Effect of Filler on Flexural Properties and Scanning Electron Microscopic Analysis of Fractured Surfaces of Nanocomposites

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Abstract—In this study, a nanocomposite containing polypropylene (PP) and nano α-Al2O3 particles was prepared using a Hake internal mixer. Flexural tests showed that flexural properties of composite enhance by addition of nano α-Al2O3 particles and dispersant agent to the polymer. Flexural analysis of PP/nano α-Al2O3 composites prepared with and without dispersant showed the maximum values of 50.5 and 55.88 MPa for flexural strength and 1954 and 2818 MPa for flexural modulus, respectively. Scanning electron microscopic observations of composite showed that fracture surface becomes more roughed by increasing the content of filler loading.

Keywords—Composite, Scanning electron microscopic, Flexural tests, polypropylene

1. INTRODUCTION

Polymer materials are, fundamentally, devised for definite relevancies because of their construction and properties. Regularly, a polymer needs modifications for a superior range of functions that may need dissimilar structural or physical properties. One modification technique is adding fillers to a polymer to generate a composite with improved properties, such as enhancement in mechanical strength, electrical conductivity or thermal stability. The utilization of nanoscale fillers to supplement the properties of polymers has led to significant improvement in polymer nanocomposites (PNC) [1, 2]. PNC is an original class of composite materials in which one of the constituents has dimensions of 1–100 nm. The nanoscale fillers used as reinforcement materials for nanocomposites contain nanofibers, nanoplatelets, nanoclays, etc. PNC exhibited superior mechanical performance and improved barrier properties at very low loading levels compared to conventional filler composites.

Improvements on mechanical properties, such as stiffness and toughness, dimensional, barrier and thermal properties as well as retardant enhancements, with respect to bulk polymer, are usually observed [3].

However, in order to extend nanocomposite materials with excellent mechanical properties, strong chemical bonding between the reinforcement is compulsory [4]. The other key issues in preparation of PNC are: uniform dispersion of nanoparticles against their agglomeration due to Vanderwaals bondling, alignment of nano particles in the matrix, volume fraction, manufacturing rate and cost effectiveness. In order to improve dispersion and interfacial property of PNC, there are at least two ways in practice; the first one is to treat the surface of nanoparticles and the second one is to modify the surface properties of Polymer [5]. The key issues in the polymer nanocomposites are: uniform dispersion of nanoparticles against their agglomeration due to vanderwaals bondling in the processing of nanocomposites, alignment of nanofiller in the matrix, volume fraction, manufacturing rate and cost effectiveness[6]. In order to improve the dispersion and interfacial property of polymer nanocomposites, there are at least two ways in practice. The first is to treat the surface of nanoparticles. The second is to modify the surface properties of PP [7]. However, in these composite systems, flexural properties and morphological properties are very important factors that affect the quality of the final products. Among different types of polymers used, Polypropylene is a semi crystalline thermoplastic that is characterized by light weight, low cost, easy processing, high mechanical strength, excellent chemical stability and electrical properties [8, 4]. In this study, nano α-Al2O3 was used to prepare nanocomposites. Nano α-Al2O3 was chosen as filler due to its special combined chemical and physical properties such as excellent resistance to heat and wear, high specific strength and good oxidation resistance. In the present study, dispersant has been used and applied for the surface treatment of nano α-Al2O3. The effects of dispersant on the mechanical behavior of PP/nano α-Al2O3 composites were investigated.

II. MATERIALS AND METHODS

2.1. Materials

The PP grade 600G (melting temperature and melt flow rate are 165°C and 11 g/min, its density is 900 kg/cm3) supplied by
Petronas Polymers Marketing and Trading Division Malaysia was used as a starting material. Nano α-Al₂O₃ (average particle size of 20-30 nm and density of 3106 kg/m³) as produced by Mirjalili [9,10]. Titanium dioxide powder with a minimum assay of 98 % supplied by Fisher Chemicals Sdn. Bhd., Malaysia was used as a coupling agent. Sodium dodecylbenzene sulfonate (SDBS) (Merck, German) was also used as a dispersant.

2.2. Surface Treatment of nano α-Al₂O₃

Ultrasonication, (KQ2200DEUltrasonic Cleanser, 100W, Kunshan of Jiangsu Equipment Company, China) which is a conventional method for dispersing the extremely entwined or aggregated nanoparticle samples, was used for preparation of miscellaneous aqueous nano-suspensions. About 0.1 g nano α-Al₂O₃, 99.8 g distilled water and 0.1 g SDBS as an anion surfactant were straight mixed in a 150 ml beaker. The suspension was then sonicated for 1 h and dried at 80 ºC for 4 hours [11].

2.3. Preparation of PP/ Nano α-Al₂O₃ Composites

Samples

PP/ nano α-Al₂O₃ composites containing 1, 2, 3, 4 and 5.0 wt% of fillers respectively were prepared for investigation. The optimal content of coupling agent (TiO₂) was considered as 2 wt % of the filler, according to the results presented in [12]. The melt blending of PP and nano α-Al₂O₃ powder was carried out using Thermo Haake Poly Drive with RheominxR600/610 blending machine at 175 ºC with rotor speed of 50 rpm. The melt–compound of PP/nano α-Al₂O₃ was then formed by Hsinchu hot press machine, in the size of 15x15 cm. The compound was preheated under the pressure of 150 kg/cm² at 180 ºC with 10 times of compression bumping. Finally, the sheet obtained was directly cooled using the cold press for 2.5 min of cooling cycle.

2.4. Characterization

2.4.1. Flexural tests

Flexural tests were performed according to the ASTM D790-98 using Instron Universal Testing Machine (Model 3365), with a load cell of 5 KN. The test process used was three-point loading system utilizing center loading. The cross-speed and span length were set to 3 mm/minute and 70 mm, respectively. The specimens were cut in to rectangular sizes with 3 mm thickness, 12 mm width and 124 mm length.

2.4.2. Scanning Electron Microscope (SEM)

Microscopic observation throughout the Scanning Electron Microscope (SEM) was achieved using Philips XL 30 ESEM operated at 20 to 30 KV.

III. RESULTS AND DISCUSSIONS

3.1 Flexural Tests

Flexural properties are considered when the maximum stress and strain occur outside of the surface of the test bar [13]. Flexural tests have more advantages than tensile tests. For instance, if a material is used in the type of a beam and if the service breakdown occurs in bending, then a flexural test is more important for designing than a tensile test. The flexural specimen is reasonably simple to organize without outstanding strain. The specimen configuration is also more difficult in tensile tests. Additional advantage of the flexural test is that at small strains, the real deformation is satisfactorily great to be measured precisely [14].

3.1.1. Flexural Strength

Flexural strength (FS) is the capacity of a material to resist the bending forces applied perpendicular to its longitudinal axis [15]. Fig. 1 shows an improvement of flexural strength for composites versus filler loading. Referring to the figure, it is noted that α-Al₂O₃ nanoparticles improve the flexural strength of composite by rising the maximum filler stress during the deformation under the constant load given. The highest flexural strength was achieved at 55.88 MPa for a composite that contains 5 wt% nano α-Al₂O₃ particles, and dispersant was used for its preparation.

Fig. 1. Flexural strength of PP/nano α-Al₂O₃ composites at various loading

It is expected that the flexural strength to be further enhanced with the addition of nano α-Al₂O₃ particles loading, since they are rigid materials and by increasing the amount of loading, rigidity of the composites are increased, and hence ductility are decreased.

3.1.2. Flexural Modulus

While flexural strength measured the bending strength of the composites, flexural modulus (FM) pointed out that the PP / nano α-Al₂O₃ composites emulate the rigidity of fillers and change from the normally flexible plastic to stronger materials. Fig.2 shows a patent indicating that nano α-Al₂O₃ particles cause the rising in flexural modulus, especially in the presence of dispersant.
Rising in flexural modulus is mostly certified to the natural rigidity of α-Al2O3 nanoparticles and limitation of the chain mobility. An improvement of about 60% was found in modulus value of the composites that dispersant was used in the preparation procedure. The improvement in modulus value of the composite was decreased to 14.45% when no dispersant was used. In case the dispersant was used for nanocomposite preparation, involvement of the larger surface area by nano α-Al2O3 particles develops better interfacial interactions with the polymer matrix, and hence leaded to a better property enhancement and enhanced the values of flexural strength and flexural modulus [16,17,18].

3.2. Morphological Analysis of PP/ Nano α-Al2O3 Composites

To study the morphologies and microstructures of the composites, Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) were carried out. The scanning electron microscope (SEM) used a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens.

3.2.1. SEM Analysis of Tensile Test Fractured Surface of PP/ Nano α-Al2O3 Composites

SEM observations on fractured surfaces of the fabricated PP/ nano α-Al2O3 composites were conducted to check the fracture behavior due to the tensile loading. Fig. 3 shows the SEM micrograph of the tensile fractured surface of pure PP samples.

Fig. 3. SEM micrograph of the fractured surface of pure PP sample

Pure PP has moderately smooth fractured surface and exhibits no symptom of plastic deformation or drawing. It is obvious that the comprehensive melting of PP was obtained while no void, hole and impurities were detected. PP pellets were suitably bonded and well continued among each other. At particularly low-grade nano α-Al2O3 particles loading (refer to Fig.4), there was a good wetting behavior between the filler and matrix. Good dispersion and wetting situations may generate desired stress distribution from the matrix to the filler throughout the tensile loading [19,20]. Agglomeration and rough dispersion of nano α-Al2O3 particles can clearly be observed particularly at higher filler loading. Therefore, a pitiable stress transfer from matrix to the filler is accounted for a decline in tensile properties with increasing of the filler loading.

Fig. 4. SEM micrograph of the fractured surface of PP/2%nano α-Al2O3 composites in the presence of dispersant (b) in the absence of dispersant

Morphology of the PP/nano α-Al2O3 composites with 5 wt% of the filler loading, shown in Fig.5.Coleman et al. [21] reported that debonding will occur when the matrix fails under the large shear stress near the interface. This condition became more agglomerated and serious with the existence of large number of voids.

Fig. 5.SEM micrograph of the fractured surface of PP/5%nano α-Al2O3 composites (a) in the presence of dispersant (b) in the absence of dispersant

These micrographs reveal that at higher filler loading, the nano α-Al2O3 particles tend to agglomerate, and therefore large holes or voids occur between nano particles and
matrix. This occurrence will permit the crack to broadcast at quicker rate (less adhesion), which affects the morphological suggestion exposed by the rougher surface as observed in Fig. 5. The results proven that the interfacial adhesion of PP/α-Al2O3 nanocomposite prepared using dispersant can distribute the higher mechanical properties, because the aggregates are shifting to the primary particles during the ultrasonication process.

III. CONCLUSIONS

Polypropylene nanocomposites containing 1-5 wt% of nano α-Al2O3 particles were prepared using a Hake internal mixer in the present study. Dispersion of nano α-Al2O3 powders through the ultrasonication process with dispersion was investigated and used for preparing PP/ nano α-Al2O3 composites. Flexural strength and modulus of PP / nano α-Al2O3 composites were improved with increasing of the nano α-Al2O3 loading. The effect of dispersant (SDBS) on mechanical properties of PP / nano α-Al2O3 composites indicated that SDBS is comparable to a surfactant that breaks up the massive agglomerates into larger ones and to support the compatibility of nano α-Al2O3 with PP. From SEM micrographs, it was established that the presence of dispersant directly affected the dispersion of particles. As a conclusion, it can be stated that the PP/ nano α-Al2O3 composites had an enormous potential to be considered in a lot of engineering requests, mainly for the purposes that need good strength, high stiffness and excellent toughness properties, as well as good physical and chemical resistance characteristics.

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REFERENCE
