

Characterization and Mechanical Properties Study of Intercalated Talc/Polypropylene Nanocomposites

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Abstract: Talc/polypropylene nanocomposite with talc contents of 10% has been prepared using an injection-molding machine. FTIR spectroscopy, X-ray diffraction (XRD) and scanning electron microscope (SEM) analysis have been used to characterize the structure. The results of these analyses showed the intercalation of PP in the interlamellar spaces of the talc. Polypropylene with talc contents of 10, 20, 30, and 40% have been prepared to study the mechanical properties. Tensile strength and impact properties have been studied as a function of talc content. The results suggested that there is an optimum talc content for enhancement of the tensile strength of the composites. With a further increase in talc content, a decrease in tensile strength was observed. The best mechanical properties in this work were obtained at a talc content of 10%.

[Masha'el Alshabanat. **Characterization and Mechanical Properties Study of Intercalated Talc/Polypropylene Nanocomposites** *J Am Sci* 2013;9(3):322-326]. (ISSN: 1545-1003). <http://www.jofamericanscience.org>. 45

Keywords: Polypropylene, Talc, Intercalated nanocomposites, Mechanical properties.

1. Introduction

Recently, nanocomposites, such as polymer-layered silicate nanocomposites, have become effective alternatives to conventional polymer composites in many applications. These systems may be classified according to the dispersion of the clay in the polymer. In general, two types of polymer-layered clay nanocomposites can be obtained, namely intercalated and exfoliated, the latter also being known as delaminated nanocomposites. Nanocomposites are defined as composites in which the dispersed particles are in the nanometer range in at least one dimension [1, 2]. The layer thickness in layered clay is around 1 nm and the lateral dimensions of these layers may vary from 300 Å to several microns, or even larger depending on the particular silicate. The layers organize themselves to form stacks with a regular van der Waals gap in between them called the interlayer [3]. Intercalates are obtained when a single polymer chain is located between silicate layers in a manner that increases layer spacing while attractive forces between the layers keep these in regularly spaced stacks. Exfoliates are obtained when the layer spacing is sufficiently increased to overcome the interactions between layers, which are then randomly dispersed in a continuous polymer matrix [4]. The X-ray diffraction (XRD) technique has been used to characterize the structures of the nanocomposites. This technique allows the determination of the spaces between structural layers of the silicate utilizing Bragg's law, $\sin \theta = n\lambda/2d$, where θ is the measured diffraction angle, λ is the wavelength of the X-ray radiation used in the diffraction experiment, and d is the spacing between diffractive lattice planes [1, 5]. By monitoring the position, shape, and intensity of basal reflections from the distributed silicate layers, the

nanocomposite structure may be identified [6]. Some authors have also used Fourier-transform infrared spectroscopy (FTIR) to elucidate the structures of the nanocomposites [7, 8]. Talc has a sheet structure with the chemical composition $Mg_3[Si_4O_{10}](OH)_8$ [9] and has become one of the most important fillers used in microcomposites of thermoplastics because of its chemical and thermal stabilities and lamellar morphology [10]. In microcomposites, the clay is acting as a conventional filler and not as a nano-dimensional material. This work aims to study the talc as a layered clay to prepare nanocomposites in intercalated structure where a polymer chain is located between the layer of clay. To achieve this objective, Polypropylene nanocomposites with 10 %wt have been prepared and the structures of which have been characterized by means of FTIR and XRD. The influence of talc content on the mechanical properties of polypropylene as a matrix has been investigated.

2. Experimental

Polypropylene (Sabic, Kingdom of Saudi Arabia) was chosen as the polymer matrix for the composites. Talc (Luzenac, France) was used as a filler in our investigation. The surfaces of the talc particles had not been subjected to any chemical treatment.

Polypropylene was mixed with various amounts of talc (10, 20, 30, and 40 wt%). Injection molding was carried out using a Battenfeld BSKN 400/100HK KS, Germany. The molding conditions are listed in Table 1. The samples of composites produced are denoted as TPP (TPP 10%, TPP 20%, TPP 30%, and TPP 40%), while neat polypropylene is denoted as PP and talc as T. The intercalated structures of the prepared nanocomposites were investigated by Fourier-transform infrared (FTIR) spectroscopy (Thermo Nicolet, FT-IR Nexus), X-ray diffraction

(XRD) analysis (Jeol X-ray diffractometer system JDX-353) and scanning electron microscopy (SEM) of the sample surface by using Jeol JSM-6360LV Scanning Electron Microscope. Tensile properties were measured using an Instron corporation machine with a series IX automated materials testing system in accordance with ASTM D 638. For tensile tests, the crosshead speed was 50.0 mm/min at a relative humidity of 50% and a temperature of 73 °C. The average value of at least five specimens was taken for each sample. Impact testing was carried out using an IZOD impact tester according to ASTM D 256. The average value of at least six specimens was taken for each sample.

Table 1. Molding conditions

Injection Pressure	500	Temperature Zone 1	180
Holding Pressure	500	Temperature Zone 2	200
Cooling Time	10	Temperature Zone 3	215
Holding Time	10	Nozzle Temperature	235
Injection Time	10	Melt Temperature	225
Total Cycle Time	33.4	Mold Temperature	40

3. Results and Discussion

3.1. Characterization

The infrared absorption spectra (FT-IR) of polypropylene (PP) and talc-filled polypropylene composite (TPP) were recorded, as shown in Fig. 1, and salient data are listed in Table 2. The infrared

spectrum of PP features strong bands near 2950, 1460, and 1380 cm^{-1} . In addition, bands of medium intensity are observed near 1155 and 970 cm^{-1} [11, 12], attributable to methyl, methylene, and methyne groups. A band is also observed at around 885 cm^{-1} , which may be due to the CH out-of-plane motions of an end group, $-(\text{CH}_3)\text{C}=\text{CH}_2$ [11]. By comparing the IR spectra of TPP and PP, it is evident that the absorption bands appearing at 2964, 2924, and 1378 cm^{-1} in the spectrum of PP are shifted towards lower frequencies of 2951, 2913, and 1373 cm^{-1} , respectively. Several peaks in the region 1299–808 cm^{-1} , as well as one at 2581 cm^{-1} , are observed in the spectrum of PP but are absent from the spectrum of TPP. A new peak seen at 671 cm^{-1} in the spectrum of TPP may be assigned to a stretching vibration of the OH group in talc. All of these changes indicate that there are interactions between the PP chains and the talc layers.

Table 2. Data of infrared spectra of PP and TPP

Absorption bands of PP (cm^{-1})	Absorption bands of TPP (cm^{-1})
2964, 2924, 2882, 2841, 2722, 2581	2951, 2913, 2882, 2840, 2723, —
1450	1455
1378	1373
1299, 1255	—
1162, 1022, 1042	1162, —, 1017
983	—
898, 841, 808	—, 841, —
—	671

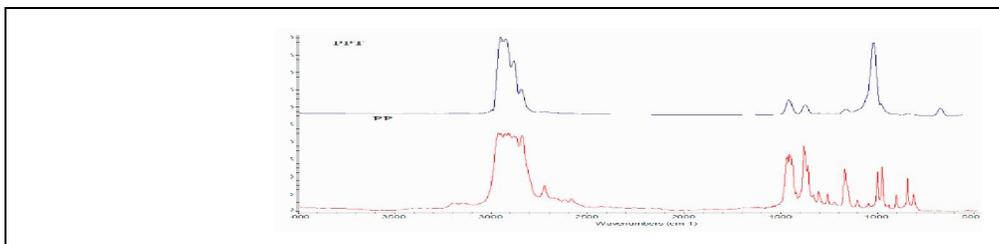


Fig. 1. The infrared spectra of PP and TPP

X-ray diffraction (XRD) provides information on the changes in the interlayer spacing of a clay upon modification. The formation of an intercalated structure should result in a decrease in the 2θ value, indicating an increase in the d -spacing, whereas the formation of an exfoliated structure usually results in the complete loss of registry between the clay layers so that no peak can be seen in the XRD trace [4]. Fig. 2 shows the XRD patterns of T and TPP, and Table 3 shows the corresponding 2θ and d spacing values. A peak at $2\theta = 9.5^\circ$ (9.302 Å) is observed for both T and TPP, indicating that the crystal structure of talc remains, although the intensity of this peak is decreased. The decrease of the peak intensity may result from the expansion of talc layers and dispersion of the talc content in PP matrix. The peaks at $2\theta = 10.5^\circ$ (8.418 Å) and 41° (2.73 Å) seen for T are absent from the pattern for TPP, while new peaks are seen for TPP at $2\theta = 18.54^\circ$ (4.77 Å), 16.375° (5.249 Å), 14.06° (6.29 Å), and 66.89° (1.397 Å), which may result from PP structure. Also, it is clear that there was a shift of the peaks that appeared at $2\theta = 18.9^\circ$ (4.681 Å) and 32.77° (2.73 Å) for T towards lower angles of $2\theta = 18.54^\circ$ (4.77 Å) and 31° (2.88 Å), thus indicating that the distance between the talc platelets had become greater and hence the intercalation structure of the polymer chain between the layers of talc. This corroborates the infrared results, which were indicative of interactions between the PP chain and the talc layers.

Fig. 3 shows the SEM image of the TPP sample. It is seen that the talc seem to be uniformly dispersed in matrix. There is no any an aggregate or a void presence, which indicates the poor adhesion between the filler and polymer.

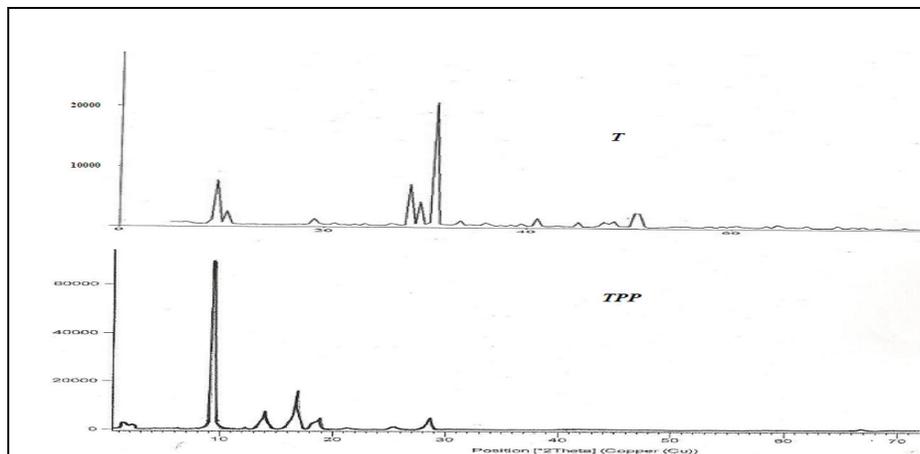


Fig. 2. The XRD spectra of PP and TPP

Table 3. The XRD Data of T and TPP

T		TPP	
$2\theta(^{\circ})$	$d(\text{\AA})$	$2\theta(^{\circ})$	$d(\text{\AA})$
9.5	9.302	9.5	9.302
10.5	8.418	—	—
41	2.73	—	—
—	—	18.54	4.77
—	—	16.375	5.249
—	—	14.06	6.29
18.9	4.681	18.54	4.77
32.77	2.73	31	2.88

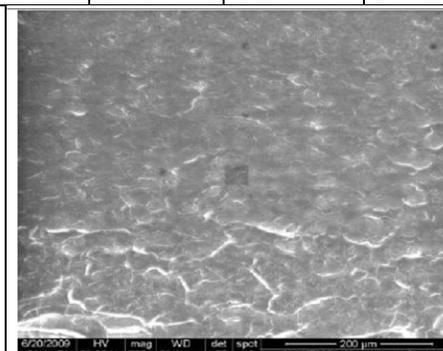


Fig. 3. SEM image of TPP sample

3.2. Mechanical properties

The values of stress at yield and strain at yield of PP and TPP are given in Table 4. Generally, it can be seen that the selected filler leads to an increase in the stress at yield to higher values compared to PP, but at the same time increasing talc content leads to a gradual decrease in the strain at yield values. Fig. 4 shows the stress at yield as a function of talc content. It is clear that there is an initial improvement in this property of PP when talc particles are added, but that there is a subsequent decrease in the stress at yield with increasing talc content. It can be seen that the highest stress at yield value for the TPP composite is obtained at an approximately 10% talc content, and amounts to an improvement of approximately 12%. This behavior is due to interfacial adhesion between

the talc particles and the PP matrix becoming weaker on increasing the talc content [13], whereupon the filler particles start to form agglomerates. These agglomerates constitute flaws and become larger in size as the talc content is increased, resulting in voids between the filler particles and the matrix such that adhesion at the interface becomes poor [14]. A similar observation has been reported by Liu and Wu [15], who studied the mechanical performance of Polyactide 66 (PA66) nanocomposites. They found that the tensile strength increased rapidly from 78 MPa for PA66 to 98 MPa for Polyactide 66–clay nanocomposites (PA66CN), but that the magnitude of the increase diminished when the clay content was above 5%. Other studies showing similar behavior have been reported by Qiu *et al.* [16], Saq'an *et al.* [17], Guerrica-Echevarria *et al.* [18], and Almeras *et al.* [19]. The results suggested that there is an optimum clay concentration for improving the tensile strength of the composites. With a further increase in clay loading, a moderate decrease in tensile strength is observed, suggesting that the relative amount of intercalation/exfoliation of the clay morphology gradually decreases with increasing clay content, since the tensile strength is usually sensitive to the degree of dispersion. The relationship between strain at yield and talc content is shown in Fig. 5. It can be seen that the lowest value of strain at yield of the composites is obtained at a talc content of around 40%. The TPP 10% composite displays lower strain at yield than PP itself, which may be attributed to the PP being substituted by the much more rigid filler particles [18]. However, it is not only the filler content that has a critical effect on the final mechanical properties, but also the characteristics of the filler. Different sizes or irregular shapes of the filler, as well as the distribution and incorporation of filler particles within the PP matrix, all have an effect on the final mechanical properties of the product, since they

contribute to the stress transfer from the polymeric matrix to the filler [14].

Table 5 shows the results of impact tests on PP and the TPP composites. There is a slight initial increase in the impact strength of the TPP composites compared to PP, but thereafter it gradually decreases with increasing talc content (Fig. 6). To rationalize this behavior, one needs to consider that when a crack is generated due to an impact it propagates towards a poor interfacial region [20]. So, as the filler content is increased, impact strength tends to gradually diminish. The presence of a filler in the matrix results in sites of stress concentration. If the filler content is low, the number of these sites and their size are small and the interfacial adhesion is stronger. At higher filler contents, however, they are more abundant, larger, and in close vicinity, and a crack, once formed, can propagate rapidly through the sample. Moreover, if the adhesion between the filler and matrix is very strong, the filler will restrict the mobility of the matrix molecules. However, some studies have indicated little or no change in the impact properties following the incorporation of clay into a polymer. For example, Liu and Wu [15] observed that the IZOD impact strength increased from 96 J/m to 146 J/m upon 5 wt% clay addition, but another study showed that the IZOD impact strength of polypropylene remained constant when the clay content was varied in the range 0–7 wt% [21].

Finally, It can be said that the content of talc play a role in the formation of polymer-talc nanocomposites. At low content, uniform distribution could be easily achieved, which could mean either that an intercalation or an exfoliation nanocomposite is formed. At high content, it is difficult to achieve uniform distribution, where the clay layers form aggregate as a microparticle, and the best description of this state is microcomposite. In our study, an optimal content of 10% was found to produce intercalation nanostructure with acceptable performance in mechanical properties. With a further increase in clay content, the clay is not well-dispersed and is acting as a conventional filler, which could mean that the microcomposit (immiscible nanocomposit) has been formed.

Table 5. Impact strengths of PP and TPP composites.

Composites	Impact strength (J/m)
PP	23
TPP 10%	31.7
TPP 20%	28.8
TPP 30%	28.3
TPP 40%	28

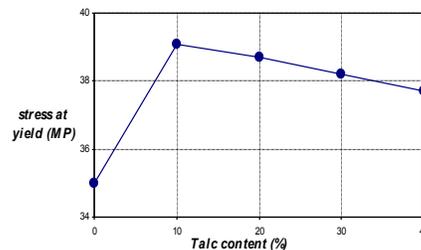


Fig. 4. The effect of filler content on stress at yield of PP and TPP composites

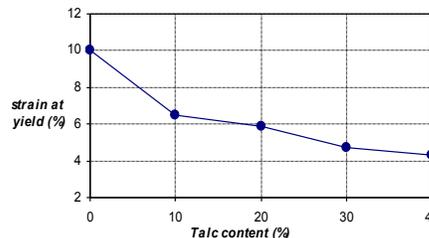


Fig. 5. The effect of filler content on strain at yield of PP and TPP composites

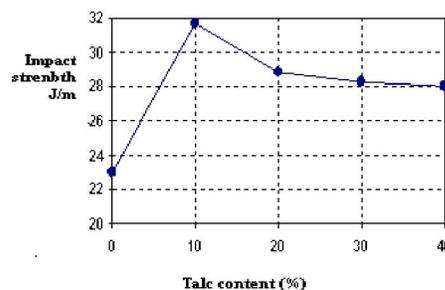


Fig. 6. The effect of filler content on impact strength of PP and TPP composites

4. Conclusion

Talc/polypropylene composites have been prepared by means of an injection-molding machine. FTIR spectroscopy, X-ray diffraction (XRD) analysis and scanning electron microscope (SEM) have been used to characterize the structure of prepared composites. The results showed that there are interactions between the talc particles and the polypropylene and the PP chain intercalated into talc layer. Polypropylene with talc contents of 10, 20, 30, and 40% have been prepared to study the mechanical properties.

Tensile and impact strengths have been studied. The results indicated that the composites became tougher and stronger, but at the same time more brittle, and a critical filler content could be defined. The addition of talc induces an increase in impact strength and yield stress, with a concomitant

decrease in yield strain, compared to PP. Microcomposite (immiscible nanocomposite) could be formed with increasing the content of talc as a result to aggregates of particles filler into polymer matrix. However, the lower talc content, 10%, was seen to be an optimal content.

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