.8–1 mm thick sheets for tensile strength measurements (standard ASTM D 638M). Flexural and impact strength tests were done for standard specimens (standards EN ISO-178 and ASTM D256 M respectively) bars produced by injection moulding (T = 180°C, P = 5 MPa, t = 3.5 min.). Technological properties (fluidity) of polymer melts were estimated with melt flow index (MFI) measurements (T = 190°C, P = 2.16 kg, standard ASTM D 1238). Water resistance measurements were done according to the standard ASTM D 570-88. Microhardness (MH) of the surface of specimens was evaluated by Vikers M-41 measurements (at load 200 g). Microstructure of the fracture surface of specimens was examined with scanning electronic microscope TM 3000. Au-sputtering was used.

3 Results and discussion

Our previous studies [13] on how different types of plywood production by-products influence unstabilized virgin polypropylene physical and mechanical properties showed, that investigated composites deformation ability decreases with increasing of the filler content, but tensile modulus and flexural modulus increase with increasing of the by-product content in polypropylene. Flexural modulus increases from 1500 MPa for virgin polypropylene up to 2600–3400 MPa for filled systems. In addition, all composites have maintained fluidity good enough: MFI values at 190°C changed from 4.6 g/10 min. for virgin PP till 0.6–1.11 g/10 min. for filled systems (filler content 40–50 wt.%) what is sufficiently to process these materials with traditional polymer processing methods. For example, for needs of extrusion process, fluidity of polymer melts about 0.1–0.3 g/10 min., but for compression moulding it could be lower than 0.1 g/10 min. Modification of the composites with interfacial modifier maleated polypropylene waxes (MAPP) considerably increases the tensile strength (2–2.2 times) and modulus, the flexural strength (1.7–2.2 times) and modulus and the impact strength (1.5–2.2 times) to compare with unmodified systems. Simultaneously, deformation ability of materials with increase of MAPP concentration in composites changes insignificantly.

Coupling agent maleated polypropylene additions remarkably improve water resistance of all investigated composites. The best water resistance was demonstrated by composites containing plywood sanding dust (PSD) and 5 wt.% MAPP. SEM studies confirmed the strengthening effects of the interfacial adhesion between wood fillers and PP matrix as a result of the action of the MAPP that evidently gives considerable increase of exploitation properties of the WPC [13].

Bearing in mind previous experimental results [13], the optimal content of the filler in composites was chosen for plywood sawdust (PSWD) exactly filler concentration 40–50 wt.%. Previous studies [13] also showed that all composites are heterogeneous systems. It means that fibres have disposition to make agglomerates during mixing process and their distribution in polymer matrix is very irregular [2]. Moreover, in filled unmodified polymer composites weak interaction on the surface between filler and polymer matrix is often observed [1–5]. In order to stabilize all chosen systems, promote dispersion of fibres in polypropylene and improve interaction between components in presented studies, optimal compositions of the composites were stabilized with thermal and oxidation stabilizer Hostanox 03 P (from preliminary investigations stabilizer concentration was chosen 1.5 wt.% from PP weight) and as interfacial modifier, one of the most effective and widely used for similar natural fibre, containing polypropylene composites maleated polypropylene waxes (MAPP) type Licocene PP MA 7452 TP granules was selected. This modifier not only improved interfacial interaction between components of the composites, but often also promoted better spread of the fillers in the volume of the polymer matrix [6–13]. The most often used concentrations of maleated polyolefin waxes as modifiers in polyolefin/wood composites are 2–8 wt.%. In our experiments [13] optimal concentration of the MAPP was 5–7 wt.%. In the beginning we approbated how addition of nanoclay particles influence previously noted optimal composition of the composite PP + 40 wt.% PSWD + 7 wt.% MAPP melt fluidity (see Table 1).

Table 1: Melt flow index (MFI) measurement results for the composites PP + 40 wt.% PSWD + 7 wt.% MAPP + nanoclay Cloisite 15A.

Content of Cloisite 15A	0	1.5 wt.%	3 wt.%	5 wt.%	7 wt.%
MFI, g/10min.	1.10	2.93	6.31	4.90	3.40

Presented results of MFI experiments show, that there exist optimal concentration of nanoclay content (3 wt.%) at which composite melts have the best fluidity and smallest viscosity. Numerical value of MFI in this case is 6.31 g/10 min that allows to process this composite by traditional polymer processing methods: extrusion and injection moulding simultaneously. That gives opportunity to considerably increase the variety of the products assortment. Therefore, for the further investigations we have chosen exactly this composition of the composite. The next step was to determine the influence of the PSWD on tensile and flexural properties of these composites (see Figure 1 and Figure 2).

From presented results it can be seen that the highest values of flexural and tensile strength (Figure 1, curves 1 and 2) shows system with 40 wt.% PSWD + 7 wt.% MAPP + 3 wt.% Cloisite 15A. Tensile modulus stays on the same level if the PSWD content increases from 40 up to 60 wt.% (Figure 2, curve 2). Only flexural modulus growth from approximately 3600 MPa till 4250 MPa was observed (Figure 2, curve 1). At the same time it is necessary to note that deformation ability of the composites decreases till 1.5% (in flexural experiments) and 2.3% (tensile experiments) with increasing of the PSWD content up to 60 wt.%. These results also show that PSWD particles obviously diminish mobility of macromolecules of the polypropylene

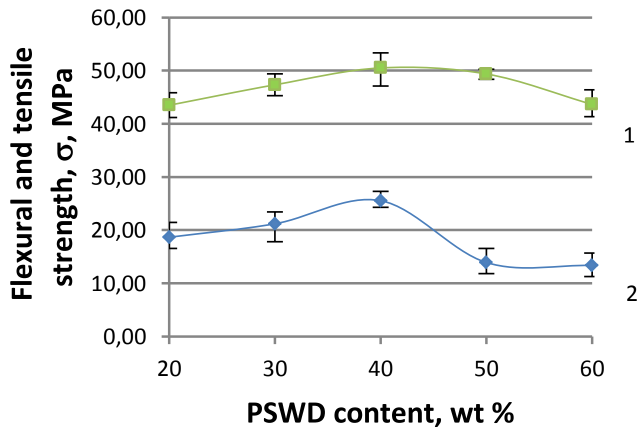


Figure 1: Dependence of flexural (1) and tensile (2) strength of polypropylene (PP) composites on the content of PSWD. Content of the interfacial modifier maleated polypropylene (MAPP) 7 wt.% and nanoclay Cloisite 15A 3 wt.%.

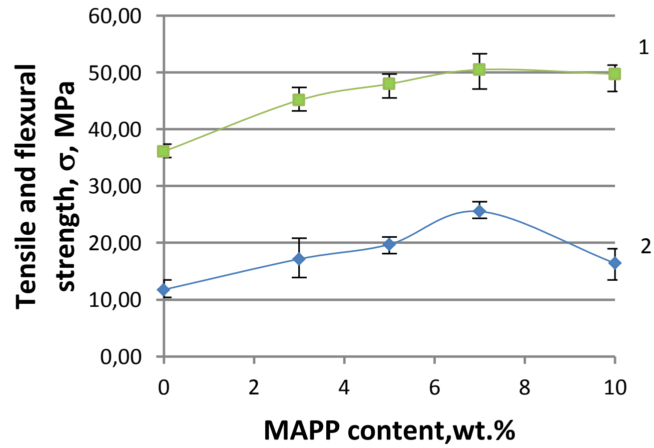


Figure 3: Dependence of flexural (1) and tensile (2) strength of polypropylene (PP) composites on the content of the interfacial modifier maleated polypropylene (MAPP). Content of the PSWD 40 wt.% and nanoclay Cloisite 15A 3 wt.%.

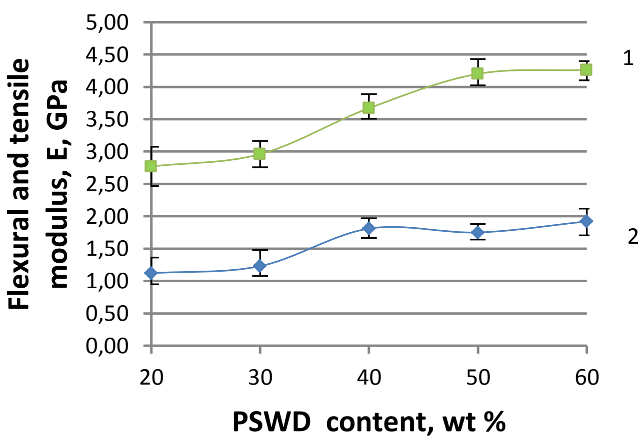


Figure 2: Dependence of flexural (1) and tensile (2) modulus of polypropylene (PP) composites on the content of PSWD. Content of the interfacial modifier maleated polypropylene (MAPP) 7 wt.% and nanoclay Cloisite 15A 3 wt.%.

matrix that could cause increasing of the flexural modulus if composites contain more than 40 wt.% PSWD. In the case of the tensile modulus measurements may be the main role begins to play with the increase of heterogeneity of the composites with 60 wt.% PSWD like in [13], what inevitably causes diminishing of the physical-mechanical properties of the WPHC.

Considering that composite with 40 wt.% PSWD also has the best fluidity, this system was selected for further studies. For determination of the MAPP content influence on flexural and tensile properties (see Figure 3) composites with different MAPP concentration were investigated. From these measurements we ascertained the fact that 7 wt.% MAPP content is the most optimal for composite with 40 wt.% PSWD + 3 wt.%. Cloisite 15A. At this MAPP concentration the maximum values of tensile strength (27 MPa, Figure 3, curve 2) and flexural strength (50 MPa, curve 1) are reached. The increase of these parameters are considerable up to 1.5–2 times. Small decrease of the flexural strength and considerable decrease (from 25.5 MPa till 16.4 MPa) of the tensile strength for composites with 10 wt.% MAPP can be explained by the plasticizing effect of the rest of interfacial modifier, which is not involved in interfacial interaction processes between components of the composite. As a confirmation of the described relationship we can mention our observations about increase of the deformation ability (from 2% up to 3%) and decrease (up to 5–10%) of elasticity of the modulus with increasing MAPP concentration up to 10 wt%. It means, that 7 wt.% of MAPP additions are enough for reaching good homogenization effect of the composites and high interfacial adhesion between components. Evidently previous determined optimal composition of the wood/ polypropylene hybrid composite (WPHC) from the point of view of the physical-mechanical properties is noted rather correctly. Summarizing previous studies, such improvements in the physical-mechanical properties of all filled materials could be explained by decreasing heterogeneity of WPC and as a result of the maleated polypropylene positive effect to increase the interfacial interaction on the surface of the fibres. MAPP also promotes better distribution of the fibres in polymer matrix [13, 19]. Optimal content of the MAPP in investigated WPHC could be 5–7 wt.%.

In order to clarify influence of nanoclay content on tensile and bending properties of WPC, concentration of Cloisite 15A in composites was varied. As an example (see Figure 4), changes of tensile and flexural modulus are pre-

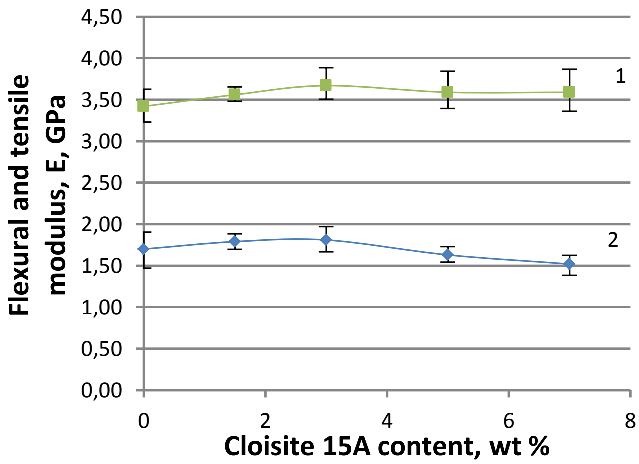


Figure 4: Dependence of flexural (1) and tensile (2) modulus of polypropylene (PP) composites on the content of Cloisite 15A. Content of PSWD 40 wt.% and interfacial modifier maleated polypropylene (MAPP) 7 wt.%.

Table 2: Impact strength (A) measurement results for polypropylene hybrid composites modified with PSWD + MAPP and 3 wt.% nano clays Cloisite 15A

7 wt.% MAPP + PSWD, wt.%					40 wt.% PSWD + MAPP, wt.%			
20	30	40	50	60	3	5	7	10
6.00	6.02	5.31	3.95	2.75	4.04	4.47	5.31	5.15

sented. In both cases, *i.e.* flexural modulus (curve 1) and tensile modulus (curve 2), small increasing of the modulus at 3 wt.% content of nanoclays in composites was observed. That again confirms the fact, that nanoclay addition improves the physical-mechanical properties of WPC. Insignificant decreasing of flexural modulus (curve 1) and small diminishing of tensile modulus (curve 2) could be explained by increasing irregular dispersion of nanoclay particles in polypropylene matrix, what extends heterogeneity of the wood plastic hybrid composites. This phenomena partly confirms our SEM investigations (see Figures 6b–6c) and experience of another researchers [15–20]. The main challenge for preparation of good quality WPHC is to get even distribution of nanoparticles in polymer matrix melts, but it is not always possible.

Very important parameter of wood plastic hybrid composites is impact strength. Results of these measurements are presented in Table 2.

Impact strength (A) measurements show that A value diminishes with increasing of the PSWD content in polypropylene hybrid composites and increases with addition of the MAPP up to 7 wt.%. For composites with 40 wt.% only small (about 11.8%) decrease of A values was observed, but for systems with 50 and 60 wt.% PSWD considerable decrease of the impact strength (corresponding about 25.6% and 56.2%) was observed compare to compositions with 20 wt.% PSWD. It means, that highly filled composites become more brittle and heterogeneity of materials increases. MAPP additions shows an opposite effect. The maximum numerical values of the impact strength are demostrated by systems with 7 wt.% MAPP. This fact once more confirms supposition regarding the plastisizing effect of the MAPP on wood plastic hybrid composites and chosen composition: polypropylene + 40 wt.% PSWD + 7 wt.% MAPP + 3 wt.% Cloisite 15A (**1**) compound is more or less optimal. Changes of the Cloisite 15A content in composite (**1**) revealed that additions of nanoclays promote diminishing of the impact strength. For example, from 9.62 kJ/m^2 (for system without nanoclays) till 5.31 and 4.01 kJ/m^2 for composites with 3 and 7 wt.% nanoclays, respectively. Results of our investigations coincide with another researchers [23] which also observed negative influence of nanoclays additions on the impact strength of WPHC. This situation forms if even distribution of nanoclay particles in polymer matrix melts has not been managed. The mentioned presumption partly also explains our SEM investigations (see pictures in Figure 6b–6c).

Frequently, for products from WPC and WPHC it is important to know their surface hardness. For example, facade and floor boards, products for automotive applications [17] and *et al.* Therefore, investigations on influence of nanoclay additions on chosen polypropylene hybrid composite microhardness (MH) also were done. Obtained results showed that nanoclay noteworthy increase surface MH of the composite samples in the rank: pure PP (MH = 87.12 MPA), PP + 40 wt.% PSWD + 7 wt.% MAPP (170.92 MPa), PP + 40 wt.% PSWD + 7 wt.% MAPP + 3 wt.% Cloisite 15A (187.2 MPa) and maximum PP + 40 wt.% PSWD + 7 wt.% MAPP + 5 wt.% Cloisite 15A (195.54 MPa). At the content of nanoclay 7.5 wt.% microhardness of the WPHC diminishes. In this case medium MH value is only 175 MPa. Similar positive influence of the nano particle additions on composite microhardness was also observed in another research [17]. Authors revealed that MH of the wood plastic hybrid composites increase up to 20%. In addition, plant fibre plastic hybrid composites surface microhardness improves if nanoparticles are better distributed in polymer matrix [15].

Adding wood to thermoplastic matrix inevitably causes an increase in moisture uptake that limits the use of these materials in exposed environments [13, 19]. Therefore, in water absorption and swell experiments it

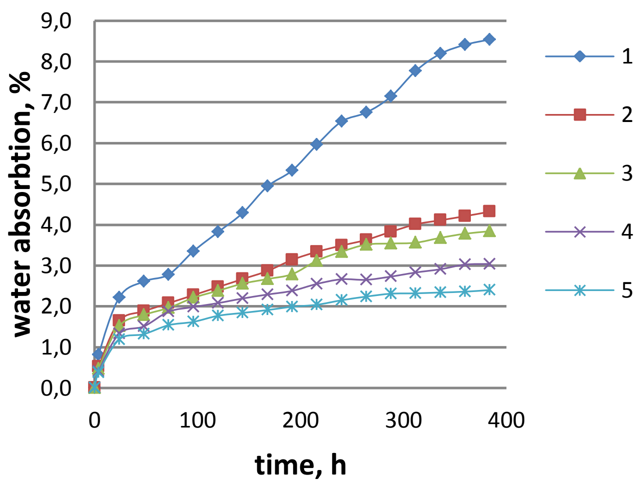
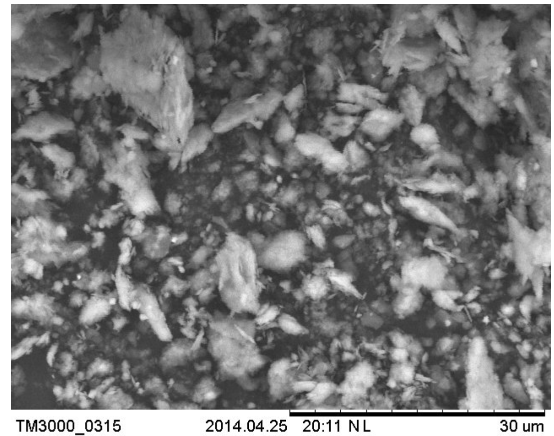


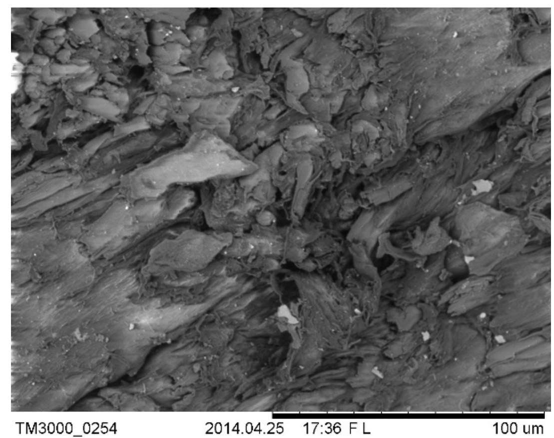
Figure 5: Water absorption kinetics of polypropylene (PP) composites. 1– PP + 40 wt.% PSWD, 2– PP + 40 wt.% PWSD + 3 wt.% MAPP, 3– PP + 40 wt.% PSWD + 5 wt.% MAPP, 4– PP + 40 wt.% PSWD + 7 wt.% MAPP, 5– PP + 40 wt.% PSWD + 10 wt.% MAPP. Content of Cloisite 15 A 3 wt.%. (curves 2–5).

is very important to evaluate behaviour of wood, containing thermoplastic composites at elevated moisture conditions. Results of water absorption tests are presented in Figure 5. Figure 5 show that MAPP addition remarkably improves water resistance of all investigated composites. During 400 h water exposure the absorbed water amount considerably decreases from 8.5% (unmodified WPHC, curve 1) till 2.3% (WPHC with 10 wt.% MAPP, curve 5). Water absorption decrease indicates that coupling agent MAPP strengthens interfacial adhesion between components [19].These results agree with our previous outcomes [13]. Sample thickness swelling also is small at presence of the MAPP. Thickness of the samples fluctuate in the range of 1–2% like in [13].

Authors [19, 24] also have shown that nano particles improve water resistance of WPHC because nanoclays have excellent barrier properties. Our studies do not confirm essential influence of nanoclay additions on composite PP + 40 wt.% PSWD + 7 wt.% MAPP water absorption properties. Composite modified with Cloisite 15A (concentration from 1.5 up to 7 wt.%) did not show considerable decrease in moisture uptake. Absorbed water amount changes were observed from 2.9 to 3.3% (after 400 h water exposure). Obviously, in our investigated WPHC composites as in [19, 20] had occured only intercalation, not exfoliation [19] of nanoclay particles in polymer matrix melts and in this case nanoclays do not show so good barrier properties than in the case of exfoliation of nanoparticles [1].



(a)



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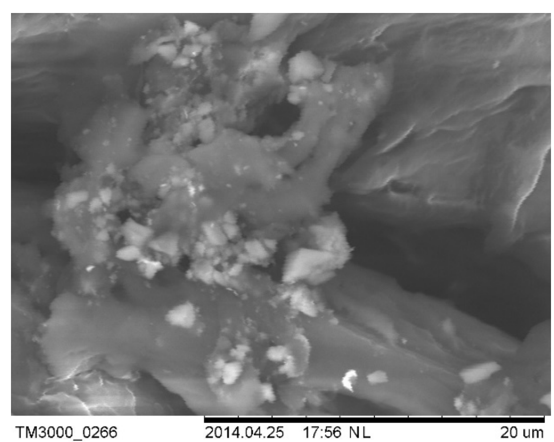

(c)

Figure 6: Scanning electron microscopy pictures (SEM) of the fracture surface of specimens made from different composites: (a) – pure Cloisite 15 A particles (magnification 2500 X), (b) – composite: PP + 40 wt.% PSWD + 7 wt.% MAPP + 3 wt.% Cloisite 15A (magnification 800X), (c) – composite: PP + 40 wt.% PSWD + 7 wt.% MAPP + 3 wt.% Cloisite 15A (magnification 5000X).

In order to determine coupling agent influence on strengthening effect of composites and fillers distribution degree in polymer matrix, a lot of researchers use scanning electron microscopy (SEM) investigations [13, 19, 24]. Some pictures from our SEM TM experimental studies are presented in Figure 6a – Cloisite 15A, 6b – modified (with 7 wt.% MAPP + 3 wt.% Cloisite 15A + PP + 40 wt.% PSWD) composite. Looking over SEM TM picture (Figure 6a) of pure Cloisite 15A particles with sizes up to 15 microns are seen. This picture show that nanoclay particles dot form agglomerates, but these particles necessary to destroy in smaller itercalated nanoclay particles, during preparing WPHC. Looking over replicas of the fracture surface of modified material sample at different magnifications (800 times, Figure 6b and 5000 times, Figure 6c), also smaller nanoclay particles (lighter regions) are observed. Furthermore, sizes and location of these regions are different. In Figure 6b partially uncovered fibre particles are observed. That indicates poor interfacial interaction between fillers and polypropylene. Fracture mechanism examination has shown prominent fiber pull-out from polymer matrix [6, 8, 12, 13, 20, 21]. Besides, fiber and more fine nanoclay particles distribution in polymer is irregular.

From previous studies and SEM TM pictures it is possible to conclude that made WPHC composites do not contain exfoliated nanoclay flat plates shape particles and in better case prepared composites are WPHC composites with intercalated nanoclay particles [1, 15, 16]. Therefore, the main problem is how to get exfoliated nanoclay particles distribution in polypropylene matrix melts [1, 14–16].

SEM studies once more confirmed the fact of strengthening the interfacial adhesion between wood fillers, nanoclay particles and PP matrix as a result of the action of the MAPP [1, 13, 16], that evidently gives the most considerable increase in exploitation properties of the wood polypropylene hybrid composites (WPHC).

4 Conclusions

1. Wood polypropylene hybrid composites (WPHC) physical mechanical properties studies showed that modification of the composites with different content of PSWD up to 40 wt.% causes increase of tensile, flexural strength and modulus. Interfacial modifier maleated polypropylene waxes (MAPP) also facilitate increasing of the tensile and flexural strength and modulus. Decrease of impact strength with increase of fiber content and increase with loading in composites MAPP additions to compare to unmodified systems. Simultaneously, small increase of flexural modulus of materials with increase of nanoclays concentration in composites was observed.
2. Coupling agent maleated polypropylene (MAPP) additions remarkably improve water resistance of all investigated composites. The best water resistance was demonstrated by composite, containing polypropylene, 40 wt.% plywood sawdust (PSWD), 3 wt.% nanoclays and 10 wt.% MAPP.
3. Melt flow index investigation showed that the best fluidity is reached by polypropylene composites with 3 wt.% nanoclays + 40 wt.% PSWD + 7 wt. MAPP.
4. SEM TM studies confirmed the strengthening effects of the interfacial adhesion between wood and nanoclays fillers and PP matrix as a result of the action of the MAPP, that evidently gives considerable increase of exploitation properties of the WPHC. Moreover, distribution of filler particles in polymer matrix is irregular.
5. From results of the presented study we can conclude, that the prepared WPHC does not contain exfoliated nanoclay particles. The main problem is high quality technology of the melt mixing process during preparing WPH nanocomposite.

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