Scientific Paper

The Adhesion Phenomena in Polypropylene/Wollastonite Composites†

Iztok Švab,[a]* Vojko Musil,[a] and Mirela Leskovac[b]

[a] *University of Maribor, FEB Maribor, Institute of Technology, Razlagova 14, 2000 Maribor, Slovenia,*
E-mail: iztok.svab@uni-mb.si
[b] *University of Zagreb, Faculty of Chemical Engineering and Technology, Marulićev trg 19, 10000 Zagreb, Croatia*

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Abstract

Modification of polypropylene (PP) with wollastonite fillers was investigated in this paper. Three types of different silane pretreated wollastonite mineral filler were used for preparation of binary PP/wollastonite composites. The composite samples were homogenized in a Brabender Plasti-Corder kneading chamber and compression moulded into plates on a laboratory press. The adhesion between the wollastonite fillers used in this study and the PP matrix was predicted on the basis of the calculated adhesion parameters (work of adhesion, interfacial free energy and spreading coefficient) obtained by the surface free energy of pure materials. The contact angle method was used to determine surface free energy of components. The obtained values of adhesion parameters at the interface in the composites were correlated with mechanical properties as well as morphology observations of corresponding composites and were proved to be in relatively good agreement with the mechanical property measurements. Stronger adhesion in investigated composites has reflected in higher yield stress and tensile strength at break but in lower elongation at break and impact resistance.

Key words: polypropylene composites, wollastonite, surface energy, adhesion, morphology, mechanical properties

Introduction

Although the use of fillers in plastics industry has been known for several decades, there is still a huge interest in all fields (academia and industry) for the development of composite materials with enhanced properties. Especially high tech products accelerate the research of advanced composites, which demand extensive knowledge of all the factors that determine final properties of polymeric composite materials. Polypropylene (PP) is one of the most widely used commodity thermoplastics, especially in automotive, electric, packaging and consumer application. The reason for this can be found in its excellent properties, such as good processing, heat distortion temperature above 100 °C, recycling ability and favourable price/performance ratio. Despite its exquisite properties, the PP is very often modified with particulate fillers and other polymers. The most common fillers used in the PP are talc, calcium carbonate, glass beads, glass fibers, mica, silica and wollastonite. Among these fillers, the wollastonite of needle shaped structure provides many processing and performance benefits, including increased stiffness and strength. Wollastonite is a calcium silicate ($CaSiO_3$), which is used as functional filler in plastics. It is the only naturally occurring white mineral which is wholly acicular. The structure of the wollastonite particles, i.e. their aspect ratio, depends not only on natural conditions but also, to a larger extent on the preparation and size reduction techniques employed.[1–3]

Mineral particulate fillers are not only used in polyolefinic composites as substitutes of a relatively costly bulk material, but are also used to improve some properties of neat polymers. The properties of the composites depend upon the characteristics of components, composition, structure and interfacial interactions. The latter factor is affected by the size of the interface and the strength of the interaction. Both, the interface and strength of the interaction could be modified to improve the wettability and adhesion between the components by surface treatment with different modifiers such as stearic acid, silane and titanate coupling agents. Silanes are by far the most popular coupling agents for filler surface treatment.[4,5] Wetting of fillers and the adhesion between the filler and the matrix is governed by the principles of the theory of adhesion based on the surface energy properties of the filler and the matrix, respectively. For this reason the measurements of the surface properties of pure materials make it possible to predict the interfacial adhesion between the filler and the matrix in composites.[6–9] To consider the characteristics of components, which influence the interfacial adhesion between the filler and polymer in the corresponding composites, the final performance of the composite material could be determined.[10–12]

The purpose of this work was to relate the mechanical properties of the PP/wollastonite composites with the surface characteristics of pure components in the composites. We considered the effect of the wollastonite filler treated with different silane coupling agents on the adhesion between the filler and the matrix and therefore on the final PP/wollastonite composite properties.

Experimental

Materials

Polypropylene (PP) used in this study was a commercial homopolymer Moplen HP501L supplied by Basell with melt flow rate (MFR) of 6.0 g/10 min, $\rho = 0.90$ g/cm^3 and $M_n = 120{,}000$ g/mol. Applied mineral fillers were proprietary (combination of silanes) surface treated wollastonite Tremin 939 300 ZST (mark W1), aminosilane surface treated wollastonite Tremin 939 300 AST (mark W2) and alkylsilane surface treated wollastonite Tremin 939 300 FST (mark W3). All types of wollastonite used with the following general characteristics $\rho = 2.85$ g/cm^3, d(50%) = 10 µm and specific surface area = 1.2 m^2/g are commercially available and were kindly supplied from Quarzwerke (Germany).

Sample preparation

Binary PP/wollastonite composites were prepared in a Brabender Plasti-Corder kneading chamber. The content of wollastonite in composites was 2, 4, 8, 12 and 16 vol.%. The components with certain proportion were kneaded for 7 min, in a chamber preheated to 200 °C, with a rotor speed of 50 min^{-1}. After kneading, the melt was rapidly transferred to a preheated laboratory press and compression molded into 1- and 4-mm thick plates. The pressing temperature was 220 °C, the pressure 100 bar and the pressing time 14 min for 1-mm, and 11.5 min for 4-mm thick plates. The plates were used for specimen preparation for morphology observation and mechanical testing.

Methods

Contact angle measurements

Surface free energies of materials were determined throughout the measurements of contact angles. Contact angles on the film of the PP and on compacts of the wollastonite fillers were measured on a DataPhysics OCA 20 Instrument. Sessile drops (2µL) of test liquids; water (distilled twice $\lambda = 1.33$ µLcm^{-1}), formamide (p.a. 99.5%, Fluka) and diiodomethane (p.a. 99%, Aldrich), at 23 °C were used for the advancing contact angle measurements. We determined the contact angle from 10 to 30 s after drop deposition.

Average values of at least five drops on different places of the same sample were taken and standard deviation was always less than 2°. Surface free energies of the PP and wollastonite fillers were calculated using harmonic mean equation:[13]

$$\gamma_{lv}(1+\cos\theta) = \frac{4\gamma_s^d\gamma_{lv}^d}{\gamma_s^d+\gamma_{lv}^d} + \frac{4\gamma_s^p\gamma_{lv}^p}{\gamma_s^p+\gamma_{lv}^p} \qquad (1)$$

where γ^d is the dispersive and γ^p the polar component of the surface free energy (surface tension), γ_{lv} and γ_s are the surface tension of liquid and surface free energy of solid respectively, while θ is the contact angle.

This evaluation method was integrated in the software (SCA 20) and was automatically carried out by the computer. The method requires the use of at least two test liquids with known surface tensions and their polar and dispersive components. Each additional liquid increases the accuracy of estimation. Surface tensions of the test liquids used for contact angle measurements are presented in Table 1.

Table 1. Surface tensions (γ_{lv}) and their dispersive (γ_{lv}^d) and polar (γ_{lv}^p) components of test liquids.

	Surface tension (mJ/m^2)		
Liquid [14]	γ_{lv}^d	γ_{lv}^p	γ_{lv}
Water	21.8	51.0	72.8
Formamide	39.0	19.0	58.0
Diiodomethane	50.8	0.0	50.8

Scanning electron microscopy

Scanning electron microscope (SEM) JEOL JSM-840A was used for the morphology observation. The samples were fractured in liquid nitrogen before they were covered with gold and examined microscopically, at an acceleration voltage of 5 kV.

Mechanical testing

Tensile properties (Young`s modulus, yield stress, tensile strength at break, elongation at break) were measured using a Zwick 147670 Z100/SN5A apparatus at 23 °C and strain rate of 2 mm/min according to ISO 527. Notched impact strength was measured according to the Charpy test (DIN 53453), using the Zwick apparatus at 25 °C.

Results and discussion

Adhesion phenomena in composites

Properties of the composites are strongly affected by the interface and/or interphase. The knowledge about the surface free energy of fillers and polymers is essential for optimising the interface and/or interphase properties of the composites. Kolarik et al.[15–17] have clearly indicated the importance of surface free energies for the resulting phase structure.

Because of the critical importance of the interface and interphase in composites, the study of adhesion is relevant to composite engineering.[18] The calculation of adhesion parameters such as the thermodynamic reversible work of adhesion (W_{mf}), interfacial free energy (γ_{mf}) and spreading coefficient (coefficient of wetting) (S_{mf}), represented in equations 2, 3, and 4 respectively,[19] enables us to predict the strength of interactions at the filler/matrix interface and to correlate them with mechanical properties of the composites.

$$W_{mf} = \gamma_f + \gamma_m - \gamma_{mf} \qquad (2)$$

$$\gamma_{mf} = \gamma_f + \gamma_m - 2(\gamma_f^d \cdot \gamma_m^d)^{1/2} - 2(\gamma_f^p \cdot \gamma_m^p)^{1/2} \qquad (3)$$

$$S_{mf} = \gamma_f - \gamma_m - \gamma_{mf} \qquad (4)$$

where subscripts *m* and *f* mean matrix and filler, respectively.

The results of the studies on the effective adhesion for a given system indicate some conditions as optimal: thermodynamic work of adhesion as a maximal, spreading coefficient as a positive value and interfacial free energy as a minimal (tends to null).[19]

In this study, contact angle measurements were used for the surface characterization of the filler and the polymer matrix. The results of surface free energies of the PP matrix and different wollastonite fillers are given in Table 2.

Table 2. The surface free energies (γ_s) and their dispersive (γ_s^d) and polar (γ_s^p) components of the PP matrix and wollastonite fillers.

Sample	Surface free energy (mJ/m^2)		
	γ_s^d	γ_s^p	γ_s
PP	31.5	1.3	32.8
W1	36.9	29.0	65.9
W2	37.4	33.5	70.9
W3	3.1	0.0	3.1

The PP matrix has an expected low polar component value of the surface free energy (Table 2), as a consequence of non-polar nature of the PP surface. As shown in Table 2, wollastonite fillers of proprietary (W1) and aminosilane surface treatment (W2) have a higher surface free energy compared to the filler treated with alkylsilane (W3). Higher values of dispersive and polar components of wollastonite fillers W1 and W2 could be correlated with the presence of functional groups (such as H_2N- in aminosilane coupling agent) at the filler surface. On the other hand, because of low wetting of filler W3 with different test liquids the contact angles were higher than 90°, which resulted in an unexpected low value of surface free energy calculated from the Equation 1. Obtained surface free energies of the components were used to calculate the adhesion parameters of the corresponding composites.

The results in Table 3 illustrate the adhesion phenomena at the interface in the PP composite with different wollastonite fillers.

Table 3. Adhesion parameters of the PP/wollastonite composites.

Composite	Adhesion parameters (mJ/m^2)		
	Work of adhesion W_{mf}	Interfacial free energy γ_{mf}	Spreading coefficient S_{mf}
PP/W1	80.3	18.4	14.7
PP/W2	81.7	22.0	16.0
PP/W3	19.8	16.1	–45.9

Higher work of adhesion value (W_{mf}) at the PP/W1 and PP/W2 than at PP/W3 composites implies higher interactions at the interface in these composites. Considerably lower value of work of adhesion for the PP/W3 composite (Table 3) implies low interactions at the interface, as a consequence of a very low surface free energy of the wollastonite filler treated with alkylsilane (Table 2). Positive values of the spreading coefficient (S_{mf}) (Table 3) indicate that the PP matrix wet the filler surface in the PP/W1 and PP/W2 composites. A negative value of the spreading coefficient for the PP/W3 composite could be an indicator of dewetting of the PP matrix from the filler surface and could also indicate low interactions at the interface. Similar results were obtained in the literature for the PVAc/$CaCO_3$ composites filled with untreated and with stearate treated filler, where the negative spreading coefficient was a result of the low interactions at interface and as authors emphasized that could be an indication of dewetting mechanism of failure.[20]

Taking into consideration the required conditions for effective adhesion, this is proved to be in case of PP/W2 composites. Therefore, the PP/W2 composites should exhibit better mechanical properties in comparison with the PP/W1 and PP/W3 composites.

Morphology

Properties of the polymer composites also depend on structural characteristics at a higher supermolecular level, especially on their phase morphology. For this reason, SEM observations were used for studying the morphology of the composites. Figure 1 (a, b and c) presents morphologies of fractured surfaces of the PP/W1, PP/W2 and PP/W3 composites, respectively. From all SEM micrographs, typically acicular, needle-shaped particles of the wollastonite filler could be seen. Furthermore, the filler particles are randomly dispersed in the PP matrix and they are mostly oriented in the direction of the melt flow in all composites due to the similar processing conditions.

As shown in Figure 1c, the visible dewetting of polymer matrix from the filler surface indicates lower adhesion at polymer/filler interface in comparison with PP/W1 and PP/W2 composites. From the morphological observation it could be expected that the observed differences in composite morphology would be confirmed with mechanical measurements.

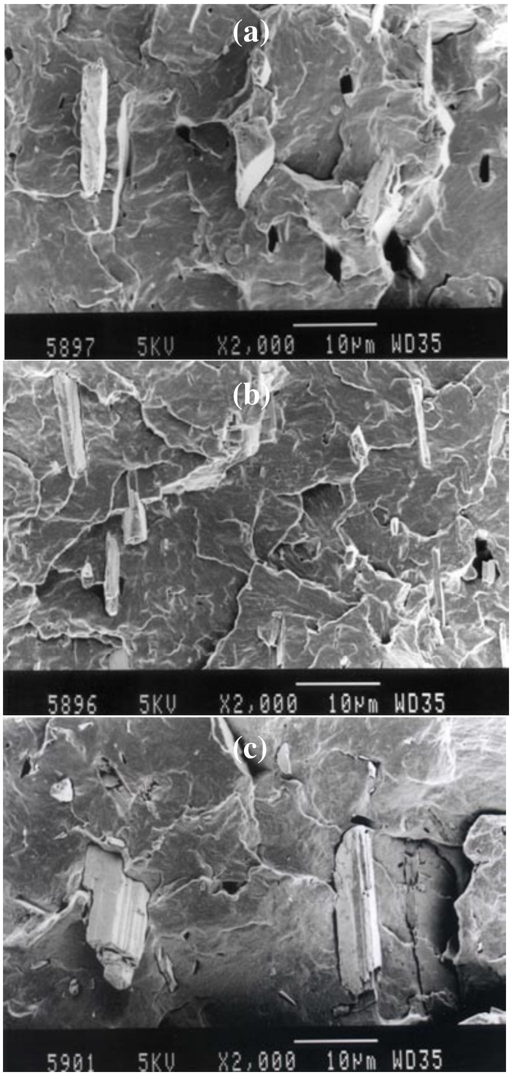

Figure 1. SEM micrographs of fractured samples of the PP/wollastonite (96/4 vol.%) composites a) PP/W1, b) PP/W2 and c) PP/W3.

Tensile properties

Young`s modulus

Interfacial interactions (adhesion) between the filler and the polymer matrix are an important factor which influences mechanical properties of the composites. The changes of the adhesion parameters in the composites as a consequence of the filler surface treatment are expected to reflect in the changes of the composite mechanical properties, presented in Figures 2–5.

Tensile properties of the composites were evaluated from the stress-strain curves. Young`s modulus is, besides yield stress, an important parameter, which characterizes the stiffness of material. The influence of the wollastonite addition to the PP matrix on Young`s modulus is illustrated in Figure 2. The composite moduli increase with the increasing wollastonite content, as would be expected because of the stiff needle-shaped wollastonite. The observed increase in modulus of the wollastonite reinforced PP can be explained in terms of reinforcement effect.[21] It can be seen that the dependence of Young`s modulus of the wollastonite content is more or less linear in the entire concentration range studied. There is only minor difference in Young`s modulus between various types of wollastonite used in this study. As was reported by others,[22] we also found out that the Young`s modulus of the composites is slightly affected by the use of the filler with different surface treatment.

The Young`s modulus data were compared with some theoretical predictions for two-phase composites. Few theoretical predictions have been derived for composites that take into account shapes, filler fraction and adhesion between the filler and the polymer matrix. Straight lines in Figure 2 were calculated by using the following equations:[9,23,24]

$$E_c = E_p(1 + 2.5\phi_f) \qquad (5)$$

$$E_c = E_p(1 + 2.5\phi_f + 14.1\phi_f^2) \qquad (6)$$

$$E_c = E_p\phi_p + E_f\phi_f \qquad (7)$$

$$E_c = \frac{E_p E_f}{E_p\phi_f + E_f\phi_p} \qquad (8)$$

where E_c, E_p and E_f are the moduli of the composite, polymer matrix and filler respectively, while ϕ_p and ϕ_f are volume fractions of the polymer and the filler. Equation 5 represents Einstein`s equation with adhesion, Equation 6 performs a modified Einstein`s viscosity equation by Guth and Gold, while Equations 7 and 8 express the parallel and series models, which represent the upper and the lower bounds of Young`s modulus prediction.

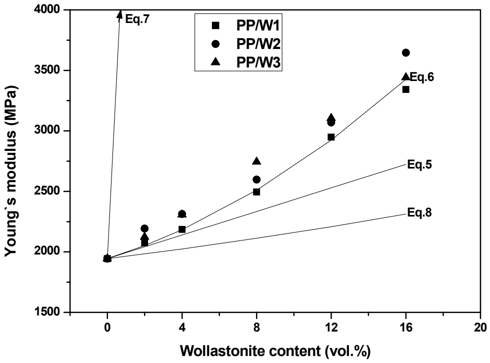

Figure 2. Comparison of experimental data with theoretical predictions of Young`s modulus for the PP/wollastonite composites in dependence of wollastonite content.

It can be seen from Figure 2 that the use of Equation 7 for the modulus prediction is the most unsuitable, especially due to very high stiffness of the wollastonite (303 GPa), resulting in very high values of predicted moduli. Equation 7 could only be applied when there is very strong adhesion between the filler and the matrix. The predicted moduli of Equations 5 and 8 possess lower values in regards to experimental values and are thus less accurate. The most suitable prediction of modulus for the PP/wollastonite composites is proven from Equation 6, where the predicted values are quite well in accordance with the experimental one.

Yield stress

Yield stress, measured at large deformations, is much more dependent on interfacial adhesion with respect to Young`s modulus, measured at small deformations. Tensile yield stress is proved to be an excellent property to correlate with interfacial interactions in heterogeneous polymer systems.[25,26] In Figure 3, the dependence of yield stress of the PP/wollastonite composites against wollastonite content is presented. The addition of different wollastonite fillers in the PP matrix results in decreasing of the yield stress values in comparison to the neat PP matrix.

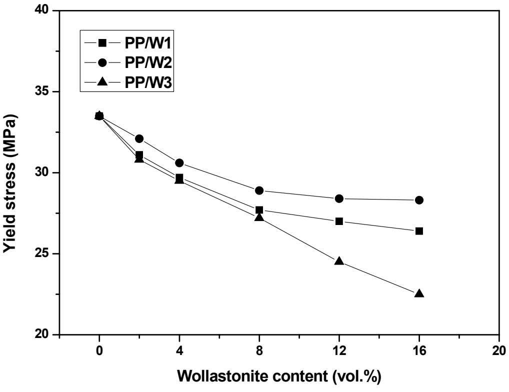

Figure 3. Yield stress of the PP/wollastonite composites in dependence of wollastonite content.

The incorporation of W1 and W2 fillers into the PP matrix results in higher yield stresses compared to the alkylsilane treated filler (W3). Similar results were obtained in literature[5] for the PP/zeolite composites, filled with untreated zeolite and a different silane treated zeolite. Better interfacial adhesion is obtained by the surface treatment of zeolite with 3-aminopropyl triethoxysilane (AMPTES) coupling agent.

As shown in Figure 3, the decreasing of yield stress is more pronounced with up to 8 vol.% of added filler, after that the decreasing is less obvious for the composites where the W1 and W2 were used. In the case of the PP/W3 composites the decreasing of yield stress is still gradual, almost linear. Possible reason for such behaviour at higher filler contents could be a more expressed interactivity on the filler/matrix interface at the PP/W1 and PP/W2 composites in comparison with the PP/W3 composites.

For the evaluation of the extent of interfacial interactions, the following equation derived by Pukanszky et al.[26,27] was used:

$$\sigma_{yc} = \sigma_{yp} \frac{1-\phi_f}{1+2.5\phi_f} \exp\left(B\phi_f\right) \qquad (9)$$

where σ_{yc} and σ_{yp} are yield stresses of the composite and the polymer matrix, respectively, while ϕ_f is the volume fraction of the filler, and *B* is a parameter which characterizes the interfacial interactions in the composite. The size of the interface (specific surface area of the filler) and the properties of the interphase, surface treatment, aggregation, anisotropy and orientation of the filler, as well as the matrix properties, influence the strength of the composites and thus the value of parameter *B*. Generally, a higher value of interaction parameter *B* reflects stronger interfacial interactions.[26] For defining the parameter *B* in the PP/wollastonite composites, $\ln\left[\left(\sigma_{yc}(1+2.5\phi_f)\right)/\left(\sigma_{yp}(1-\phi_f)\right)\right]$ was plotted against ϕ_f. The slopes of the straight lines give values of the interaction parameter *B*, which are given in Table 4.

Table 4. Interaction parameter *B* for the PP/wollastonite composites.

Composite	B
PP/W1	1.50
PP/W2	1.93
PP/W3	0.66

Higher value of the interaction parameter *B* in Table 4 confirms higher interfacial interactions between the PP matrix and wollastonite filler W1 and W2 and corresponds to the higher work of adhesion at the interface of the composites in comparison with W3 filler

(Table 3). However, decreasing of yield stress and quite low B values of the corresponding composites indicate the lack of chemical reactions at the interphase. It can therefore be assumed that only secondary forces take part in the investigated composites, in spite of using reactive aminosilanes[28,29] The filler surface treatment with alkylsilane (W3) lowers the interactions and corresponds to the low value of W_{mf} and negative S_{mf} (Table 3) and thus to lower parameter B, resulting in the composite weakening and dewetting at the interface (Figure 1c). The highest value of parameter B for the PP/W2 composites, $B=1.93$, implies the strongest interfacial interactions in comparison to the PP/W1 and PP/W3 composites.

Tensile strength at break and elongation at break

Determination of the ultimate tensile properties often takes place when characterizing polymer composites. By evaluating the ultimate tensile properties, geometric and physical effects should be taken into consideration separately. Decrease of specimen cross-section during elongation, strain hardening, decrease of the effective load-bearing cross section due to the presence of filler and interface interactions are the most important factors, which determine tensile strength at break and elongation at break of a polymer composites.[26] In Figure 4, tensile strength at break of the PP/wollastonite composites against the wollastonite content is shown. From the Figure 4 it could be seen that at small amounts of wollastonite filler (up to 4 vol.%) there is little difference between the values of tensile strength at break of composites with different wollastonite types used. A much more pronounced difference is observed at higher filler loadings (8 vol.% and more), where tensile strength at break of the PP/W1 and PP/W2 composites does not change much. But tensile strength at break values drastically decrease with the filler content in the case of the PP/W3 composites. For integrity of the ultimate tensile composite properties, Figure 5 presents the elongation at break in dependence of filler content of the PP/wollastonite composites. The use of different types of wollastonite in PP affects elongation at break very similarly as tensile strength at break. There is also a small difference between the various types of wollastonite used at low filler contents, a much more expressed difference is observed at higher filler loadings (8 vol.% and more), where elongation at break is much smaller in case of the PP/W1 and PP/W2 composites in comparison with the PP/W3 composites. Evaluation of tensile strength at break and elongation at break showed that small differences at lower filler content are a consequence of small impact of filler on the PP matrix due to the small interfacial area. In this case, tensile strength at break and elongation at break values are primarily influenced by the decrease of specimen cross-section during elongation or strain hardening. At higher filler loadings, the effect of filler on the PP matrix is much more expressive due to the enlargement of the interfacial area and the formation of increased interfacial interaction between the filler and matrix.

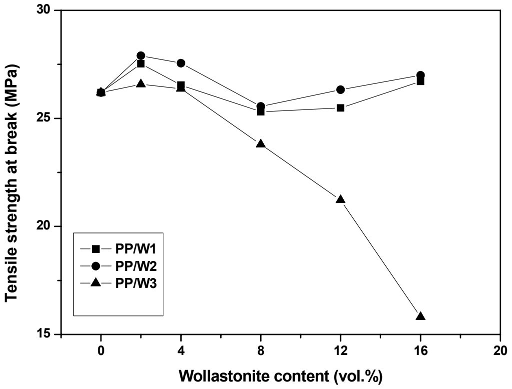

Figure 4. Tensile strength at break of the PP/wollastonite composites in dependence of wollastonite content.

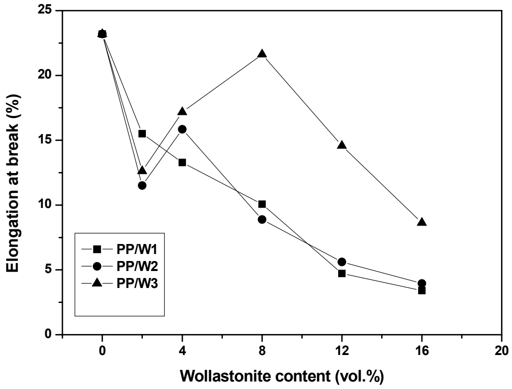

Figure 5. Elongation at break of the PP/wollastonite composites in dependence of wollastonite content.

As a consequence of strong interactions between the phases in composite, tensile strength at break increases and elongation at break decreases.[26] The same fact was established from our results, where the PP/W3 composites have lower values of tensile strength at break and higher values of elongation at break in comparison with the PP/W1 and PP/W2 composites, as a consequence of weaker adhesion. This is also in accordance with higher work of adhesion and improved wetting in composites with fillers W1 and W2, compared to filler W3, where low work of adhesion and negative spreading coefficient indicate weaker adhesion.

Notched impact strength

Notched impact strength of the material expresses its toughness, which reflects material resistance against fracture. Fracture resistance of composites is determined by several factors. Some of them, the matrix properties, the amount of the dispersed component, aggregation and orientation of anisotropic filler particles and interaction between filler and the matrix, are the most influencing ones. Figure 6 shows dependence of the notched impact strength of the wollastonite content in the PP/wollastonite composites. It can be clearly seen that the notched impact strength decreases with wollastonite content for all types of wollastonites used. The decrease of notched impact strength is more pronounced at higher filler content, probably due to the increased aggregation of the filler particles. Although the PP/W3 composites prove to have the weakest adhesion regarding the calculated adhesion parameters and parameter B, they have somewhat higher values of notched impact strength with regard to the PP composites with W1 and W2 as fillers used. As was also conducted by others,[30,31] the result of stronger adhesion, which among others also affected the interphase properties, could be in the reduction of the impact strength. This indicates the fact that stronger adhesion could lead to a brittle composite. The reduction of the impact strength when incorporating fillers into the PP matrix is always an undesired phenomenon. The use of different impact modifiers to balance the relation between stiffness and toughness is a well known method for the compensation of the deterioration effect on toughness when filler is added to the PP matrix.

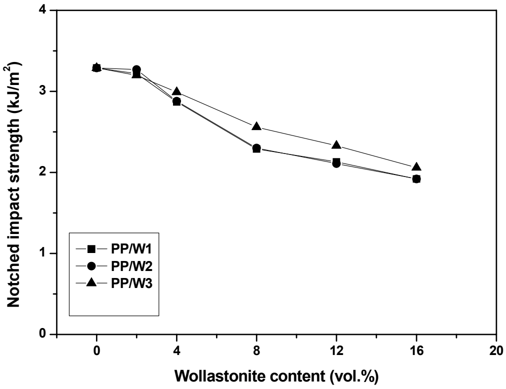

Figure 6. Notched impact strength of the PP/wollastonite composites in dependence of wollastonite content.

Conclusions

An interface and/or interphase in composites play a crucial role for composite properties. In this study the correlation between calculated adhesion parameters, based on the surface free energies of pure components using the contact angle method and mechanical properties of the corresponding composites was investigated. Proprietary (W1) and aminosilane (W2) surface pretreated wollastonite assure higher interactions with the PP matrix in comparison to alkylsilane treated filler (W3). Stronger interactions in composites with higher surface free energy wollastonite increase thermodynamic work of adhesion at the interface and lead to improved tensile properties of composites. Surface treatment of wollastonite filler with alkylsilane lowers the surface free energy and leads to weaker adhesion at the interface and thus to deterioration of tensile properties of composites. Stronger adhesion in the PP/wollastonite composites reflects in higher yield stress and tensile strength at break, but in lower elongation at break and impact resistance.

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Povzetek

Preučevali smo modifikacijo polipropilena (PP) z wollastonitom kot polnilom. V ta namen smo uporabili tri različne tipe wollastonitov, obdelanih z različnimi silani. Vzorce kompozitov smo pripravili v talini v Brabenderjevem gnetilniku in s stiskanjem v plošče v laboratorijski stiskalnici. Predvideli smo jakost adhezije med uporabljenimi polnili in PP matriko na osnovi izračunanih adhezijskih parametrov (adhezijsko delo, prosta energija medpovršine in koeficient omočenja), ki smo jih izračunali iz prostih energij površine posameznih komponent. Za določanje proste energije površine komponent smo uporabili metodo kontaktnega kota. Dobljene vrednosti adhezijskih parametrov na medpovršini smo korelirali z mehanskimi lastnosti in morfološkimi raziskavami preučevanih kompozitov in pri tem ugotovili njihovo dobro skladnost z rezultati mehanskih lastnosti. Močnejša adhezija v preučevanih PP/wollastonit kompozitih se je odražala v višjih vrednostih meje plastičnosti in pretržne trdnosti, medtem ko so bile vrednosti raztezka ob pretrgu in zarezne udarne žilavosti nižje.