Research Article

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Miscibility studies of polystyrene/polyvinyl chloride blend in presence of organoclay

https://doi.org/10.1515/chem-2019-0095
received March 28, 2019; accepted July 8, 2019.

Abstract: Polystyrene (PS)/polyvinyl chloride (PVC) blends of different compositions, with and without organoclay (OC) were prepared by a solution casting method. PS and PVC were combined in ratios of 100/0, 90/10, 10/90, and 0/100. Local clay was treated with 0.5M NaCl to generate sodium clay (Na-clay). The Na-clay was subsequently modified using the cationic surfactant, cetylpyridinium chloride (CPC), at a concentration of 0.5 times that of Na-clay. The CPC-modified clay was denoted as organoclay (OC). The organoclay was dispersed in PS/PVC blends (90/10 and 10/90) with different weight percentages of OC (0%, 1%, 5%, and 10%). To evaluate the miscibility of PS/PVC blends, the blends with and without OC were characterized using Fourier-transform infrared (FTIR) spectroscopy, differential scanning calorimetry (DSC), X-ray diffraction (XRD), and scanning and transmission electron microscopy (SEM and TEM). The addition of OC to the PS/PVC blends produced exfoliated nanocomposites, as proven by XRD. The SEM and TEM micrographs showed that the PS/PVC(10/90) blend components were more miscible than those of the PS/PVC(90/10) blend, leading to favorable morphology.

Keywords: Polystyrene; poly vinyl chloride; organoclay; morphology; nanocomposites.

1 Introduction

Polymer blend nanocomposites have recently attracted attention, with attempts being made to improve the miscibility between various immiscible polymers. In this regard, it was suggested that modification of clay with a cationic surfactant can contribute to improve the compatibility of blends based on immiscible polymers, in turn leading to enhanced properties [1,2,3]. To achieve compatibility between two polymers, the interfacial tension between the phases must be lowered. Furthermore, coalescence of the particles during blending must be prevented. It is postulated that organoclay (OC) may play in both polymer blends and water treatment [4]. The first attempt was to use organoclay as a compatibilizer was demonstrated by Gelfer and coworkers [5]. They studied the impact of organoclay on thermal and rheological properties and morphology of polystyrene (PS)/Polymethyl methacrylate (PMMA) [8]. The preparation of nanocomposites using organoclay can be achieved via different routes, including solvent casting, in situ polymerization, and melt compounding [5,6]. Among others, Polystyrene is regarded as one of the best polymers with various uses. There are several ways of modifying its properties, for instance through physical blend with various materials or copolymerization. Currently, physical modification through blending with other polymers is widely employed. For the majority of cases, the stability of PS is merely fair due to its low resistance to heat. Untreated organoclay have several drawbacks due to their layered structure with low hydrophilic sites, low surface area, agglomeration of clay particles and mineral impurities. To improve these properties of organoclay is used acid treatment and other depends upon their application. The broader application as fillers in membranes technology to improve the barrier performance and others used in various polymer technology.[9-12]

Poly(vinyl chloride) (PVC) is another important polymer that is extremely vulnerable to heat, thus stimulating interest in research toward achieving higher stability of the polymer. The rapid expansion and consumption of PVC is due to its lower cost and greater availability. The thermal stability of both polymers in combination is better than that of PVC or PS alone.

To improve the thermal stability of PVC and PS blend in addition of nanoparticle. Literature survey reveals that the work on Saudi clay not used in PVC and PS blend for
miscibility. Therefore, this work aims to study the effect of modifying Saudi clay with the cationic surfactant, cetylpyridinium chloride (CPC), on the miscibility of the PS/PVC blend [13]. The influence of the organoclay content on the morphology of the PS/PVC blends is characterized using Fourier-transform infrared (FTIR) spectroscopy, differential scanning calorimetry (DSC), X-ray diffraction (XRD), and scanning and transmission electron microscopy (SEM and TEM).

2 Experimental

2.1 Materials

Polystyrene (PS) and poly(vinyl chloride) (PVC) from Saudi Basic Industries Corporation (SABIC) were utilized in the form of a matrix. Polystyrene is available under the brand-name PS125, and poly (vinyl chloride) is retailed as PVC (67S). Table 1 provides details of the polymers. Raw clay was bought from the northern region of Jeddah (Kholais region) in Saudi Arabia. The chemical composition of the clay sample was previously investigated (Table 2) [14]. Cetylpyridinium chloride and tetrahydrofuran(THF) were from BDH Chemicals, England.

2.2 Methods

The structure of Na-clay, OC, and the PS/PVC and PS/PVC/OC blends was investigated using FTIR spectroscopy, Spectrum 100(Perkin Elmer Instrument, USA), Perkin Elmer, USA). The structure of Na-clay, OC, and the PS/PVC blends was analyzed using X-ray diffraction (XRD; PANALytical X-pert PRO X-ray diffractometer, Holland), and the intensity data were collected in the 2θ range of 3–50° using a Cu-Kα source (λ = 1.54 Å) operated at a generator current of 40 mA and a generator tension of 45 KV. Differential scanning calorimetric analysis of the PS/PVC/MM blends with and without OC was performed using a TA instruments SDT2960 Simultaneous DSC analyzer. The samples were heated from 25°C to 200°C; the rate of scanning was 10°C·min⁻¹ under a flow of nitrogen. Transmission electron microscopy images were recorded for investigation of the morphology of the PS/PVC/OC blend using a JEOL JSM-6060LV transmission electron microscope. The accelerating voltage used for the TEM analysis was 100 KV, where small drops of dilute suspensions of 0.1 g of the clay samples (Na-clay and OC) in 5 cm³ of double distilled water were placed on mesh grids of copper that were covered with a thin carbon film. A scanning electron microscope (JEOL JSM7600F) was used to determine the surface morphology of the blend. The microscope was operated at 5 kV with a working distance of 4.5 mm.

2.3 Washing and saturation of clay

The clay sample was ground to dimensions of less than 60 µm using a mortar and pestle. The clay fraction with a fine particle size was separated by fractionation [14]. The separated clay was dispersed in solution with shaken for 24 h and again immersed in 0.5 mol L⁻¹ NaCl solutions. This step was repeated five successive times. Excess salt was removed by centrifugation and subsequent washing with excess deionized water until the AgNO₃ tests were negative for Cl⁻. The thus-obtained clay saturated with Na⁺ was dried at 120°C for 1 h, and finally ground.

2.4 Modification of clay sample

The organoclay was prepared using the ion exchange method. Na-clay (0.5 g) was immersed in 500 mL of distilled water at room temperature for 24 h and again immersed in 0.5 mol L⁻¹ NaCl solutions. This step was repeated five successive times. Excess salt was removed by centrifugation and subsequent washing with excess deionized water until the AgNO₃ tests were negative for Cl⁻. The thus-obtained clay saturated with Na⁺ was dried at 120°C for 1 h, and finally ground.

Table 1: Molecular weight and glass transition temperature of PS and PVC.

| Sample  | Mass polymerization | Mₙ (gmol⁻¹) | Mₚ (gmol⁻¹) | Tg (°C) |
|---------|---------------------|-------------|-------------|---------|
| PS 125  | Mass polymerization | 143,000     | 259,000     | 89.33   |
| PVC 67S | Suspension polymerization | 59,000     | 160,000     | 84.61   |

Table 2: The chemical composition of Organoclay(OC).

| SiO₂      | Al₂O₃ | Fe₂O₃  | CaO    | MgO   | Na₂O    | K₂O    | TiO₂     | Impurities |
|-----------|-------|--------|--------|-------|---------|--------|----------|------------|
| 70.30%    | 15%   | 7.75%  | 2.30%  | 1.45% | 1.20%   | 0.30%  | 0.30%    | 0.10 wt.%  |
Miscibility studies of polystyrene/polyvinyl chloride blend in presence of organoclay

added to the dispersion of Na-clay with stirring, and the mixture was stirred for a further 12 h. The cation exchange reaction occurs rapidly, and the clay subjected to cation exchange was washed with distilled water and filtered until there was no detection of chloride ions using a 0.1 M AgNO_3 solution. The resulting OC was dried under vacuum at 60°C for 24 h and ground with a mortar and pestle to obtain finely powdered OC [15].

2.5 Preparation of polymer blends (PS/PVC)

Polymer blends of PVC and PS were developed using THF as a solvent. The polymer solution was developed by dissolving PS/PVC in different weight ratios (100/0, 90/10, 10/90, and 0/100 wt%) in 20 mL of THF. The solutions were mixed at a temperature of 40-45°C and stirred for 18 h. The solutions of PS and PVC were sonicated in an ultrasonic bath for 0.5 h and cast into Petri dishes, and the THF was allowed to evaporate [16,17]. The thus-obtained thin films were removed from the glass plates after 24 h at room temperature.

To prepare the PS/PVC/OC blends with different OC contents (1, 5, and 10wt% of OC), the components were placed into a 100 mL beaker, and 0.25 g of OC was added with the inclusion of 10 mL THF. This suspension was stirred magnetically for 1 h at room temperature and the PS/PVC blend was added to this suspension. The mixture was stirred magnetically for 6 h with heating at 40-45°C, then sonicated for 0.5 h. The mixture was cast into 100 × 20 mm Petri dishes and the THF was allowed to evaporate at room temperature. The film was detached from the glass plate after 24 h.

Ethical approval: The conducted research is not related to either human or animal use.

3 Results and Discussion

3.1 FTIR analysis

The FTIR spectrum of Na-clay (Figure 1) indicates that clay was the dominant mineral phase. The bands at 3625 cm^{-1} and 3430 cm^{-1} are due to stretching of the -OH groups of Al-OH and Si-OH. The band at 3430 cm^{-1} corresponds to the -OH stretching band of interlayer water. The absorption band was at 1636 cm^{-1}, associated with the -OH bending mode of absorbed water. The bands at 1033 cm^{-1} and 693 cm^{-1} are attributed to the Si-O stretching (in plane) vibration of layered silicates. The FT IR band at 693 cm^{-1} is attributed to Si-O stretching of quartz, and that at 915 cm^{-1} is characteristic of Al-OH-Al bending vibrations. The characteristic band at 527 cm^{-1} is due to Si-O-Si bending, and Al-O-Si bending was observed at 465 cm^{-1} [18]. The pure CPC surfactant presented characteristic absorption peaks of the C-H bonds at 2915 and 2850 cm^{-1}, which are attributed to C-H asymmetric and symmetric stretching vibrations of the surfactant, respectively. The third band around 1468 cm^{-1} is assigned to the CH_{2} vibration. The presence of these peaks in the spectrum of OC confirms intercalation of the surfactant molecules with the silica layers in OC. Therefore, the clay was successfully modified, making it hydrophobic.

3.2 X-ray diffraction analysis

Figure 2 shows the XRD patterns of Na-clay and OC. Upon intercalation of CPC, the d-spacing (001) of OC shifted to a higher value than that of Na-clay. The d-spacing of 12.562 Å (2θ = 7.036°) for Na-clay increased to 22.891 Å (2θ = 3.86°) for OC. The increase in the d-spacing after modification of Na-clay with the surfactant may reflect intercalation of the surfactant cations between the silicate layers after exchange with sodium (Na^{+}). The X-ray data reveal that Na-clay was successfully modified by CPC.

The XRD pattern of pure PS, as shown in Figure 3, exhibits a broad hump with two halos at 2θ = 10.200°
Jumaa Aseeri et al.

The X-ray diffraction (XRD) pattern of pure PVC (Figure 4) was characterized by two broad humps centered at 20 = 17.76° (4.99 Å) and 20 = 24.22° (3.67 Å). The XRD pattern of the PS/PVA (90:10) blend sample exhibited the characteristic humps of pure PS at the same positions (20 = 10.20° (9.03 Å) and 20 = 20.0° (4.62 Å)) with disappearance of the characteristic humps of PVC. This provides evidence of the complexation of PVC and PS in the blends and of partial miscibility of these species in the blends. The XRD pattern of PS/PVA (90:10) showed one broad peak at 20 = 20° (4.62 Å), instead of the two humps observed for pure PVC. This indicates greater miscibility of the components of the PS/PVA (90:10) blends than for the PS/PVA (90:10) blend. This may be because the addition of PS disrupted the arrangement in the polymer backbone of PVC and increased the amorphous phase of PVC [20]. The changes in the XRD patterns of this blend clearly indicate coordination between the components of the blend [21, 22]. From the XRD data, it can be concluded that when 10% PS was added to the PVC matrix, the blend became more homogenous and PS was miscible with the PVC chains, unlike the case when 10% PVC was added to the PS matrix.

Figure 4 shows the X-ray diffraction patterns of the PS/PVA (90:10) and PS/PVA (90:10) blends with different contents of OC (1, 5, and 10 wt%). The characteristic peaks of OC (as shown in Figure 2) disappeared in the spectra of all the PS/PVC/OC blends. The absence of the characteristic peaks of OC in the polymer blends indicates the formation of an exfoliated structure in the PS/PVC/OC blend, as confirmed by TEM.

3.3 Evidence of miscibility of the PS/PVC blend components

The glass transition temperature (Tg) of a polymer blend is often used as a criterion to determine the miscibility of the blend components [23]. DSC is regularly utilized to determine the Tg. The samples were heated from ambient temperature to 200°C at a rate of 10°C·min⁻¹ under a flow of nitrogen and were cooled back to room temperature and again heated to 200°C at the same rate. The Tg of the samples was determined from the second heating run. The Tg of the PS/PVC blends was determined from the midpoints of the transitions. Figure 5 shows the DSC thermograms of pure PVC, PS, and the PS/PVC blends. The Tg values for pure PS and PVC were about 89.22°C and 84.62°C, respectively. Two distinct Tgs were observed at 91.99°C and 105°C for PS/PVA(10:90), corresponding to the phases of PS and PVC, showing that phase separation occurred in the polymer blend, whereas the PS/PVA(90:10) blend showed a single Tg at 89.85°C. According to Merfeld and Paul [24], a single Tg intermediate between the Tgs of the pure components is indicative of molecular homogeneity, while the appearance of the Tg reflects phase separation of
the blend components. However, Zakaria and coworkers [20] claimed that the glass transition temperatures for all the blends of PVC/PEMA lie between those of these two polymers. The DSC results reveal that PS/PVA (10:90) is an immiscible blend (shows two glass transitions), whereas PS/PVA (90:10) is a miscible blend.

Figure 5 shows the DSC thermograms of PS/PVA (10:90) and PS/PVA (90:10) with different contents of OC (0, 1, 5, and 10 wt%). A single $T_g$ was observed for the PS/PVA (10:90)/OC and PS/PVA (90:10)/OC nanocomposites, close to the glass transition of neat PS/PVA (10:90) or PS/PVA (90:10). Organoclay may be useful for compatibilizing polymer blends. Addition of OC to PS/PVA (10:90) or PS/PVA (90:10) can decrease the interfacial tension between the two polymeric phases; thus, the interaction between PS and PVA was improved, which lowers the Gibb’s free energy of mixing and increases the miscibility of the component phases.

### 3.4 Electron microscopy (SEM and TEM)

The images obtained from TEM analysis of Na-clay and OC are shown in Figure 6. The TEM analysis supports the findings from XRD and FTIR. The image of Na-clay shows multilayered silicate platelets aligned as ordered, parallel, and straight lines (dark image), whereas the TEM image of OC shows dispersed silicate platelets (bright image). The bright image indicates intercalation of the CPC cation between the interlayers of clay compared to that of the Na-clay sample. This result indicates the successful intercalation of the CPC ion between the clay layers.

Although the PS/PVA (90/10) blend showed a single $T_g$, this does not definitely indicate miscibility on the molecular scale; however, SEM (and especially TEM) provides the unique opportunity to illustrate the miscibility of polymer blends [20]. The morphology and the structure of the clay layer dispersion in the PS/PVC...
Figure 5: Differential Scanning Calorimetry (DSC) thermogram of Pd1(PS/PVA:10/90), Pd1/1%OC, Pd1/5%OC, Pd1/10%OC, Pd2(PS/PVA:90/10), Pd2/1%OC, Pd2/5%OC, Pd2/10%OC.

Figure 6: Transmission Electron Microscope (TEM) images of A) Na-clay and B) Organoclay (OC).

blends were further analyzed by SEM and TEM. Figure 7 presents the TEM images of PS/PVA (10/90), PS/PVA (90/10), PS/PVA (10/90)/10% OC, and PS/PVA (90/10)/10% OC. In the case of PS/PVA (10/90) and PS/PVA (90/10), phase separation of nearly-spherical PVC and PS droplets suspended in PS/PVA (10/90) and PS/PVA (90/10) could respectively be observed. However, when OC was added to PS/PVA (10/90), the domain size of the PVA phase in the PS matrix was reduced compared to the domain size before addition of OC. This result is attributed to the fact that two immiscible polymer chains can exist together between the interlayer of OC; thus, these OC platelets reduced the interfacial tension and improved the miscibility of PVC in the PS matrix [3, 25]. On the other hand, the TEM image of PS/PVA (90/10)/OC (Figure 7) showed silicate layers homogenously dispersed in the polymer matrix, indicating the formation of fully exfoliated nanocomposites. The exfoliated clay plates effectively prevent coalescence of the PVC domains in the PS matrix. It can be concluded that when the polymer blend is rich in a polar polymer such as PVC in the PS/PVA (90/10) blend, there is a strong interaction between the polar group (such as in PVC) and OH groups on the clay surfaces. This interaction facilitates complete miscibility of PS in the PVC matrix.

The SEM images (Figure 8) of the PS/PVA (10/90) and PS/PVA (90/10) blends clearly demonstrate two phases (phase separation). The minor phase is dispersed as a spherical domain phase in the polymer-rich matrix. In addition, the micro voids surrounding PVC in the PS-rich or PS in the PVC-rich phase droplets indicate weak interfacial
Figure 7: Transmission Electron Microscope (TEM) images of Pd1(PS/PVA:10/90), Pd1/10% Organoclay(OC), Pd2(PS/PVA:90/10) and Pd2/10% Organoclay(OC).

Figure 8: Scanning Electron Microscope (SEM) images of Pd1(PS/PVA:10/90), Pd1/10% Organoclay(OC), Pd2(PS/PVA:90/10) and Pd2/10% Organoclay(OC).
adhesion in the blend [26]. Interestingly, addition of 10 wt% OC to the PS/PVA (10/90) blend resulted in a great decrease in the PVC phase domains in the PS matrix. With the addition of 10 wt% OC to the PS/PVA (90/10) blend, the morphology of the domain of the PS phase changed to a continuous structure (one-phase), in good agreement with the TEM data.

4 Conclusions

Organoclay from Saudi region was successfully prepared by cation exchange method using cetylpyridinium chloride as a cationic surfactant. The different layers of exfoliated clay were improved the miscibility of PS and PVC polymer. By using thermal analysis and morphological analysis, it is proved the PS and PVC blends are easily miscible by organoclay as compare to without clay.

Conflicts of Interest: The authors declare that there is no conflict of interest regarding the publication of this paper.

Acknowledgments: This Project was supported by King Saud University, Deanship of Scientific Research, College of Sciences Research Centre.

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