

# THE EFFECT OF CONDUCTIVE FILLERS AND POLY (ETHYLENE GLYCOL) DIGLYCIDYL ETHER **ON THE PROPERTIES OF POLY (VINYL** CHLORIDE)/POLY (ETHYLENE OXIDE) **CONDUCTIVE FILMS**

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SITI HAJAR BINTI MOHD DIN 1430411278 Thisitem

by

A thesis submitted in fulfillment of the requirements for the degree of Master of Science in Materials Engineering

> **School of Materials Engineering UNIVERSITI MALAYSIA PERLIS**

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SITI HAJAR BINTI MOHD DIN

# **UNIVERSITI MALAYSIA PERLIS**

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3.2	Conductivity, σ	58
Ő	Conductivity, or Conductivity, or Conduc	

## LIST OF ABBREVIATIONS OR NOMENCLATURES

Argentum
Acrylonitrile butadiene styrene
American Society for Testing and Materials
Carbon black
Coconut fibers
Carbon nanotubes
Dibutyl phthalate
Dodecyl benzene sulfonic acid
Dodecyl amine modified montmorillonite
Dioctyl adipate
Dioctyl phthalate
Dioctyl sebacate
Dodecyl amine modified montmorillonite Dioctyl adipate Dioctyl phthalate Dioctyl sebacate Dioctyl terephthalate
Dioctyl azelate
Differential scanning calorimetry
Emeraldine base
Ethylene carbonate
Electromagnetic interference shield
Electrostatic discharge
Fourier transform infrared spectroscopy
High density polyethylene

	HIPS	High impact polystyrene
	HTPB	Hydroxyl terminated polybutadiene
	LEB	Leucoemeraldine base
	$M_{w}$	Molecular weight
	MA	Maleic anhydride
	Ni	Nickel
	NMR	Nuclear magnetic resonance
	NR	Natural rubber
	PA6	Polyamides 6
	PAni	Polyaniline
	PB	Pernigraniline base
	PC zeo	Polycarbonate
	PDMS	Poly (dimethyl siloxane)
	PE S	Polyethylene
	PC PDMS PE PEG entreprotected PEG	Polyethylene glycol
	PEGDE	Poly (ethylene glycol) diglycidyl ether
C	PEGDME	Poly (ethylene glycol) dimethyl ether
	PEO	Polyethylene oxide
	PET	Polyethylene terephthalate
	РММА	Poly (methyl methacrylate)
	PP	Polypropylene
	PPE	Poly (oxy-2, 6-dimethyl-1, 4-phenylene)
	РРу	Polypyrrole

PS	Polystyrene		
PU	Polyurethane		
PVC	Poly (vinyl chloride)		
PVPr	Polyvinyl propionate		
SAN	Styrene-acrylonitrile		
SEM	Scanning electron microscopy		
SiC	Silicon carbide		
$T_5$	Temperature at 5% weight loss		
T <sub>50</sub>	Temperature at 50% weight loss		
Tg	Glass transition temperature		
T <sub>m</sub>	Melting temperature		
TGA	Thermogravimetric analysis		
THF	Tetrahydrofuran		
TiB <sub>2</sub>	Titanium diboride		
$T_m$ TGA THF TiB <sub>2</sub> TiO <sub>2</sub> TSAS	Titanium dioxide		
TSAS	Toluene sulfonic acid		

### Kesan Pengisi- Pengisi Konduktif dan Poli (Etilena Glikol) Diglisidil Eter Terhadap Sifat-Sifat Filem Konduktif Poli (Vinil Klorida) / Poli (Etilena Oksida)

#### ABSTRAK

Dalam kajian ini, poli (vinil klorida) / polietilena oksida (PVC/PEO) dengan pengisi konduktif seperti karbon hitam, polianilina dan polipirrol telah disediakan secara teknik tuangan dengan kelajuan 400 rpm. Komposisi 5, 10, 15, 20, 25, 30% berat pengisi dicampurkan dalam campuran PVC/PEO 50/50. Sifat-sifat tegangan, konduktor morfologi, analisis inframerah spektroskopi (FTIR), kekonduksian elektrik dan analisis permeteran gravity haba (TGA) telah dikaji. Filem konduktif Poli (vinil klorida) / poli (etilena oksida) / karbon hitam (PVC/PEO/CB) pada komposisi yang berbeza telah disediakan. Kekuatan tegangan dan modulus tegangan meningkat sebanyak 25.33% dan 16.66% setiap satu dengan penambahan 25 % berat pengisi karbon hitam. Walau bagaimanapun, pada 30% berat kandungan karbon hitam, kekuatan tegangan dan modulus keanjalan adalah sedikit menurun kepada 15.87% dan 10.7% setiap satu. Kestabilan haba dan kekonduksian elektrik meningkat apabila jumlah karbon hitam didalam PVC/PEO meningkat. Dalam system yang lain, polianilina (PAni) diisi campuran PVC/PEO menjelaskan bahawa peningkatan jumlah kandungan PAni mengurangkan sebanyak 61.24 % kekuatan tegangan dan sebanyak 20.44% modulus keanjalan tetapi menunjukkan peningkatan positif kepada kekonduksian elektrik dan kestabilan haba filem konduktif PVC/PEO/PAni Kesan poli (etilena glikol) diglisidil eter (PEGDE) terhadap filem konduktif PVC/ PEO / CB menurun 55.65% untuk kekuatan tegangan dan kestabilan haba tetapi meningkat 6.45% untuk modulus keanjalan dan kekonduksian elektrik. Walau bagaimanapun, penambahan PEGDE dalam filem konduktif PVC/PEO/Pani telah meningkatkan 1.21% kekuatan tegangan, 6.45% modulus keanjalan dan kekonduksian elektrik tetapi kestabilan haba menurun. Morfologi filem konduktif setelah direndam menunjukkan penambahan PEGDE menyebabkan serakan pengisi yang lebih baik kepada filem konduktif PVC/PEO/CB dan PVC/PEO/PAni. Interaksi fizikal dalam campuran PVC/PEO, PEGDE dan pengisi dikenal pasti menggunakan spektroskopi FTIR. Dalam kajian itu, polypirrol (PPy) diisi dalam filem konduktif PVC / PEO pada 15 peratus berat dengan tambahan PEGDE menunjukkan komposisi lebih baik dari segi kekonduksian elektrik yang lebih tinggi dan kestabilan terma. Walau bagaimanapun, kekuatan tegangan dan modulus keanjalan adalah lebih rendah berbanding dengan filem konduktif PVC/PEO/CB dan PVC/PEO/PAni dengan dan tanpa kehadiran PEGDE.

# The Effect of Conductive Fillers and Poly (Ethylene Glycol) Diglycidyl Ether on the Properties of Poly (Vinyl Chloride)/Poly (Ethylene Oxide) Conductive Films

### ABSTRACT

In this research, the conductive films based on poly (vinyl chloride)/poly (ethylene oxide) (PVC/PEO) with conductive fillers like carbon black, polyaniline and polypyrrole were prepared by solution casting method at the speed of 400 rpm. The composition of 5, 10, 15, 20, 25, 30 wt % of filler loading were incorporated in PVC/PEO: 50/50 blends. The tensile properties, morphology, Fourier Transform Infrared spectroscopy (FTIR) analysis, electrical conductivity and thermogravimetric analysis (TGA) were studied. Poly (vinyl chloride)/poly (ethylene oxide)/carbon black (PVC/PEO/CB) at different compositions were prepared. The tensile strength and modulus of elasticity were increased until up to 25 wt% with the addition of the CB by 25.33% and 16.66% respectively. However, at 30 wt% of CB loading, tensile strength and tensile modulus were slightly decreased to 15.87% and 10.7% respectively. The thermal stability and electrical conductivity of PVC/PEO/CB conductive films are increased with the increasing of CB loading. In another system, polyaniline (PAni) filled in PVC/PEO blends explained that the increasing amount of PAni content reduced the 61.24 % of tensile strength and 20.44 % of modulus of elasticity but showed positive increment on electrical conductivity and thermal stability of PVC/PEO/PAni conductive films. The effect of poly (ethylene glycol) diglycidyl ether (PEGDE) on PVC/PEO/CB conductive films decreased 55.65 % of tensile strength and thermal stability but increased 6.45% modulus of elasticity and electrical conductivity. However, addition of PEGDE has increased 1.21% of tensile strength, 6.45% of modulus of elasticity and electrical conductivity but decreased the thermal stability of PVC/PEO/PAni conductive films. The morphology of soaked conductive films indicated that the addition of PEGDE imparted better filler dispersion to the PVC/PEO/CB and PVC/PEO/PAni conductive films. The physical interaction between PVC/PEO blends, PEGDE and conductive filler presented in conductive films was identified by FTIR spectroscopy. In the study, polypyyrole (PPy) filled PVC/PEO conductive films at 15 wt% with the addition of PEGDE exhibited better composition in terms of higher electrical conductivity and thermal stability. However, the tensile strength and modulus of elasticity was lower compared to PVC/PEO/CB and PVC/PEO/PAni conductive films with and without the presence of PEGDE.

### **CHAPTER 1**

### INTRODUCTION

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### **1.1 Research Background**

In recent year, conductive polymer may find new applications and the properties of polymeric materials can be simply transformed by combining more than one polymer by incorporation with additives, reinforcement fiber, conductive fillers and so on (Richard et al., 1993). In industry, the usage of filler in thermoplastic has been broadly experienced to enhance certain properties of the matrix. This approach offers various grades of polymer and to exhibit properties that are superior to any of component alone (Supri et al., 2011). The incorporation of inorganic filler recently gets wide attention in consideration of economic and properties modification factors such as mechanical, electrical and thermal properties (Gonzalez et al., 2002).

Current developments in the area of electrical power generation and storage system, polymer are widely used as electrical conductors, ionic conductors, polymer electrolytes, static dissipative materials and etc. The conductivity of the insulating polymer is in the range of  $10^{13}$ - $10^{16}$  [ $\Omega$  cm]. Owing to these merits, polymers are widely used as electrical insulators in many applications. Polymeric materials are naturally insulators and conductivity of the composite materials depends on the content and properties of the filler

such as the size and shape of the filler particles in addition to their distribution matrix, beside the interaction between the filler surface and polymeric matrix (Bishay et al., 2011).

A wide spread attention has been growing in manufacturing polymeric materials as conductive material without compromising the enviable mechanical and processing properties. Such area include the development of conductive polymers having the advantage of lower density than metals and able to perform well in corrosive environments. These can be attained by blending the conductive components where as generally the conductive fillers are used including metal powders, flakes or fibers, carbon black and carbon fibers. These electrically conductive polymer composites are widely used in antistatic products and components for electronic interconnects fuel cells, electromagnetic shielding and conducting adhesives in electronic devices (Kiraly & Ronkay, 2013).

Conductive polymer composites can be fabricated to obtain the desired properties for specific applications. One of the most useful applications for conductive polymer composites is as electromagnetic interference shields (EMI) (Strumpler & Glatz, 1999; Bouchet et al., 2000; Barba et al., 2006). Since electronic devices play a role in everyday life, EMI regulators will allow electronics to work in the intended fashion without any loss of performance. The intent of EMI shields is to protect electronic devices and circuits against interference from external electromagnetic fields of different frequencies. The main requirements for EMI devices are that they should be light weight, contain high-quality mechanical properties, are easy to process and most importantly, cost efficient. Meeting all of these requirements is what makes conductive polymer composites ideal for these applications (Barba et al., 2006). The composites are geared towards containing a conductive network that can be interrupted with an outside source. Applications that require this phenomenon are self regulating heaters, over-current protectors and chemical sensors (Strumpler, 1996; Chen & Tsubakawa, 2001). Heaney (1996) studied carbon black polymer composites. In his study, he observed in a sample with the same filler content that the resistance of the sample increased by over eight decades when the temperature went from  $25^{\circ}$ C to  $180^{\circ}$ C. This large change in resistance is known as the positive temperature coefficient of resistance. Strumpler (1996) studied temperature sensors and observed the same effect in epoxy composites containing Ni, Ni coated Ag, and TiB<sub>2</sub>. Chemical sensors work in the same fashion; instead of the change in temperature, the vapors from the chemical causes the polymers to swell (Chen & Tsubakawa, 2001).

Poly (vinyl chloride) is one kind of general purpose polymer materials with mature production technology, low price and the overall excellent performance. It can be widely used in construction, transportation, packaging, electronic, electric appliance and automobile, etc. However, the surface resistivity of PVC materials is high to $10^{14} \sim 10^{17}\Omega$ . For its good electrical insulation performance, in some cases, it is easy to produce static electronic and cause vacuuming, shock and other bad phenomenon. To a certain degree, the accumulated electrostatic still can produce a series of harm, cause great loss. The complex antistatic PVC materials which can be obtained by blending, filling and compounding with the other materials, it also can be easily shaped. They are economic, suitable for mass industrialized production, and have great market potential and broad prospect of application (Hakkarainen, 2003) The most broadly used material to improve conductivity is carbon black. Carbon black (CB) is composed about 97–99% elemental carbon. The other major constituents are hydrogen and oxygen. The hydrogen is distributed as hydrocarbon throughout the CB. These atoms are set into several organic functional groups such as -OH (hydroxyl), -COOH (carboxyl) on its surface. Besides the oxygen and hydrogen groups, CB may contain very small amounts of nitrogen and sulfur depending upon the nature of hydrocarbons used in the manufacture (Jiang et al., 2012).

Carbon black is used to reinforce elastomers to enhance mechanical properties such as modulus, hardness, tensile strength, and abrasion resistance. Payne (1965) stated that for reinforcing elastomers, CB has little influence on the temperature-frequency relationship of the dynamic modulus. In addition, the glass transition temperature Tg cannot be changed by CB loading (Kraus, 1970). Thus, geometry parameters of CB such as surface morphology and interactions between the CB and the polymer are the most important factors that can influence the mechanical properties of the composite.

Although, the conductivity of composites can be enhanced by the incorporation of conductive filler such as CB into the polymer matrix (Huang 2002; Mamunya 2001; Zois et al. 2001), some mechanical properties such as elongation and impact strength may decrease, which means the composite becomes more brittle. Therefore, mechanical properties of polymer composites such as polyethylene-CB and poly (vinyl chloride)-CB are important issues that need to be investigated. Recently, researchers (Liang et al. 2009; Yuan et al. 2010) had investigated the influence of CB content on the mechanical properties of low density polyethylene (LDPE), linear low density polyethylene (LLDPE), and high density polyethylene (HDPE). The tensile fracture strength and the elongation at break

increased significantly by the addition of less than 5% weight fraction of CB. On the other hand, the electrical conductivity with increasing volume fraction of CB. Therefore, choosing the appropriate loading of CB and improving the CB-matrix interaction are important to enhance the mechanical properties of the polymer composite.

According to Chiu et al., (2011), the addition of CB can be promising conductive filler for PPy/NBR composites because it has synergistic effect and compounding conductivity in the mechanical properties. The increasing of CB loading from 10 phr until 40 phr leads to improvement in of mechanical properties. Tantawy et al. (2002), the conductivity of epoxy reinforced CB composites increased continuously with increased of CB content until up to 6 wt% of CB loading. When CB content is lower than 4 wt. %, the conductivity of composites changes slightly and the composites will present insulator properties. This can be explained by the fact that the conductive CB particles in the epoxy matrix are widely separated and formed a finite cluster in epoxy matrix. Wen et al. (2012) have reported that the thermal decomposition temperature for PP/CB nanocomposites shifted significantly to a higher temperature range than for neat PP which indicates an improvement of thermal stability. Both  $T_{5 wt\%}$  increased with increasing of CB content. This was due to formation of three dimensional filler network structure by CB in PP which responsible for the improvement in thermal stability.

According to the Chiu et al., (2011), the electrically conducting nitrile rubber (NBR) containing electro-conductive carbon black and polypyrrole as conducting modifier showed the increase in thermal stability, conductive pathways and synergistic properties on thermal stability. However, electro conductive carbon black is limited by issues of hardness