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EFFECT OF WATER ABSORPTION ON THE MECHANICAL PROPERTIES OF SILANE-TREATED WOOD FLOUR POLYPROPYLENE COMPOSITES

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ABSTRACT

The behavior of the wood flour-polypropylene composites prepared with various contents of untreated and silane-treated wood flour by immersion in water was studied, as well as the effect of the water absorbed by the samples on their mechanical properties. The highest degree of water absorption was observed for the composites containing 30 – 50 mass% alkali pre-treated wood flour. For the materials with 50 mass% untreated, silane-treated and alkali pre-treated wood flour, the water absorption measured was 8.76%, 7.84% and 13.91%, respectively, after 15 days immersion in water. The value of the thickness swelling calculated for the samples prepared with 50 mass% silane-treated wood flour – polypropylene was the lowest – 1.29%. It was proved that the absorption of water molecules results in change of the tensile profile of the thermoplastic polypropylene composites filled with hygroscopic filler.

KEYWORDS: Polypropylene, wood flour, surface treatment, water absorption, mechanical properties.

INTRODUCTION

The materials filled with natural fillers, like wood-polymer composites, are some of the most widely used composites. They are of certain interest due to their possibility to replace the classic composites. Nevertheless, the polymer-wood flour composites have some disadvantages, e.g. incompatibility between the filler and the matrix, low stability of dimensions and resistance in presence of water (Smith and Wolcott 2006) which limits their application.

Despite the fact that the hydrophobic thermoplastic matrix of polypropylene (PP) retards the water absorption, the moisture intake is a major disadvantage of the polymer-wood composite materials (Lomelí-Ramírez et al. 2009). The repeated swelling and shrinking in the course of operation leads to a decrease of their strength over time (Schirp and Wolcott 2005). For this reason, it is important to study the behavior of the composite materials containing wood particles by water absorption and the influence of the moisture in their mechanical properties.

Wang et al. (2020) reported that the quantity of absorbed water is connected with the wood powder content in the composite materials based on PP matrix. Furthermore, the same authors established that the water absorbed in the samples worsens their mechanical properties. The tensile strength and the modulus of the composites filled with wood powder decreased, compared to the dry samples (Wang et al. 2020). Stark (2001) also confirmed that the wood flour (WF)-polypropylene composites with 40% WF absorbed more moisture than these with 20% WF. The materials containing 20% WF did not show significant decrease of their properties. Only for composites immersed in a water bath the tensile properties and impact strengths decreased (Stark 2001).

Ferede (2020) however, found out that the increase of the wood flakes contents up to 40% in PP composites improved the tensile, bending, impact and compressive strengths of the composites. At wood flakes content higher than 40%, the mechanical properties worsened and the degree of water uptake increased with the increase of the amount of wood flakes. 40% content of wood flakes in the matrix the optimal water absorption and mechanical properties were achieved.

Various methods have been used to reduce the hygroscopicity of the wood particles and improve the compatibility between composites components. These include chemical modification of the wooden fillers (Pelaez-Samaniego et al. 2013) like coupling with functional silanes, enzymatic, heat, esterification, mercerization, acetylation treatment, coating the wood flour with stearic acid, etc. (Zhou 2018, Farsi 2010, Zelča et al. 2016, Sobczak et al. 2013, Al-Mutairi 2014). Most of these methods require relatively complex treatment and quite high cost.

The addition of binding agents like silanes (Chen et al. 2017, Abdelmouleh et al. 2007, Zahari et al. 2015) which contain hydrophobic alkyl group is one of the methods to improve the water repellency of the polymer-wood composites. Beside reduction of the percentage of water uptake by the PP matrix, the preliminary treatment with silanes helps improve their mechanical properties (Chen et al. 2017, Zahari et al. 2015, Ichazo et al. 2001).

In the present work, the wood flour-polypropylene composites obtained containing different amounts of untreated and silane-treated wood flour were subjected to study of their behavior by water uptake and the effect of the water absorbed in the samples on composites' mechanical properties was investigated.

MATERIAL AND METHODS

Materials

Polypropylene (Buplen 6531); wood flour (Kronospan Bulgaria ltd); sodium hydroxide and vinyltrimethoxysilane, (Sigma-Aldrich) were used.

Preparation and surface treatments of the filler

Preparation of the wood flour filler and its fraction composition were described in our previous work (Ilieva and Kiryakova 2023). Wood flour surface alkali (NaOH) and silane (vinyltrimethoxysilane) treatments were carried out according the procedures (Zahari et al. (2015), and Ichazo et al. (2001). Untreated and treated wood flour samples were designated as WF (untreated), Na-sWF (alkali-silane treated) and sWF (silane treated), respectively.

Preparation of wood flour polypropylene composites

The series of WF-PP compositions prepared with 1, 3, 5, 10, 20, 30, 40 and 50 mass% treated and untreated wood flour were mixed and pressed under the conditions described in (Ilieva and Kiryakova 2023). The composites obtained were designated according to the type of wood flour used, followed by a number that corresponds to the filler content in the PP matrix, i.e., sWF40-PP means PP composite with 40 mass% silane-treated wood flour.

Characterization of the composite materials

The water absorption (*WA*, %) after 1, 3, 6, 7, 8, 11, 13 and 15 days immersion in water and the thickness swelling (*TS*, %) values of the PP composites were calculated using the equations (Kord 2011).

The tensile properties for the initial polypropylene and its composites with untreated and silane-treated wood flour before and after immersion in water for 15 days were measured according to EN ISO 50527-1 on a dynamometer INSTRON 4203.

The hardness by Shore A was also determined for the obtained materials.

RESULTS AND DISCUSSION

One of the main parameters used as indicator of the quality of wood-polymer materials is their ability to absorb water. Water uptake is considered as disadvantage of the composite materials as it might result in disruption of the boundary surface between wood flour and PP (Venukadasula 2011, Panthapulakkal et al. 2005), affect their stability, cause changes of their mechanical properties and decrease their strength (Venukadasula 2011, Espert et al. 2004).

The influences of the wood four contents and the immersion time on the water absorption of the PP-wood flour composites obtained are shown in Fig. 1. Pure polypropylene is hydrophobic polymer and absorbs small quantities of water so the water absorption of the PP matrix can be ignored compared to that of the composites filled with WF. As can be seen in Fig. 1, the water absorption of the composites increased with the increase of filler content and immersion time (Wang et al. 2020, Espert et al. 2004, Dhakal et al. 2007, Lin et al. 2002). The increased water absorption was expected since the absorption of composites obtained with non-polar polymers depends on the type and nature of the fillers they contain. With the increase of the wood flour content in the composite, the amount of –OH groups present in wood particles structure also increases. The hydrophilic WF absorb water by forming hydrogen bonds between the water molecules and the –OH on WF surface (Ferede 2020, Das et al. 2018, Turku et al. 2017, Kaewkuk et al. 2013) and thus they are responsible for the increased water absorption.

PP samples containing untreated (WF) and silane-treated wood flour (Na-sWF and sWF), showed water uptake curves of similar shape (Fig. 1). For all the samples studied containing up to 20 mass% WF, regardless of the surface treatment of the filler, the dependence of the amount of absorbed water on the immersion time is almost linear. When higher amounts of these fillers were used (30, 40 and 50 mass%) in the initial stage of the *WA*, water absorption rapidly increased with the immersion time. For example, the materials WF50-PP, sWF50-PP and Na-sWF50-PP showed *WA* 4.77, 3.79 and 5.04%, respectively, after immersion time of 1 day. The same composites immersed for 3 days increased the values of their water absorption to 8.03, 7.32 and 12.75%. The increase of the same indicator for the samples from Na-sWF50-PP was 2.5 times. After immersion period of 3 days, the water absorption increased slowly. On the 15th day the *WA* determined was: 8.76% for WF50-PP (Fig. 1a), 7.84% for sWF50-PP (Fig. 1b) and 13.91% for Na-sWF50-PP (Fig. 1c).

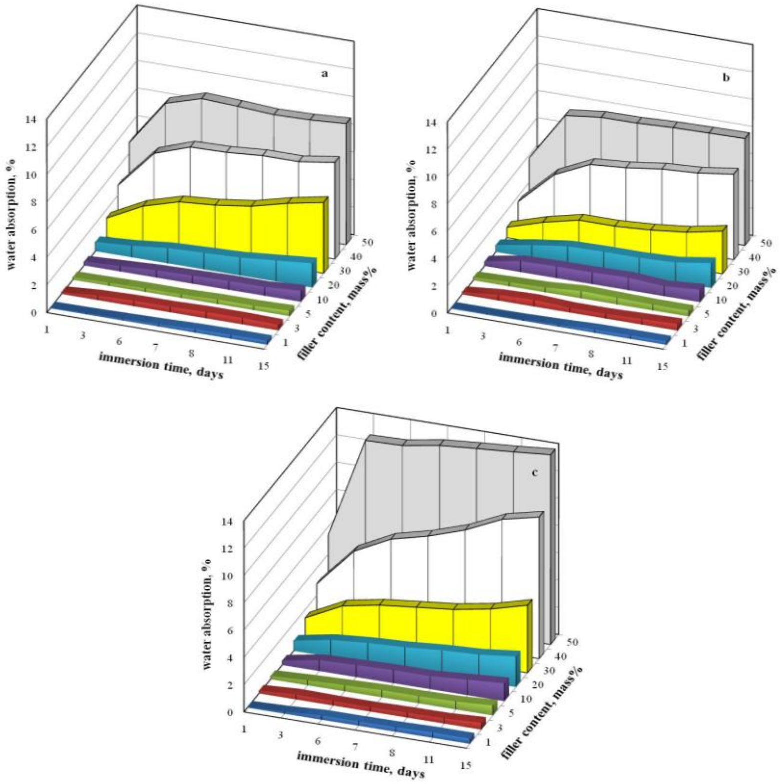

Fig. 1: Dependence of water absorption on the WF (a), sWF (b) and Na-sWF (c) content in polypropylene composites after immersion in water.

It was established that the use of only vinyltrimethoxysilane in the treatment of WF surface resulted in a decrease of the water absorption (Fig. 1b). The higher *WA* was obtained for the composites with untreated WF (Fig. 1a), while the highest degree of water absorption was observed for PP composites containing 30 – 50 mass% alkali pre-treated Na-sWF (Fig. 1c). As it has been found in our previous work, the difference in the WA can be attributed to the fact that the alkali pre-treatment of the wood flour creates porous and coarse surface, which can easily absorb water (Ilieva and Kiryakova 2023).

Tab. 1 shows the thickness swelling (*TS*) and Hardness by Shore of WF-PP, sWF-PP and Na-sWF-PP composites with 50 mass% contents of filler after immersion in water for 15 days. Results in Tab. 1 showed that the composites where the filler used was untreated WF had thickness swelling – 4.57%.

Tab. 1: Thickness swelling and hardness by Shore of wood flour polypropylene composites after immersion in water.

Parameter	50 mass% filler content		
	WF	sWF	Na-sWF
Thickness swelling, %	4.57	1.29	5.23
Hardness by Shore	89	93	91

Despite the high content of sWF filler, the use of coupling agent vinyltrimethoxysilane (VTMS) gave decreased *TS* of the samples, similar to *WA*. After immersion in water for 15 days, both indicators (*WA* and *TS*) did not exceed 8% and the calculated value of *TS* for the sWF50-PP compositions was the lowest – 1.29%. It suggests that the materials on their basis possess the best anti-swelling performance (Mohebb et al. 2010). The probable reasons for the low values of *TS* are that, on one hand, the wood flour surface is covered with a layer of VTMS and on the other hand – the wood flour is wrapped additionally in PP matrix which improves the compatibility between the matrix and sWF. In accordance with the increased *WA*, the highest thickness swelling (5.23%) was determined for the composites containing 50 mass% alkali pre-treated Na-sWF.

Since the harder materials are more resistant to friction and wearing (Kord 2011, George et al. 2001, Ghasemi and Kord 2009, Stark and Rowlands 2003), the hardness by Shore was used to estimate the wear resistance of the materials obtained. As can be seen from Tab. 1, the hardness of the composites containing 50 mass% WF was 89. The same value for the PP with silane treated WF was 93 while for Na-sWF50-PP it was 91. The differences of the Hardness by Shore observed for the materials with sWF and Na-sWF can be attributed to the treatment with NaOH which probably causes excessive delignification of the filler. It can damage the treated wood flour and negatively affect the hardness by Shore.

The studies of the water uptake of polymer composites with natural fillers report in the literature confirm that the mechanical properties of the composites are affected by the amount of absorbed water (Perrier et al. 2017, Machado et al. 2016, Shen et al. 2019, Lu and Vuure 2019, Yang et al. 2018). The influence of the WF, sWF and Na-sWF content in polypropylene composites before and after immersion in water on the tensile strength (σ, MPa) was studied and the results obtained are summarized in Fig. 2.

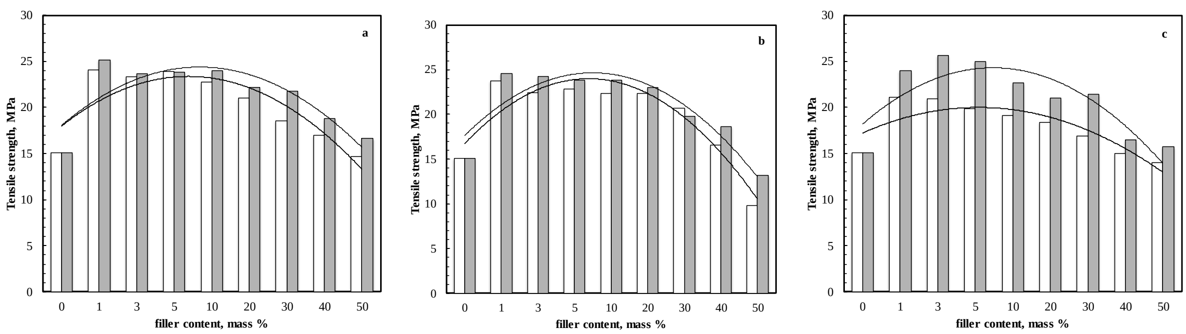

Fig. 2: Dependence of the tensile strength on the WF (a), sWF (b) and Na-sWF (c) content in polypropylene composites before □ and after ▩ immersion in water.

It can be clearly seen in Figs. 2a and b that the immersion of samples in water for 15 days did not result in noticeable change of the tensile strength of the composites containing untreated and VTMS treated wood flour. The highest values of the tensile strength of 24.08 and 23.73 MPa were measured for the composites containing 1 mass% filler (WF and sWF). The immersion in water for 15 days of the same compositions resulted in insignificant increase of the tensile strength to 25.11 and 24.60 MPa, respectively. At 40 mass% content of untreated and silane-treated WF, σ had values close to these of the initial PP (~17 MPa) and then began to decrease. More significant was the decrease of the tensile strength (to 9.78 MPa) for the materials prepared from sWF50-PP because of the water absorption – Fig. 2b. The decrease of the tensile strength of the wood flour-PP composites observed is in accordance with the results reported in the literature (Bouza et al. 2009).

It is well known that the mechanical properties of the polymer composites filled with natural fillers can be improved by modification of the filler or the polymer matrix (Wang et al. 2020, George et al. 2001, Cai et al. 2016). The surface treatment might weaken the effect of moisture on some mechanical properties (Espert et al. 2004, Wang et al. 2019). The study of the change of the tensile strength (σ, MPa) of the composites based on Na-sWF-PP revealed certain “strengthening” effect after the immersion in water for 15 days – Fig. 2c. The increase of the tensile strength may be due to absorption of water molecules resulting in stronger intermolecular interactions filler-matrix accompanied by formation of cross-links (Vlaev et al. 2009). For instance, σ of the materials containing 3 mass% Na-sWF reached maximum value of 25.63 MPa after 15 days immersion in water. For comparison, the same value for the samples of Na-sWF3-PP before immersion in water was 20.95 MPa. The strengthening effect observed persists, including for the samples with high content of filler – 30 mass%.

The elongation at break (ε, %) of the composite materials based on Na-sWF-PP (Fig. 3c) showed a tendency of weaker decrease, regardless of the water absorption. For Na-sWF1-PP and Na-sWF3-PP, before and after water absorption, ε had values of about 20 – 26% and then gradually decreased. The same value measured after introduction of 1 mass% WF or sWF in the PP matrix was smaller – 19 and 10%, respectively, Fig. 3 a and b. For all the materials studied, the use of higher amounts of filler resulted in additional decrease of the elongation at break. At 20 – 50 mass% filler content, ε had values in the interval 2 – 8% regardless of the wood flour surface treatment and 15 days immersion in water. The decrease of ε by the introduction of natural fillers in PP matrix is in accordance with the results reported in the scientific literature by other authors (Bouza et al. 2009, Demir et al. 2006).

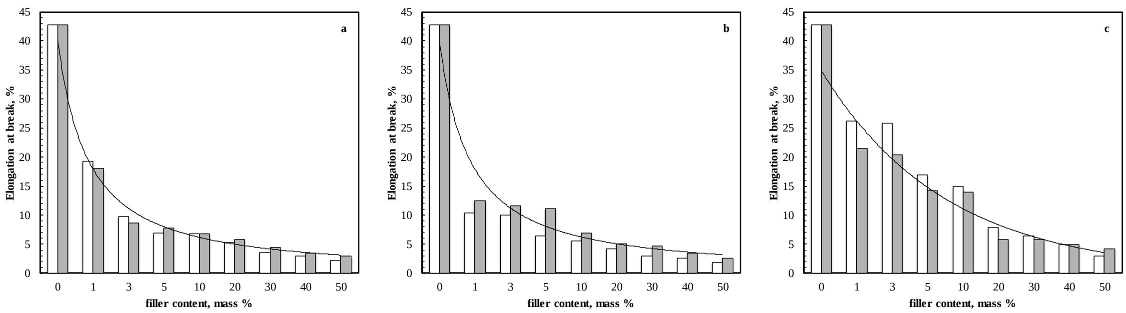

Fig. 3: Dependence of the elongation at break on the WF (a), sWF (b) and Na-sWF (c) content in polypropylene composites before □ and after ▒ immersion in water.

It is known that the values of the elasticity modulus of the filler is one of the main factors determining the modulus of the composite materials obtained (Demir et al. 2006, Ansari and Ismail 2009, Gironès et al. 2007). The high elasticity modulus of the wood flour was probably the reason for the increase of the values of the property studied for all the compositions containing non-treated and silane-treated WF – Fig. 4a and b. As can be seen in Fig. 4a, the immersion in water for 15 days had insignificant effect on the elasticity modulus of the samples. The maximum value of 1250 MPa was reached for sWF40-PP (Fig. 4b). The increase of the modulus characteristic for the materials with 50 mass% sWF allows obtaining composites of high hardness even after immersion in water (Tab. 1).

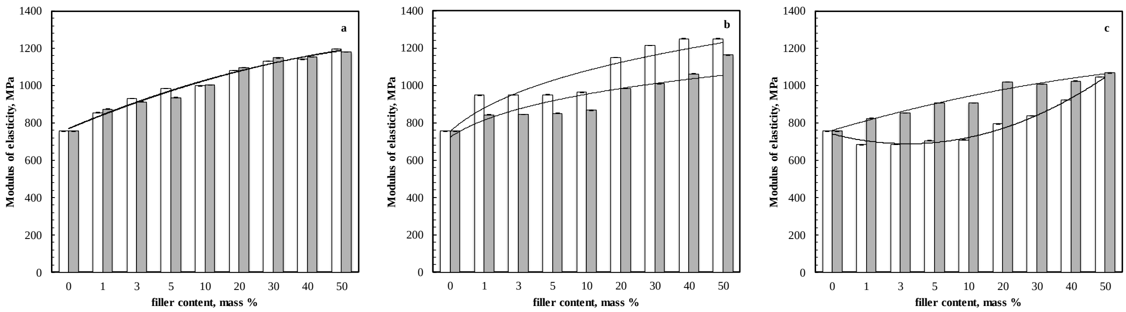

Fig. 4: Dependence of the modulus of elasticity on the WF (a), sWF (b) and Na-sWF (c) content in polypropylene composites before □ and after ■ immersion in water.

The preliminary treatment of WF with NaOH may be the reason for the lower values of the elasticity modulus (Fig. 4c) resulting from the delignification of the filler by the NaOH treatment. Only when using significantly higher amounts of Na-sWF (higher than 20 mass%) the modulus exceeded that of the polymer matrix (756 MPa). It should be noted for the Na-sWF-PP based materials that after immersion in water for 15 days the values of the modulus showed an increasing trend. The increase observed was significant for the compositions with Na-sWF content lower than 30 mass%. For instance, the elasticity modulus established for Na-sWF5-PP was 705 and 908 MPa before and after immersion, respectively. For Na-sWF40-PP, the modulus was still by 102 MPa higher than that of the same samples before immersion while at 50 mass% content of Na-sWF in the compositions the values of the modulus were very close (about 1050 MPa).

CONCLUSIONS

It was found that the use of only coupling agent, vinyltrimethoxysilane for surface treatment of the wood flour results in decrease of the water absorption of the polypropylene based compositions. The immersion of the samples in water for 15 days did not result in noticeable change of the tensile strength of the composites containing untreated and vinyltrimethoxysilane treated wood flour. The study of the change of the tensile strength of the composites with alkali pre-treated wood flour showed certain “strengthening” effect after immersing the samples in water for 15 days. For the materials with 3 mass% alkali pre-treated wood flour, for example, the tensile strength reached the maximum value of 25.63 MPa after

15 days immersion in water. For comparison, the same value before the immersion in water was 20.95 MPa. The strengthening effect observed persists including for the samples with high filler contents up to 30 mass%. The highest elasticity modulus of 1250 MPa was found for the material of polypropylene with 40 mass% silane functionalized wood flour. The increase of the elasticity modulus characteristic for the materials with 50 mass% silane-treated wood flour allows obtaining materials of high hardness even after immersion in water.

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