



Effects of Ferric Chloride as Secondary Dopant on the Properties of Polyethylene Oxide / Polyvinyl Chloride / Polyaniline Conductive Films

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ABSTRACT

The potential uses of conductive films in flexible electronic devices have attracted a lot of attention to their development. The mechanical and electrical properties of polyethylene oxide (PEO) and polyvinyl chloride (PVC) have been investigated as a matrix in electrically conductive polymer composites. However, it is still difficult to increase their electrical conductivity without sacrificing their mechanical integrity. To solve these problems, the use of conductive polymers like polyaniline (PAni) has showed potential. Despite progress, nothing is known about how different PAni concentrations with the inclusion of secondary dopants such ferric chloride (FeCl_3) will affect the overall performance of the PEO/PVC conductive films. The purpose of this work is to examine how the tensile strength, electrical conductivity, and morphological structure of PEO/PVC based conductive films are affected by the addition of PAni and FeCl_3 . The results offer guidance on how to best optimize the mixture for PEO/PVC blends with 2.5 wt.% - 10 wt.% PAni loadings were fabricated with and without the presence of FeCl_3 . PAni was doped by dissolving it with 6 wt.% FeCl_3 in toluene to initiate the secondary doping. Then, the doped PAni will be utilized as conductive filler in PEO/PVC/PAni- FeCl_3 conductive films. The conductive films were prepared by solution casting method using tetrahydrofuran as the solvent at ambient temperature. The electrical conductivity, tensile strength and X-ray diffraction (XRD) analysis of the conductive films was analyzed. The study found that higher loading of PAni increased the electrical conductivity of the conductive films and further improved with the addition of FeCl_3 . The XRD analysis study supported the findings by revealing the modified structure of PAni, with FeCl_3 as the oxidizing agent. The tensile strength, however, decreased with the addition of PAni and FeCl_3 in the conductive films due to poor agglomeration of filler.

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1. Introduction

Polymers are well known typically as insulators as they are generally organic molecules made of carbon, hydrogen and many other elements with covalent bonds. Materials with covalent bond are generally non-conductive due to the absence of free electrons. However, polymer technology is growing rapidly with lots of studies on the blending of commercial polymer with conductive polymer have been reported in recent years [1-3]. Polymer blending is a widely preferred approach to establish new polymeric materials. This method is valuable to develop polymers with an extensive range of characteristics such as the blending of conductive polymer with the commercialized - non conducting polymers.

Intrinsically conductive polymers such as PANi, polypyrrole, and polythiophene are among the most studied classes of conducting polymers [4]. PANi has been broadly reviewed for several factors, such as its economic feasibility, environmental stability and the easiness of the electrical conductivity to be altered by modifying the oxidation state and degree of doping of the backbone. PANi is a promising material for various techno-commercial applications, and hence the electrical properties, thermal and environmental stability of PANi conductive film is highly dependent on the nature and the size of its dopant, as demonstrated in a previous study [5].

Interestingly, PANi can undergo second time doping processes to further improve its electrical conductivity. In 1990, Heeger and his fellow researchers revealed the breakthrough of secondary doping effects in PANi systems [6]. They reported that the exposure of camphor sulfonic acid-doped PANi to m-cresol improved the electrical conductivity of the sample from 1–2 to almost 400 S/cm. Previous studies [7] have shown that solvents, due to their ability to efficiently enhance the electrical conductivity of PANi, are commonly referred to as secondary dopants. Lately, there's been plenty of attention given to improve the electrical conductivity of PANi salts by post polymerization process with a suitable secondary dopant such as m-cresol solvent. On the other hand, the electrical conductivity of PANi has also been reported to be reliant on the solvent used for casting or solvent they have been exposed to. The solvent generates a modification in the polymer conformation which causes greater electrical conductivity. This phenomenon is also named secondary doping, as demonstrated in previous studies by Beygisangchin *et al.*, [8].

It has been reported that the doping process may employ oxidants or also referred to as doping agents to complement the doping process. FeCl₃, ammonium persulfate, hydrogen persulfate and cerium (IV) sulfate are among the favored oxidants available. Among them, FeCl₃ emerges as one of the most studied materials. FeCl₃ carries the smallest oxidation potential amongst the widely available oxidizing agents as per the prior report by Doblhofer *et al.*, [9]. Its low oxidation potential, E⁰, of 0.77 V minimizes the risk of over-oxidation, effectively preventing polymer chain scission or excessive oxidation that could otherwise compromise the mechanical and electrical properties of the material. In contrast, stronger oxidizing agents, such as ammonium persulfate or hydrogen peroxide, may lead to undesirable side reactions, including structural degradation and loss of performance in PANi.

Previous studies [10,11] have shown that it can be used as an etching medium, such as for etching copper, or as an oxidant for conducting polymers. FeCl₃ became influential after studies revealed that polyacetylene electrical conductivity improved with the treatment of FeCl₃-nitromethane solutions. Mamma and his team of researchers [7] studied the effect on Poly

(C₆H₅NH₂) emeraldine salt by FeCl₃ and KMnO₄ as secondary dopants and found out that the electrical conductivity has dramatically increased. The secondary doping causes structural rearrangement of PANi, from a compact-coil form to a more expandable conformation. In addition, it is also reported that the π conjugation is also increased due to the secondary doping.

In this paper, PEO and PVC were selected as matrix materials and were blended with the PANi filler to produce PEO/PVC/PAni conductive films. PVC has been widely exploited in polymer industry and attracts many researchers to explore various potentials of the polymer. PVC offers many benefits such as low cost, good processability and compatibility with numerous plasticizers. It has been widely used as construction pipes, flooring materials and cable sheaths. However, the amount of research involving the investigation of PVC for more advanced fields such as in electronic application is rising from year to year. For several decades, various approaches have been developed to improve electrical conductivity of PVC, as highlighted in previous studies [12,13]. It includes blending PVC with conductive polymer, coating PVC surfaces with conductive layers, mixing the PVC with liquid anti-static agents, or even by adding conductive inorganic fillers into the PVC, as demonstrated in previous studies [14].

PEO is a polymer of great interest which offers superior solvating ability for salt while establishing better stability with inorganic salts, as highlighted in previous studies [15]. Moreover, PEO shows sufficient thermal and chemical stability, and the spacing between the ether groups along the PEO backbone is ideal for cation solvation. Papers on PEO/PVC blend have been widely reported by fellow scholars. The vital point to keep in mind when constructing new polymer blends is the miscibility of a blend, as per previous findings [16,17]. Ramesh and Arof [18] investigated the PVC/PEO miscibility at various ratios and concluded that the blends were miscible. In addition, Turcanu [19] and Hajar *et al.*, [20] also came out with the same finding as they concluded that PVC/PEO blends were miscible in their study.

While PEO and PVC blends have been studied for their mechanical and electrical properties, the impact of incorporating varying concentrations of Pani with the use of FeCl₃ as a secondary dopant remains underexplored. There is a lack of comprehensive understanding regarding the trade-off between enhanced electrical conductivity and the potential reduction in mechanical strength in these blends.

Although the properties of PEO and PVC have been investigated, little is known about the effects of adding different amounts of PANi and FeCl₃ utilisation as a secondary dopant. Comprehensive knowledge of the trade-off between these composite possible decrease in mechanical strength and improved electrical conductivity is lacking. Valuable knowledge regarding optimizing PEO/PVC blend composition for the foundation of flexible electronics applications is provided by this study. The research helps create materials that balance electrical conductivity and mechanical integrity by examining the combined impacts of PANi and FeCl₃ on tensile strength and electrical conductivity. This opens the door for improvements in future conductive films.

In this study, we have analyzed the effect of FeCl₃ addition and PANi loading on the PEO/PVC/PAni conductive films where the PANi used was already prepared in emeraldine salt form. Although a significant number of papers have discussed the effect of doping on PANi, there is still limited work reported on doping a PANi in emeraldine salt form. Besides, the method of using oxidants, FeCl₃, without using any type of acidic solvent can be considered as a novelty in

this field of research. The main objective of the work was to study the tensile strength, electrical conductivity, morphology and chemical structure of the conductive films with and without the presence of FeCl_3 . The effects of PANi loading on the structure and properties of the conductive films were also reported.

2. Methodology

2.1 Materials

PEO powder with a molecular weight of 100,000 g/mol was used in this study. It has a glass transition temperature of -53°C to -45°C and melting temperature of 65°C . PVC powder had a molecular weight of 220,000 g/mol, glass transition temperature of 84°C and melting temperature of 100°C to 160°C . Both PEO and PVC were provided by AR Alatan Sdn. Bhd., Alor Star, Malaysia. PANi (emeraldine salt with 20 wt.% of carbon black) with particle size of $21\ \mu\text{m}$ and approximated molecular weight of 5000 was manufactured by Sigma Aldrich. FeCl_3 , with molar mass of 162.2 g/mol and density of $2.9\ \text{g/cm}^3$ was supplied by AR Alatan Sdn. Bhd. Tetrahydrofuran with molecular weight of 72.11 g/mol and dioctyl terephthalate (DOTP) with molecular weight of 390 g/mol were used in this research. Finally, ethanol, $\text{C}_2\text{H}_5\text{OH}$, with molar mass of 46.07 g/mol and density of $0.79\ \text{g/cm}^3$ was supplied also by AR Alatan Sdn. Bhd.

2.2 Conductive Films Preparation

The samples fabrication was carried out by solution casting method. Both the PEO powder and PVC powder were dissolved in a separate conical flask with THF solvent. After that, the PEO and PVC solution were mixed in a single conical flask before it was then stirred to become homogeneous. Next, DOTP and PANi were added into the homogeneous solution. To accomplish a suitable viscosity and decent homogeneous condition, the solution was stirred via magnetic stirrer at 400 rpm for 4 hours. Lastly, the solution was cast onto a glass mold and left until it dried slowly inside a fume cupboard.

2.3 Preparation of Secondary Doping of PANi with FeCl_3

PANi were dissolved with 6 wt.% of FeCl_3 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 PANi powders were grinded to fine powders and ready to preserve for sample preparations.

2.4 Electrical Conductivity Test

The electrical conductivity of the conductive films was analyzed via Keithley Model 4200-SCS semiconductor characterization system. Each sample's electrical conductivity was carried out with four-point probe with voltage varied from 0 to 10 V. The value of electrical conductivity was calculated using its correlation with resistivity. With the probes centered on a very wide and very thin sample, the electrical conductivity can be calculated *via* Eq. (1) and (2):

$$\rho = \left(\frac{\pi}{\ln 2}\right) t \left(\frac{V}{I}\right) \quad (1)$$

Where ρ = resistivity, t = sample thickness, V = voltage between two inner probes, I = current flows between two outer probes and the electrical conductivity, σ , was calculated using the Eq. (2):

$$\sigma = \frac{1}{\rho} \quad (2)$$

2.5 Tensile Strength

The samples were cut into dumbbell shapes by using an ASTM D638 sample cutter. The tensile test was conducted under ambient temperature (25°C) by using a universal testing machine model AG-XD plus (Shimadzu, Kyoto, Japan) with a crosshead speed of 10 mm/min. The average values of five (5) samples from each type of sample will be calculated to determine the average of all results.

2.6 X-Ray Diffraction (XRD)

The crystallinity of the conductive films was determined by using a Shimadzu XRD-6000 Analyzer at an acceleration voltage of 35 kV and 25 mA with $\text{CuK}\alpha$ ($\lambda = 1.5406$ nm) radiation source, in the range of 10-80 (2θ). This analysis was performed at ambient temperature with scan speed of 10 °C/min.

3. Results And Discussion

3.1 Electrical Conductivity

The effect of PANi loading on the electrical conductivity of the conductive films was monitored. As seen in Figure 1, the electrical conductivity of the PEO/PVC/PANi conductive films was further improved with the addition of FeCl_3 . For example, both PEO/PVC/PANi- FeCl_3 and PEO/PVC/PANi- FeCl_3 /PEGDE conductive films achieved their percolation threshold of 9.16×10^{-04} S/mm and 4.50×10^{-04} S/mm as compared to 2.48×10^{-04} S/mm for PEO/PVC/PANi conductive films. The result is aligned with Li *et al.*, [21] as they reported that the electrical conductivity of the synthesized PANi- FeCl_3 was higher than the single PANi. In addition, there were several reports [22,23] by other scholars who proposed that FeCl_3 can perform as dopant and oxidant at the same time as FeCl_3 , being an acidic salt, offers protons in aqueous solution by hydrolysis. No acidic solvent (secondary dopant) was used in this research to dope the PANi. The ability of the oxidant (FeCl_3) to perform as secondary dopant has been utilized and it resulted in higher electrical conductivity compared to the conductive films without FeCl_3 .

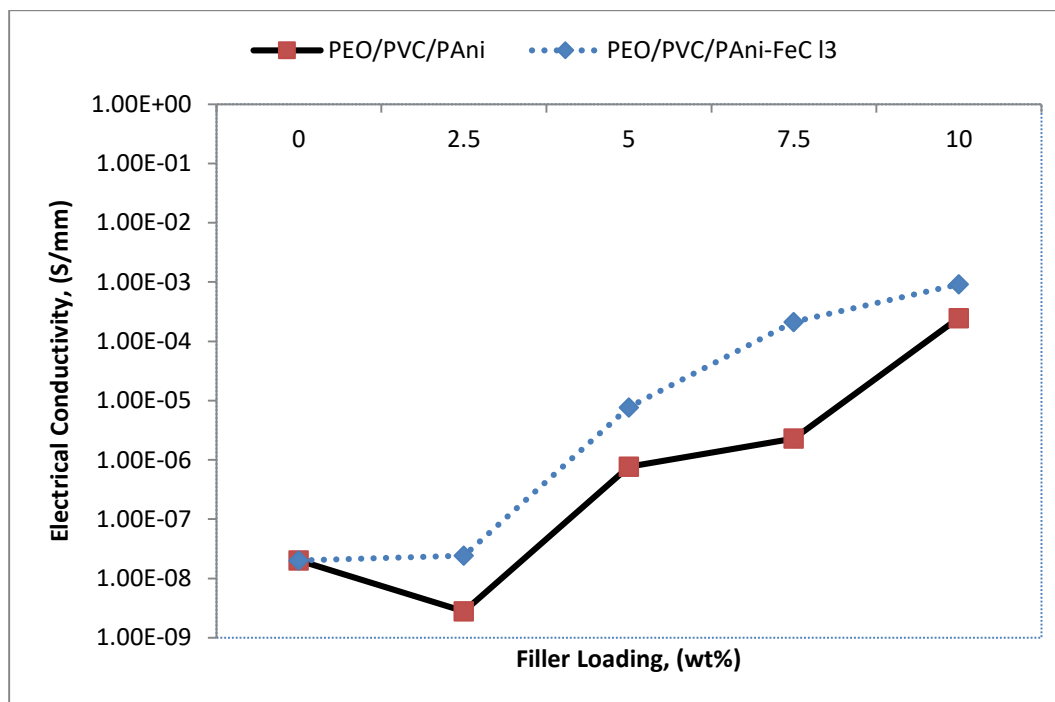


Fig. 1. Electrical conductivity of the PEO/PVC/PAni conductive films with varying filler loading and effect of FeCl₃

3.2 Tensile Strength

Tensile strength of the PEO/PVC/PAni conductive films containing FeCl₃ were studied. Figure 2 illustrates the tensile strength of PEO/PVC/PAni and PEO/PVC/PAni-FeCl₃ conductive films. Tensile strength of both conductive films decreased as filler loading increased from 0 wt.% to 10 wt.%. This is common because lower mechanical characteristics might result from filler disruption of the polymer matrix. As noted in previous studies [24], increased filler loading may cause voids to occur at the filler-matrix interface, reducing stress transmission from the matrix to the fillers and lowering the tensile strength values. According to a prior polymer doping study by Wang *et al.*, [25] chemical doping can cause a polymer chain to reorganize and result in a more crystalline structure. It is anticipated that the ordered structure will improve π - π stacking between intermolecular chains, leading to improved electrical conductivity and high carrier mobility.

However, the dopants have an impact on the surface and structural characteristics of CPs in addition to their electrical conductivity. Large dopants can alter the density of the polymer, which in turn can alter the surface topography and physical characteristics of the polymer, according to Le *et al.*, [26]. This may lead to aggregation within the polymer matrix, creating surface irregularities and voids that weakened tensile strength. The claim is comparable to the previous findings by Yazid *et al.*, [24] where the agglomeration of PAni-FeCl₃ filler seems to be greater as compared to PAni filler. As a result, this weakens the ability of load transfer from the matrix to the filler, thereby reducing the tensile strength of the conductive films.

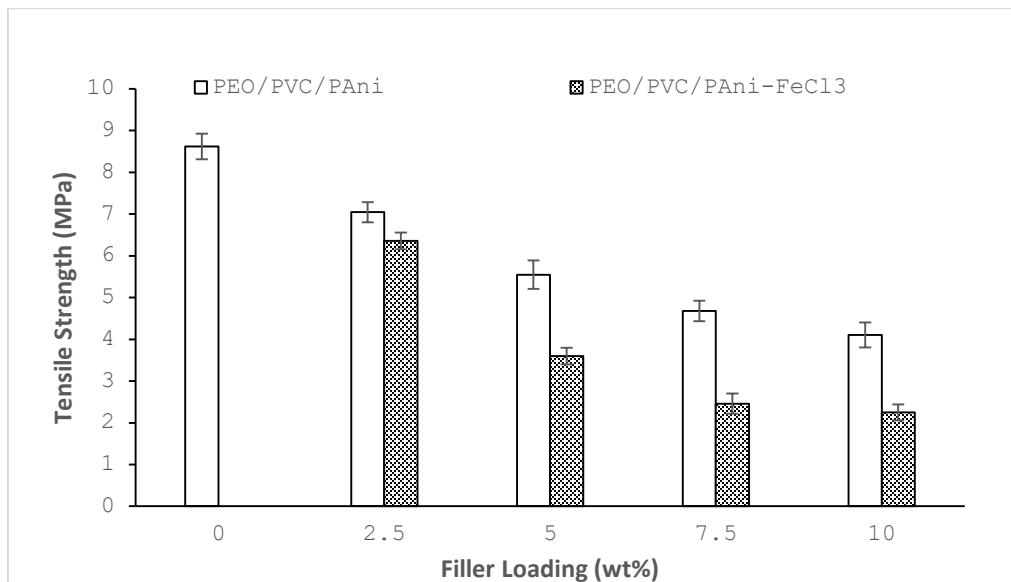


Fig. 2. Tensile strength vs filler loading of PEO/PVC/PAni conductive films and PEO/PVC/PAni-FeCl₃ conductive films

3.3 X-ray Diffraction Analysis

The X-ray diffraction patterns of the PAni and PAni-FeCl₃ are shown in Figure 3. The X-ray diffraction pattern of PAni and PAni-FeCl₃ have the same amorphous nature with broad diffraction shoulder. However, the diffraction from the doped PAni sample (PAni-FeCl₃) was more intense as compared to the PAni (un-doped), showing that the doping process has enhanced the crystallinity of the filler. This result is also supported by Wang *et al.*, [25] where the doped PAni shows higher crystallinity than the undoped PAni. A significantly higher intensity of PAni-FeCl₃ peak at around $2\theta = 25^\circ$ compared to PAni confirms a change in the crystalline form of the sample. The outcome is also supported by Zhang *et al.*, [27] as they published that FeCl₃ is an efficient oxidant to prepare highly crystalline PAni. They also stated that the highly crystalline PAni-FeCl₃ resulted from a low growth rate of the filler, caused by low redox potential of FeCl₃ (0.77 V) as the oxidant.

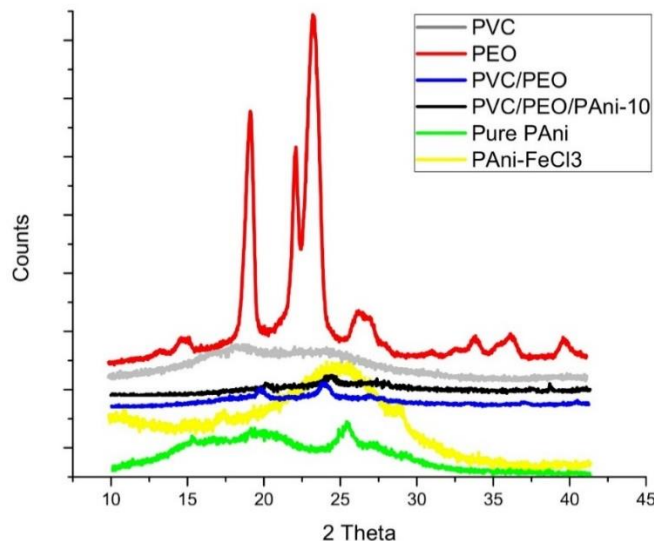


Fig. 3. XRD diffractogram of PVC, PEO, PAni, PAni-FeCl₃, PEO/PVC and PEO/PVC/PAni-10 conductive films

4. Conclusion

PEO/PVC/PAni and PEO/PVC/PAni-FeCl₃ conductive films were prepared by solution casting technique. The electrical conductivity of the conductive films was found to be increased with higher loading of PAni and further improved with the introduction of FeCl₃. XRD analysis supported the findings by showing that the crystallinity of PAni increased after the doping process. Hence, the purpose of using FeCl₃ as secondary dopant is seen to increase the electrical conductivity of PAni. On the other hand, the tensile strength of the conductive films decreased with the introduction of the secondary dopant due to filler agglomeration in the conductive films.

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