Effect of Graphite Loading and Addition of Ferric Chloride (FeCl$_3$) on the Properties of Poly (Vinyl Chloride)/Poly (Ethylene Oxide)/Graphite Polymer Films

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Effect of Graphite Loading and Addition of Ferric Chloride (FeCl₃) on the Properties of Poly (Vinyl Chloride)/Poly (Ethylene Oxide)/Graphite Polymer Films

M D Siti Hajar¹, P L Teh¹, A W M Kahar¹, M I M Yazid¹, H N Hamzah¹
¹School of Materials Engineering, Kompleks Pusat Pengajian Jejawi 2, Taman Muhibbah, Universiti Malaysia Perlis (UniMAP), Jejawi, 02600, Arau, Perlis, Malaysia.

*Email: sitihajahmohddin@gmail.com

Abstract. In this study, films consisting of a blend of poly (ethylene oxide)/poly (vinyl chloride) (PEO/PVC) and a conductive filler, graphite with the addition of FeCl₃ as a doping were prepared and characterized for their mechanical and electrical properties. Solid polymer blend films based on PVC/PEO (50/50 wt%/wt%) with different graphite loading were prepared by using solution casting technique. Electrical conductivity results discovered the conductivity increased with increasing of filler loading and addition of FeCl₃. However, the addition of FeCl₃ in graphite loading led to a decreased in tensile strength of PEO/PVC/Graphite polymer films. The dispersion of graphite and mechanism of conductive path in the polymer films were also investigated by scanning electron microscopy (SEM). The morphology of the PEO/PVC/Graphite polymer films shows that agglomeration occurred to complete the connection of conductive path, thus improving the conductivity behavior of the polymer films.

1. Introduction

Basically, polymeric materials either organic or inorganic are electrical insulators in their nature. Therefore limits its potentials as smart material under electrical conducting application. Efforts to promote from insulating polymer to conductive polymer composites bring to the rising of nanocarbons filler like carbon nanofibers [1], carbon nanotubes [2], and graphene [3]. Due to their excellent mechanical, thermal and electrical properties 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 is crystal form of a pure carbon much resembling of mica-sheets of strongly linked atoms, with very weak bonds between the sheets. Graphite generally used as electronically conducting filler because it is economical and abundant naturally [4]. It has excellent electrical properties and well dispersion in polymer matrix [5]. In this study, the electrical properties, mechanical properties and morphology of PVC/PEO blends containing graphite-FeCl₃ filler were investigated.
2. Experimental

2.1. Materials
PVC powder with molecular weight of 220 000 g/mol and PEO powder with molecular weight of 100 000 g/mol as the matrix for conductive polymer film was supplied by AR Alatan Sdn. Bhd., Alor Star, Malaysia. Graphite with molecular weight of 12.02 g/mol and particle size of <50 µm was supplied by R&M Chemicals. Plasticizer used in this research was Dioctyl terephthalate with molecular weight of 390 g/mol and tetrahydrofuran with molecular weight of 72.11 g/mol was provided from AR Alatan Sdn. Bhd.

2.2. Composites Preparation
The PEO/PVC/Graphite polymer films were processed by solution casting method. Initially, PEO and PVC are dissolved in tetrahydrofuran solution (THF) and were blended together to get homogeneous solution. The DOTP as was incorporated at the same amount for all composition. After that, conductive filler were added in the solution. The PEO/PVC/Graphite polymer films solutions were poured and casted on a glass mould after 4 hours and let it to evaporate in a fume cupboard at room temperature. The PEO/PVC/Graphite polymer films were formed when THF are fully dried. The same process was repeated to produce films which consist of different graphite content.

2.3. Preparation of Secondary Doping of Graphite with FeCl₃
Graphite were dissolved with 6 wt% of ferric chloride in toluene and then stirred for 24 hours. Next, the doped mixture was washed with distilled water and dried in an oven for 5 hours at 60°C. Finally, the doped graphite powders were grinded to fine powders and ready to preserve for sample preparations.

2.4. Scanning Electron Microscopy (SEM)
The tensile properties of the samples were assessed by using Universal Testing Machine Instron 5569 according to ASTM D638. Rectangular shaped specimens were tested at relative humidity (30 ± 2) °C and ambient temperature (25 ± 3) °C with the crosshead speed of 30 mm/min. The tensile strength and Young’s modulus data were acquired from the average values of five samples for each samples.

2.5. Electrical Conductivity Test
The conductivity measurement of PEO/PVC/Graphite polymer films was conducted using I-V measurement systems. The conductivity was tested by Keithley Model 4200 Semiconductor Characterization System. The samples were conducted with voltage varied from 0 V to 10 V. The
electrical conductivity was determined using its relationship with resistivity and conductivity as shown in Equation 1 and Equation 2 below:

\[
\rho = R \cdot \left(\frac{w \times t}{l}\right) 
\]  

(1)

Where \( R \) = resistance of the films, \( w \) = width, \( t \) = thickness, \( l \) = length between the probe contact.

\[
\sigma = \frac{1}{\rho} 
\]  

(2)

3. Results and Discussion

Resistance, \( R \) of PVC/PEO/Graphite and PVC/PEO/Graphite-FeCl\(_3\) conductive polymer films with filler content 5-25 wt% prepared by solution casting method was measured at room temperature by two point probe. Their conductivities, \( \sigma \) are plotted against filler loading as shown in Figure 2. Electrical conductivity of both PVC/PEO/Graphite and PVC/PEO/Graphite-FeCl\(_3\) polymer films are increased with further increase in the amount of filler. Poor electron mobility in the polymer film was observed due to lower graphite loading. At this point, the graphite filler is covered by PVC/PEO matrix where the polymer film does not form conductive pathways for electron mobility to reach percolation limit.

The addition of FeCl\(_3\) content in PVC/PEO/Graphite-FeCl\(_3\) polymer films results in improvement in the electrical conductivity. The conductivity of PVC/PEO/Graphite-FeCl\(_3\) is higher than PVC/PEO/Graphite conductive polymer films and was increased by numerous orders of magnitude. The electrical conductivity of PVC/PEO/Graphite polymer films increased to \( 10^2 \) S/cm compared to \( 10^-4 \) S/cm at 25 wt% of graphite loading with the presence of FeCl\(_3\) in the polymer films. From the previous study by J Jin et al. [6], addition of LiCl in Graphite/polyethersulphone composites has increased the electrical conductivity due to the formation of lithium ion-graphite intercalation compound (Li-GIC). The densities of holes in the composites are greater than pure graphite. So, the high density and the high mobility of holes contributed to the high electrical conductivity of the doped composites.

![Figure 2: Electrical conductivity of PVC/PEO/Graphite and PVC/PEO/Graphite-FeCl\(_3\) polymer films at different graphite loading.](image-url)
The tensile strength of PVC/PEO/Graphite and PVC/PEO/Graphite-FeCl$_3$ polymer films is shown in Figure 3. From the Figure 3, it can be observe that the tensile strength decreased gradually with the increasing of graphite content. The addition of graphite fillers attributes a restrictive effect over the PEO/PVC polymer films by deformation of their molecular chains and restricting the motion. This can be explained by the weak filler-matrix interaction between the graphite and PVC/PEO matrix in the polymer films. Too high concentration of conductive filler could lead to material redundancy and detrimental mechanical properties. At similar filler loading, PVC/PEO/Graphite polymer films with the presence of FeCl$_3$ showed lower tensile strength compared to PVC/PEO/Graphite polymer films. The additions of FeCl$_3$ have given negative effect in mechanical properties on PVC/PEO/Graphite polymer films.

![Figure 3: Tensile strength vs. graphite loading of PVC/PEO/Graphite polymer films with and without presence of FeCl$_3$.](image)

The SEM morphologies of PVC/PEO/Graphite and PVC/PEO/Graphite-FeCl$_3$ polymer films are represented in Figure 4. The analysis was conducted to visualize the effect of graphite loading and presence of FeCl$_3$ on the properties of the films. Figure 4 (a)-(c) shows the surface morphology of PVC/PEO polymer films with addition of 5 wt%, 15 wt% and 25 wt% of graphite contents respectively. The surface morphology appears in the SEM images to be almost entirely filled at high graphite content as it surface are become rough and polymer matrix become darker colour. The effect of addition of FeCl$_3$ on the PVC/PEO/Graphite polymer films are shown in Figure 4 (d)-(f). The surface morphology of PVC/PEO/Graphite polymer films containing FeCl$_3$ exhibited poor surface morphology compared to polymer films with the absence of FeCl$_3$. Furthermore, the presence of FeCl$_3$ displayed agglomeration of the filler as a results of FeCl$_3$ ion reducing the PVC/PEO/Graphite miscibility and weaken the interaction between them. This proves that the tensile strength results are poor with the addition of FeCl$_3$ in the polymer films. However, the existences of agglomeration contributed to the high electrical conductivity as the electric charge are easy to transfer between graphite filler.
4. Conclusion
PVC/PEO/Graphite and PVC/PEO/Graphite-FeCl$_3$ polymer films were prepared by solution casting technique. The electrical conductivity of the films were found to be increased with higher loading of graphite and further improved with the introduction of FeCl$_3$. It was observed that the tensile strength of the films was reduced with the addition of graphite. At a similar loading, the presence of FeCl$_3$ reduced the tensile strength of the films. On the other hand, SEM morphology of PVC/PEO/Graphite and PVC/PEO/Graphite-FeCl$_3$ polymer films supported the tensile results by showing more voids and agglomeration as more graphite content incorporated as well as when the FeCl$_3$ were introduced into the films.
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