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Preparation, morphology, and thermomechanical properties of coal ash/polyethylene oxide composites

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Coal ash/polyethylene oxide (ash/PEO) composite films were prepared by the solution casting technique. Scanning electron microscopy (SEM) micrographs indicated that the ash particles were dispersed and embedded well within the polymer matrix. The size of the ash particles in the PEO matrix was less than 3 μm . The polarized optical microscopic (POM) analyses revealed that the pure PEO, upon crystallization, showed distinct crystalline spherulites of a considerable size. The size of the spherulites was decreased by the incorporation of ash particles into the polymer matrix, which might be due to the nucleation role of ash in ash/PEO composite, and resulted in small-sized spherulites. The differential scanning calorimetry (DSC) thermograms showed that the melting temperature (T_m) of pure PEO was about 66 °C, while the T_m peak shifted to a slightly lower temperature when ash was incorporated in the polymer matrix. The mechanical properties of the ash/PEO composites were enhanced up to an optimum level (ash wt. %) as compared to pure PEO. The thermograms from the thermogravimetric analyzer (TGA) indicated that the thermal stability of the ash/PEO composite shifted to a higher temperature, about 30 °C higher than pure PEO.

Key Words: Coal ash, polyethylene oxide, spherulites, SEM, TGA

Introduction

Polymer-matrix composites represent attractive materials because the incorporation of fillers in the polymer matrix increases the mechanical stability (due to the reinforcing effect of the filler particles) and thermal stabil-

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ity, but decreases the thermal expansion coefficient.¹ There are a number of studies available that incorporate various kinds of reinforcing materials, including clays, carbon fiber, graphite, and carbon nanotubes, into the polymer matrix. Various properties like the mechanical, electrical, and thermal stability of the polymer are enhanced significantly by the addition of a small quantity of the reinforcing materials. Burmistr et al.² prepared polymer-clay composites by melt blending (blended organobentonite with polyamide and polystyrene) and reported that the organoclay improved the mechanical properties and thermal stability of the pure polymers. Saeed et al.³ and Chen et al.⁴ prepared CNTs/nylon and CNTs/poly(caprolactone) nanocomposites, respectively, and found that the nanocomposite material had higher electrical, thermal, and mechanical properties than pure polymers. Yasmin et al.⁵ and Saeed et al.⁶ used graphite and titanium oxide as reinforced materials for the enhancement of the mechanical and thermal stability of epoxy and polyacrylonitrile, respectively. Wu et al.⁷ prepared poly(ethyleneterephthalate)-silica/polystyrene nanocomposite films and reported that the heterogeneous dispersion of inorganic nanoparticles in the polymeric matrix reduced their properties, including mechanical and thermal stability. For homogeneous dispersion in a polymer matrix, the inorganic nanoparticles are usually modified to improve their compatibility with the polymer matrix, which results in enhanced properties of polymer composites.

In the present study, we used ash as a reinforcing material in a PEO matrix. PEO is an important water-soluble polymer, used for a variety of purposes in textile industries, cosmetics, antifoaming agents, chemical intermediates, ink and dye solvents, demulsifiers, plasticizers, and so on.⁸ In our study, we chose ash as a reinforcing material because of its low cost and easy availability. The main objective of this work was to study the effect of ash on the properties of PEO, because ash is the waste product obtained after burning coal. In order to utilize these materials in a proper way, we made the ash/PEO composites via the solution casting technique. The ash/PEO composites were analyzed for various properties using a variety of techniques, including SEM, POM, a universal testing machine, DSC, and TGA.

Experimental

Materials

The PEO (average molecular weight of 600,000) was purchased from Aldrich. The Cherat coal sample was kindly supplied by the Pakistan Mineral Development Corporation (PMDC). The coal lumps were crushed, ground, and sieved through a 212- μm mesh screen.

Preparation of ash

The Cherat coal sample was heated in a muffle furnace at 750 ± 25 °C until all of the carbon particles disappeared. The obtained ash was then sieved through a screen (mesh size of 60 μm) and stored for further use.

Preparation of ash/PEO composites

A known weight of PEO was first dissolved in 25 mL of distilled water, and then 1% (wt.) of ash was added to the polymer solution and stirred for 2 h. The ash/PEO solution was then placed in a petri dish, and a thin

film of ash/PEO composite was obtained after the evaporation of the solvent at room temperature. The same method was used for the preparation of 3%, 5%, and 7% (wt.) ash/PEO composites.

Instrumentation

The SEM micrographs of the gold-coated fractured surfaces (broken in the liquid nitrogen) of the ash/PEO composites were analyzed using a JEOL JSM-5910 SEM. The POM photographs were obtained using a BX-51 POM. The sample was melted on a heater and squeezed between 2 glass slides at 150 °C for 10 min. The thermal properties of the PEO and the ash/PEO composites were studied by TGA and DSC. The DSC analyses were carried out using a Diamond Series DSC PerkinElmer apparatus. A specific amount of sample was sealed in an aluminum sample pan and prepared by compression molding. DSC thermograms were obtained at heating and cooling rates of 10 °C/min under a nitrogen atmosphere in order to diminish oxidation. The TGA thermograms of the composites were obtained in a nitrogen atmosphere at a heating rate of 20 °C/min between 25 °C and 600 °C, using a Diamond TG/DTA PerkinElmer apparatus. The tensile properties were measured using a universal testing machine (Model 100-500KN, Testometric Inc., UK). The tests were carried out at room temperature with a crosshead speed of 50 mm/min. The sample dimensions were 70.9 (gauge length) × 27 (width) × 0.1 (thickness) mm.

Results and discussion

Morphology of PEO

The SEM micrographs of ash (sieved through a screen with a mesh size of 60 μm) and the ash (5% wt.)/PEO composite are shown in Figure 1. Figure 1a shows agglomerates of irregular-shaped ash particles with relatively smooth surfaces. The size of the ash particles was several micrometers. Figure 1b presents the fractured surface (broken in the liquid nitrogen) of the ash (5% wt.)/PEO composite, which indicates that the ash particles were embedded within the polymer matrix. The size of the ash particles in the PEO matrix was less than 3 μm . It was also found that the ash particles were not agglomerated, but were present in a dispersed form in the polymer matrix.

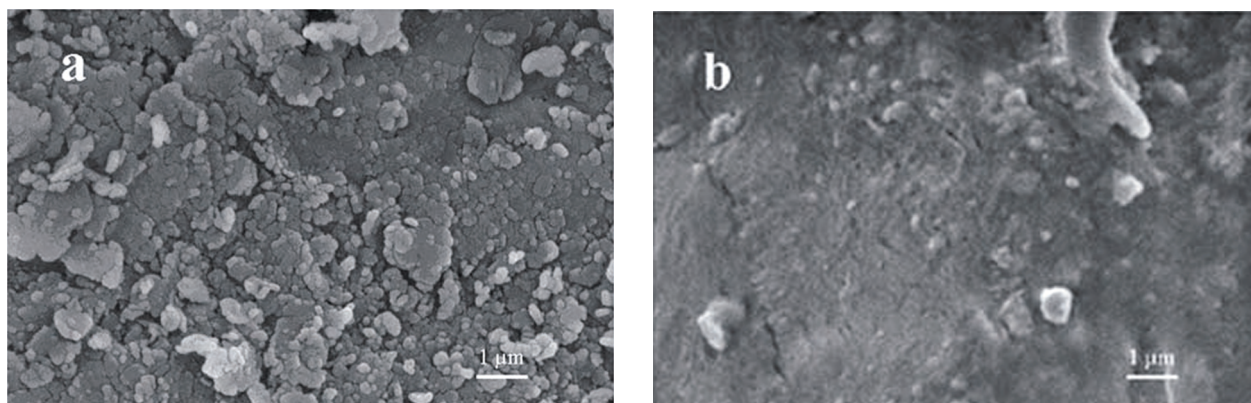


Figure 1. SEM images of the fractured surface of a) ash and b) ash (5% wt.)/PEO.

Crystallization of PEO and ash/PEO

Figure 2 shows the POM microphotographs for pure PEO and the ash (5% wt.)/PEO composite, which were prepared by cooling from the melted state (100 °C). It can be seen that the pure PEO, upon crystallization, showed a considerable size of distinct crystalline spherulites (Figure 2a). The observed spherulites for PEO were similar to the reported pattern.⁹ The POM microphotograph (Figure 2b) showed a smaller size of spherulites for the ash (5% wt.)/PEO composite (ash hinders the crystallization of PEO). The nucleation role of ash in the ash/PEO composite might have caused the small size of spherulites. The crystallization behavior for clay/PEO nanocomposites was studied by Ratna and Chen et al.,^{9,10} and they also found that the size of PEO spherulites decreased when clay was incorporated into the polymer. They reported that the crystallinity initially increased with the incorporation of clay, acting as a nucleation agent, and decreased with the further addition of clay, due to the hindrance offered by randomly oriented clay. Loyens et al.¹¹ reported that the crystallinity of PEO was decreased even by the incorporation of small amounts of clay (less than 1% wt.).

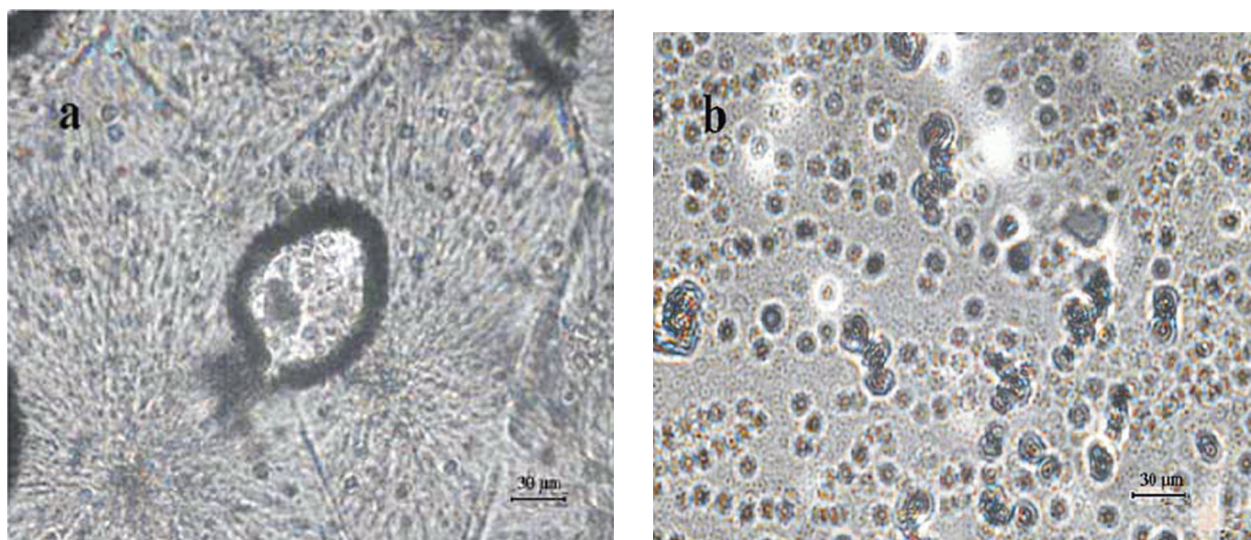


Figure 2. POM micrographs of a) pure PEO and b) ash (5% wt.)/PEO.

Mechanical properties of PEO and ash/PEO

The mechanical properties of polymer composites are associated with the interface strength between the inorganic and the organic phases. In most cases, the inorganic additives show more improved mechanical properties than the organic polymer.¹² The Table shows the mechanical properties of the PEO and ash/PEO composite. The Young's modulus of the pure PEO and the ash/PEO (3% and 7% wt. ash) composites were 225.9, 849.9, and 189.9 N/mm², respectively. The Young's modulus of the composite was enhanced greatly by the incorporation of 3% (wt.) ash, which might be due to the good dispersion of ash in the polymer matrix as well as the improved interfacial adhesion between the PEO and ash particles. However, by the incorporation of 7% (wt.) ash, the modulus was decreased, which might be due to the agglomeration of the ash particles at high concentrations. The elongation at the break behaved as the modulus, which was also increased by the incorporation of 3% (wt.) ash, followed by a decrease with up to 7% (wt.) ash. The elongations at the break

of the pure PEO and the 3% and 7% (wt.) ash/PEO composites were 13.6%, 53.4%, and 26.9%, respectively. The obtained mechanical results indicated that the incorporation of ash up to an optimum level resulted in a considerable reinforcing effect. The increase in the mechanical properties by incorporation of clay in PEO, followed by a decrease at high concentrations, was also observed by Ratna et al.⁹

Table. Mechanical properties of pure PEO and ash/PEO composites (3% and 7% wt. ash).

Sample	Young's Modulus (N/mm ²)	Elongation at break (%)
PEO	225.9	13.6
3% ash/PEO	849.9	53.4
7% ash/PEO	189.9	26.9

Thermal properties of ash/PEO composite

DSC analysis is generally one of the most suitable techniques for the evaluation of first order transitions like melting and crystallization. The composite samples with different amounts of ash were subjected to DSC analyses, and the obtained thermograms of pure PEO and the ash/PEO composites are shown in Figure 3. The DSC curve (Figure 3a) showed that the melting temperature (T_m) of pure PEO was about 66 °C, while the melting temperature peak was decreased slightly with the incorporation of ash in the polymer matrix (Figures 3b-3d). The decrease in T_m was also reported by Maria et al.¹³ in the case of clay/poly(ethylene terephthalate) nanocomposites. They suggested that the nanoscale interactions between the polymer and the clay surface might be results of the formation of less stable crystals during the crystallization from the melting. This study was also supported by Ou et al.¹⁴ and Burnside et al.¹⁵ They presented findings that the melting temperature of poly(ethylene terephthalate) slightly decreased in the presence of nanoparticles, and, furthermore, no reduction in T_m was observed when the amount of nanoclay exceeded 1% (wt.).

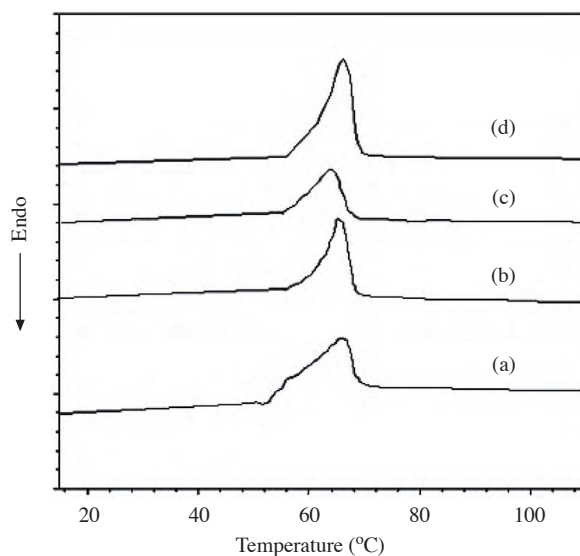


Figure 3. DSC T_m of pure PEO and ash/PEO composites; wt. % of ash: a) 0%, b) 1%, c) 3%, and d) 5%.

Figure 4 shows the TGA curves of the pure PEO and the ash (3% wt.)/PEO composite. The TG curve of the pure PEO showed that mass initially remained constant, up to 200 °C, then turned and decreased abruptly. The pure PEO started its weight loss at about 210 °C and completely decomposed at about 410 °C. The thermogram of the ash (3% wt.)/PEO composite showed that the composite started its weight loss at about 240 °C, which was about 30 °C higher than the pure PEO. The ash (3% wt.)/PEO composite also decomposed completely at about 450 °C. The residual amount, which was contributed to by the ash in the composites, remained at higher temperatures. The TGA thermograms indicated that the incorporation of ash enhanced the thermal degradation of PEO.

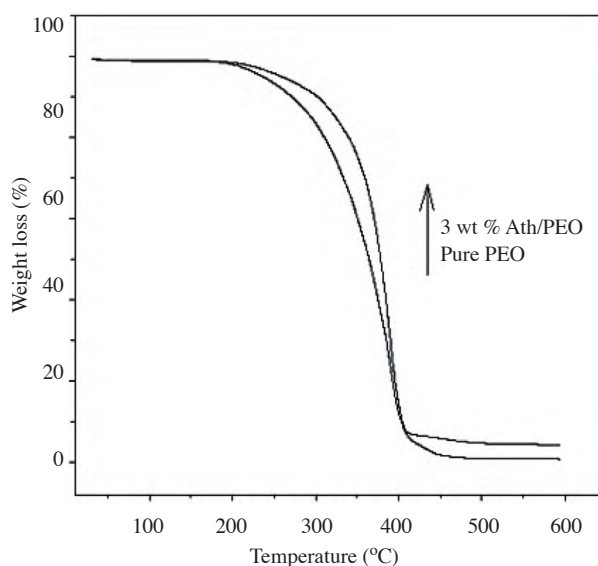


Figure 4. TGA thermograms of a) pure PEO and b) ash (5% wt.)/PEO.

Conclusion

Ash/PEO composite films were prepared by the solution casting technique. The SEM micrographs indicated that the ash particles were disperse and embedded well within the polymer matrix. The size of the ash particles in the PEO matrix was below 3 μm . The size of the spherulites and the T_m of the ash/PEO composites decreased compared to the pure PEO. The mechanical properties (stress and Young's modulus) of the ash/PEO composites were higher than pure PEO, which decreased as the amount of ash in the polymer matrix increased. The decrease in the stress and Young's modulus might be due to the agglomeration of the ash particles at high concentrations. The thermal stability of PEO was improved by the incorporation of ash in the polymer matrix.

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