PARTIAL DISCHARGE AND BREAKDOWN STRENGTH OF CROSS-LINKED POLYETHYLENE NANOCOMPOSITES CONTAINING PLASMA-TREATED SILICON DIOXIDE NANOPARTICLES

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy

School of Electrical Engineering
Faculty of Engineering
Universiti Teknologi Malaysia

AUGUST 2022

DEDICATION

Specially dedicated to

My beloved family members for the support and encouragement to complete this project.

Father and Mother,

Mat Saman Bin Abd Wahab and Fatimah Binti Ibrahim,

Siblings,

Nurhafizah Binti Mat Saman

Nurhafiza Binti Mat Saman

Norhafizi Bin Mat Saman

Nurhafehasnita Binti Mat Saman

And

My supervisor who always guided and encouraged me through the research's hardship.

My friends for their support and sincerity.

ACKNOWLEDGEMENT

Bismillahirahmanirrahim.

Alhamdulillah, first and foremost, all praise Allah S.W.T for the guidance and blessings given to me to complete my Ph.D. study. Peace be upon the Prophet of Muhammad S.A.W., the messenger of Allah S.W.T. Secondly, I would express my deepest gratitude to the supportive supervisors, Ts. Dr. Mohd Hafizi Ahmad and Prof. Dr. Mat Uzir Wahit for their advice, guidance, and support until this research was completed successfully. They always give wonderful ideas and information that benefit me to carry out this research effectively. Their provision and support are valuable in completing my study.

Furthermore, I am greatly indebted to the Ministry of Higher Education Malaysia (MOHE), Universiti Teknologi Malaysia (UTM), and Majlis Amanah Rakyat (MARA) for the sponsorship and granting the funds which financially support me in my Ph.D. study. Special appreciation goes to all my friends and research partners for their kind cooperation and moral support throughout the period of completing this research. Moreover, in completing my Ph.D. study, I contacted many researchers, academicians, and practitioners. I would like to express sincere gratitude to all who continuously support me in terms of mental, physical, knowledge, and constant guidance during this research duration.

The highest appreciation and thankfulness are also addressed to the staff of the Institute of High Voltage and High Current (IVAT) and the School of Electrical Engineering (SKE) for their help and technical support in various ways throughout the experimental work in the laboratory.

Last but not least, I would like to dedicate my sincere gratitude to my family, especially my beloved parents and all of my siblings, for their continuous prayer, love, support, patience, understanding, and encouragement in accomplishing my Ph.D. study. This research would not be completed without the support, assistance, and precious advice from every one of them, as aforementioned. Each of them gave all of the guidance and support are truly priceless for me to complete my Ph.D. successfully. May Allah S.W.T bless all of us.

ABSTRACT

Polymer nanocomposites are promising formulations and an inspiring route for developing innovative polymeric-based insulating materials. However, the agglomeration of nanoparticles within the polymer matrix is the main factor that restricts the enhancement of insulation characteristics because the non-uniform dispersion of nanoparticles consequently reduces the interfacial area and creates weak polymer-nanofiller interfacial bonds. Conventionally, the chemical surface functionalization and calcination techniques have been introduced to modify the surface of nanoparticles in enhancing the compatibility between the nanoparticles and polymer matrix. However, these techniques were unsuitable for implementation because they required complex sequential steps. Thus, this research proposed an alternative technique of nanoparticle surface modification using atmospheric pressure plasma (APP) to improve the surface compatibility of nanoparticles and polymer matrix, consequently leading to the enhancement of dielectric properties such as partial discharge (PD) resistance and alternating current (AC) breakdown strength. This thesis also explores the effect of plasma treatment and its correlation to the aforementioned dielectric properties. In this study, the optimum operating parameters of APP, such as voltage supply, excitation frequency, and working gas flow rate have been characterized to acquire homogeneous and stable plasma discharge, which is then used to treat the surface of silicon dioxide (SiO_2) nanoparticles in enhancing its compatibility with cross-linked polyethylene (XLPE) matrices. The weight percentages of untreated and plasma-treated SiO_2 nanoparticles dispersed into XLPE were manipulated to 1 wt%, 3 wt%, and 5 wt%, as well as the duration of treatment that manipulated to 1 minute, 3 minutes, and 5 minutes to identify the most effective formulation of XLPE/SiO_2 nanocomposites. As aforementioned, the optimum operating parameters for producing homogeneous and stable plasma discharge were 0.5 kV, 20 kHz, and 0.8 L/min, respectively. In comparison with unfilled XLPE, the most effective formulation of XLPE nanocomposites was shown by the sample with 3 wt% of 5-minute plasma-treated SiO_2 nanoparticles with the highest PD resistance with the reduction of PD magnitude up to 2000 pC, the reduction of PD number up to 220512, and the reduction of surface roughness due to PD attacked up to 1.04 μm. Meanwhile, the same formulation of XLPE nanocomposites also indicated the most significant enhancement of AC breakdown strength up to 26.29 kV/mm compared to unfilled XLPE. Plasma has been found to be an alternative technique to improve the PD resistance and AC breakdown strength of XLPE nanocomposites by exciting the formation of the more substantial interfacial regions through the formation of interfacial bonds and the reduction of the size and number of agglomerated clusters. It is inferred that the plasma treatment method is appropriate for producing nanocomposites with improved surface compatibility and enhancing polymer nanocomposites' dielectric properties for high voltage insulation material applications.

ABSTRAK

Nanokomposit polimer adalah rumusan yang mengalakkan dan laluan inspirasi untuk membangunkan bahan penebat berasaskan polimer yang inovatif. Walau bagaimanapun, aglomerasi nanozarah dalam matriks polimer adalah faktor utama yang menyekat peningkatan ciri penebat kerana penyebaran nanozarah yang tidak seragam mengakibatkan pengurangan kawasan antara muka dan mewujudkan ikatan antara muka polimer-nanozarah yang lemah. Secara konvensional, teknik rawatan kimia dan teknik pengkalsinan telah diperkenalkan untuk mengubah suai permukaan nanozarah dalam meningkatkan keserasian antara nanozarah dan matriks polimer. Walau bagaimanapun, teknik ini tidak sesuai untuk pelaksanaan kerana memerlukan langkah berurutan yang kompleks. Oleh itu, penyelidikan ini mencadangkan teknik alternatif pengubahsuaian permukaan nanozarah menggunakan plasma tekanan atmosfera (APP) untuk meningkatkan keserasian permukaan nanozarah dan matriks polimer, seterusnya membawa kepada peningkatan sifat dielektrik seperti rintangan nyahcas separa (PD) dan kekuatan pecahan arus ulang alik (AC). Tesis ini juga memperincikan kesan rawatan plasma dan kaitannya dengan sifat dielektrik yang disebutkan di atas. Dalam kajian ini, parameter operasi optimum APP seperti voltan bekalan, kekerapan pengujaan, dan kadar aliran gas kerja telah dicirikan untuk memperoleh pelepasan plasma yang homogen dan stabil, yang kemudiannya digunakan untuk merawat permukaan nanozarah silikon dioksida (SiO_2) dalam mempertingkatkan keserasiannya dengan matriks polietilena berkait silang (XLPE). Peratusan berat nanopartikel SiO_2 yang tidak dirawat dan dirawat plasma yang disebarkan ke dalam XLPE telah dimanipulasi kepada 1 wt%, 3 wt%, dan 5 wt%, serta tempoh rawatan yang juga dimanipulasi kepada 1 minit, 3 minit, dan 5 minit untuk mengenal pasti formulasi nanokomposit XLPE/SiO_2 yang paling berkesan. Parameter operasi optimum untuk menghasilkan plasma dengan nyahcas yang homogen dan stabil seperti yang dinyatakan di atas masing-masing adalah 0.5 kV, 20 kHz, dan 0.8 L/min. Berbanding dengan XLPE yang tidak diisi, formulasi nanokomposit XLPE yang paling berkesan ditunjukkan oleh sampel dengan 3 wt% daripada nanozarah SiO_2 yang dirawat plasma selama 5 minit dengan rintangan PD tertinggi dengan pengurangan magnitud PD sehingga 2000 pC, pengurangan bilangan PD sehingga 220512, dan pengurangan kekasaran permukaan akibat serangan PD sehingga 1.04 µm. Sementara itu, formulasi nanokomposit XLPE yang sama juga menunjukkan peningkatan kekuatan pecahan AC yang paling ketara sehingga 26.29 kV/mm berbanding XLPE yang tidak diisi. Plasma telah didapati sebagai teknik alternatif untuk meningkatkan rintangan PD dan kekuatan pecahan AC nanokomposit XLPE dengan mengujakan pembentukan kawasan antara muka yang lebih besar melalui pembentukan ikatan antara muka dan pengurangan saiz dan bilangan gugusan beraglomerasi. Dapat disimpulkan bahawa kaedah rawatan plasma adalah sesuai digunakan dalam pengeluaran nanokomposit untuk meningkatkan keserasian permukaan nanozarah dan perumah polimer, sekaligus meningkatkan sifat dielektrik nanokomposit polimer sebagai bahan penebat untuk aplikasi voltan tinggi.

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•H - Hydrogen radical species
•O - Oxygen radical species
•OH - Hydroxyl radical species
-COOH - Carboxyl group
-C(=O)OH - Carboxyl functional group
-OH - Hydroxyl functional group
0-D - Zero-dimensional
1-D - One-dimensional
2-D - Two-dimensional
3-D - Three-dimensional
AC - Alternating current
AEAPS - 3-aminopropyltrimethoxysilane
Al - Aluminium
AlO_6 - Alumina octahedral
Al_2O_3 - Aluminium oxide
APP - Atmospheric pressure plasma
APPJ - Atmospheric pressure plasma jet
APPJs - Atmospheric pressure plasma jets
APTES - 3-aminopropyltrimethoxysilane
BE - Binding energy
BN - Boron nitride
BNNPs - Boron nitride nanoparticles
BNNSs - Boron nitride nanosheets
BS - Breakdown strength
C - Carbon atom
CAS - Chemical abstracts service
CCC - Current-carrying conductor
CH_2 - Methylene
CH_3 - Methyl group
DBD - Dielectric barrier discharge

DC - Direct current
DMAc - Dimethylacetamide
e^- - Free electron
e^* - High-energy species of the electron
ER - Epoxy resin
FESEM - Field emission scanning electron microscopy
FTIR - Fourier transform infrared spectroscopy
H - Hydrogen atom
HDPE - High density polyethylene
He - Helium atom
He^+ - Positive helium ion
He^* - High-energy species of helium atom
HF - High frequency
HMDS - Hexamethyldisilazane
HNO_3 - Nitric acid
HO_2^- - Hydroperoxyl functional group
H_2O - Water molecule
H_2SO_4 - Sulfuric acid
HV - High voltage
HVAC - High voltage alternating current
HVDC - High voltage direct current
IVAT - Institute of High Voltage and High Current
Kα - K-alpha
LDPE - Low-density polyethylene
LPP - Low-pressure plasma
LROP - Largest repeatedly occurring pulse
MARA - Majlis Amanah Rakyat
Mg - Magnesium
MJIIT - Malaysia-Japan International Institute of Technology
MMT - Montmorillonite
MOHE - Ministry of Higher Education
MW - Microwave
MWCNT - Multi-walled carbon nanotube

N_2	-	Nitrogen gas
N_2^+	-	Positive dinitrogen ion
N_2^*	-	High-energy species of nitrogen molecules
O	-	Oxygen atom
O_2	-	Oxygen gas
O^{2-}	-	Oxygen ion
O_2^{-2}	-	Peroxide functional group
O_2^{*-}	-	Superoxide anion
OH^-	-	Hydroxyl functional group
PD	-	Partial discharge
PDs	-	Partial discharges
PDEV	-	Partial discharge extinction voltage
PDIV	-	Partial discharge inception voltage
PEO	-	Polyethylene oxide
Ph.D.	-	Doctor of Philosophy
PJ	-	Plasma jet
PP	-	Polypropylene
PPMU	-	Pusat Pengurusan Makmal Universiti
PRPD	-	Phase-resolved partial discharge
Q-V	-	Charge-voltage
RF	-	Radio frequency
ROS	-	Radical oxygen species
Si	-	Silicon
Si^{1+}	-	Silicon substrate with one number of sub-oxide
Si^{2+}	-	Silicon substrate with two number of sub-oxides
Si^{3+}	-	Silicon substrate with three number of sub-oxides
Si^{4+}	-	Silicon substrate with four number of sub-oxides
SiO_2	-	Silicon dioxide
SiO_4	-	Silicate tetrahedral
SiR	-	Silicone rubber
SKE	-	Sekolah Kejuruteraan Elektrik
SWCNT	-	Single-walled carbon nanotube
TES	-	Triethoxyvinylsilane

TiO_2	-	Titanium dioxide
UATR	-	Universal attenuated total reflectance
UTM	-	Universiti Teknologi Malaysia
USA	-	United States of America
V-I	-	Voltage-Current
VLF	-	Very low frequency
VTMS	-	Vinyltrimethoxysilane
XLPE	-	Cross-linked polyethylene
XPS	-	X-ray photoelectron spectroscopy
ZnO	-	Zinc oxide
ZrO_2	-	Zirconium dioxide

LIST OF SYMBOLS

°	-	Degree
°C	-	Degree Celsius
$-\delta$	-	Negatively charged surfaces
γ	-	Gamma
τ	-	Transition time
μ	-	Micro
η	-	Efficiency
Ω	-	Ohm
+	-	Positive
-	-	Negative
%	-	Percent
Å	-	Angstrom
A	-	Ampere
atm	-	Standard atmosphere
$A_{Lissajous}$	-	Passive phase area of the Lissajous figure
α	-	Weibull’s scale parameter
β	-	Weibull’s shape parameter
cm	-	Centimetre
cm^3	-	Cubic centimetre
C	-	Capacitance
C_d	-	Dielectric capacitance
C_g	-	Gap capacitance
C_t	-	Total load capacitance
C_{total}	-	Total capacitance
dQ	-	Infinitesimal change in charges
dQ_h	-	Infinitesimal change in charges emitted for horizontal-component
dQ_v	-	Infinitesimal change in charges emitted for vertical-component
dV	-	Infinitesimal change in voltage

dV_h - Infinitesimal change in voltage applied for horizontal-component

dV_v - Infinitesimal change in voltage applied for vertical-component

eV - Electronvolt

exp - Exponential

E - Electric field

f - Frequency

F - Farad

g - Gram

G - Giga

Hz - Hertz

i - The progressive order of failure tests

I - Current

I_c - Capacitive current

I_{Dis} - Discharge current

I_{in} - Input current

I_r - Resistive current

k - The total number of tests

k - Kilo

K - Kelvin

L/min - Liter per minute

M - Mega

m - Metre

m^2 - Square metre

m^3 - Cubic metre

mA - Milliampere

mg - Milligram

mm - Millimetre

mm^2 - Square millimetre

mΩ - Milliohm

mbar - Millibar

ms - Millisecond

mW - Milliwatt

mol - Mole

nC - Nanocoulomb

nm - Nanometre

ns - Nanosecond

Pa - Pascal

pC - Picocoulomb

pF - Picofarad

P_{in} - Input power

P_{out} - Output power

$P(E)$ - The cumulative probability of failure according to the electric field applied

$P(E;\alpha,\beta)$ - The cumulative probability of failure according to the electric field applied which affected by the scale and shape parameters

Q - Charge

Q_h - Horizontal-component charge

Q_v - Vertical-component charge

Qmax - Maximum charge of partial discharge magnitude

R - Resistance

rpm - Revolution per minute

s - Second

S_a - Average surface roughness

$S(E)$ - The cumulative probability of survival according to the electric field applied

$S(E;\alpha,\beta)$ - The cumulative probability of survival according to the electric field applied which affected by the scale and shape parameters

t - Time

tons - Tonnes

V - Voltage

V_c - Voltage across capacitance

V_d - Voltage across the discharge gap

V_E - Interception voltage at the negative cycle

V_F - Interception voltage at the positive cycle

V_h - Horizontal-component voltage

V_{in} - Input voltage

V_r, V_R - Voltage across resistor

V_v	-	Vertical-component voltage
Vrms	-	Root mean square voltage
Vmax	-	Maximum voltage
W	-	Watt
wt%	-	Weight percentage

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CHAPTER 1

INTRODUCTION

1.1 Research Background

In the 1940s, organic insulating mediums based on polymeric materials were introduced intensively, especially in high and medium voltage applications due to the superior characteristics of polymers, such as outstanding mechanical, thermal, and electrical properties [1]. The maturity in the research trend of polymer-based electrical insulation has prompted the scientific exploration focusing on polymer nanocomposites for the last two decades. Polymer nanocomposite is a composition formed by dispersing nanometer-sized particles into a polymer matrix [2]. Polymer nanocomposites are encouraging formulations and an inspiring route for developing innovative polymeric-based insulating materials. Recently, multiple studies of polymer nanocomposites have been executed vigorously to obtain a high-quality insulating medium with superior performance. Insulation failure in an electrical system is the main factor that leads to power supply interruption. Thus, designing effective insulating materials is required to maximize insulation properties, subsequently minimizing power supply interruption. The series of polymer nanocomposites research revealed that incorporating nano-meter-sized fillers into the polymer matrix is a practical approach to improving the polymer's insulating properties.

In 1973, Nielsen *et al.* [3] revealed that dispersing the nanoparticles into the polymer matrix has improved the thermal conductivity of the polymer. However, the studies on polymer nanocomposites have only been carried out intensively in the past 20 years to improve the performance of polymer insulating materials. A particular focus lies in insulation characteristics such as prolonging the electrical treeing, partial discharge resistance, dielectric field strength, dielectric constant, dielectric losses, space charge, etc. Based on the collective study of polymer nanocomposites, the insulation properties can be improved significantly even by incorporating a small number of nanoparticles into the polymer matrix. The trend study of polymer nanocomposites was initiated by manipulating the materials used as nanofillers and polymer hosts. In 2004, Nelson *et al.* [4] studied partial discharge characteristics on epoxy resin nanocomposites filled with titanium dioxide, followed by numerous researchers that manipulated the type of nanofiller, polymer host, and the processing techniques used to prepare the composition. Most researchers have come to the same inference that polymer nanocomposites has better insulation properties than pure polymer.

Several types of base polymer are typically used in high and medium-voltage applications, such as silicon rubber, epoxy resin, low-density polyethylene, high-density polyethylene, and cross-linked polyethylene. Nowadays, cross-linked polyethylene has been used predominantly as the insulating material for the underground cable in the power delivery system [5]. It has been a selective material by cable manufacturers due to its outstanding characteristics, such as permitting high conductor operating temperature, reducing short circuits, and high dielectric field strength [6]. However, the insulating properties of XLPE can be further enhanced by introducing nanoparticles into the matrix. Among the insulating properties promoted to be improved are partial discharge and dielectric field strength [7]. Partial discharge is a pre-breakdown phenomenon that indicates the electrical discharge that partially bridges the dielectric medium between two conduction parts. Meanwhile, the dielectric breakdown strength represents the maximum electric field stress that the insulator can withstand. In some studies, the collective review showed that XLPE nanocomposites have better insulation properties, as pointed out by Tanaka *et al.* [8], where the partial discharge resistance and dielectric field strength of XLPE nanocomposites filled with silicon dioxide showed significant improvement over pure XLPE.

Conventionally, the preparation of XLPE nanocomposites typically utilizes the direct mixing technique without concerning the effective dispersion of nanoparticles within the polymer matrix. The study trend involves XLPE nanocomposites has been conducted aggressively in the last few years by manipulating the types of nanoparticles, processing technique, and electrical test measurement. Widely, oxide-based nanoparticles have drawn the attention of researchers to be added to the XLPE matrix due to their prominent role in enhancing the insulation properties of XLPE. Among the oxide-based nanoparticles, silicon dioxide is a selective compound to be dispersed into polymer matrix due to its comprehensive characteristics such as outstanding hardness, solid and stable molecular structure with a gigantic covalent bond [9]. Besides, silicon dioxide also has a great potential to be utilized as nanoparticles in producing large-scale polymer nanocomposites since it is commonly found in nature as quartz and the cell walls of diatoms [10]. Therefore, this study is focused on the insulation characteristics such as partial discharge resistance and dielectric field strength of the composition based on XLPE and SiO_2 as a base polymer and nanofiller, respectively.

Incorporating nanometer-sized silicon dioxide into the polymer base material prominently improved the partial discharge resistance and dielectric field strength by increasing the contact surface area of the composition [11], which possibly creates a sort of hindrance to the accumulation of space charge due to the elimination of the local electric field distortion [12]. In obtaining effective polymer nanocomposites, the finding factors are directed to the several characteristics of the composition, such as huge interaction zone, proximity, and ample spaces for the trapping of charge carriers, in realizing the mechanism of nanoparticles to inhibit PD activities by preventing and reducing the drift and movement of charge carriers, respectively [13]. However, some of the studies claimed that the ineffective dispersion of nanoparticles into the XLPE matrix had been the crucial issue that limits the insulation performances of XLPE nanocomposites [14]. Therefore, the study on the surface interaction of polymer nanocomposites has been executed to determine the factors which affect the polymer-nanofiller interfacing region.

Through the rigid findings, the agglomeration of nanoparticles within the polymer matrix is the main factor that restricts the enhancement of insulation characteristics because the non-uniform dispersion of nanoparticles consequently reduces the interfacial area and creates weak polymer-nanofiller interfacial bonds [15]. The previous studies revealed that different chemical characteristics between nanofiller and base polymer also being the factor that causes the formation of the poor polymer-nanofiller interfacial region. For instance, a base polymer is generally an organic compound, whereas a nanofiller like silica is inorganic. Previous studies reported that uniform dispersion of nanoparticles within a polymer matrix could be achieved by treating the surface of nanoparticles for the composition of organic-inorganic polymer nanocomposites [16].

Conventionally, several methods have been introduced to modify the surface of nanoparticles in enhancing the compatibility between the nanoparticles and the matrix of the base polymer. These approaches are typically known as the surface modification or surface treatment of nanoparticles. The most favourable technique among the researchers is the chemical surface functionalization method. This method is preferred because the outcomes are impressive and promise the excellent potential to functionalize a specific chemical functional group on the surface of nanoparticles such as carbonyl, carboxyl, amines, hydroxyl, hydroperoxyl, and peroxide functional groups.

The effectiveness of the chemical surface functionalization method also seems able to improve the uniformity dispersion of nanoparticles within the polymer matrix, resulting in overcoming the agglomeration issue. However, there have certain limitations regarding this method. This method uses chemical agents and/or solvents such as dimethylacetamide and propyltrimethoxysilane for coupling, swelling, and exfoliating purposes. Therefore, this method is unsuitable in terms of eco-environmentally [17]. In addition, the process of chemical surface functionalization is also complex and challenging to implement since this method also involves a few other processes, such as exfoliating, swelling, and doping [18]. Thus, due to the complexity, the chemical surface functionalization method is unsuitable to be chosen as a surface modification technique for the nanoparticles.

Furthermore, heat treatment, also known as the calcination technique, is another method used to treat the surface of nanoparticles. However, the effectiveness of this method in overcoming the agglomeration issue is still being explored among researchers. Thus, this technique seems unsatisfactory in enhancing the insulation characteristics of polymer nanocomposites, such as partial discharge resistance, dielectric field strength, and dielectric constant. In addition, this method is also difficult to be applied because complex and controllable systems are required to control the amount of heat exposure to the surface of nanoparticles.

As mentioned, the performance of XLPE nanocomposites still does not achieve a satisfactory level due to the factor of the incompatible surface structure of nanoparticles and XLPE matrix, which led the nanoparticles to agglomerate, resulting in restricting the enhancement of insulation characteristics [14], [15], [19]. Besides, the conventional techniques used to improve the surface compatibility of nanoparticles and polymer matrix, such as chemical surface functionalization and heat treatment, appear to be less effective with various drawbacks.

Thus, plasma treatment is an alternative method introduced to provide a modification mechanism on the surface of nanoparticles, promising an improvement in the dispersion of nanoparticles within the polymer matrix [17]. Hence, the insulation characteristics of XLPE nanocomposites, such as partial discharge resistance and dielectric field strength, are expected to be enhanced by incorporating plasma-treated nanosilica into the XLPE matrix. Since this method is still being explored at an early stage, the effectiveness of this method and the specific design of plasma treatment systems in treating nanoparticles have not been formally reported in the previous study. In 1993, Friedrich *et al.* [20] pointed out that plasma treatment is an effective method to maximize the adhesion of chemical bonds on the surface of polymer substrates. However, no additives or fillers were included in the study. Hence, no significant justification was made regarding the polymer incorporated with plasma-treated fillers.

In the next few years, Shi *et al.* [21] experimented that coating nanoalumina's surfaces with ultra-thin pyrrole films using the plasma polymerization technique. The study found that coating the surface of nanoalumina using plasma polymerization technique could effectively produce energetic stable polymorph. However, the study did not include the insulation characteristics of the polymer nanocomposites. In 2013, Yan *et al.* [22] conducted intensive research on polymer nanocomposites modification using plasma polymerization. The study revealed that plasma polymerization is a promising technique for enhancing the insulation characteristics of the polymer nanocomposites, such as partial discharge resistance and dielectric field strength. The plasma polymerization method has been introduced for the same purpose as other surface modification techniques: to improve the compatibility between polymer-nanofiller interfaces by coating the thermoplastic material on nanofiller surfaces.

Plasma polymerization is the preferable technique to replace the chemical surface functionalization method in overcoming the agglomeration issue because the surface modification mechanism becomes more effective with the ionization process provided by the plasma discharge. The main idea of how this technique works is that the coating agent was coated on nanofiller surfaces using plasma discharge. The plasma discharge will ionize the background gas molecules and produce a bombardment of electrons, forming electronegative charges on the nanofiller surfaces. This will excite and form new covalent bonds between the nanofillers and coating agents, strengthening the molecular structure between nanofiller-coating agents [22]. The coated nanofillers could have better surface compatibility with the base polymer because they have almost similar surface functional groups that match and self-encouragement merged.

However, the plasma polymerization technique also has several drawbacks that deny the applicable potential. Even though this technique could obtain high effectiveness of polymer nanocomposites with the excellent agreement of insulation properties, the chemical agent is still required to be used in the process of polymerization. For instance, Liu *et al.* [23] used a chemical solution to synthesize the coated nanofillers into the polymer matrix. Besides, some of the coating agents themselves are harmful solvents, which may cause the coated nanofillers to become toxic. In addition, plasma polymerization is a complex technique because it simultaneously requires complicated plasma and chemical system handling. Furthermore, the by-product produced through plasma polymerization is also thought to contain poison and is excessively activated in unforeseen circumstances.

Two classes of plasma have been employed in the previous study to modify the surface morphology of nanofillers operated under low-pressure and atmospheric pressure conditions. Low-pressure plasma is a good discharge process in treating the surface of nanofillers with the high capability to prevent the nanofillers from being contaminated with undesired molecules. However, the treatment chamber of low-pressure plasma is complex due to its operating condition typically being lower than atmospheric pressure. Thus, the low-pressure plasma treatment must be operated inside a closed vacuum system. The complexity and difficulty of controlling the vacuum system were the reasons why low-pressure plasma is not preferred in this study. In addition, low-pressure plasma is also inapplicable to be implemented at the industrial level because it requires a high cost for the vacuum and controlling system [24].

Previous researchers also intensively used atmospheric pressure plasma because it is easier to develop and operate under atmospheric pressure at ambient temperature. Since APP operates under an atmospheric pressure condition, thus vacuum system is not required for this category of plasma treatment. Therefore, an effective APP treatment system will be designed, developed, and characterized in this research by concerning optimum input parameters such as voltage supply, operating frequency, and flow rate of discharge gas. Besides, the effectiveness of APP treatment in improving the insulation characteristics of XLPE nanocomposites is also required to be observed and justified in terms of partial discharge resistance and dielectric field strength. In addition, the optimum configuration of weight percentage and duration of plasma treatment of the nanosilica also be identified in this study. The material characterization is also included in this study to distinguish the physical and chemical structure of plasma-treated nanosilica and XLPE nanocomposites which is related to the enhancement of partial discharge resistance and dielectric field strength.

1.2 Problem Statements

In specific, insulation characteristics can be further enhanced by resolving the agglomeration issue to acquire a compatible surface structure between the nanofiller and base polymer using the surface modification technique. Conventionally, the surface modification technique used is the chemical surface functionalization method and heat treatment. However, these methods are not suggested to be implemented, especially at the manufacturing level, due to their complexity. Therefore, plasma treatment is an effective technique over the conventional methods with a straightforward surface modification process on the nanoparticles in resolving the agglomeration issue by enhancing the compatibility between the nanoparticles and base polymer [11], [17], [25]-[32]. In the last few years, Yan *et al.* [25]-[27], Musa *et al.* [17], [28], and Awang *et al.* [11] have utilized atmospheric pressure plasma treatment techniques to modify the surface of silicon dioxide nanofiller in achieving uniform dispersion of the nanofiller within the matrix of epoxy resin, silicon rubber, and low-density polyethylene, respectively. However, their studies are limited to characterizing certain insulation parameters on the specific polymer base materials filled with silicon dioxide nanoparticles. Besides, these studies are only limited to plasma treatments with the filamentary mechanism of the non-uniform discharge process. Moreover, these studies also do not focus on developing a specific APP treatment system. According to the literature review, no investigation formally reported the characteristics of partial discharge and dielectric field strength of the XLPE nanocomposites added with silicon dioxide nanoparticle that has been modified using the glow mechanism of APP treatment. Therefore, the collective limitations in their studies have become the gap that will be gratified in this study by conducting the effectiveness of glow mechanism plasma treatment with a uniform discharge process in enhancing the partial discharge resistance and dielectric field strength of XLPE nanocomposites. Supplementary, this study also includes developing an APP treatment system with a glow discharge mechanism to be explicitly used in modifying the surface of nanoparticles by considering the input parameters in plasma production.

1.3 Research Objectives

The objectives of this research are drawn as listed:

(a) To characterize the optimum input parameters in producing atmospheric pressure plasma discharge to modify the surface of silicon dioxide nanoparticles.

(b) To characterize the morphology of the nanoparticles affected by plasma treatment.

(c) To analyze the effective formulation of XLPE nanocomposites in improving partial discharge resistance and dielectric field strength characteristics by distinguishing the composition added with untreated and plasma-treated silicon dioxide nanoparticles.

1.4 Research Scopes

This research has been conducted according to the following scope:

(a) The development of an atmospheric pressure plasma treatment system aims to produce glow plasma with a uniform discharge mechanism. The mechanism of plasma discharge highly relies on the input parameters such as supply voltage, operating frequency, and flow rate of discharge gas which are comprehensively considered in this study. Thus, this study was focused on optimizing the input parameters to produce homogenous plasma discharge by characterizing the discharge mechanism according to discharge current waveform and Lissajous figure analysis.

(b) The uniform discharge of APP treatment was conducted on the surface of silicon dioxide nanoparticles under the different treatment durations of 1, 3, and 5 minutes. The treatment time was varied to determine the effect of treatment duration on the insulation characteristics of XLPE nanocomposites. The chemical analyses on the morphology of the untreated and plasma-treated silicon dioxide nanoparticles using x-ray photoelectron spectroscopy (XPS), Fourier transforms infrared (FTIR) spectroscopy, and field emission scanning electron microscopy (FESEM) were included in this study.

(c) The formulation of polymer nanocomposites is focused on the XLPE as the material of the polymer host with the addition of 1 wt%, 3 wt%, and 5 wt% of fillers which are the untreated and plasma-treated silicon dioxide nanoparticles.

(d) The experimental investigations were carried out to identify the effective formulation of XLPE nanocomposites according to the characteristics of partial discharge resistance and dielectric field strength measurements. Concurrently, the effectiveness of APP treatment in enhancing these insulation characteristics of the XLPE nanocomposites is also involved in this study.

(e) The composition of XLPE with the untreated and plasma-treated silicon dioxide nanoparticles was characterized by using FTIR and FESEM to analyze the chemical composition and uniformity of the filler dispersion within the XLPE matrix, respectively.

1.5 Research Limitation

The following were limitations of this study:

(a) In this study, the weight percentage of fillers was only limited to 1 wt%, 3wt%, and 5wt% of silicon dioxide nanoparticles. According to the literature, the insulation characteristics of polymer nanocomposites typically have the potential to be enhanced at these amounts of nanofillers [8], [33].

(b) This exploration only focussed on a single type of nanoparticle, silicon dioxide, due to its outstanding properties such as high electrical resistance and strong molecular bond. Besides, this nanoparticle is also frequently reported in previous research as a comprehensive type of filler to enhance the insulation characteristics of the polymer.

(c) The development of an APP treatment system with a glow discharge mechanism is only limited to the effects of input parameters to acquire uniform plasma discharge in treating the surface of nanoparticles.

(d) Besides, this study is only limited to identifying APP treatment’s effectiveness in enhancing the insulation characteristics of XLPE nanocomposites according to the partial discharge resistance and dielectric field strength.

1.6 Research Contribution

The contributions of this research are as listed:

(a) Atmospheric pressure plasma treatment with a uniform discharge has drawn an encouraging improvement in the insulation characteristics of XLPE nanocomposites. This has been achieved by overcoming the agglomeration of silicon dioxide nanoparticles within the XLPE matrix. Homogenous dispersion of silicon dioxide nanoparticles within the XLPE matrix has been acquired by functionalizing the hydroxyl, hydroperoxyl, and peroxide functional groups on the surface of nanoparticles using the APP treatment technique. For the first time, this research work offers improvement of insulation characteristics of XLPE nanocomposite such as partial discharge resistance and dielectric field strength by enhancing the compatibility between the silicon dioxide nanoparticles and the XLPE matrix using APP treatment technique with glow discharge mechanism.

(b) The investigation of effective XLPE nanocomposites formulation based on the weight percentage and plasma treatment time of silicon dioxide nanoparticles also made this research work significant and highly impacted in terms of finding and information. In particular, the evaluation of effective XLPE nanocomposites formulation is made according to the insulation characteristics mentioned.

(c) This study found that the addition of plasma-treated silicon dioxide nanoparticles into the XLPE matrix has increased the partial discharge resistance and dielectric field strength. The enhancement of insulation characteristics of XLPE nanocomposites based on the uniform APP treatment on the surface of silicon dioxide nanoparticles indicates the contribution of this study due to the limited publication has been reported, such as the study mentioned. Thus, this configuration of exploration is the main research contribution.

(d) Besides, the optimization of input parameters in producing glow-based surface modification through APP treatment has also been explored. This study found that homogeneous plasma discharge consists of a single pulse discharge current in each cycle of supply voltage with a constant value of discharge capacitance.

(e) In addition, the improvement of insulation characteristics achieved in this study by using homogeneous plasma discharge treatment is also in line with the nanomaterial evolution as a comprehensive initiative in designing a new insulating material, thereby can be utilized in high voltage equipment with a longer lifetime at once has potential to reduce the maintenance costs.

1.7 Thesis Outline

The thesis is organized as follows:

Chapter 2 consists of a review of the chemical and physical structure of polymer nanocomposites. Besides, the surface modification of nanofillers, partial discharge phenomenon, and dielectric field strength of polymer nanocomposites with its classification and influencing factors are also described in this chapter. In particular, the characteristics of partial discharge resistance and dielectric field strength of polymer nanocomposites from the previous studies were also reviewed accordingly. This chapter also includes a review of the limitations of conventional surface modification methods in enhancing the insulation characteristics of polymer nanocomposites. Besides, this review also focused on the previous studies on surface modification using the atmospheric pressure plasma technique.

Chapter 3 presents the research methodology that explains the experimental setup, procedures, and methods of data collection executed in this study. The experimental setup of atmospheric pressure plasma treatment on the silicon dioxide nanoparticles has been described. The descriptions of the samples to be tested and the types of tests conducted have also been presented. It details the measurement procedure of the partial discharge and dielectric field strength measurement. In addition, the procedure of morphological analysis of each sample is also elaborated in this chapter.

Chapter 4 discusses and analyses the characteristics of a plasma discharge to optimize the input parameters in producing a homogeneous plasma discharge according to the discharge current and Lissajous figure analysis. The characteristics of atmospheric pressure plasma used to modify nanoparticles' surfaces, such as the discharge capacitance, discharge power, and discharge efficiency, are also elaborated in this chapter. Furthermore, morphological analyses of the untreated and plasma-treated silicon dioxide nanoparticles based on the results from XPS, FTIR, and FESEM have been discussed thoroughly. This chapter also explains the insulation characteristics of XLPE nanocomposites incorporated with the untreated and plasma-treated silicon dioxide nanoparticles in terms of partial discharge resistance and dielectric field strength. Moreover, the morphological analysis by using FTIR and FESEM on the samples of XLPE nanocomposites has also been discussed thoroughly in this chapter. The mechanism of surface modification on the nanoparticles using APP treatment to enhance the insulation characteristics of XLPE nanocomposites is also described accordingly.

Chapter 5 concludes and summarizes the findings acquired in this study. This chapter provides some recommendations related to this work. This chapter also provides suggestions for future studies related to this region.

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LIST OF PUBLICATIONS

Journal Paper

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2. **N. M. Saman**, M. H. Ahmad, and Z. Buntat, "Experimental Analysis of Cold Plasma with Glow Discharge Mechanism Under a Variety of Input Parameters," *IEEE Transactions on Plasma Science*, vol. 50, no. 7, pp. 2110–2125, Jul. 2022. (Q3, IF: 1.222)
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