



**UNIVERSITI PUTRA MALAYSIA**

***PREPARATION AND CHARACTERIZATION OF ANTI-CORROSIVE  
COATINGS BASED ON SILANE FUNCTIONALIZED GRAPHENE OXIDE/  
EPOXY RESIN NANOCOMPOSITES***

**AUJARA KABIRU MUSA**

**FS 2022 23**



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**By**

**AUJARA KABIRU MUSA**

**Thesis Submitted to the school of Graduate Studies, Universiti Putra Malaysia, in  
Fulfillment of the Requirements for the degree of Doctor of Philosophy**

**December 2020**

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## DEDICATION

I hereby dedicate this thesis to the Almighty Allah, who has been my help, sustainer, provider, guide, encouragement, keeper, and my all in all throughout the course of my studies and also to my parents (Alhaji Musa Ibrahim and Hajiya Fatima Bintu Usman), My wife Fauziyya Musa and my beloved and beautiful daughters (Rabi'at, Fatima, Amina, and Nana Aisha) whose prayers and support have kept me going. Finally, to my brothers and sisters who are always by my side and ready to render any kind of support and assistance



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Doctor of Philosophy

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**December 2020**

**Chairman : Associate Professor Nor Azowa binti Ibrahim, PhD**  
**Faculty : Science**

Epoxy resins are generally used to protect metal substrates, however, there is a need for improvement of anti-rust performance and mechanical properties. The addition of nano-sized fillers such as graphene to produce nanocomposites can overcome the shortage of polymeric materials and has remarkable mechanical, electrical, and gas barrier properties. In this study, graphene oxide and functional-GO were incorporated into epoxy resins to provide a protective layer of the metal substrate. Functional-GO is synthesized using environmentally friendly gamma irradiation techniques, which are a simple and clean alternative approach to alter the structural and physicochemical properties of graphene oxide (GO). Graphene oxide obtained by Hummer method was modified by incorporating 3-Aminopropyltriethoxysilane (APTES) and 3-Glycidyloxypropyltrimethoxy silane (GPTMS) to its surface by radiation from gamma ray. The nature of GO and functional-GO is characterized by various techniques such as Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction spectroscopy (XRD), field release scanning electron microscope (FESEM), Raman spectroscopy, and thermogravimetric analysis (TGA). The FT-IR spectrum reveals chemical interactions between the cross section which shows significant weakening of the -OH, COOH and C-O-C with the appearance of chemical bonds due to the withdrawal of oxygen functional groups on the GO surface where crystal surface changes and surface defects due to modification are determined by XRD which shows the gradual weakening with simultaneous disappearance of graphite peak as the oxidation process proceeds with the corresponding appearance of diffraction peak at about  $2\theta=9.8$  leading to an increased in the interlayer spacing from 0.34nm to 0.90nm. Similarly, the Raman spectroscopy indicate an increase in  $I_D/I_G$  from 0.90 for GO to 1.21 and 1.18 for AGO-150 and GGO-150 respectively. The TGA thermograms showed peaks at various temperature regions (i.e. 30-120°C, 120-300°C and 300-650°C) which can be attributed to the degradation oxygen functional groups and chemically bonded silane on the GO surface. Within these temperature regions the AGO-150 exhibits highest thermal stability with the lowest water evaporation (5.5%), lowest decomposition of unreacted silanes (9.36%) and thermal-oxidative decomposition of grafted silane (32.17%). Preparation of the

protective material of the steel substrate begins by ultrasonically dispersing the 1 wt.% GO in the solvent before mixing it by shearing it into an epoxy matrix and adding a hardener. XRD showed the existence of GO morphology, functional-GO intercalation and exfoliation throughout the matrix shown by the existence of broad diffraction peak of epoxy between  $2\theta=5-28^\circ$  centered at  $2\theta=18^\circ$  indicating the dispersion of functionalized-GO in the matrix. SEM on the sample surface layer determined during the tensile test showed microscopic and homogeneous functional-GO dispersion in the matrix. Thermogravimetric analysis (TGA) through the analysis of some thermodynamic parameters such as  $T_{on}$ ,  $W_{350^\circ C}$ ,  $T_{max}$  and  $T_{500^\circ C}$  all revealed improved thermal stabilities of nanocomposites coatings fabricated with functionalized-GO. The dynamic mechanical analysis (DMA) was used to investigate the thermomechanical properties of nanocomposite coatings. Thermodynamic parameters such  $E'$  and  $T_g$  of the nanocomposites coatings showed a significant increase in storage modulus and a gradual increase in glass transition temperature with the dispersion of functionalized-GO, where EAG-150 exhibits highest  $E'$  of 3414.90 MPa and  $T_g$  of  $90.49^\circ C$ .

The physical properties of coatings such as adhesion, hardness, flexibility, and chemical properties are also assessed. Corrosion resistance of nanocomposite layers in NaCl solution (3.5% by mass) was also assessed using Open Circuit Potential (OCP), potentiodynamic polarization, and Electrochemical Impedance Spectroscopy (EIS). The results showed that the functional nanocomposite layer-GO is capable to block the penetration of electrolytes between the metal surface and the coating. This study shows that Functional-GO /epoxy nanocomposites provide better corrosion protection and can act as an excellent corrosion barrier on lightweight steel substrates due to improvement of some electrochemical properties such as shifting of  $E_{corr}$  to more positive value i.e., from  $-0.853554V$  for EP to  $-0.18488$  for EAG-150 and decreasing of  $I_{corr}$  value from  $8.349 \times 10^{-7}$  for EP to  $5.281 \times 10^{-8}$  for EAG-150. This improvement in corrosion protection behavior can be attributed to the GO two-dimensional (2D) structure and outstanding performance, which can hinder the penetration of corrosive media to a certain extent, thereby enhancing the anti-corrosion properties of the coatings.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

## **PENYEDIAAN DAN PENCIRIAN LAPISAN ANTI KARAT BERASASKAN GRAPHEN BERFUNGSIONAL SILAN/EPOKSI NANOKOMPOSIT**

Oleh

**AUJARA KABIRU MUSA**

**Disember 2020**

**Pengerusi : Profesor Madya Nor Azowa binti Ibrahim, PhD**  
**Fakulti : Sains**

Resin epoksi secara amnya digunakan untuk melindungi substrat logam, namun, terdapat keperluan untuk peningkatan prestasi anti karat dan sifat mekanikalnya. Tambahan pengisi bersaiz nano seperti grafin untuk menghasilkan nanokomposit dapat mengatasi kekurangan bahan polimer dan mempunyai sifat mekanikal, elektrik, dan gas yang utuh. Pada kajian ini, grafin oksida dan fungsional-GO dimasukkan ke dalam resin epoksi bagi menyediakan lapisan pelindung substrat logam. Fungsional-GO disintesis dengan menggunakan teknik penyinaran gamma yang mesra alam, dimana merupakan pendekatan alternatif mudah dan bersih untuk mengubah sifat struktur dan fizikokimia grafin oksida (GO). Grafin oksida yang diperolehi melalui kaedah Hummer telah diubahsuai dengan mencantumkan 3-Aminopropiltriethoksi silana (APTES) dan 3-glisidiloksipropiltrimetoksi silana (GPTMS) ke atas permukaan grafin oksida menggunakan radiasi dari penyinaran sinar gamma. Sifat GO dan fungsional-GO dicirikan melalui pelbagai teknik seperti spektroskopi inframerah transformasi Fourier (FT-IR), spektroskopi difraksi sinar-X (XRD), mikroskop elektron pengimbasan pelepasan medan (FESEM), spektroskopi Raman, dan analisis termogravimetrik (TGA). Spektrum FT-IR mendedahkan interaksi kimia antara keratan rentas yang menunjukkan kelemahan ketara -OH, COOH dan C-O-C dengan kemunculan ikatan kimia kerana tarikan kumpulan fungsional oksigen pada permukaan GO yang membawa perubahan permukaan kristal dan kerosakan permukaan disebabkan untuk pengubahsuaian ditentukan oleh XRD yang menunjukkan kelemahan secara beransur-ansur dengan hilangnya puncak grafit secara serentak ketika proses pengoksidaan diteruskan dengan penampilan puncak difraksi yang sepadan pada sekitar  $2\theta = 9,8$  yang menyebabkan peningkatan jarak antara lapisan dari 0.34nm kepada 0.90nm. Seterusnya, spektroskopi Raman juga menunjukkan peningkatan  $I_D/I_G$  dari 0.90 untuk GO ke 1.21 dan 1.18 untuk AGO-150 dan GGO-150g. Termogram TGA menunjukkan puncak julat bagi suhu (30-120 °C, 120-300 °C dan 300-650 °C) yang dikaitkan dengan degradasi kumpulan fungsi oksigen dan ikatan kimia silan pada permukaan GO. Pada kawasan suhu ini, AGO-150 menunjukkan kestabilan terma tertinggi dengan penyejatan air terendah (5.5%), penguraian terendah silan yang tidak berinteraksi (9.36%) dan penguraian terma-oksidatif silan cantuman (32.17%). Penyediaan bahan pelindung substrat keluli

dimulakan dengan penyebaran ultrasonik GO seberat 1% ke dalam pelarut sebelum mencampurkannya dengan mengkikis ke dalam matriks epoksi dan menambahkan bahan pengeras. XRD menunjukkan adanya morfologi GO dan interkalasi dan pengelupasan GO-fungsional di seluruh matriks yang ditunjukkan oleh adanya puncak difraksi epoksi luas antara  $2\theta = 5-28^\circ$  berpusat pada  $2\theta = 18^\circ$  menunjukkan penyebaran fungsional-GO dalam matriks. SEM pada lapisan permukaan sampel yang ditentukan semasa ujian tegangan menunjukkan penyebaran fungsional-GO mikroskopik dan homogen dalam matriks. Analisis termogravimetri (TGA) melalui analisis beberapa parameter termodinamik seperti  $T_{on}$ ,  $W_{350^\circ C}$ ,  $T_{max}$  dan  $T_{500^\circ C}$  semuanya menunjukkan peningkatan kestabilan terma lapisan nanokomposit yang dibuat dengan fungsional-GO. Analisis mekanikal dinamik (DMA) digunakan untuk menyiasat sifat termomekanik pelapis nanokomposit. Parameter termodinamik seperti pelapisan nanokomposit  $E'$  dan  $T_g$  menunjukkan peningkatan modulus penyimpanan yang ketara dan peningkatan suhu peralihan kaca secara beransur-ansur dengan penyebaran fungsional-GO, di mana EAG-150 menunjukkan  $E'$  tertinggi 3414.90 MPa dan  $T_g$  90.49 °C.

Sifat fizikal lapisan seperti kelekatan, kekerasan, kelenturan, dan sifat kimia juga dinilai. Ketahanan kakisan lapisan nanokomposit dalam larutan NaCl (3.5% jisim) juga dinilai menggunakan Potensi Litar Terbuka (OCP), polarisasi potensiodinamik, dan Spektroskopi Impedansi Elektrokimia (EIS). Hasil kajian menunjukkan bahawa lapisan nanokomposit berfungsi-GO mampu menyekat penembusan elektrolit antara permukaan logam dan lapisan. Kajian ini menunjukkan bahawa nanokomposit fungsional-GO/epoksi memberikan perlindungan hakisan yang lebih baik dan dapat bertindak sebagai penghalang hakisan yang sangat baik pada substrat keluli ringan kerana peningkatan beberapa sifat elektrokimia seperti peralihan  $E_{corr}$  ke nilai yang lebih positif iaitu dari -0.853554V untuk EP ke -0.18488 untuk EAG-150 dan penurunan nilai  $I_{corr}$  dari  $8.349 \times 10^{-7}$  untuk EP kepada  $5.281 \times 10^{-8}$  untuk EAG-150. Peningkatan tingkah laku perlindungan hakisan ini dapat dikaitkan dengan struktur dua dimensi (2D) GO dan prestasi luar biasa, yang dapat menghalang penembusan bahan hakisan kepada tahap tertentu, sehingga meningkatkan sifat anti-karat permukaan.



## ACKNOWLEDGEMENTS

In the name of Allah, the most gracious and the most merciful who thought man by pen. All thanks to Allah, who has allowed me to see this day, the day I have been dreaming of, may the peace and blessings of Allah be upon his noble prophet Muhammad (SAW).

First of all, I would like to use this opportunity to express my heartfelt thankfulness to my supervisor, Assoc. Prof. Dr. Nor Azowa Bint Ibrahim, for her continues advice, support, guidance, and care for the successful conduct of this research work despite multiple challenges. I appreciate your immense contribution, and I pray that may Allah continue to shower blessing on your path. I would also like to express my gratitude to my co-supervisors, Dr. Norhazlin Zainuddin, Dr. Siti Mariam Mohd Nor, and Chantara Thevy Ratnam, for their great contribution, precious suggestions, and critical assessment toward smooth conduct of this research. May God continue to bless them, and may He light all their ways of achievements. I will also like to acknowledge my sincere gratitude to the Malaysian Nuclear Agency (Nuklear Malaysia) for providing resources, especially radiation instrument for this study. A similar gesture goes to Dr. Chieng Buong Woei of the Polymer research laboratory and Dr. Teo Ming Ting (Nuclear Malaysia) for their guidance for the smooth conduct of the research. I will also wish to express my appreciation to all my colleagues and lab-mates in the polymer research laboratory, Isyaku Saleh, Ahmed Chalabi, for their help, contributions, support, and understanding they rendered during the conduct of this research. More so, to all lecturers and members of staff in Faculty Sciences for their support in making this research a successful one. Words cannot express my gratitude to my wife, Fauziyya Musa and my daughters Rabi'at, Fatima, Amina, and Nana Aisha for their support and encouragement towards the success of this research. They are always beside me during the happy and hard moments to motivate me. I would also like to extend my sincere gratitude to my father, Alhaji Musa Ibrahim and Hajiya Fatima Bintu Usman, for their prayers, love, care, support, and generosity. May Allah grant them all his best in Jannatul Firdaus. My gratitude also goes to the entire members of my family for their encouragement, advice, and support as well. May Allah bless them all.

My appreciation also goes to Tertiary Education Trust Fund (Tetfund) via Jigawa State Polytechnic, Dutse, Jigawa State.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirements for the award of Doctor of Philosophy degree. The members of the Supervisory Committee were as follows:

**Nor Azowa binti Ibrahim, PhD**

Associate Professor  
Faculty of Sciences  
Universiti Putra Malaysia  
(Chairperson)

**Norhazlin binti Zainuddin, PhD**

Senior Lecturer  
Faculty of Sciences  
Universiti Putra Malaysia  
(Member)

**Siti Mariam binti Mohd Nor, PhD**

Senior Lecturer  
Faculty of Sciences,  
Universiti Putra Malaysia  
(Member)

**Chantara They Ratnam, PhD**

Senior Research Scientist  
Malaysian Nuclear Agency  
Bangi, Malaysia  
(Member)

---

**ZALILAH MOHD SHARIFF, PhD**

Professor and Dean  
School of Graduate Studies  
Universiti Putra Malaysia

Date: 21 July 2022

## Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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Signature: \_\_\_\_\_  
Name of  
Chairman of  
Supervisory  
Committee: Associate Professor Dr. Nor Azowa binti Ibrahim

Signature: \_\_\_\_\_  
Name of  
Member of  
Supervisory  
Committee: Dr. Norhazlin binti Zainuddin

Signature: \_\_\_\_\_  
Name of  
Member of  
Supervisory  
Committee: Dr. Siti Mariam binti Mohd Nor

Signature: \_\_\_\_\_  
Name of  
Member of  
Supervisory  
Committee: Dr. Chantara Theyv Ratnam

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## LIST OF ABBREVIATIONS

0D	0 Dimensional
$^{135}\text{Cs}$	Cesium-137
1D	1 dimensional
2D	2 dimensional
3D	3 dimensional
$^{60}\text{Co}$	Cobolt-60
APTES	3-Aminopropyltriethoxysilane
ASTM	American Society for Testing and Materials
BISE	1,3-bis[3-(4,5-epoxy-1,2,3,6-tetrahydrophthalimido)propyl] tetramethyldisiloxane
BPA	Bisphenol A
CAE	Cycloaliphatic epoxy
CNT	Carbon nanotube
CR	Corrosion rate
CVD	Chemical Vapor Deposition
DETA	Diethylenetriamine
DGEBPA	Diglycidyl Ether of Bisphenol-A
DMA	Dynamic Mechanical Analysis
DMF	Dimethylformamide
DTA	Deferential Thermal Analysis
DWCNT	Double-walled carbon nanotube
EAG	APTES functionalized-GO/Epoxy coatings
ECH	Epichlorohydrin
$E_{corr}$	Corrosion potential

EG	Expanded graphite
EGG	GPTMS functionalized-GO/Epoxy coatings
EGO	GO/Epoxy coatings
eV	Electron volts
EVA	Ethylene-vinyl acetate
FESEM	Field Emission Scanning Electron Microscopy
f-GO	Functionalized-GO
FRA	Frequency Response Analysis
FT-IR	Fourier-transform infrared spectroscopy
GDP	Gross Domestic Product
GGO	GPTMS functionalized-GO
GNP	Graphene Nano Platelets
GO	Graphene Oxide
GPTMS	3-glycidylloxypropyltrimethoxy silane
Gy	Gray
H <sub>12</sub> MDI	4,4'-dicyclohexylmethane diisocyanates
HDI	Hexamethylene diisocyanates
HHPA	Hexahydrophthalic anhydride
<i>I<sub>corr</sub></i>	Corrosion current density
IPDI	Isophorone diisocyanates
ISO	International Organisation for Standardization
KBr	Potassium Bromide
KGy	Kilo Gray
LDH	Layered Double Hydroxide

MEK	Methyl Ethyl Ketone
MHHPA	Methyl hexahydrophthalic anhydride
MMT	Montmorillonite
IMPACT	International Measures and Prevention, Application and Economics of Corrosion Technologies
MTHPA	Methyl Tetrahydrophthalic Anhydride
MWCNT	Multi-walled carbon nanotube
NACE	National Association of Corrosion Engineers
OCP	Open Circuit Potential
PA	Phthalic Anhydride
PDI	Pentamethylene diisocyanates
PDP	potentiodynamic polarization
PE-g-MA	polyethylene-grafted maleic anhydride
PMMA	polymethylmethacrylate
PNC	Polymer Nanocomposites
PS	Polystyrene
PVP	polyvinylpyrrolidone
rGO	Reduced Graphene Oxide
RM	Ringgit
$R_p$	polarization resistance
RT	Room Temperature
SCE	Saturated Calomel Electrode
SEM	Scanning Electron Microscopy
SRI	Standard research institute
SWCNT	Single-walled carbon nanotube

TEM	Transmission electron microscope
TETA	triethylenetetramine
$T_g$	Glass transition temperature
THF	tetrahydrofuran
THPA	tetrahydrophthalic anhydride
TMXDI	tetramethyl xylylene diisocyanates
US	United States
UV	Ultra violet
XDI	xylylene diisocyanates
XRD	X-ray diffraction
$\gamma$ -ray	Gamma-ray

# CHAPTER 1

## INTRODUCTION

### 1.1 Research Background

Corrosion is a process that leads to the deterioration of the performance of a material; it is a physicochemical interaction leading to a significant reduction of the functional properties of either the material or/and the environment in which it has interacted (Marcus and Maurice, 2009). Corrosion is more commonly understood to involve a chemical or electrochemical process that involved the typical transfer of electron due to the natural tendency for most metals to return to their natural state. Typically, metals are produced by extracting them from nature by applying energy to the mineral and ore. In an open environment, these metals are not thermodynamically stable and therefore tend to return to their lower Gibbs free energy forms as hydroxides, oxides, etc. These chemical and electrochemical processes through which metals go back to their lower Gibbs free energy states are termed corrosion. Corrosion tends to deteriorate metal surface through charge transfer reaction at an ambient environment (Cai et al., 2016).

Corrosion is a spontaneous process, and just like any other natural phenomenon, it drives the material to its lowest possible energy state. Metals and alloys have a natural tendency to combine with oxygen and water in the surrounding environment and return to their most stable form. When iron and steel interact with the environment, they return to their native and stable oxide. Like any other natural disaster such as weather change and earthquake, corrosion results in an unprecedented and economic challenge. National Association of Corrosion Engineers (NACE) International established the International Measures and Prevention, Application, and Economics of Corrosion Technologies (IMPACT) in October 2014. The NACE-IMPACT report is regarded to be the first assessment of global corrosion costs. Its report published in 2016 estimates the cost of corrosion at US\$2.5 trillion, equivalent to 3.4% of global GDP (Prasad et al., 2020). To make a global assessment, classification was made based on economic region to represent the total economy according to the World Bank's economic status for the countries. The economic regions comprised of the United States (US), China, European region, India, the Arab world, Russia, Japan, the four Asian tigers + Macau, and the rest of the world.

Furthermore, the industrial economic sector, services, and agricultural sectors in each of these countries were considered. A substantial economic impact was logged for the Arab world, sacrificed 5% of GDP for corrosion costs in different economic sectors. The least affected economic region is Japan, with total corrosion cost equivalent to 1% of GDP. The report, however, excludes the cost associated with individual safety and environmental consequences even though its essential in a real sense for implementing effective corrosion management strategies (Koch et al., 2016). The lifetime of products containing metallic components is often limited by the corrosion of their metallic parts. This cost could be saved with a better understanding and control of corrosion phenomena (Marcus and Maurice, 2009). Loss of production, inefficient operation, high maintenance



are among the expensive problem associated with metal corrosion (Karthik et al., 2015). Its therefore imperative to note that total elimination of the corrosion process is impossible; hence, various techniques are utilize in multiple fields such as marine equipment, pipelines, and construction to attenuate the intensity and severity of corrosion. These techniques include anodic or cathodic protection (Shen et al., 2005), corrosion inhibitors (Zheludkevich et al., 2006), and protective coatings (Hong, 2018).

Coatings are usually applied to improve the surface properties of a substrate, corrosion resistance, wettability, and adhesion. Applications of these coatings is a smart way to combine the mechanical properties of metals with the surface characteristics of the coatings (Ammar et al., 2016). The coating industry has been driven to seek new technologies and materials to improve the efficiency of coatings by economic benefits and growing environmental concerns. Several factors affect the effectiveness of a coating against all the possible damaging sources: they are the quality of the coating, the substrate characteristics, the properties of the coating/substrate interface, and the corrosiveness of the environment (Kiran et al., 2017). Organic coatings are widely used in the protection of the metallic structures against corrosion due to their high barrier and superior mechanical properties (Alam et al., 2013).

Various organic coating systems such as polyurethane, acrylic coatings, vinyl-based coatings, epoxy coatings, etc., are available in the market as it is generally assumed that these polymeric coatings play as a physical barrier between the metal substrate and corrosive environment. Among these polymeric coatings, Epoxy coatings have been widely used for protecting metal structures from environmental and corrosion attacks due to their outstanding toughness and durability (Brostow et al., 2010)

Epoxies are noted for their versatility, high resistance to chemicals, outstanding adhesion to a variety of substrates, toughness, high electrical resistance, durability at high and low temperatures, low shrinkage upon cure, flexibility, and the ease with which they can be poured or cast without forming bubbles and relatively cheap (Wang et al., 2018). These properties make them eligible for use in various applications such as protective coatings that can reduce the corrosion of metal/alloy substrates by acting as an excellent physical barrier layer because it also effectively prevents the invasion of aggressive chloride ion, hydrogen ion, and oxygen in a wet environment (Jin et al., 2017). But pure epoxy coatings have some deficiencies, which include micro-pores, bubbling, poor resistance to cracks propagation. Also, epoxies have hydrophilic hydroxyl groups and free volumes in their structure, which make them permeable to aggressive agents (i.e., water, oxygen,  $\text{Cl}^-$ ), which consequently accelerates the corrosion of the metallic substrate over time (Ji et al., 2006). To overcomes this problem, various efforts were made by researchers to improve the properties of the epoxy by the addition of various types of additives such as carbon-based nanofillers (Frigione and Lettieri, 2020), thereby enhancing barrier performance. The incorporations of these additives that are miscible into the resin occupy cavities, thereby reducing the porosity and zigzagging the diffusion path for the deleterious species and consequently enhancing the integrity and durability of the coating (Chen et al., 2017). Apart from increasing the length of the diffusion pathways for the corrosive agents, these additives also offer electrochemical anodic or cathodic protection effects. Also, properties of the coating such as UV blocking, scratch and abrasion

resistance, hardness, and other mechanical properties were reported to be improved by the addition of these fillers (Rahman et al., 2015).

Graphene related materials have attracted tremendous attention and have been employed in various field, especially in the improvement of the properties of polymeric coatings, due to its two-dimensional structure, high aspect ratio and specific surface area, superior gas barrier properties, and permeability to small ions and molecules (Xia et al., 2016). This property consequently makes the reinforced composites coatings to have an excellent anti-corrosive performance in a severely corrosive environment.

However, the electrostatic interaction,  $\pi$ - $\pi$  stacking, and high specific surface area make graphene aggregates easily, thereby resulting in heterogeneous dispersion in the polymer matrix. The tendency of graphene to agglomerate and its subsequent stacking resulted in graphite-like with low surface area pose a challenge in obtaining homogeneous dispersion and efficient interfacial interaction of graphene in organic coating and subsequent nanocomposites with optimal properties (Wang et al., 2012).

However, one of the approaches to improve the dispersion of graphene sheets in organic coatings is utilizing graphene oxide (GO), which has functional groups including hydroxyls, epoxides, and carboxyl on its structure that alter the van der Waals interactions, making them easily dispersed in the polymer matrix. Recently, researchers show that graphene oxide (GO) as a two-dimensional nanofiller of  $sp^2$ -hybridized carbon atoms, remarkably improves the corrosion resistance of polymeric coatings via increasing the diffusion path of corrosive agents as well as decreasing the coating porosity (Mo et al., 2015). However, to ensure adequate dispersion of GO in polymer coatings and its subsequent strengthening of the interfacial bonding between GO and polymer matrix, surface modification of GO is an alternative choice to prevent GO agglomeration and hence improved corrosion resistance of the coating. The functional groups at the basal plane and edges of GO provide the reactive sites for covalent functionalization. Amongst various functionalization methods considered for improving GO dispersion in a polymer matrix, silane modification is a promising method for enhancing the properties of the composites due to its bifunctional structure (Zhang et al., 2018). The coupling process is accomplished via a chemical reaction between the silane molecules alkoxy groups and the hydroxyl group's presence on the surface of the graphene, at the same time, the other functional groups of silane molecules, which are generally amine, ethylene, epoxy, trihydroxy, etc. remained. These surviving functional groups can provide a chemical bonding site of graphene with the polymer matrix, leading to enhanced properties (Li et al., 2015). Thus, the performance of epoxy coatings will ultimately be enhanced by utilizing the advantages of functionalized-GO (f-GO).

So far, there have been several methods for the functionalization of GO, one of which is chemical techniques that typically require a long reaction time, toxic reagent, strict reaction conditions that restrict large-scale production (Ahmad et al., 2017). Hence, a new route combining the economic advantage and convenience of chemical synthesis with excessive reduction efficiency has therefore been an import goal for the preparation of functionalized GO. Gamma-ray irradiation has recently been investigated as a safe, clean and simple way to alter the nanostructure and properties of carbon materials and

facilitate chemical reactions on their surface (Ansón-Casaos et al., 2014). Not only because it is an environmentally friendly procedure, but also because it is practical, cost-effective and can be performed at room temperature, the method has attracted interest among researchers. However, the study of inducing grafting onto the surface of GO using gamma-ray irradiation is still an emerging field.

## 1.2 Problems statement

Corrosion is a disturbing phenomenon in which chemical or electrochemical reactions occur that lead to the deterioration of metals materials. Apart from economic losses associated with corrosion, the process is a threat to the safety of industrial production. However, various corrosion protection methods have been developed which includes protective coatings, environmental modifications, cathodic and anodic protection, application of inhibitors or any combination of these. Protection of metallic surface from corrosion employing polymeric coatings are the commonest approach due to their excellent anti-corrosion performance coupled with their low cost. Among them, epoxy coatings have captivated the interest of researchers due to their excellent mechanical properties and adhesion to metallic substrates. Nevertheless, epoxy coatings fail over prolong exposure to permeable aggressive corrosive media, in addition to its low resistance to crack propagation; epoxy contains hydrophilic hydroxyl groups in their cured network leading to poor resistance in humid condition. Hence, the initiation and propagation of cracks in the coating allow the penetration of corroding agents through the defect thus reduces adhesion and enhancing corrosion.

To overcome this problem, nanoparticles such as graphene oxide is incorporated as a nanofiller. Nonetheless, graphene oxide sheet agglomeration due to the strong van der Waals forces among their sheets and its weak compatibility with most of the polymer matrices have been fundamental roadblocks that restrict its potentials. Surface modification by adding functional groups such as organo silane compounds is an effective way to reduce the tendency to agglomerate. Also, functionalization increases the graphene compatibility with specific polymers thereby improving the reinforcing effect. Previous studies focus on the functionalization of graphene derivatives through various techniques that are often complex with long reaction time, toxic reagents with specific reaction conditions. However, this study focus on the  $\gamma$ -irradiation method of graphene functionalization, which is cost-effective, environmentally friendly, and can be conducted at low temperatures.

## 1.3 Objective of the study

To prepare and characterized an anti-corrosive coating based on silane functionalized Graphene Oxide (f-GO) / Epoxy resin nanocomposites

Specific Objectives:

- To prepare, functionalized and characterized Graphene oxide with Organosilane, i.e. 3-Aminopropyltriethoxysilane (APTES) and 3-

Glycidyloxypropyltrimethoxy (GPTMS) via radiation assisted process using gamma-ray irradiation at various radiation doses.

- To prepare and characterize nanocomposites coatings for morphology, coatings surface performance and chemical resistance performance
- To investigate thermomechanical properties of the nanocomposites coatings via Thermogravimetric Analysis (TGA) and Dynamic Mechanical Analysis (DMA).
- To investigate the anti-corrosion performance of the AGO/Epoxy coatings (EAG's) and GGO/Epoxy coatings (EGG's) via Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic polarization.

#### **1.4 Significance of the Study**

Several studies in this field have been inspired by the excellent properties of graphene. Up till now, the performance of polymer/graphene nanocomposites have been extensively investigated so far in applications such as solar cells, supercapacitors, lithium-ion batteries, electrochemical sensing, and membrane-based separation applications. Currently, most of the research of graphene/Epoxy nanocomposites is largely focusing on their electrical conductivity features in fuel cells, transistors, and photocatalytic applications. Up till now, no research is being reported on the incorporation of gamma-irradiation functionalized-GO nanomaterials as nanofillers in the fabrication of epoxy nanocomposites for anti-corrosion coatings. Since various researches findings have shown that graphene exhibited anti-corrosion properties, the present study would concentrate on the functionalization of GO with hydrophobic silane molecules such as APTES and GPTMS to increase the properties of GO. This can, in turn, heighten the performance of GO as a coating material.

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