

Preparation of Montmorillonite Modified with Long Hydrocarbon Tail-Bearing CTA⁺ Ions-Polystyrene Nanocomposites (MMPS) and Investigation of Their Thermal and Mechanical Properties

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HIGHLIGHTS

- > Polystyrene (PS)/organo-clay nanocomposites have been prepared via the melt intercalation by using two different organo-clays synthesized with the simple and the long-tailed CTA⁺ ions.
- > The glass transition temperature and thermal stability of the synthesized composites by using long-tailed CTA⁺ ions are higher than those of the virgin polymer and other composites.
- > The effective adhesion behavior between the organo-clay surface and the polymer chains led to the development of the exfoliated dispersion.

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ABSTRACT

Polymer nanocomposites have significantly taken lots of attention over the last decade. The reason for this attention is thanks to the substantial improvements in thermal and mechanical properties of these composites which are obtained even with a small addition of clay, compared to conventional composites. This study aims to prepare nanocomposites of organo-clay and polystyrene by melt intercalation method and to investigate their mechanical and thermal properties. Organo-clay was synthesized by solution intercalation method using aqueous dispersions of a long-chain hydrocarbon dispersed in aqueous medium by cetyltrimethylammonium bromide (CTAB) as a surfactant, with montmorillonite clay. The structural, thermal and mechanical characteristics of the nanocomposites were also investigated as a function of the content of the organo-clay. The organo-clay and the PCNs synthesized were characterized via XRD, HRTEM, FTIR and DSC techniques. The XRD patterns and HRTEM images show that in both cases, the organo-clay platelets have predominantly dispersed as tactoids (stacks of parallel clay platelets at about 100 nm separation) and also partially exfoliated into the polymer matrix. It was also found that the mechanical and thermal properties of the nanocomposites were significantly improved compared with pure polymer. The presence of the infrared bands of CTAB-montmorillonite in the PS/montmorillonite nanocomposite signifies that the clay tactoids were turned into fine particles and homogeneously dispersed in the PS matrix. DSC results also show that glass transition temperature and the thermal stability of the synthesized composites are higher than those of the pure polymer and other composites.

1. Introduction

Polymer nanocomposites have significantly taken lots of attention over the last decade. The reason for this attention is thanks to the substantial improvements in thermal, and

mechanical features of these composites which are obtained even with a small addition of clay when compared to conventional composites [1]. As the clay platelets are exfoliated and dispersed randomly in the polymer matrix, the enhancements of the properties such as higher modulus, increased thermal stability and flame resistance, and more

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efficient gas barrier are maximized [2]. Interfacial interactions between polymer molecules and the ultrafine individual clay platelets causes such improvements [3,4]. The thermal stability of the organo-clay is an important issue, especially if the nanocomposite is prepared via melt compounding, because the polymer is exposed to high temperatures in melt processing. Surfactant molecules can decompose thermally or exude from the organo-clay galleries, under melt processing conditions. By products obtained during thermal decomposition can catalyze polymer decomposition and thus cause deterioration of polymer properties [5,6]. Furthermore, thermal decomposition/exudation of surfactant molecules could reduce the interlayer spacing and cause an increase in the hydrophilicity of the organo-clay. The overall result is the reduction of the probability of polymer diffusion into the clay galleries. Moreover, free surfactant molecules (usually with low molecular weight) could cause undesirable plasticization effects, the production of smoke during processing or odors in the products [7,8]. In these types of composites, polymers have been used as a matrix; and clay minerals act like reinforcement material. Different types of clay are available in nature which is able to be used in the preparation of a polymer-clay nanocomposite. Generally, montmorillonite (MMT)-type mineral found in bentonite clay is used for the preparation of a nanocomposite [9,10].

MMT is a 2:1 sheet-layered silicate, which contains two-dimensional layers with around 1 nm layer thickness and lateral dimension ranging from 300 Å to several microns [11–14]. A large-scale work has been done in the field of polymer-clay nanocomposites over the past ten years with microns [15–18]. Remarkable studies focused on synthesizing PS-clay nanocomposites have been reported, which includes the synthesis of both exfoliated and intercalated polystyrene-clay nanocomposites microns [19,20]. Polystyrene (PS) is a commodity polymer that is used in a number of commercial products microns [21]. It is a very appropriate and functional manufacturing material and, a strong plastic which can be easily injected, blow-molded, or extruded. Polystyrene-clay nanocomposites have been produced in different ways: direct melt intercalation [22], in-situ polymerization [23], and solution blending method [24].

In this study, it is aimed to prepare nanocomposites of organo-clay and polystyrene by melt intercalation method and to investigate their mechanical and thermal properties

2. Material and method

2.1. Materials

For the synthesis of composites and organo-clay were mainly used a layered clay, Bentonite, polystyrene (PS), and Cetyltrimethylammonium bromide (CTAB). Cationic surfactant, CTAB, was used for synthesizing organophilic clay and for preparing the aqueous dispersion of a long chain hydrocarbon agent which will be used to produce the long-tailed CTA⁺ ions. The layered clay including the minerals, such as 26 percent of smectite, 20 percent of chlorite, 17 percent of illite, 14 percent of kaolinite, 11 percent of analcime, 7 percent of calcite, 3 percent of feldspar, and 3 percent of quartz was provided from Erzurum in Turkey. The chemical composition of the clay used is shown in Table 1.

Table 1. The chemical composition of the layered clay used

SiO ₂	45.12
Al ₂ O ₃	13.70
CaO	7.48
MgO	7.29
Fe ₂ O ₃	5.63
K ₂ O	2.62
Na ₂ O	2.37
TiO ₂	0.53
SO ₃	0.44
P ₂ O ₅	0.25

The methylene blue test was used for determining the cation exchange capacity (CEC) of the clay (ANSI/ASTM C837-76) [25]. Tables 2 and 3 also show the several features of the layered clay and long-chain hydrocarbon agent, respectively. Merck is the supplier for all chemicals used in the study.

Table 2. Some physical properties for the layered clay used

CEC (meg/100g)	48.9
d (g/cm ³)	2.61
OMC (%)	5.10
Liquid limit, w _L (%)	102.00
Plastic limit, w _P (%)	35.00
Plasticity index, I _p	67.00
a (m ² /g)	64.20

(CEC) Cation exchange capacity, (d) Specific gravity, (OMC) Organic matter content, (a) Specific surface area

Table 3. Some physical and chemical properties for the long-chain hydrocarbon agent used in this study

Density (15 °C), kg/m ³	990.7
Calorific value MJ/kg	42.74
Flash point °C	105.8
Water by distillation, wt %	0.1
C	83.4
H	11.9
N	0.8
S	1.5
Ash	0.03

2.2. Preparation of Nanocomposites

2.2.1. Preparation of long hydrocarbon tail-bearing CTA⁺ ions and organo-clay modified with them

The layered clay, which was purified by washing method [26] was dried at a vacuum oven and sieved to obtain a 38-85 µm size fraction using sieves of ASTM. 12.5 g of the long chain hydrocarbon agent was dispersed in 25 L of CTAB aqueous solution (160 mg/L) and mechanically stirred at a rate of 50 min⁻¹ for 30 min. Then, 50 g of raw clay was added to the prepared dispersion, and mixing was continued for 30 minutes at a speed of 200 min⁻¹. In the end, the filtered mixture was dried for 2 hours in a vacuum oven at 110 °C. The CTAB concentration and the adsorbed amount of the corresponding CTA⁺ ions were determined, allowing the surface potential of the clay to approach 0 mV.

For this, we determined CTA^+ concentrations in the supernatant after the adsorption via a double-beam UV spectrophotometer at 375 nm along with 0.40 mL of 1,2-dichloroethane added per 1.0 ml of the supernatant and 0.02 mL of 0.1% picric acid in 0.002 M NaOH [27]. The concentrations in solution before and after adsorption were used to compute the amount of adsorbed CTAB. The measurements of the zeta potential of the modified clay were made by using Zeta meter 3+ instruments.

2.3. Preparation of Polystyrene/Organo-Clay Nanocomposites

2.3.1. Melt Intercalation Method

A given amount of the synthesized organo-clays via two different ions, using the simple CTA^+ and long hydrocarbon tail-bearing CTA^+ Ions, was evenly mixed with PS, and then the mixture was put into the single-screw extruder for additional melt blending at a temperature range of 190–210 °C and a screw speed of 145 rpm. In the end, the strands, which are obtained from the extruder, were mechanically pressed. The ratios of organo-clay of the nanocomposites were determined as 5.0, 10.0, 15.0 and 20.0 % wt., respectively.

2.4. Characterization of Polystyrene (PS)/organo-clay nanocomposites

A lot of techniques of characterization, such as XRD, HRTEM, FT-IR, and DSC were used to determine characteristics of the nanocomposites and to evaluate their thermal stabilities in this research. For XRD measurements of the organo-clay samples, Rigaku 2200D/max (Rigaku Corporation, Tokyo, Japan) powder diffractometer equipment with a $\text{CuK}\alpha$ radiation source was used a Perkin-Elmer Spectrum-One and KBr pelleting method for a range of 4000–400 cm^{-1} at a scanning rate of 2°/min was used for FTIR spectra for the composites. An average of 100 scans and a resolution of 1 cm^{-1} were chosen for all measurements. In order to forecast the framework rearrangement of nanocomposites, a JEOL 2100 high-resolution transmission electron microscope (HRTEM) LaB 6 filament operated at 200 kV was used for examining the specimens taken from the samples.

A differential scanning calorimeter (DSC7020) under nitrogen atmosphere was used to observe the curing behaviors of nanocomposites. The typical weight of the sample was about 10 mg and the scan rate was 200 °C/min.

For mechanical characterization of the composites, their Shore hardness which is a measurement method of the resistance a material has by penetrating a needle under a determined spring force was measured. It is defined with a number ranging from 0 to 100 on the scales A or D. If the number is high, this means the higher hardness. Flexible types are shown with the letter A and the letter D means rigid types. Measurements were carried out at room temperature. We used the Shore D hardness scale and the average of 10 hardness values was calculated.

3. Results and Discussion

3.1. XRD Analysis

XRD patterns for the composites, which were prepared using two different organo-clays: the simple and the long-tailed CTA^+ ions, have been shown in Fig. 1.

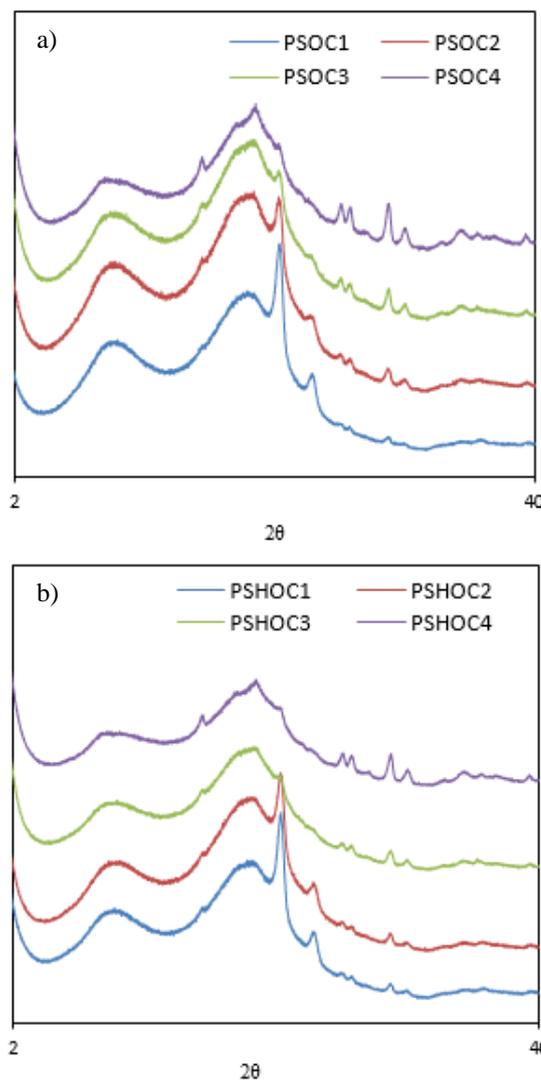


Figure 1. XRD patterns for the composites, which were prepared at the different ratios of the organo-clays, (a) (PSOC1:5.0%; PSOC2:10.0%; PSOC3:15.0% and PSOC4:20.0%), (b) (PSHOC1:5.0%; PSHOC2:10.0%; PSHOC3:15.0% and PSHOC4:20.0%), using two different organo-clays, the simple and the long-tailed CTA^+ ions.

From these figures, it can be seen that in both cases, the organo-clay platelets have predominantly dispersed as tactoids (stacks of parallel clay platelets at about 100 nm separation) and also partially exfoliated into the polymer matrix. The fact that the clay platelets may be dispersed in the PS matrix, even at very high ratios, has been shown by this result. In contrast to the expected behavior, it is monitored that the exfoliation or dispersion degree of the organo-clay platelets increased with increasing clay content. The increase in the exfoliation degree took place because of facilitating diffusion of PS chains to interlayer regions along with increasing clay content facilitates, which can be concluded.

3.2. FTIR Analysis

FTIR spectra for the composites, which were prepared at the different ratios of the organo-clays, using two different organo-clays synthesized with the simple and the long-tailed CTA⁺ ions, are given in Fig 2.

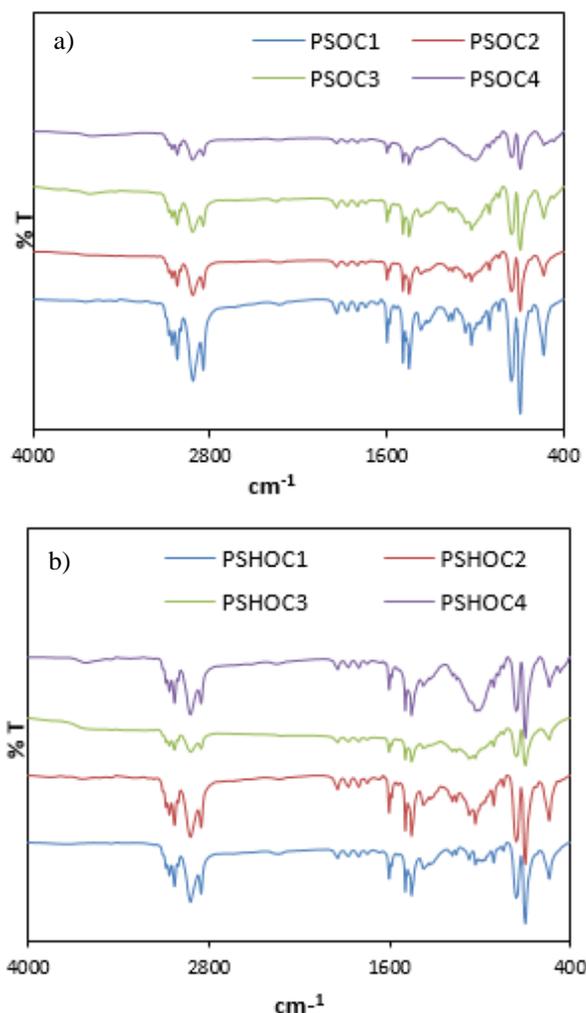


Figure 2. FTIR spectra for the composites, which were prepared at the different ratios of the organo-clays, (a) (PSOC1:5.0%; PSOC2:10.0%; PSOC3:15.0% and PSOC4:20.0%), (b) PSHOC1:5.0%; PSHOC2:10.0%; PSHOC3:15.0% and PSHOC4:20.0%), using two different organo-clays synthesized with the simple and the long-tailed CTA⁺ ions.

Figure 2 shows that in both cases, the peaks at 3600 cm⁻¹ that are associated with OH stretching vibrations only appear at the high organo-clay ratios of 15.0 and 20.0%. Also, all composites exhibit the peaks associated with C–H asymmetric and symmetric stretching vibrations in the alkyl chains of the ammonium salt at 2924 and 2850 cm⁻¹, respectively and also the peaks at 1600 cm⁻¹ and 466 cm⁻¹ that are associated with the aromatic stretching vibration of C=C and Si-O bending vibrations, respectively [28]. The bands at 3062, 3033 cm⁻¹ are assigned to C-H stretching of the aromatic ring in benzyl groups [29]. As can be seen from Fig. 2, in the case of the prepared composites by using the organo-clay modified with the long-tailed CTA⁺ ions, the intensity of peaks at 2849 cm⁻¹ that is associated with aliphatic stretching vibration decreases with the increasing clay content. These results clearly indicate.

3.3. DSC Analysis

Differential scanning calorimetric measurements have been extensively applied in the study of a variety of phenomena which occurs throughout the heating of organo-clays and polymer/clay nanocomposites or nanotubes, involving glass transition (T_g), crystallization, melting and curing [30]. Thermal behaviors of the prepared PS/the organo-clay composites were evaluated via DSC analysis Fig. 3 shows the thermograms belonging to the prepared composites. As organo-clay platelets do not experience any transitions in the temperature range of the experiment, only thermal transitions polystyrene is observed. Firstly, samples are heated from room temperature to 190°C and this is performed for removing their thermal history. Then, they are heated to higher temperatures to analyze other irreversible thermal behaviors the phase conversion. There is a clear inflection in the heating path attributed to the glass transition temperature of the samples and, there is no exothermic peak in the higher temperatures, which indicates that the structure of polystyrene and its nanocomposites are chiefly amorphous and polystyrene chains are not crystallized [31]. DSC results also show that the glass transition temperature and thermal stability of the synthesized composites by using long-tailed CTA⁺ ions are higher than those of the virgin polymer and other composites.

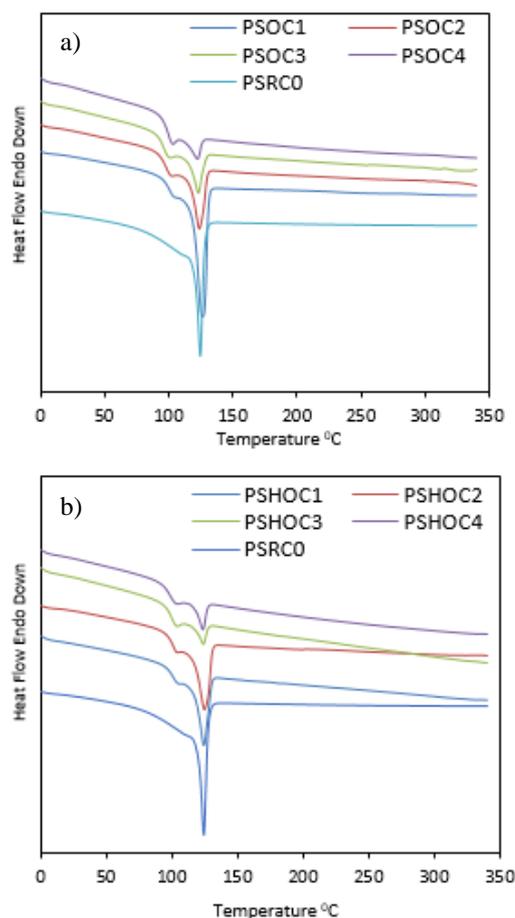


Figure 3. DSC thermograms for the composites, which were prepared at the different ratios of the organo-clays, (a) (PSOC1:5.0%; PSOC2:10.0%; PSOC3:15.0% and PSOC4:20.0%), (b) PSHOC1:5.0%; PSHOC2:10.0%; PSHOC3:15.0% and PSHOC4:20.0%), using two different organo-clays synthesized with the simple and the long-tailed CTA⁺ ions.

3.4. HRTEM Analysis

HRTEM was used for the exfoliation or dispersion behaviors of the clay platelets in the nanocomposites, as Fig. 4 showed. The dark lines coincide with the silicate platelets in the polymer matrix [4]. Dispersion and delamination of individual silicate layers in the polymer matrix are available in the literature; however, as seen in the HRTEM micrographs, commonly intercalated stacks with a range of gallery distances are obtained [32]. As stated in the HRTEM image, some individual tactoids, and more stacks with a small number of platelets, can be monitored. Again, this from figures, it could be understood that random dispersion of partially exfoliated clay stacks, as well as predominantly, tactoidal dispersion of organo-clay platelets, in the polymer matrix has emerged.

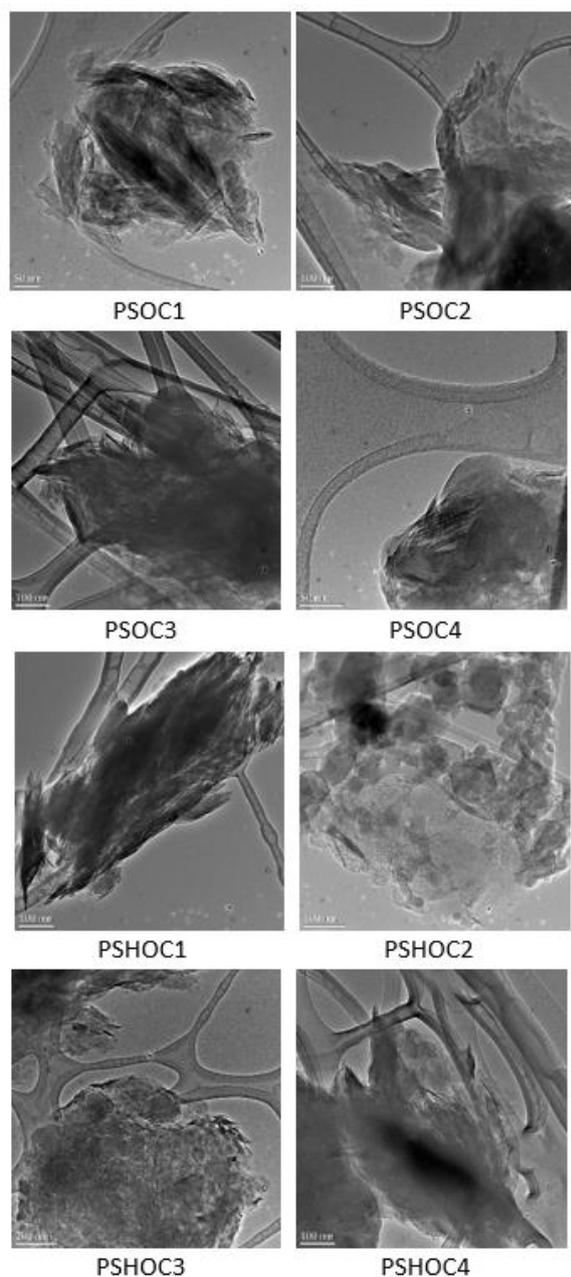


Figure 4. HRTEM images for the composites, which were prepared at the different ratios of the organo-clays, (PSOC1:5.0%; PSOC2:10.0%; PSOC3:15.0% and PSOC4:20.0%, and PSHOC1:5.0%; PSHOC2:10.0%; PSHOC3:15.0% and PSHOC4:20.0%), using two different organo-clays synthesized with the simple and the long-tailed CTA⁺ ions.

3.5. Hardness Measurements

Mechanical properties (Fig. 5) depend in great measure on the dispersion degree of clay plates [33] and the development of adhesion between clay surface and the polymer chains [34].

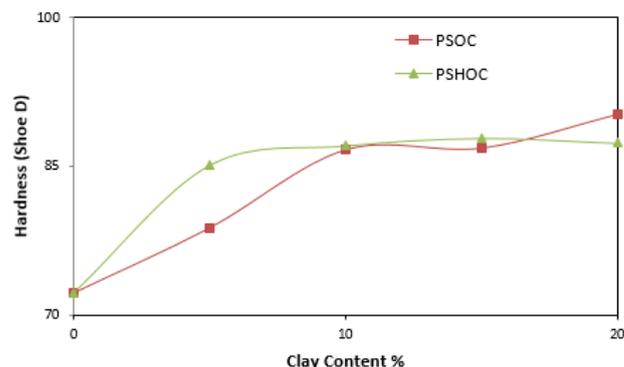


Figure 5. The variation of Hardness (Shore D) values belonging to the composites prepared by using two different organo-clays synthesized with the simple and the long-tailed CTA⁺ ions, with clay content.

As seen from the figure, the hardness values of the composite, with increasing clay content, increased first and then remained constant. Extremely high hardness values indicate that the inclusion of organo-clay into the polymer matrix, the formation of a quite durable and relatively rigid structure provides.

3.6. Conclusion

The main findings of the study can be summarized as follows:

- The XRD patterns and HRTEM images show that in both cases, the organo-clay platelets have predominantly dispersed as tactoids (stacks of parallel clay platelets at about 100 nm separation) and also partially exfoliated into the polymer matrix. It was also found that the mechanical and thermal features of the nanocomposites were significantly improved compared with pure polymer.
- The presence of the infrared bands of CTAB-montmorillonite in the PS/montmorillonite nanocomposite signifies that the clay tactoids were turned into fine particles and homogeneously dispersed in the PS matrix
- DSC results also show that glass transition temperature and the thermal stability of the synthesized composites are higher than those of the pure polymer and other composites.
- Hardness measurement shows that the adhesion between the clay surface and the polymer chains was developed.

Compliance with Ethical Standards

There is no conflict of interest to disclose.

Conflict of Interest

The author(s) declares no known competing financial interests or personal relationships.

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