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Dynamic Mechanical and Roughness Analyses of Polypropylene/Kaolin Nanocomposite

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ABSTRACT. The topography and dynamic mechanical properties of polypropylene (PP) incorporated with kaolin were studied to appraise their true engineering functions. Atomic force microscopy (AFM) and dynamic mechanical analysis (DMA) were accomplished to investigate the effects of different kaolin loadings on the topography and dynamic mechanical properties of the PP/kaolin nanocomposite. The PP was discovered to be inside the interparticle space of the kaolin as revealed by AFM. The PP/kaolin nanocomposite demonstrated a fine and smooth surface roughness (R_a) of the matrix with increase kaolin loading by maximum of 87.68% and hence displayed improved adhesion properties within matrix and the kaolin; that was accountable for the enhancement of the tribological properties of the nanocomposites. Increase in kaolin loading can significantly enhance the storage modulus and reinforcing effect of PP/kaolin nanocomposite.

Keywords: *Polypropylene, Kaolin, Roughness, Dynamic mechanical analysis;*

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1. INTRODUCTION

Polymer nanocomposites involve developing area of contemporary research and have established considerable devotion from industries and academia. Nanocomposites reveal unforeseen hybrid properties. Several features of these composites are discovered by numerous scientists. The nanocomposites produced from clays as fillers have been progressively innovated and explored by many researchers. They have shown extraordinary upgraded properties, including superior barrier property, thermal stability, enhanced mechanical properties and reduced flammability [1]. Specifically, PP is an essential class of polymer material due to its remarkable features and distinct applications. The development of nanocomposites require intercalation of clay layers into organic compounds, causing a stretched and organophilic layered structure [2]. Melt intercalation and in situ polymerization are common techniques by which organoclays are incorporated into the polymer matrix. The advances in substantial enhancements of mechanical properties are offered by these methods through dispersal of the clay in the polymer matrix [3,4]. The desired materials of high-technology instruments and applications are therefore obtained from these novel nanocomposite materials.

The role of PP/clay composites is motivated as a result of their potential uses in the automotive industry. Little investigation on PP/clay nanocomposites are conducted by Toyota group researchers and shows that dynamic storage moduli of the Polypropylene (PP) clay hybrids (PPCHs) remained greater than those of PP lesser at approximately 150 °C [3]. Dynamic mechanical properties of PP composites and dispersion rate were reported after synthetizing PPCH via bare melt-blending of three constituents; PP, maleic anhydride improved polypropylene oligomers (PP-MA), and clays which are incorporated into stearylammonium [3]. The reports done by Wang et al., [5] investigated the impacts of using different loading of three fillers silica (SiO_2), calcium carbonate ($CaCO_3$) and phenolic microsphere (PFM) in producing PP composites. They found that the PP composites displayed 1.4 times greater impact strength property than the pristine PP.

However, earlier studies of PP/clay nanocomposites on dynamic mechanical properties have been frequently dedicated to montmorillonite, and only few studies were established using kaolin. Kaolin is the utmost common 1:1 (two layer sheet) natural clay mineral with its fundamental unit containing tetrahedral sheet of SiO_2, siloxane units and an octahedral sheet of alumina ($AlO_2(OH)_4$) [6]. When modified, kaolin particles can become uniformly dispersed in the polymer matrix and be used as a functional filler for polymers due to its light colour, distinct stratified configuration, and obtainability [7,8]. In this work, studies on enhancement of dynamic mechanical properties as a result of PP/kaolin nanocomposites produced by melt intercalation of PP, kaolin and PP-MA as compatibiliser at different loadings of kaolin were established. AFM studies were conducted to ascertain the surface morphology and roughness of the PP/kaolin nanocomposite.

2. MATERIALS AND METHODS

2.1 Materials. Polypropylene (PP) copolymer of Titanpro SM-240 resin grade used in the present research, was provided by Lotte Chemical Titan (M) Sdn. Bhd. The SM-240 was in pellet shape with density of 0.894 gcm^{-3}, and melt flow index (MFI) of 230 ºC/2.16 kg as measured according to ASTM D1238. The coupling agent, Polypropylene grafted maleic anhydride (PP-g-MA) was supplied by Sigma Aldrich. Whereas kaolin was acquired from Kaolin (Malaysia) Sdn. Bhd. The distinct kaolin samples were sieved to particle size of 45 µm. The kaolin particles exhibit chemical composition in wt. % as: SiO_2 57.633, Al_2O_3 37.766, Fe_2O_3 0.86, MgO 0.596, CaO 0.346, K_2O 1.801, TiO_2 0.605 and P_2O_5 0.311. Lastly, the di-amine carbide powder (urea) was obtained from Bendosen Chemical Company, China.

2.2 Sample Preparation. Method of Valaskova et al. [9] and Suzi et al. [10] were used to conduct the surface treatment of kaolin. Planetary milling of 250 g of kaolin with 165 g of urea was homogeneously carried out for 15 min using a planetary mill (Fritch Pulverisette 6). Prolonged heating of regulated mixture was carried out at 95 ˚C in a laboratory oven (MummertLinn High Therm) for 48 hours to accomplish intercalation of urea into kaolin interlayer. Ultrasonic bath (Elmasonic E30H) was applied for 5 hours long at low temperature and delamination of kaolin was achieved. The separation of urea from the mixture was carried out by mixing 50 g of treated kaolin with 1.5 L of distilled water in a beaker and stirred. Sedimentation was used to detach the liquid portion from solid portion and subsequently dried at 60 ˚C for 24 hours in an oven.

2.3 Preparation of PP/K Nanocomposite. The nanocomposites were produced by melt intercalation method with heated two-roll mill model PW 3000 for 20 min, with speed of 10 rpm. The temperature rolls are 190° and 177 °C for front and back respectively. The coupling agent used was PP-g-MA. The nanocomposites consists of 1, 3, 5 and 7 wt.% of kaolin as filler.

2.4 Methods. Samples were analysed using dynamic mechanical test (TA instruments DMA Q800) at temperature of 25 °C - 170 °C, heating rate 5 °C/min and vibration frequency of 1 Hz. The verification of surface roughness and topography of samples were carried out by Atomic force microscopy (AFM) of SPM microscope (XE-100 Park Systems) on a contact mode.

3. RESULTS AND DISCUSSION

3.1 Dynamic Mechanical Analysis (DMA). DMA was used in this work to determine the effect of loading kaolin nanoparticle on dynamic mechanical operation of PP/K nanocomposites. Díez-Pascual et al [11] reported that viscoelastic properties of the polymers are affected when polymer matrix are incorporated with dispersed inorganic fillers. Fig.1 displays the temperature dependence on the loss tangent (tan δ), storage modulus (E′) and loss modulus. PP and its nanocomposites consisting of different kaolin concentration are extremely advantageous in determining the operation of samples under stress and temperature.

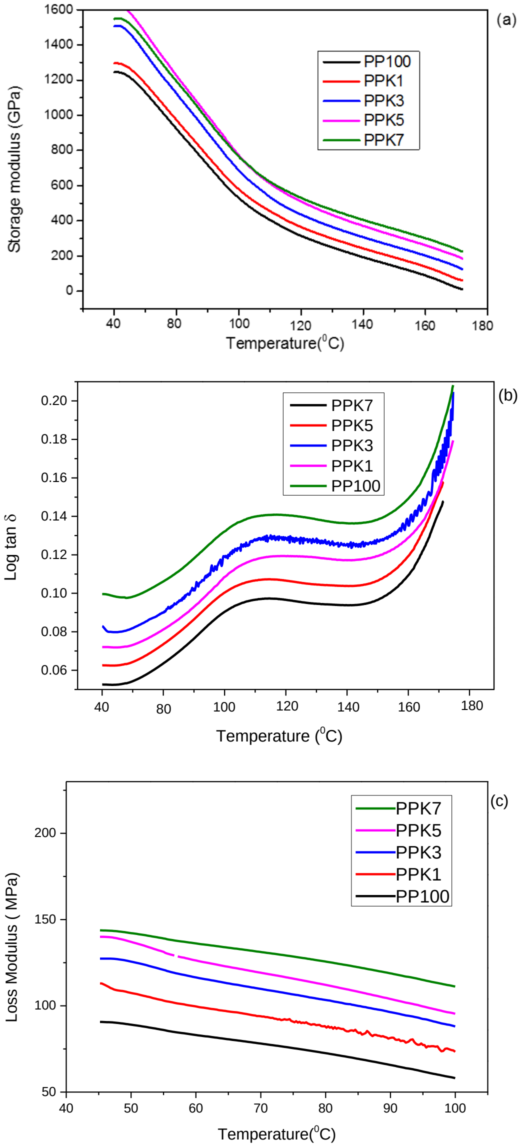

Fig. 1 DMA spectra of pristine PP and PP/K nanocomposites (a) Storage modulus, (b) Tan δ and (c) Loss modulus

The ability of a material to store input mechanical energy and elasticity is usually illustrated by storage modulus [12]. The storage modulus of the nanocomposite is greater than that of the pristine polymer as illustrated by DMA. The storage modulus obtained with pristine PP was 1200 GPa at 40 ºC, and it reduces over the whole temperature range. Relaxation of the amorphous region is related to the plateau region detected at range of temperatures of 100 to 110 °C. The more kaolin loading, the higher the storage modulus at various temperature range. Upon loading different wt % of kaolin, the storage modulus at 43 °C rise to 12,200, 12,500, 12550 and 12,600 GPa for nanocomposites compositions of 1, 3, 5 and 7 wt.% kaolin respectively, which is virtually 9.5% boost of storage modulus compared to that of pristine PP crystals. Hence, PP/K nanocomposites display greater storage modulus through the evaluated temperature range. The result is in agreement with Cabedo et al. [13]. Indentification in the percent of crystallinity is usually due to the rise in the storage modulus. Thus, the rise in the storage modulus might be ascribed to the reinforcing effect of kaolin complemented by the homogeneous dispersion (lacking of agglomerates) of kaolin within the polymer matrix.

Measurements of the energy dissipation (damping) capacity of the material can be investigated by tan δ curve, which exhibits peak at 105 °C and additional broad peak around 149 °C for pristine PP. The glass transition temperature (Tg) is correlated to the initial peak at 105 °C and damping within the crystalline lamellae occurs in subsequent peak at 149 °C. At about 170 °C an intense fall occurs in the storage modulus and a rapid rise in tan δ owing to the onset of melting of PP crystals.

Fig. 1c displays the deviation of the temperature as a function of loss modulus E″. The loss modulus E″, expresses the sticky feature of the constituent together with ability of the material to dissipate energy. Conforming to Fig. 1c, pristine PP has lower loss modulus compared to PP/K nanocomposites. PP nanocomposite reformation with modified nanoclay affected rise of E″ at low temperature zone. Loss modulus decreases at higher temperature region. Similar result was obtained by Benmesli and Riahi [14].

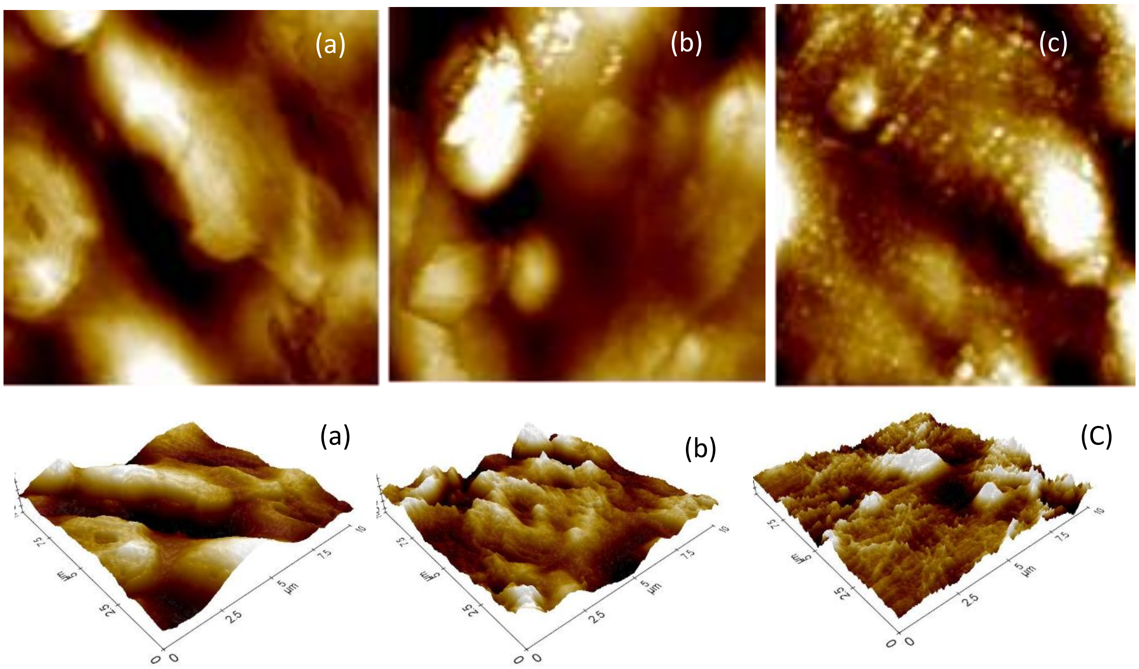

Fig. 2 AFM topographic images of (a) PP, (b) PP/K 3%, and (c) PP/K 5 %

3.2 Atomic Force Microscopy. Fig. 2 illustrates the surface roughness properties of pristine PP and PP/K nanocomposites consisting 3% and 5% nanoclay particles in 2d and 3d topography with uniform dispersal of kaolin in PP matrix. The PP/K nanocomposite shows higher surface roughness (Fig. 1c) compared to pristine PP which exhibit smoother surface (see Fig. 1a). In order to amend surface and bulk properties of functioning materials, inorganic additives are constantly incorporated in polymer matrix [13-15]. Typically, owing to numerous reasons such as size, mobility, end group functionalities, relative composition and molecular structure; organic additives are capable to move to the surface of polymers. Significant higher roughness of PP nanocomposite surface occurs by the increase in kaolin loading as filler, hence increase roughness to about 86.7%. This result is confirmed by reports of improvement in mechanical properties through DMA studies above. These findings are in agreement with Parvinzadeh et al. [16] & Hajiraissi and Parvinzadeh [17].

4. SUMMARY

The study highlighted the role of kaolin in PP nanocomposite in enhancing dynamic mechanical properties and increasing surface roughness to about 86.7%. The integration of kaolin into PP matrix with increase kaolin loading can highly upgrade the storage modulus. It has proven the exhibit of a laudable reinforcing effect to PP/K nanocomposite for the benefit of technological developments.

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