Jurnal Teknologi

INFLUENCE OF SONICATION ASSISTED DISPERSION METHOD ON THE MECHANICAL AND ELECTRICAL PROPERTIES OF NYLON 66/NANO-COPPER NANOCOMPOSITE

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> Accepted 13 March 2017

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Graphical abstract Without With Sonication Sonication 3E-13 1.6E-15 $\sigma \approx 10^{-13}$ $\sigma pprox 10^{-15}$ 2.5E-13 1.4E-15 1.2E-15 2E-13 1E-15 1.5E-13 8E-16 6E-16 1E-13 4E-16 5E-14 2E-16 Percentage of nano Percentaga of nanocopper (wt%) Electrical Conductivity (S/m)

Abstract

Nylon 66 is a well known engineering polymer with excellent mechanical and thermal properties. However, its poor electrical conductivity restricts its application for conductive material. In this study, nano-copper particles are added into nylon 66 polymer matrix to enhance the electrical conductivity value of the nanocomposite. Sonication assisted dispersion method was used to achieve well dispersed nanomaterials through reasonable exfoliation of the nano-copper particles in the nylon 66 polymer. The impact of sonication on the mechanical performance and the electrical conductivity of nanocomposite were evaluated. The sonication was found to effectively reduce agglomeration of nano-coppers in nylon 66, and improved both mechanical performance and electrical conductivity of the nano-copper amount, nanocomposite with sonication-treated nano-copper consistently showed higher hardness and impact strength than nanocomposite without sonication. The electrical conductivity increased by two orders of magnitude from 10⁻¹⁵ to 10⁻¹³ for the nanocomposite added with sonication-treated nano-copper compared to that without sonication treatment.

Keywords: Nylon 66, nano-copper, nanocomposite, sonication, EMI shielding

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1.0 INTRODUCTION

Development of miniaturized and portable electronic telecommunication system leads to higher demands for electromagnetic shielding material [1-3]. Electromagnetic interference (EMI) is defined as a process by which disruptive electromagnetic energy is transmitted from one electronic device to another via radiated or conducted paths or both, causing upset in operation. EMI problems have impacted almost all electrical and electronic systems from daily life, to military activity, to space exploration and may leads to the loss of valuable time, energy, resources, money or even life [4,5]. Protecting electronic devices from EMI is very important in order to mantain the devices functionality and integrity, while controlling EMI emissions of electronic devices is essential to avoid product failure as to comply with electromagnetic

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compatibility standards imposed by governmental agencies. Furthermore, EMI is not only disruptive to electronic devices but also may cause harmness to human as well.

Metals are good candidate to be applied for EMI shielding material due to their high conductance except, there are heavy weight, physical rigidity, easy corrosion and poor processability in corner and tips. For those reasons, new candidates like carbon materials and conjugated polymers have been widely explored for EMI shielding efficiency owing to their light weight and corrosion resistance [6, 7].

In general, nylon 66 has an excellent mechanical resistance resulting from the mutual attraction of their long chains due to hydrogen bonds and their crosslinking [8]. Nylon composite has been widely used as the housing material for portable computer and notebook casing for years. Nylon 66 is often used as insulator because it has low thermal and electrical conductivity properties. It also has excellent thermal, physical and mechanical properties.

To form conductive composite, conductive fillers have to be added to the polymer matrix. If small conductive particles can be incorporated in the network within the composite, lower concentration of filler is needed to form conductive composite. Thus, the most practical method of producing a conductive network with fillers at a low concentration is to use nanosized filler particles. The addition of conductive fillers such as nanosized copper filler to insulative polymer such as nylon 66 can induce electrical conductivity. Copper is widely known as a promising material regarding to its high conductivity and lower cost. In particular, copper nanoparticles have received much attention due to their good performance to be applied in nanomaterials, thermal conducting applications, lubrication, nanofluids and also catalysts [9].

Ultrasonic is a well-established method for particle size reduction in dispersions and emulsions. Ultrasonic processors are used in the generation of nano-size material slurries, dispersions and emulsions because of the potential in the deagglomeration and the reduction of primaries. Through the particle size reduction, the surface area of the material can be increased. Due to this, a higher percentage of the atoms can interact with the polymer matrix. Surface activity is a key aspect of nanomaterials.

Agglomeration and aggregation blocks surface area from contact with other matter. Only welldispersed or single dispersed particles allow the full utilization of the beneficial potential of the matter. In other words, good dispersion reduces the quantity of nanomaterials needed to achieve the same effects [10, 11].

In this paper, we report the impact of sonication on the mechanical and electrical properties of nylon 66/nano-copper nanocomposite of various compositions.

2.0 METHODOLOGY

Nylon Zytel® 101F NC010 (Dupont) was used without any purification. Nano-copper powder (Nanocorp) was purchased from Terra Techno Engineering. The nano-copper filler concentration was varied from 1 to 5 wt%. For ultrasonically assisted mixing, nano-copper was first dispersed by ultrasonic instrument (Fisher Scientific Sonic Dismembrator) at frequency of 40 Hz. Ethanol was used as solvent to dissolve the nanocopper.

Nylon 66 composites were prepared by mixing of nano-copper filler with nylon 66 by means of dry mixing method using a table top high speed mixture at room temperature for 5 minutes.

All mixtures of nylon 66/nano-copper were extruded by co-rotating twin screw extruder (Sino PSM 30). The materials passed through heated barrels zones and exited at the nozzle holes. The barrel temperature setting were kept constant (255-280°C) for all sample formulations. Extruded samples were then cooled by water.

Subsequently, the samples were cut into pelletized form by pelletizer machine. The pellets were kept dry to prevent excessive moisture. Finally, the pellets were injection moulded to produce test specimens using an injection moulding machine (Battenfeld HM 80-350). The barrel temperatures were set at 260-300°C.

Durometer hardness was used to determine the relative hardness of the nano composites. The test measures the penetration of a specified indentor into the material under specified conditions of force and time. The Zwick/Roell Hardness test was carried out according to ASTM D2240 using a shore D Hardness probe.

The Izod impact test method was used to measure the impact resistance of PA66/nano copper composites. This impact test is use to compare the relative impact resistance under controlled laboratory conditions and often used for material selection or quality control. The test method is according to ASTM D256. Notched specimens were tested at room temperature.

Electrical conductivity or specific conductance is the reciprocal quantity, and measures a material's ability to conduct an electric current. The resistivities of samples were measured using high resistance meter (Hewlett Packard 4339A) and converted to the electrical conductivity(σ) using the following relation: r = 1/tdR, where t, d, and R are the thickness, diameter, and resistivity of sample films, respectively. The dimension of samples was 60 mm in diameter and 3 mm in thickness [12].

Dispersion of nano-copper in solvent and nylon 66 (without and with aid of ultrasonic) were examined by transmission electron microscope (TEM) (JEOL JEM-2100).

3.0 RESULTS AND DISCUSSION

The incorporation of nanoparticles into a polymer matrix can lead to a simultaneous improvement of different material properties. For this reason, the nano particles have to be dispersed into the polymer matrix in order to have their high specific surface interact with the polymer, i.e. the agglomerates have to be broken up and the particles should be distributed homogeneously within the matrix. TEM images (Figure 1 & 2) show the differences between dispersion of nano-copper in ethanol solvent with and without using ultrasonic method. Ultrasonic gives significant effect for a good dispersion of nano-copper by breaking up the agglomeration of nano-copper particles and this will lead to the improvement of the properties of composites.



Figure 1 Nano-copper dispersion in ethanol solution without sonication at (a) 6,000X magnification and (b) 20,000X magnification

Figure 1(b) shows the diameter of nano-copper without sonication method that ranges between 80-100 nm. With sonication, the diameter of nano-copper was reduced to 20-50 nm as shown in Figure 2(b). Ultrasonic application managed to break the agglomeration of nano-copper into individual particles of nano-copper as clearly demonstrated in Figure 2.



Figure 2 Nano-copper dispersion in ethanol solution with sonication at (a) 6,000X magnification and (b) 30,000X magnification

The shore hardness is an important parameter for evaluating or designating nanocomposite materials. The values of hardness for nylon 66 added with various amount of nano-copper are shown in Figure 3. Pristine nylon 66 showed the lowest value of hardness at 72 Shore D. It can be clearly observed that the hardness value increases with the increase of nano-copper amount in the nylon 66. However, the increase is larger for the sonicated nanocomposite compared to the nanocomposite without sonication. At 5wt% of nanocopper addition, the nanocomposite with sonication showed 6.3% of increase, as compared to a merely 2.3% increase for the nanocomposite without sonication.

The results indicate that the sonication of nanocopper prior to its introduction into the polymer matrix effectively improves the hardness of nylon 66/nanocopper composite. It seems that the rigidity changes considerably with the incorporation of the metallic nanoparticles leading to a considerable increase in hardness compared to neat polymer as reported previously [13].



Figure 3 Hardness of nylon 66/nano-copper nanocomposite containing various amount of nano-copper



Figure 4 Impact strength of nylon 66/nano-copper nanocomposite containing various amount of nano-copper

Figure 4 shows the impact of nano-copper amount and sonication on the impact strength of nylon 66. From Figure 4, loading of nano-copper resulted in an increase of impact strength up to 43% as compared to pristine nylon 66. Furthermore, nanocomposite with sonication consistently shows higher impact strength than nanocomposite without sonication, irrespective of the nano-copper amount.

The significant improvements in both impact and hardness of the nanocomposites with sonication can be explained by the reinforcement effect of nanocopper on the matrix due to improved dispersion of the fillers. The results indicate that homogenous dispersion of the nano-copper in nylon 66 contributes to the increased mechanical properties of nylon 66.

Electrical conductivies of nylon 66/nano-copper without and with sonication are shown in Figure 5 and 6, respectively. The electrical conductivity showed a significant increase when sonication was applied, in which it increased by two orders of magnitude (10⁻¹⁵ to 10⁻¹³). Furthermore, nanocomposite with sonication showed almost exponential increase in electrical conductivity as the the increase became greater as the amount of nano-copper was increased. The results can be associated with the improved dispersion of nano-copper in nylon 66, that enables it to reach the percolation threshold, thus enhances the electrical properties of the nanocomposite [14-16].



Figure 5 Electrical conductivity of nylon 66/nano-copper nanocomposite without sonication



Figure 6 Electrical conductivity of nylon 66/nano-copper nanocomposite with sonication

4.0 CONCLUSION

The effects of nano-copper loading and sonication on the nylon 66/nano-copper nanocomposite were studied. Ultrasonic was introduced as a method to achieve a good dispersion of nano-copper filler in polymer matrix. In general, incorporation of nanocopper as filler to nylon 66 successfully enhanced the mechanical and electrical properties of nylon 66/nano-copper nanocomposites. The sonication was found to effectively reduce agglomeration of nanocoppers in nylon 66, improving the dispersion as observed in the TEM images. The effect is reflected in the higher improvement of mechanical and electrical properties for the nano composites with sonication. A good dispersion leads to better properties of nanocomposites due to improved homogeneity of the mixture.

Acknowledgement

The authors would like to acknowledge the Ministry of Science & Technology (MOSTI) for funding this research under Science Fund grant (03-03-01-SF0165). The authors also would like to thank the Ministry of Education Malaysia for the financial support through FRGS/2013/FKP/TK04/02/4/F00164. Furthermore, the authors also sincerely thank the Faculty of Manufacturing Engineering of UTeM and Malaysian Nuclear Agency for providing the equipment and technical supports which made this work possible.

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