Study on Mechanical and Morphological Properties of Polyoxymethylene/Al$_2$O$_3$ Nanocomposites

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Abstract – Polyoxymethylene (POM)/Al$_2$O$_3$ nanocomposites were prepared by melting-blend with a twin screw extruder. The effect of Al$_2$O$_3$ on mechanical and morphological properties of POM/Al$_2$O$_3$ nanocomposites and the dispersion state of Al$_2$O$_3$ particles in POM matrix were investigated. The results showed that the tensile strength and Young’s modulus the nanocomposites increased by adding Al$_2$O$_3$ particles in a range of 0.5-1 wt% and then decreased. POM/Al$_2$O$_3$ nanocomposite with increasing filler content showed a decreased in the impact strength. The microstructure of neat POM and POM/Al$_2$O$_3$ nanocomposites was observed by scanning electron microscopy (SEM). It can be found that the dispersion of Al$_2$O$_3$ nanoparticles was relatively good in a low content, but poor dispersion in high content. Moreover, the agglomeration of Al$_2$O$_3$ nanoparticles in POM matrix increased with increasing Al$_2$O$_3$ content. This observation was confirmed by the result of the mechanical properties.

Keyword: Polyoxymethylene, Aluminium Oxide, Polymer Nanocomposites, Morphology, Mechanical Properties
1. Introduction

The resin-based nanocomposites are a new class of materials where nanoscale particles are finely dispersed on a resin matrix. They have been reported to exhibit markedly improved properties as compared to the pure polymers [1-4]. The addition of inorganic component have improved the mechanical, thermal, barrier properties, electric conductivity of polymer [5-8]. For example, Wang et al. [7] studied the mechanical properties of calcium carbonate (CaCO₃)/low-density-polyethylene (LDPE) nanocomposites. The results showed that the tensile property and the flexural modulus of the system evidently increased by the addition of CaCO₃. The CaCO₃ particles were dispersed in the matrix in the nanometer scale. Wang et al. [8] studied the properties of CaCO₃/acylonitrile-butadiene-styrene (ABS) nanocomposites and CaCO₃/ethylene-vinyl acetate copolymer (EVA)/ABS nanocomposites. The results showed that CaCO₃ nanoparticles could increase flexural modulus of CaCO₃/EVA/ABS nanocomposites and maintain or increase their impact strength for a certain nano-CaCO₃ loading range. The tensile strength of the nanocomposites decreased by adding CaCO₃ nanoparticles. The microstructure of pure ABS and the nanocomposite was observed by scanning electron microscopy (SEM). It can be found that CaCO₃ nanoparticles were well-dispersed in ABS matrix at nanoscale. The morphology of the fracture surfaces of the nanocomposites revealed that when CaCO₃/EVA/ABS nanocomposites were exposed to external force, CaCO₃ nanoparticles initiated and terminated crazing, which can absorb more impact energy than neat ABS.

Al₂O₃ is the most cost effective and widely used material in the family of engineering ceramics. But its use as a nanomaterials for reinforcement is limited. The raw materials from which this high performance technical grade Al₂O₃ is made are readily available and reasonably priced. In addition to this it has excellent properties like hard, wear-resistant, excellent dielectric properties, resists strong acid and alkali attack at elevated temperatures, good thermal conductivity, excellent size and shape capability, high strength and stiffness, etc. With this excellent combination of properties and an attractive price, it has a very wide range of applications [9].

The properties of the nanocomposites used Al₂O₃ as filler have been studied. Kar et al. [9] prepared the nanocomposites of ABS and α-alumina (Al₂O₃). The results found that the ABS/Al₂O₃nanocomposites had slightly higher Young’s modulus, but lower tensile strength, strain% at break, flexural and impact strength than the virgin ABS. But its flexural modulus increases with increasing Al₂O₃ content in ABS matrix. The fractured surfaces of tensile test samples were also examined through SEM and showed that the ductile fracture of ABS is converted to brittle fracture with addition of Al₂O₃. Siengchin et al. [10] compared the mechanical, thermal, and rheological properties of polystyrene (PS)/Al₂O₃ composites in which the Al₂O₃ particles were micro and nanoscaled dispersed. The stiffness, measured in dynamic-mechanical thermal analysis (DMTA) and tensile tests, of the nanocomposites was markedly higher than that of the microcomposites. The tensile strength and the heat distortion temperature (HDT) values were also enhanced.

Polyoxymethylene (POM) resin is a kind of widely used engineering thermoplastic material, which exhibits good fatigue resistance, creep resistance and high impact strength. POM and POM composites have been widely used as self-lubricating materials in many fields such as automobile, electronic appliance and engineering [11].

The addition of such nanoparticles to polymers leads to a significant improvement in mechanical properties like stiffness, tensile and shear strength, and fracture toughness; in addition, a certain dependence on the nano-filler amount, size and morphology has been reported [12]. So this work investigated the effect of Al₂O₃ on the mechanical and morphological properties of POM nanocomposites filled with Al₂O₃. POM/Al₂O₃ nanocomposites were prepared by melting-blend with a twin screw extruder.

2. Research Methodology

2.1. Materials

POM was produced by Polylastics Co, Ltd, under the trade name of “DURACON M90-44”. The melting temperature of the POM was around 165 °C. Melt flow index of POM equal to 8.9 g/10 min. Al₂O₃ in the form of a white powder with average particle sizes of 36.9 nm was produced by Ajax Finechem Co., Ltd.

2.2. Preparation of POM/Al₂O₃ Nanocomposites

POM pellets and Al₂O₃ particles were dried in an oven at 110 °C for 3 h before melt extrusion. The POM/Al₂O₃ nanocomposites were melt-compounded in the desired compositions (0.5, 1, 2, and 4 wt% of Al₂O₃) in a twin screw extruder at temperatures in a range of 150-200 °C and a screw speed of 50 rpm. After compounding, the nanocomposites were compression-molded into the standard dumb-bell tensile bars and rectangular bars at temperature 180 °C for 20 min.

2.3. Characterization of POM/Al₂O₃ Nanocomposites

Tensile tests were conducted using a universal tensile testing machine (LR 50K, Lloyd instruments). The tensile tests were performed at a crosshead speed of 50 mm/min. The tensile strength, the percent strain at break
and Young’s modulus was determined using dumbbell shaped specimens. Each value reported, represented the average of five samples.

Impact strength is defined as the ability of a material to resist breaking under a shock loading or the ability to resist the fracture under stress applied at high speed [9]. The impact strength of the nanocomposites was carried out in Pendulum impact testing (Zwick/material testing August-Nagelstr.11.D-89079 Ulm) at room temperature. Five specimens were prepared for each test and the data reported were the average of five tests.

The dispersion quality of the Al2O3 nanoparticles was determined by SEM. All specimens were coated with gold before SEM study. The fractured surfaces from impact test were also studied by using SEM.

3. Results and Discussion

3.1. Mechanical properties of the nanocomposites

The evolution of tensile stress and strain curves at different Al2O3 contents is shown in Fig. 1. All the curves display a linear Hookean range at low strain (<25%) then the plastic deformation under roughly constant stress was maintained until the failure occurs. As the filler loading increased in a range of 0.5-2.0 %wt, the nanocomposites became more ductile. As the filler loading increased, the fraction of thermoplastic polymer decreased and interfacial area increased, which increased the toughness. The increase of the Al2O3 content, both the modulus and the tensile strength increased until 1 wt%, while the percent strain at break decreased after adding Al2O3 more than 1 wt%. These different effects are better depicted in Figs. 3-4.

POM/Al2O3 nanocomposites. It can be seen that the percent strain at break increased at 0.5-1.0 wt% Al2O3 and decreased at high Al2O3 content. The percent strain at break showed a maximum at 1 wt% of Al2O3. The addition of Al2O3 more than 1 wt% showed the percent strain at break of the POM nanocomposites was lower than neat POM. The decreased ductility of the nanocomposites, as compared to neat polymer, is ascribed to the presence of fillers or agglomerates which act as stress concentrators: when the defects initiated at the filler-matrix interface become larger than the critical crack size, failure occurs [12].

Fig. 2 shows the tensile strength of POM/Al2O3 nanocomposites. Notice that Young’s modulus of virgin POM was lower than that of nanocomposites after adding 0.5-1 wt% of Al2O3. A rise in the Al2O3 concentration from zero to 1 wt% led to small improvement in Young’s modulus. The reinforcement effect caused by Al2O3 in nanocomposites was due to a good dispersion of Al2O3 nanoparticles at low content in the matrix supported from SEM micrograph. With further addition of Al2O3 (2 wt%) in nanocomposites there was no improvement in Young’s modulus, but at 4 wt%, decreased in Young’s modulus is observed and shown in Fig. 4.

Fig. 3 shows the percent strain at break of POM/Al2O3 nanocomposites. It found that the tensile strength increased when the addition of Al2O3 was 0.5-1 wt%. With further addition of nanosized Al2O3, tensile strength decreased and was lower than pure POM.

Fig. 4 presents Young’s modulus POM/Al2O3 nanocomposites. Notice that Young’s modulus of virgin POM was lower than that of nanocomposites after adding 0.5-1 wt% of Al2O3. A rise in the Al2O3 concentration from zero to 1 wt% led to small improvement in Young’s modulus. The reinforcement effect caused by Al2O3 in nanocomposites was due to a good dispersion of Al2O3 nanoparticles at low content in the matrix supported from SEM micrograph. With further addition of Al2O3 (2 wt%) in nanocomposites there was no improvement in Young’s modulus, but at 4 wt%, decreased in Young’s modulus is observed and shown in Fig. 4.

Fig. 5 shows the variation of impact strength with addition on Al2O3 nanoparticles in POM thermoplastic. The impact strength of virgin POM was approximately...
8.6 mJ/mm². But with addition of nanoparticles, the impact strength decreased with increasing Al₂O₃ content. This decrement is due to the brittle failure of nanocomposites confirmed by SEM study. Thus the addition of Al₂O₃ did not improve the impact properties of POM.

![Fig. 4. Young’s modulus of pure POM and POM/Al₂O₃ nanocomposites.](image)

**Fig. 4.** Young’s modulus of pure POM and POM/Al₂O₃ nanocomposites.

![Fig. 5. Impact strength of pure POM and POM/Al₂O₃ nanocomposites.](image)

**Fig. 5.** Impact strength of pure POM and POM/Al₂O₃ nanocomposites.

### 3.2. SEM study

In order to understand the role of Al₂O₃ on the toughness of POM, the impact-fractured surface of pure POM and POM/Al₂O₃ nanocomposites were characterized by SEM, and the results are shown in Fig. 6. For pure POM, the impact-fractured surface was flat and smooth, which is the typical character of brittle fracture during the impact process.

From Fig. 6(b-e), it was clear that the fracture section of the nanocomposite has extensive plastic deformation, leading to a ductile fracture. Al₂O₃ nanoparticles, as stress concentration sites, initiated and terminated the crazing in impact testing [8]. At the same time, they caused POM chain to create shear yielding, which improved the toughness of the nanocomposites. However, if the agglomerates of Al₂O₃ nanoparticles formed in the matrix as shown in Fig. 7(c-d), it caused decreased the impact strength of POM/Al₂O₃ nanocomposites.

It is difficult to disperse Al₂O₃ nanoparticles well in thermoplastics since the surface of Al₂O₃ nanoparticles has hydrophilic groups, and these nanoparticles have a strong tendency to agglomerate due to a large surface energy. The dispersion of the Al₂O₃ nanoparticles in POM matrix is presented in Fig. 7. The observation found that the dispersion of Al₂O₃ nanoparticles was good at low Al₂O₃ content but the aggregates appeared at high loading. Fig. 7(c,d) displayed the aggregate was bigger with a large content of the Al₂O₃ nanoparticles.

![Fig. 6. SEM micrographs showed the impact-fractured surface of pure POM and POM/Al₂O₃ nanocomposites.](image)

**Fig. 6.** SEM micrographs showed the impact-fractured surface of pure POM and POM/Al₂O₃ nanocomposites.

(a) pure POM (b) POM/Al₂O₃ 0.5 wt% (c) POM/Al₂O₃ 1 wt% (d) POM/Al₂O₃ 2 wt% (e) POM/Al₂O₃ 4 wt%
that the impact-fractured surface of pure POM was flat and smooth, which is the typical character of brittle fracture during the impact process. And the fracture section of the nanocomposite has extensive plastic deformation characterizing brittle fracture. The dispersion of Al$_2$O$_3$ particle in POM matrix was good at low Al$_2$O$_3$ content but the aggregates appeared at high loading.

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