POLARIZATION AND DEPOLARIZATION CURRENT OF LINEAR LOW DENSITY POLYETHYLENE-NATURAL RUBBER NANOCOMPOSITE SUBJECTED TO ELECTRICAL TRACKING AND MOISTURE

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Polymeric nanocomposites are widely used for high voltage outdoor insulating application due to their efficient electrical performance. Recently, SiO$_2$, TiO$_2$ and MMT nanofillers are used as fillers because they are listed as the main nanofiller commonly used in electrical engineering due to the increase of effective activation energy. Natural rubber (NR) is used because the nature of the interphase is found to affect viscoelasticity and it develops several interphases with the Linear Low-Density Polyethylene (LLDPE) matrix. This thesis presents the outcome of an experimental study which has been carried out to determine the conductivity level and dielectric response function of the LLDPE-NR compound, filled with different amount of SiO$_2$, TiO$_2$ and MMT nanofillers using Polarization and Depolarization Current (PDC) measurement technique. LLDPE and NR with the ratio composition of 80:20 were selected as a base polymer. The PDC testing was done on samples at various conditions: without any defect (normal condition), wet condition (moisture absorption) and after electrical tracking effect condition based on BS EN 60587:2007 standards. One of the problems associated with outdoor polymeric insulators is the tracking of the surface which can directly influence the reliability of the insulator. The amount of water content can be used to monitor the dielectric quality of insulation and as an indicator of possible deterioration for outdoor degradation. Besides PDC, surface morphology analysis using FESEM was also conducted to study the changes in physical structure which can explain the conductivity and response function. This research found that an addition of certain weight percentage of nanofiller and NR into the LLDPE improved conductivity level of the insulator. LLDPE-NR/TiO$_2$ at 5 wt% has become the best sample in terms of the lowest conductivity in normal and under electrical tracking effect. Besides, this sample has the fastest time response under electrical tracking and moisture condition. This investigation has successfully identified the PDC pattern, conductivity and dielectric response function of LLDPE-NR nanocomposite. Results show that different compositions as well as the surface physical conditions affect the PDC measurement results.
ABSTRAK

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<td>Aluminium</td>
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<td>ATH</td>
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<td>ASTM</td>
<td>American Society for Testing and Materials</td>
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<td>BS EN</td>
<td>British Standard European Norm</td>
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<tr>
<td>FESEM</td>
<td>Field Emission Scanning Electron Microscopy</td>
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<tr>
<td>HDPE</td>
<td>High Density Polyethylene</td>
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<tr>
<td>HV</td>
<td>High Voltage</td>
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<tr>
<td>IEEE</td>
<td>Institute of Electrical and Electronics Engineers</td>
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<td>IPT</td>
<td>Inclined Plane Tracking</td>
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<td>LabVIEW</td>
<td>Laboratory Virtual Instrumentation Engineering Workbench</td>
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<td>LC</td>
<td>Leakage Current</td>
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<tr>
<td>LDH</td>
<td>Layered Double Hydroxide</td>
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<tr>
<td>LLDPE</td>
<td>Linear Low- Density Polyethylene</td>
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<td>Layer Silicates</td>
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<td>MV</td>
<td>Medium Voltage</td>
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<td>MDH</td>
<td>Magnesium Dihydroxide</td>
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<td>MMT</td>
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<td>NR</td>
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<td>O</td>
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<td>OLTC</td>
<td>On-Load Tap Changer</td>
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<td>Organically Modified Montmorillonite</td>
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<td>PDC</td>
<td>Polarization and Depolarization Current</td>
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<td>Extruded Cross-Linked Polyethylene</td>
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<td>XRD</td>
<td>X-Ray Diffraction</td>
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<td>ZnO</td>
<td>Zinc Oxide</td>
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LIST OF SYMBOLS

$I_p(t)$ - Polarization current
$I_d(t)$ - Depolarization current
$U_0$ - Potential difference
$C_0$ - Geometric capacitance
$C_m$ - Measured capacitance
$f(t)$ - Dielectric response function
$\sigma$ - DC conductivity
$\varepsilon_0$ - Dielectric constant
$\varepsilon_r$ - Relative static permittivity
$\rho$ - Density
$wt$ - Weightage
# LIST OF APPENDICES

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CHAPTER 1

INTRODUCTION

1.1 Background of the Research

Nanocomposite material application in the field of dielectric and electrical insulation has attracted many researchers’ attention recently. Polymer nanocomposite materials were applied as insulation material since the 21st century and were given more attention because of the material properties that can be drastically improved by adding a few percent of nano-sized filler. This nanocomposite can be used as nanodielectric. This material had widely been used as power apparatuses insulator and cables insulator. Polymers nanocomposites are made by adding nanometer sized fillers and homogenously dispersed into the matrix of polymers composite. The filler is dispersed homogenously in the composite matrix by a certain weight percentages (wt%). The fillers that are added to the matrix are done in just a small quantity, typically less than about 10 wt%. Polyethylene or epoxy resin are widely used as insulators in industrial applications as the base material of many recent studies, while SiO$_2$, Al$_2$O$_3$, MgO, TiO$_2$ or layer silicates (LS) and nano clay(MMT) function as typical nanofillers [1-4].

Lately, significant attention has been given into the monitoring of the condition of electrical apparatus such as transformer, cable and rotating machine insulation system. These equipments are important units in power system. Hence, these equipment insulation systems should be monitored frequently to prolong the
equipment’s life time. For this reason, several new techniques for monitoring high voltage (HV) equipment through its insulation have been developed in recent years.

One of these techniques is Polarization and Depolarization Current (PDC) measurement. PDC is the time domain based techniques that are widely accepted by many utilities due to the advancement in hardware and software interpretation schemes [5]. Results from these techniques are severely influenced by several environmental factors, predominantly the temperature and aging process of the electrical insulation. Polarization and conduction phenomena occur in every electrical insulating material. Each problem in the dielectric is produced by the mechanism of polarization and conduction. Polarization takes place in all molecules of a dielectric and causes chemical change or deterioration in the material.

This dielectric technique has been considered by many researchers for power system insulations condition assessment due to its ability to provide information on the conductivity and moisture contents. Conductivity can be estimated within the initial periods (seconds) and the moisture content for long periods after a DC step voltage application.

Polymer nanocomposites are the 21st century engineering materials with wide range of markets such as transportation, electrical and electronics, food packaging and building industries. They are particularly attractive as advanced dielectrics and electrical insulation in the viewpoint of their inherent excellent properties. Polymeric nanocomposites have gained importance due to their material properties in providing enhanced performances, namely through their electrical and mechanical properties. But until now, there are no investigations done on the conductivity and response function in time domain of linear low-density polyethylene when it blends with natural rubber (LLDPE-NR) nanocomposite as a new insulation.

One of the objectives of this research in the field of LLDPE-NR nanocomposite dielectrics is to obtain new materials using improved dielectric properties of HV insulation by adding SiO$_2$, MMT and TiO$_2$ nanofiller. This research
presents results of an experimental activity which is aimed to find the conductivity variation and dielectric response functions of LLDPE-NR nanocomposite that was focused on normal condition, the effect of electrical tracking and the effect of moisture absorption. This research is focused on formulations of natural rubber blends nanocomposites obtained from polyethylene filled with nanoparticles of SiO$_2$, MMT and TiO$_2$. The conductivity and dielectric response function was done by using Polarization and Depolarization Current (PDC) measurement technique. Study on these parameters is required as an increasing number of power utilities nowadays choose polymer nanocomposite as a new insulation due to its unique properties. The study on conductivity is important in discovering how well the material can conduct the movement of charges when there was voltage supplied to the electrode. The lower the resistance, the greater is its conductivity because conductivity is the inverse of electrical resistance. Response function in time domain is important to determine how fast or slow the dipole would take to relax and return to the original state when there was no voltage supply.

1.2 Problem Statement

Polyethylene such as linear low-density polyethylene (LLDPE) is obtained via the copolymerization of ethylene with various alpha olefins. They have grown in importance because of the specific properties that can be obtained by varying co-monomer content and polymerization condition [6]. Several studies have been conducted on the LLDPE-NR and found that the composition of 80:20 ratio has shown to be the best composition due to the lowest carbon track development as compared with virgin LLDPE (Piah et al., 2003). Natural rubber was used because the nature of the interphase is found to affect viscoelasticity. It develops several interphases with the LLDPE matrix. The current project used LLDPE-NR as base material because it has shown significant improvement in terms of degradation due surface tracking and moisture effect. SiO$_2$, TiO$_2$ and MMT nanofillers are being used as filler because they are listed as the main nanofiller commonly used in electrical engineering due to the increase in effective activation energy.
Most of the high voltage apparatus’ polymer insulation such as transformer insulation and cable insulation face degradation due to surface tracking and water trees aging. Faults usually occur in the bodies of oil-paper cables, high voltage circuit breakers, surge arresters, power capacitors and transformers. One of the factors that affect the life cycle of a electrical equipment is the operating state of its insulation system. There are many insulation defects in power system equipment such as the presence of partial discharging (corona) in insulator, external flashover of bushings due to salt pollution and degradation of insulation due to ingress of moisture and surface tracking. This project was focused on degradation condition subjected to electrical tracking and moisture of LLDPE-NR nanocomposite because these conditions leave the most effect on insulation properties and they can represent outdoor aging. Carbon track is a process that takes place in the surface of insulator due to the surface tracking phenomena. The presence of carbon in most of polymers causes possible carbon track to occur.

Most of previous researches performed morphology studies, breakdown strength test, space charge formation and dielectric loss test to determine the electrical properties of polymer nanocomposite [7-9]. These studies showed physical structure in static condition, breakdown strength and electrical strength. However, these dielectric measurements are not adequate because they do not reveal information on the polarization current, depolarization current and the variation conductivity of nanocomposite under the defect of electrical tracking and under moisture condition. Nonetheless, without the accurate information of these parameters, it is difficult to determine the cause for the degradation of the insulator.

Condition monitoring techniques such as PDC measurement technique can be used to predict the remaining life of the electrical apparatus and also reduce unscheduled outages, improve maintenance planning and increase system reliability. Analysis of the fundamental dielectric processes has shown that the polarization phenomena are strongly influenced by the morphology and degradation of the polymeric insulation. Therefore, research into the dielectric properties of polymer nanocomposites based on PDC measurement would provide invaluable information to further understand the electrical insulating performance of polymer
nanocomposites used in high voltage applications. PDC value is very sensitive to moisture and electrical tracking which happen under wet contamination. Conductivity and dielectric response of the insulating material can be determined by using the relevant PDC technique. Response function in time domain is important to determine the response for the dipole when it is relaxed and returned to the original state when there is no voltage supply.

1.3 Aim and Objectives of the Research

The aim of this research is to identify the best LLDPE-NR nanocomposite uses for HV insulation by determining the conductivity and dielectric response function in normal condition and after degradation factors: electrical tracking and moisture effect. In order to achieve this aim, the study was divided into three objectives. The objectives of this research are:

i. To study the characteristics of electrical tracking test and moisture absorption effect of the newly developed LLDPE-NR nanocomposite materials. The effect of different weight percentages of nanofillers on material was also investigated.

ii. To analyse the Polarization and Depolarization Current (PDC) and conductivity trend of the newly developed LLDPE-NR nanocomposite materials, LLDPE-NR nanocomposite effect of moisture absorption and LLDPE-NR nanocomposite effect of electrical tracking condition.

iii. To determine the dielectric responsive function of the newly developed LLDPE-NR nanocomposite materials, LLDPE-NR nanocomposite effect of moisture absorption and LLDPE-NR nanocomposite effect of electrical tracking condition.
1.4 Scope of the Research

This research focused on the improvement of LLDPE-NR nanocomposite material. The scopes and limitation of this research are listed as below:

i. Insulating material that is used focused on polymer nanocomposite material. Linear Low Density Polyethylene (LLDPE) is selected as a base polymer and Natural Rubber (NR) grade SMR CV 60 with the ratio composition of 80:20. LLDPE-NR is filled with a small weight percentage of TiO$_2$, SiO$_2$ and MMT nanofillers.

ii. The wt % of nanofiller used are 1 wt %, 3 wt %, 5 wt % and 7 wt %.

iii. PDC testing is done on sample of the new material at the condition of: without any defect (normal condition), wet condition (moisture absorption) and after applied an electrical tracking condition based on BS EN 60587:2007 standard [10].

1.5 Significance of the Study

Polymer nanocomposites possess promising high performance characteristic as engineering materials if they are prepared and fabricated properly. Some work has been recently done on such polymer nanocomposites as dielectrics and electrical insulation [11]. One of the targets in this field is to obtain new materials with improved dielectric properties for high voltage (HV) insulation application. In general, a good insulator must have extremely low conductivity level. All the samples have undergone degradation process of surface tracking and moisture absorption. These conditions can represent major outdoor degradation. By considering this condition, the conductivity performance in normal and degraded conditions of all the samples studied can be determined.

PDC patterns of mineral oil and paper as transformers’ insulation have been studied by many researchers [12, 13]. PDC analysis is a non-destructive dielectric
testing method for determining the conductivity and dielectric response function. PDC analysis is normally used to determine the water content in the oil-paper insulation barrier and also its oil conductivity. However, there are no investigations on PDC done for LLDPE-NR nanocomposite. Study on the PDC pattern of LLDPE-NR nanocomposite is required as it is a newly introduced insulation material for electrical equipment. Others dielectric measurements are not focused on important parameters such as information on the polarization current, depolarization current, variation conductivity and dielectric response function of nanocomposite. Nonetheless, without the accurate information of these parameters, it is difficult to determine the cause for the degradation of the insulator. PDC is also very sensitive to moisture condition. For this reason, PDC was selected as an accurate testing method to determine the conductivity and response function in time domain.

Therefore in this work, PDC tests were performed on insulation of LLDPE-NR nanocomposite samples at various conditions, namely the normal condition, the moisture absorption condition and the electrical tracking effect condition. These conditions are chosen because electrical tracking is one of the major problems in outdoor insulation systems and it similarly occurs due to degradation of aging factor. Effect of moisture absorption is considered as one of the major insulation degradation cause. The amount of water content can be used to monitor the dielectric quality of insulation and as an indicator of possible deterioration. Deterioration will adversely affect its electrical properties and this will eventually cause dielectric breakdown. The higher the moisture contents in the insulation, the higher the conductivity will be.

The test data are used to find the dielectric responsive function and maximum conductivities of LLDPE-NR nanocomposite. PDC is carried out to find the dielectric responsive function and trend of this material conductivities level based on the test conditions. In order to estimate the dielectric response function \( f(t) \) from a depolarization current measurement, it is assumed that the dielectric response function is a continuously decreasing function in time, then if the polarization period is sufficiently long, as in \( f(t+t_c) \approx 0 \), the dielectric response function \( f(t) \) is proportional to the depolarization current.
The present thesis consists of six chapters. Chapter One provides an introduction to the problem statement, objectives, scope of the research and significance of the research. Chapter Two outlines the literature review which covers topic of polymer nanocomposite, DC conductivity and moisture content of polymer nanocomposite, dielectric response, Polarization and Depolarization Current (PDC) and electrical tracking. Chapter Three outlines some of the test methods used to investigate the performance of insulation materials. Detailed explanation of experimental setup, procedure and technique employed in this research include material preparation, morphological test, capacitance test, polarization and depolarization current test and electrical tracking test. In Chapter Four, all experimental results are presented and discussed. Chapter Five consists of maximum conductivity and response function analysis. Finally, Chapter Six draws conclusions and makes recommendations for future research.
CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, key subjects that are related to this research are summarised, firstly by providing an outline of the history of polymer nanocomposite, then focusing on the electrical properties and literature on polymer nanocomposite for high voltage insulation. This chapter also reviews the Polarization and Depolarization Current (PDC) measurement technique and electrical tracking effect on insulation, moisture effect on insulation and polymer nanocomposite, theories, technologies and research done which are briefly explained and discussed thereafter. This part presents comprehensive review and analysis on conductivity value based on the experimental results from previous researchers to find patterns and trends of the test material. The review and analysis from previous researches and studies on current and conductivity using different methods and samples are also compared.

2.2 Polymer Nanocomposite

In the 1990s, the commercialisation of Polyamide 6/clay nanocomposite that was manufactured as engineering plastics by applying polymer nanocomposite for engineering level has encouraged researchers to study the possibility of combining different polymers with different nano inorganic fillers. Since then, much effort has been made to develop and apply polymer nanocomposites in electrical and
electronics engineering, transportation, food package and industries. Polymer nanocomposites which are newly developed advanced materials have wide application potential [11].

Nanodielectrics for the electrical insulation field have commenced in year 1994 when John Lewis published a paper entitled “Nanometric Dielectrics” in a publication titled IEEE Transactions on Dielectrics and Electrical Insulation [14]. The role of nanometric interfacial processes in initiating electrical breakdown in insulating materials has gained much attention since. Studies showed that the attainment of equilibrium or a steady state in the interface of dielectric and conductive is a requirement. In general that charge is transported towards or away from it through the bulk phases on either side that depend on their electrical conductivities. If one or both possess a highly insulating characteristic, the time to achieve this attainment can be exceedingly long [14].

In 2002, first experimental data were produced on nanometric dielectrics by a researcher [15]. It was proved that nanometric fillers mitigate the interfacial polarization characteristic of conventional materials and hence, the internal field accumulation was reduced. Large interaction zone in reduced mobility (free volume) nanofilled polymers should lead to a significant change in electrical properties. Studies of electrical behaviour thus have provided an opportunity for both a fundamental study of this interaction zone, as well as an opportunity for optimising performance for specific and critical applications. Nanotechnology was overviewed with a focus on material potentialities, implications and development which concluded that a new class of materials, nanodielectrics, is emerging [16].

Since 2002, some works that have been done in IEEE focusing particularly on nanocomposite. Several reviews were made on polymer nanocomposites for electrical insulation and high voltage dielectric application. A multi-core model which has a far-distance effect and is closely related to an “interaction zones” was proposed by considering electrical and chemical structures mesoscopic analysis of an existing interface with finite thickness [11]. To have a deeper understanding of
nanodielectrics in general, further studies were done toward future research and development of the material [17].

The second generation resins that are used in insulation engineering are called polymer nanocomposites. These materials consist of polymers that are filled with many micron-sized inorganic fillers in the order of 50 wt%. The recently produced polymer composites can be defined as a small amount of nanofillers that fill in a substance of polymers. The size of the nanofillers varies from 1 to 100 nm, while the content varies from 1 to 10 wt%. Nanofillers should be dispersed in the polymer matrix homogeneously. Although nanofillers have a small content percentage (low wt %), they possess very large surface areas if compared to those of microfillers [11]. These newly developed polymer nanocomposite advanced materials have a wide application in food packaging, building, electrical and electronics engineering, transportation and other fields of industries. The materials are used in application of coating materials, foamed materials, barrier-functional materials, and flame-retardant materials. The most used product in the market is polyolefin.

When polymer nanocomposite can change one or several of its dielectric properties, it can be used as nanodielectric [18]. Nanodielectric is widely used in rotating machine insulations, HV cables, solid insulation in switchgear, and outdoor insulation for power electronic devices packaging. Besides that, it is also used in rotating machine insulations [19]. It has been proved that nano-sized filler improved the material dielectric properties for power system insulation application [20].

A lot of research was conducted to study the polymer nanocomposite dielectric properties [21-27]. Most of these studies focused on epoxy nanocomposite which has different nanofiller influencing the dielectric properties. Most of these researchers measured electrical properties of the material such as resistivity of dc volume, strength of ac dielectric, permittivity of dielectric and tan delta (400 Hz - 1 MHz). Results from these studies showed the dielectric behaviours of nanocomposites. It was also found that some electrical properties of these materials are unique and very useful to be used in existing and potential electrical systems at
certain loadings of nanoparticle. Ar [28] has performed dielectric strength, volume resistivity and surface resistivity testing on HDPE clay nanocomposite based on ASTM D149 and ASTM D257 standards. Observation showed that there was significant improvement in nanoclay when organoclay was treated with titanate and silane compounds. Nanofiller mixture composite can greatly improve the discharge surface resistance and breakdown strength of insulation [29, 30]. According to [31], from various nanoparticulates, clay minerals and carbon nanotubes are more preferable and often used to enhance physical, mechanical and thermal properties of polymers. In terms of material, the four main groups that were significantly used in nanocomposite for electrical engineering are silicone oxides (silica), metallic oxides and hydroxides (Al₂O₃, TiO₂, MgO, ZnO etc), nanoclays (Montmorillonite, hectorite etc) and carbon nanotubes [4, 32]. Pettarin et al. [33] in their testing prepared their base material with high-density polyethylene (PE), an organically modified montmorillonite (OMMT), while ethylene/methacrylic acid copolymer (EMAA) was used to melt compounding. The composites morphology was examined by XRD, SEM and TEM and the mechanical properties were evaluated under static conditions.

Another research [34] used morphology and thermal stabilisation mechanism to study LLDPE/MMT and LLDPE/LDH nanocomposites. The research found that all nanocomposites show significantly enhanced thermal stability compared to virgin LLDPE due to the increase of effective activation energy during the degradation process. According to [35], the dynamic mechanical properties of LLDPE/nano-SiO₂ composites were improved compared with the corresponding value for LLDPE/untreated SiO₂ composites. Some researchers conducted studies on silica nanoparticles as insulators [3, 30, 35-38]. Researcher [39] performed morphology, water treeing and electrical behaviour test on a linear low density polyethylene (LLDPE) with silica nanoparticles surface treatment. This research found that samples with silane-treated nanoparticles have improved the dielectric properties compared to the unsurface-treated sample. Researchers [40] developed nanocomposites formed from isotactic polypropylene (iPP) filled with MMT. Thus, this material is regarded as a candidate for power cable insulation material with
enhanced properties. The research found that, the material has increased loss factor and permittivity compared to unfilled iPP.

2.3 DC Conductivity of Polymer Nanocomposite

Extensive studies on polymer nanocomposites for HV insulation were conducted in these recent years because it has unique properties and able to enhance electrical performance. Polymer nanocomposites electrical conduction is used as one of the components to monitor its dielectric behaviour. Many studies were done to investigate the electrical properties of this material, such as strength of electric breakdown, resistance of partial discharge, space charge and conduction current measurement [9, 41-43].

The result shows that DC conductivity exhibits significant reduction with the addition of any type of nanofiller in a certain amount. However exact percentage of the nanofiller addition is not the same for different types of base materials. This was due to the decreasing of initiation probability of short circuit treeing in each material. The conductivity analysis done found that the addition of nanofiller into the polymer at certain percentage has improved its insulation properties.

Ciuprina and Plesa conducted a research on DC conductivity and the variation of the real part of complex conductivity with the frequency for three formulations of nanocomposites obtained from polyethylene filled with nanoparticles of Al$_2$O$_3$, SiO$_2$ and TiO$_2$ [41]. Three types of polyethylene nanocomposites were prepared by researchers [41] with nanofillers of SiO$_2$, TiO$_2$ and Al$_2$O$_3$. Nanoparticles content of the tested formulations were recorded at 2, 5 and 10 wt%. The average diameter of the nanoparticle for Al$_2$O$_3$ was 40 nm while for SiO$_2$ and TiO$_2$ the average diameter was 15 nm.

Similar research were also conducted by [42] on electrical conduction at various applied electric fields. It was found that the conduction current shows a
minimum reading at a 1% b.w. concentration nano alumina particle. Researchers [9, 43] studied DC conduction of MgO/LDPE nanocomposite. Through the DC conduction pattern, they found that the addition of MgO nanofiller leads to the improvement of DC electrical insulating properties of LDPE.

Researchers [42] have studied the electrical conduction processes in linear low density polyethylene (LLDPE) filled with nano alumina particles. LLDPE used in this study is a commercial linear low density polyethylene from Atofina. It has a density of 0.934 g/cm$^3$ at a melt index of 0.87 g/10 min. Nanoparticles of aluminium oxide from Degussa with a particle size of about 13 nm was used as filler.

Researchers [9, 43] studied DC conduction of MgO/LDPE nanocomposite. The employed polymer was LDPE mixed with MgO nanofiller. MgO nanofiller has an average diameter of tens of nm. MgO nanofiller went through surface treatment with a silane coupling agent, and then treated with the jet grinding treatment before kneading is done. High concentration of MgO nanofiller master batch was prepared first by the twin-screw extruder.

Researchers [44] studied surface potential decay and dc conductivity of TiO$_2$-based Polyimide Nanocomposite Films. In-situ dispersion polymerization process with a thickness of 70 μm was used to prepare Polyimide/TiO$_2$ (PI/TiO$_2$) nanocomposite films containing surface modified nano-TiO$_2$ particles by employing silane coupling agent. Researcher [38] found that, the conduction via electron tunnelling will occur between polymer-covered particles under the influence of an electric field only within the range of 1 - 4 nm. Due to the large surface area of the nanofiller, conductive pathways throughout the composite can be lead [45].

2.3.1 Conduction Current Measurement System

Figure 2.1 shows the electrode configuration and the experimental setup for the conduction current measurement used by [9, 43]. A gold electrode of 40 mm in
diameter was formed by vacuum evaporation on one side of the film. On the other side, a gold electrode of 26 mm in diameter was formed as the main electrode and a gold electrode consisting of 32 mm in inner diameter and 40 mm in outer diameter was formed as the guard electrode. The conduction current measurement was performed at 303 Kelvin at room temperature. The conduction current at 10 min after the DC voltage application was employed to determine the volume resistivity.

![Figure 2.1: Conduction current measurement system [9, 43, 46]](image)

Researchers [44] have done the DC conductivity test in the oven, with 20 mm diameter gold electrodes on both sides at 6.5 kV for 1 hour. The testing temperature was fixed at 30 °C to avoid the influence of small temperature fluctuation in the room on measured current [44].

Both conduction current measurements set up by researchers [9, 43] and researcher [44] showed different DC measurement setup with different electrode configuration. The injected voltage and measurement period also differed from both experiments. Even though there are some differences, results from both experiments can be used to determine the DC conductivity of the samples.
2.3.2 Current and Conduction Analysis

Current and conductivity had been applied by many researchers to study the best concentration percentage of nanofiller. Researcher [44] measured current for the TiO2 based polyimide nanocomposite at different added % amounts and these values are shown in Figure 2.2. From the results, 3% is seen to possess the best insulation property, 1% is slightly better than 5%, while 7% is noted to be the worst. It can be concluded that adding nanoparticles into dielectrics can improve their electrical properties. However, different amount of nanofillers will yield different results. Larger amount of nanofiller can create agglomeration inside a material which can contribute to larger overlapping interaction zone. This will cause the charge moving increase when the content of nanofiller is increased. As 1% and 3% samples reduce the conductivity to the lowest point, it can be known that a small amount of nanofillers is separated inside the dielectric with a certain wide distance, which can be known as extra traps for the dielectric, and therefore improve the insulation property [44]. These nanofillers will fill the empty zones inside the material which can contribute to function as the extra trapping mechanism for the dielectric. Nanofillers may align together and help charge moving inside the material.

However, if more nanoparticles are added into the dielectrics, for example, a 5% sample has better insulation property than a pure sample, but its conductivity is higher than 1% and 3% samples. This is because some of the nanofillers are too close to each other, and each nanoparticle has an interaction zone around it, which results to some overlap within the interaction zones. For the 7% sample or higher amount nano dielectrics, the probability of interaction zones overlap is higher, and therefore, the nanoparticles may align with each other, which help the charges to move across the dielectric [44].
Figure 2.2: Current for TiO$_2$ based polyimide nanocomposite at different % amount added [44]

Figure 2.3 shows the influence of the MgO nanofiller content on the volume resistivity under the field application of 40 or 80 kV/mm. The volume resistivity was calculated by multiplying the applied field and the main electrode area to the reciprocal of conduction current. The open sign is each value of three measurements is in each condition. The volume resistivity of the film with MgO nanofiller under each applied field was higher than that without MgO nanofiller. Above the MgO nanofiller content of 1 or 2 phr, the volume resistivity of the film with MgO nanofiller showed a saturated value [9, 43].
Figure 2.3: The influence of the MgO nano-filler content on the volume resistivity [9, 43]

Figure 2.4 shows I-V characteristic in alumina nanoparticles filled LLDPE samples. The conduction current in PE-1 sample is lower than the sample without any alumina nanoparticles. This means that the addition of a small amount of alumina hinders the movement of charge in the bulk of the material. As the amount of alumina increases the conduction current in PE-5 increases significantly compared to PE-0. The conduction current in PE-10 sample shows significant reduction at lower voltages but becomes very high once the applied voltage exceeds 5 kV [42].
Based on the research output highlighted earlier, it can be concluded that DC conductivity measurement can be used to compare nano dielectrics properties. Different types of nanofiller and the percentage of concentration will result to different values of current and conductivity. The conduction current increases when the filler content increase up to certain wt%. The presence of nanofillers influences the conduction, especially for high electric fields [46]. Besides, the volume resistivity of LDPE can be increased by addition of MgO nanofiller.

2.4 Moisture Absorption in Polymer Nanocomposite

Some research was done to study the moisture absorption of nanocomposite polymer at different level of water content [38, 47-49]. They discussed how water absorption by nanofillers can significantly affect the material’s electrical properties. Researchers [50, 51] conducted tests for thermal properties, moisture absorption and dielectric properties of pure epoxy and epoxy composites with micron-sized and nano-sized filler. The test results found that nanocomposite studied has a higher moisture absorption compared with microcomposite. These results were based on the

![Figure 2.4: I - V characteristics in alumina nanoparticles filled LLDPE samples at 20°C [42]](image-url)
filler’s density, shape, as well as its surface treatment. This indicated that the effectiveness of the dielectric constant of the composite was dependent on the dielectric constant ratio between the filler and polymer and also the degree of interaction between them. Different amount of nanofiller can effect the interaction between filler and polymer and dielectric constant from the dissolving the nanofiller uniformly inside the material. With the experimental data and molecular dipole polarization calculations, it was shown that the addition of silane coupling agent could form chemical bonding and improve the interphase interaction in the composite so that the interphase dielectric constant can be increased [50].

Figure 2.5: Moisture absorption of three materials as aging time [50]

Figure 2.5 shows the moisture absorption of composites with 20 wt% filler as a function of time after the moisture uptake was normalised to the polymer weight percentage. From the figure above, the moisture absorption behaviour of the polymer matrix is not affected by the micron-sized filler. On the other hand, a rapid increase in moisture uptake was shown by the nanocomposite. It is believed that the moisture was attracted by the hydrophilic nature of the nano-sized silica surface. The nanocomposite has a larger interface area between the resin and the filler compared to the microcomposite. Thus, the larger numbers of sites attract moisture. Since the surface becomes more hydrophilic, the interfacial tension between the surface and
water is reduced [4]. And finally, the increased polymer dynamics in the nanocomposite also helps the moisture diffusion. The particles with spherical shape have a surface constituted of hydroxyl [32]. This can contribute to higher moisture absorption.

2.5 Polarization and Depolarization Current (PDC)

Polarization and Depolarization Current (PDC) test were used to monitor condition of many electrical apparatus such as machine stator, transformer and power cables. PDC measurement with time domain polarization based technique is widely accepted by many utilities due to the advancement in hardware and software interpretation schemes [5]. Also this technique is very useful for estimating the conductivity and moisture contents of the insulations. PDC test was used for the maintenance and diagnostic tests periodically conducted on machine stator insulation systems to continuously monitor both charge and discharge current during a step voltage test [52, 53].

Researcher [54] has applied the PDC analyser for the insulation assessment of power cables since 2002. He found that the most advantageous aspect of this technique is its easy identification between “conduction” and “polarization” phenomena in a dielectric. Researches [55-57] applied PDC technique for XLPE insulation that was subjected to wet aging. These testing were done using AVO Megger S1-5010 at the medium voltage (MV) underground power cables with extruded cross-linked polyethylene (XLPE) insulation. Furthermore, there was a research done to describe the polarization and depolarization current responses from XLPE cable insulation with and without water trees [55]. This researcher used apparent conductivity which was based on difference between polarization and depolarization currents and degree of nonlinearity factor which is the ratio of apparent conductivity at different voltage as the parameter to determine the condition of the cables. From the results obtained, it showed that the difference of polarization and depolarization current is proportional to the conductivity of the insulation and
can therefore be as a first indicator of insulation aging. Conductivity is the ability of the material to conduct an electric current. Equation (2.4) shows that PDC value is proportional to conductivity. The higher is the difference between these currents or the higher the conductivity, the greater is the degradation of the cable insulation [56]. Researchers [58, 59] too assessed the insulation condition of an underground cable by measuring its polarization and depolarization currents. PDC measurements can be used to determine the severity of insulation degradation. The level of the conductivity can represent the severity of degradation in term of moisture absorption. A higher content of moisture absorption can yield to a higher level of conductivity. This is because PDC is very sensitive to moisture content and outdoor degradation. Polarization and conduction phenomena occur in every electrical insulating material.

Research also has been conducted by [60] on PDC analysis for power transformer. The scope of the analysis was not only limited to insulation between windings, but also on insulation systems of winding-to-ground. Sometimes this can reveal trouble in the transformer accessories, such as on-load tap changer (OLTC) which focuses on water and contaminant in a new OLTC, moisture and surface humidity and free water in a refurbished transformer. This technique had also been already applied as a quality assurance tool for the assessment of refurbishment efficiency of power transformers by researcher [61] and moisture assessment of transformer bushings by researchers [62]. Estimation of water content and conductivity in a power transformer that is focused on paper insulation also were done using PDC measurement by researcher [12, 63-72]. Moisture and aging strongly influence the dielectric properties of oil-paper insulation system of power transformer. Moisture accelerates the aging process in the oil-paper insulation of the transformer. PDC analysis was also done by researcher [73 and 74] on the influence of thermal transients and thermal runaway of oil-paper insulation. These researches were done to identify and locate overheating/thermal fault in the transformer solid insulation using PDC analysis [75]. There was also research done on paper oil insulation under conditions of copper corrosion using PDC technique [76].
The PDC patterns of mineral oil, biodegradable oil and paper as transformer insulations were studied by many researchers such as [12, 13, 63]. Researches [13, 68, 73, 74, 77, 78] were done to investigate the moisture content and conductivity of the oil insulation that is focused on transformer oil. There was also research carried out to find the dielectric responsive function and maximum conductivities of biodegradable and mineral transformer oils with different moisture levels (dry, normal, or wet) for comparative analysis by researcher [12]. This research examined the condition of the insulation oils at different moisture levels. Comparison on oils conductivity level (σ) and measured capacitance value (Cm) in each condition with the PDC fingerprint of the oils during the initial installation was done. The trends of the PDC response were found to be different for those two insulation oils. They extended the research for faults identification of biodegradable oil-filled transformers by comparing them with the PDC pattern [79]. The PDC technique provides significant information that can be used in the evaluation of the transformer condition. Each fresh insulation oil has its own unique PDC fingerprint response [12].

Polarization and depolarization currents analysis were done in biodegradable polymers that can be used in electrical apparatus and cable [80]. For instance, one research presented and discussed the data on polarization and depolarization characteristics of thermally degraded epoxy mica composites [81]. Besides, researchers [82] obtained conduction currents results using PDC for epoxy resin nanocomposites. A DER 332 epoxy resin was chosen as matrix and nanosilica and/or Boron Nitride were chosen as fillers. They obtained the dielectric response in the frequency domain.

Other researches were done to monitor insulation condition on transformer oil, XLPE cable insulation, rotating machines insulation and biodegradable polymers using PDC test. Similarly, a lot of research were done on application of PDC technique in assessing the conductivity and moisture content of the insulation of the electrical equipment. These studies examined the effects of moisture level, aging and water trees of the insulation.
In conclusion, the PDC method is a simple and reliable technique based on time domain analysis to monitor HV insulation systems. From the PDC results, dielectric responsive function of each sample was determined to examine the dielectric response based on time domain. This is a natural response of the material itself due to the depolarization current that was measured from the sample.

In this research, PDC measurement was performed on polymer nanocomposite as an insulating material at various conditions. The viability and reliability of the PDC method in providing information on the conductivity and moisture content of polymer nanocomposite has not been explored and done before, and thus it was investigated in this research.

### 2.5.1 Basic Principle of PDC

The measurement principle of polarization and depolarization current is based on applications of DC voltage across the test object for a long time. According to [41], the test voltage at 500 V has led to satisfactory results on measurement performed on LDPE nanocomposite for DC conductivity. Most of the researchers in PDC testing used 1000 V in the same test with duration of 10,000 seconds. During the test time, the current arising from the activation of the polarization process with different time constant that corresponded to different insulation materials due to the conductivity of the object was measured. Then the voltage was removed and the test object was short circuited. The activated polarization process then gave rise to the discharging current in the opposite direction, where no contribution of the conductivity was present. The charging and discharging currents were influenced by the properties of the insulating materials as well as by the geometric structure of the insulating system.

According to [12, 13, 52, 83, 84], polarization and depolarization currents measurement can be used to investigate the polarization process of a dielectric material in the time domain analysis. The test object can be a single dielectric
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