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Preparation of Conductive Polymer Graphite (PG) Composites

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Abstract. The preparation of conductive polymer graphite (PG) composites thin film is described. The thickness of the PG composites due to slip casting method was set approximately ~0.1 mm. The optical microscope (OM) and fourier transform infra-red spectroscopy (FTIR) has been operated to distinguish the structure–property relationships scheme of PG composites. It shows that the graphite is homogenously dispersed in polymer matrix composites. The electrical characteristics of the PG composite were measured at room temperature and the electrical conductivity (σ) was discovered with respect of its resistivity (Ω). By achieving conductivity of 10^3 S/m, it is proven that at certain graphite weight loading (PG_{20}, PG_{25} and PG_{30}) attributes to electron pathway in PG composites.

1. Introduction

Bio- polymer composites are classified as multifunctional materials due to its biological and chemical properties [1,2] . The polymer composite materials take place in many industrial applications as it is a biocompatible, biodegradability, renewable-resource based origin and low cost material. In Sustainable Polymer Engineering, Advanced Manufacturing and Materials Center (SPEN-AMMC) itself, the polymer is progressively studied for foams [3] , thin film [4,5] coating [6] etc. Basicly, polymeric materials either organic or inorganic are electrical insulators in their nature. Therefore limits its potentials as smart material under electrical conducting application. Attemps to promotes from insulating to conductive polymer composites contribute the rising of nano-carbons filler like carbon nanotubes [7] carbon nanofibers [8,9] and grapheme [10,11]. Due to their excellent mechanical, thermal and electrical properties [12] in composites materials leads to new research area in polymer matrix composites (PMC). There are a lot of advantages by using conductive polymers composites compared to metallic conductors. For instance, they can be easily converted into low cost technologies, light weight, provide corrosion resistance and also offer a wide variety of electrical conductivities.

Graphite, well known as graphene-based filler is naturally abundant and is reported as the best conductive filler for its excellent conductive properties and well dispersion in polymer matrix [13,14]. Recently, Shih, et al., found that graphite incorporated with polydimethylsiloxane (PDMS) shows highest temperature sensitivity and higher stability than the composites using carbon fillers as sensor [15]. In addition, polymer graphite composites also widely used as memory and energy storage [16], plastic chip electrode [17,18] etc.

In this work, conductive polymer graphite (PG) composite thin film were prepared through slip casting method by mixing the graphite filler into the polymer matrix with varying graphite weight loading (wt.%). The influence of graphite weight loading in electrical properties of the polymer composites is studied. The morphological- structural properties are investigated by employing optical microscopy (OM) and fourier transform infra-red spectroscopy (FTIR) in order to understand their structure–property relationships scheme.

2. Methodology

2.1. Graphite preparation

Flake graphite mixture was placed into a flask in the ultrasonic cleaning bath at room temperature. After that, the graphite mixture was washed to neutrality in which confirmed via pH paper and dried in an oven up to 100 °C for 60 min. Noted that this method has been adopted from Jihui Li in 2007 [19].

2.2. Monomer preparation

Monomer was prepared from renewable resources of virgin cooking oil (VCO). VCO were obtained and chemically manipulated at laboratory scale using less than 1L/ run. The monomer conversion begins with the catalyst preparation whereby to generate the epoxies from the unsaturated fatty compound. The second reaction is the acid-catalyst ring opening of the epoxies to form the final product, polyols [20].

2.3. Conductive polymer graphite composites preparation

Thin films of PG composites were prepared by mixing the monomer with Methylene Diphenyl Diisocyanate (MDI) as crosslinker and prepared graphite with varying graphite weight loading 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.%, 25 wt.%, 30 wt.% (PG_5, PG_{10}, PG_{15}, PG_{20}, PG_{25}, PG_{30}) [21] and neat (PG_0) using mechanical stirrer and cast into square container which are then dried at ambient temperature for at least 6 hours. The resulting substrate films were peeled off and digital micrometre was used to measure the thickness of the sample at particular point ranging ~0.1 mm.

2.4. Measurement and Characterization

The morphology of the composites was observed using an optical microscope instrument. Perkin Elmer Fourier Transform Infrared Spectroscopy (FTIR) spectrometer was used to identify the types of chemical bonds (functional groups) of the PG composites. Measurements of current-voltage characteristics of the prepared samples at 9 points were carried out using Keithley 6517A electrometer. The resistances R were determined from the slopes of the current–voltage characteristics. Electrical resistivity ρ and conductivity σ were calculated from the expression;

$$\rho = \sigma - 1 \, RLl/g \tag{1}$$

l/g is the electrode geometrical factor (l is the total length of the electrode width and g is the electrode distance), L is the film thickness [21].

3. Results and Discussion

3.1 Film morphology

The properties of PG composites appear promising as determined from OM images upon 200x as referring to figure 1 where (a) neat, PG_0, (b) PG_5, (c) PG_{10}, (d) PG_{15}, (e) PG_{20}, (f) PG_{25} and (g) PG_{30}, with its cross-sectional view respectively. As the graphite matrix loading increased from 5 wt% till up to 30 wt%, the composite appears in the OM images to be almost entirely filled with graphite as it turn the light yellowish of polymer host to darker colour (even at low loading of graphite). The effect of graphite in polymer matrix contributes to 'mirror-like' or 'visual' effect on its cross-sectional view (on the right images). This is due to the enormous surface area of the PG composites. Thus conclude that the graphite is homogenously dispersed in the polymer matrix.

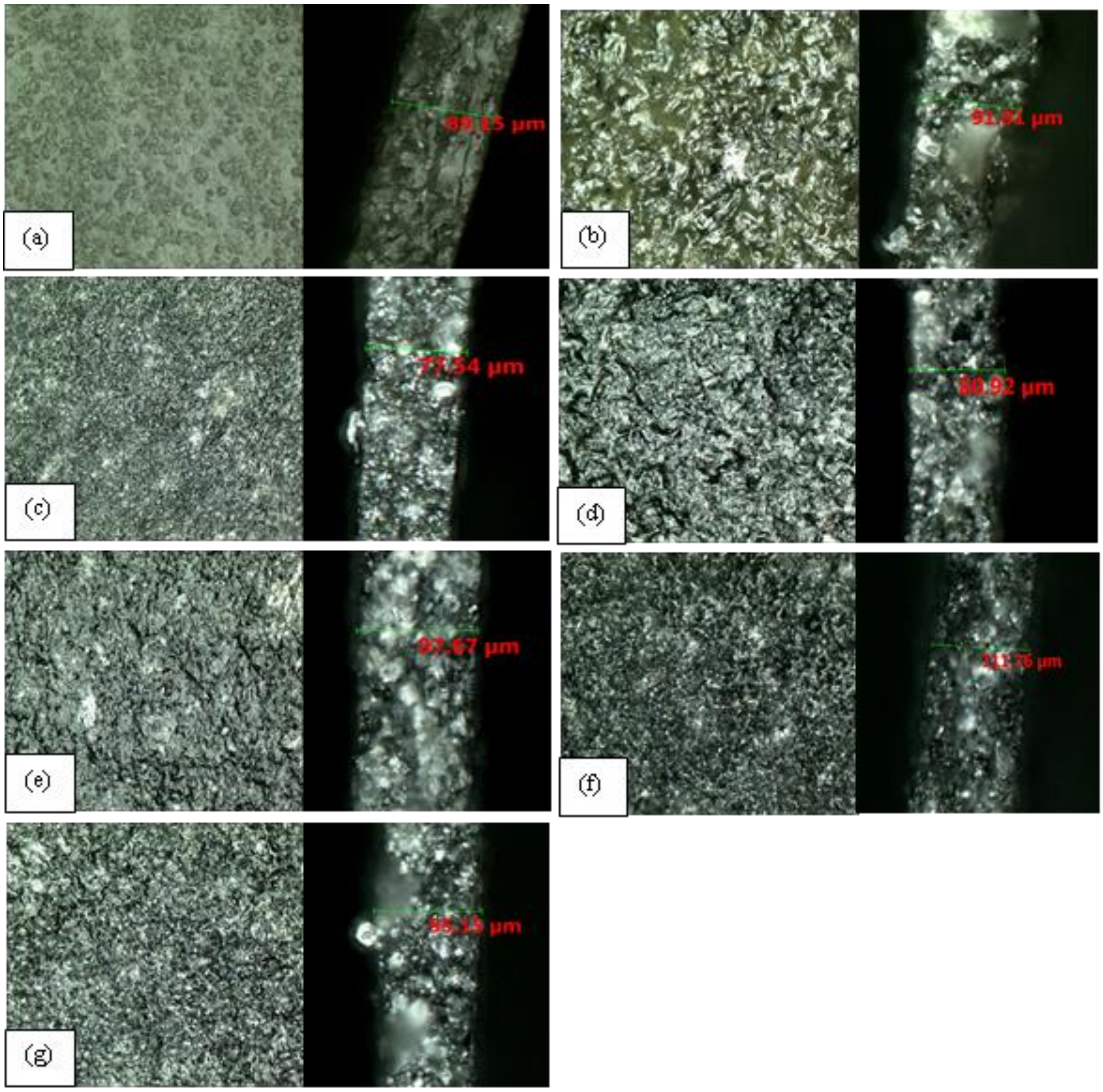

Figure 1. Optical microscope images 200x magnification of conductive organic polymer graphite composites (a) neat, PG_0, (b) PG_5, (c) PG_{10}, (d) PG_{15}, (e) PG_{20}, (f) PG_{25} and (g) PG_{30}, with its cross-sectional view respectively.

3.2 Functional groups characteristic

The FT-IR spectra of PG composites are shown in figure 2. The spectra illustrates no significant changes between those bands, exceptional for: shoulder at range 3200 cm^{-1} and at 2650.47 cm^{-1} , the decrease of polyether bands (C-O-C) at 1300 cm^{-1} – 1100 cm^{-1}, and increasing aromatic bands (C=C) at peak range 1600 cm^{-1} – 1400 cm^{-1}. The carbonyl group at peak 1733.70 cm^{-1} is weaker hinting to the formation of (NHCOO) bond whereby the disappearance of the peak at around 2260 cm^{-1}which indicating that all the diisocyanate are consumed in the reaction.

The stretching vibration absorption peak at 3200 cm^{-1} shows the hydroxyl group in the structure of PG composites. This is might be due to the hygroscopicity of the original graphite, graphite oxide (GO), and pretreated graphite [22] in the composites.

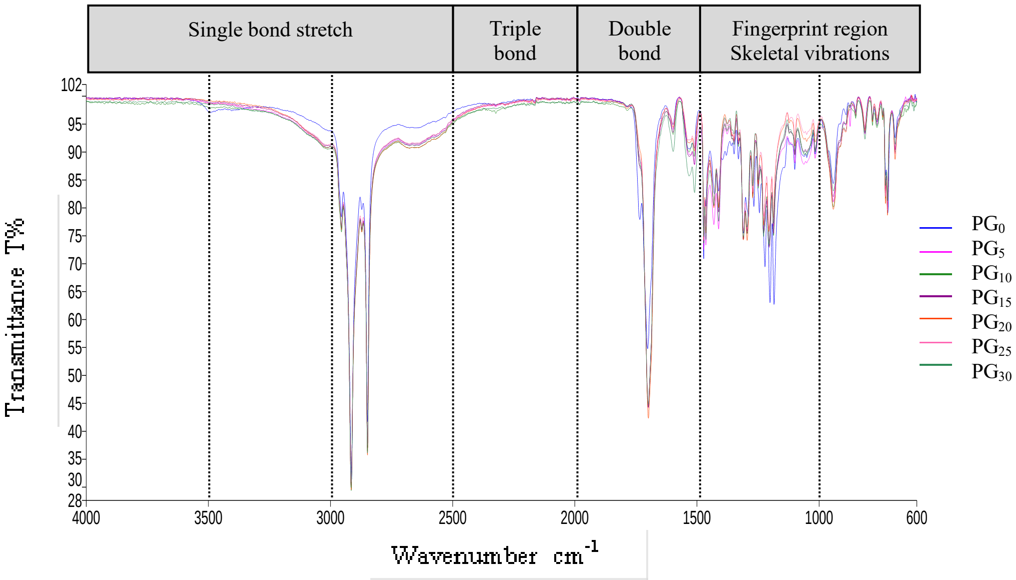

Figure 2. FTIR spectrum of conductive organic polymer graphite composites of PG_0, PG_5, PG_{10}, PG_{15}, PG_{20}, PG_{25} and PG_{30}

3.3 Electrical characteristic

The electrical conductivity (σ) and resistivity (Ω) values of PG composites are plotted in figure 3. The calculated electrical conductivities from the reciprocal of the resistivity were increased by numerous orders of magnitude from 60- 70 x 10^3 S/m upon PG_{20}, PG_{25} and PG_{30} composites in which comparable with 10^{-7}- 10^{-5} to 30- 40 S/m of graphite in PA6/PC composites [12].

Poor electron mobility in the composites was observed due to lower graphite loading (PG_5, PG_{10} and PG_{15}) in PG composites. Having said that at this point, the graphite is covered by PG_0 matrix where the composite does not form a conductive pathway for electron mobility to reach the percolation limit. Sometimes, it is difficult to validate or invalidate the expected conduction mechanism by direct analytical measurements because of low currents implied. It is suggested that different measurement techniques should be applied such as DC conductivity, thermally stimulated DC current etc. in understanding the relationship between their electrical properties [23].

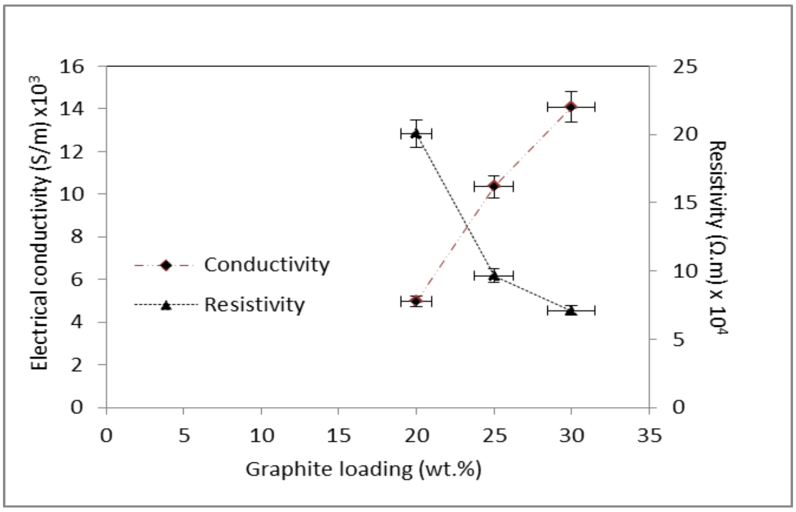

Figure 3. The electrical characteristic of conductive organic polymer graphite composites of PG_0, PG_5, PG_{10}, PG_{15}, PG_{20}, PG_{25} and PG_{30}

4. Conclusion

Related to the conductivity of the PG composites, the interaction in polymer matrix implies a homogenous dispersion, thus assigned the insulating polymer host to conductive polymer composites. Reporting that electron mobile in PG_{20}, PG_{25} and PG_{30} of high graphite loading (wt.%) composites gives conductivity up to 60- 70 x 10^3 S/m. This mechanism is in good agreement with results obtained for graphite filled polymer matrix.

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