## **UNIVERSITI TEKNOLOGI MARA**

# UNSATURATED POLYESTER-GRAPHENE COATINGS TREATED SILANE COUPLING AGENT FOR METAL CORROSION PROTECTION

# HASNIRAAIMAN BINTI ABDUL HAMID

MSc

June 2020

### **AUTHOR'S DECLARATION**

I declare that the work in this dissertation was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student	:	Hasniraaiman Binti Abdul Hamid
Student I.D. No.	:	2017170365
Programme	:	Master of Science (Science and Polymer Technology)– AS761
Faculty	:	Applied Sciences
Thesis Tittle	:	Unsaturated Polyester-Graphene Coatings Treated Silane Coupling Agent for Metal Corrosion Protection
		Hasniva
Signature of Student	:	~~~
Date	:	June 2020

### ABSTRACT

This study has been develop to reduce the corrosion and improved the protection of the offshore platform by using unsaturated polyester (UPE) resin reinforced graphene (GR) primer coatings. The study was done by firstly; modifying GR loadings from 0-8% to determine the optimum loading, secondly; the optimization studies were undergone by using different mixing techniques (ultrasonication and mechanical stirring) and different mixing times at 30, 60, and 90 minutes to determine the optimum condition and lastly; the effect of silanization of GR with different loading of silane coupling agent, 3-aminopropyltriethoxysilane (3-APTES) from 1-7% to improve the dispersion of GR into the UPE resin. The primer coatings have been coated onto the carbon steel plate by using hand brush. Fourier Transform Infrared Spectroscopy (FT-IR) characterization technique is successfully done to analyze the functional group present and evaluate the UPE-GR and treated GR interaction. The Field Emission Scanning Electron Microscope (FE-SEM) analysis also has been done to study the surface morphology of the coatings. Corrosion rate for the preliminary studies shows the optimum results at 2% of GR loadings at 0.586 millimetre per year (mmpy) and no corrosion was found on the coating for immersion test in seawater and salt solution for 9 days. Whilst, mechanical properties which are pencil hardness, cross-cut adhesion strength and pull-off adhesion strength also shows the optimum properties at 2% of GR loading with 5H of pencil hardness, 0% of area has been removed from cross-cut adhesion test, and 3.35MPa of adhesion for pull-off adhesion strength test. The incorporation of 2% of GR loadings in UPE primer system successfully improved the mechanical properties and inhibits the corrosion of metal substrate due to well dispersion of GR into the UPE resin. The ultrasonication and mechanical stirring method in optimization studies shows the optimum anti-corrosive and mechanical properties at 60 minutes mixing times. The optimum corrosion rate for ultrasonication method is 0.375 mmpy whilst for mechanical stirring method is 0.536 mmpy. Both methods shows the optimum pencil hardness at 5H and 0% of coating being detached from the substrate for cross-cut adhesion strength. The optimum pull-off adhesion strength has been observed for ultrasonication method and mechanical stirring method at 3.78MPa and 3.58MPa, respectively. The optimum condition was found at 60 minutes mixing time by using ultrasonication method. This is due to the ultrasonication method produced more uniform dispersion of GR compared to the mechanical stirring method as the ultrasonication possessed a good vibration energy dissipation to enable a better disentanglement of GR. The enhancement of UPE-GR 2% using 3-APTES with different loading (1, 3, 5 and 7%) by using ultrasonication method at 60 minutes mixing time was also successfully conducted. The results showed the optimum properties with improved mechanical and anti-corrosive behaviour at 3% of APTES. The corrosion rate is found at 0.073 mmpy and the corrosion has not been formed on the substrate after immersion test. The conductivity also was found at 1.289 x  $10^{-8} \Omega.cm^2$ . The mechanical properties also showed the optimum condition at 3% of treated GR with 5H of the pencil hardness, 0% of area being detached from the substrate, pull-off adhesion strength at 4.19MPa and 160.23° of contact angle. This is attributed to the 3-APTES that acted as a dispersion agent and enhanced the interaction of GR with UPE resin as well as improved the mechanical and anti-corrosive properties of the coatings.

### ACKNOWLEDGEMENTS

Firstly, I wish to thank Allah for giving me the opportunity to embark on my research study and for completing this journey successfully. My gratitude and thanks go to my superwoman supervisor Dr Zuliahani Binti Ahmad, and cosupervisors, Prof. Ts. Dr Mohd Azlan Bin Mohd Ishak and Dr Azniwati Binti Abd Aziz for guiding me in doing my research and for being such a great supervisors. Thank you for sharing the times, dedications, knowledge, support, patience and invaluable ideas in assisting me with this project. Deepest gratitude is also to the postgraduate team members and beloved roommates, without their knowledge, motivation and assistance, this study would not have been enjoyable and meaningful. I would also like to express thanks to all FBERG members, PJI staffs, UiTM staffs, USM, and UiTM Perlis Postgraduate Society for helping me with my studies. Deepest love and thanks are also due to my beloved husband, siblings, colleagues, lab assistants, lecturers, juniors and friends for all of your contributions, efforts and prayers. May Allah increase all of you in your affairs for the better, aamiin. Finally, this thesis is dedicated to the heroes in my life, my late father Abdul Hamid Bin Ismail and princess, my late mother Che Amnah Binti Hj Noor for their inspiration, spirit and endless love that give me unbelievable strength, motivation and spirit to further my study and go through all of the challenges bravely throughout the duration of my studies. I want to make you proud of me. Everything I want to be all you think about and anything you dream about. This piece of victory is dedicated to both of you. Alhamdulillah.

## **TABLE OF CONTENT**

<b>CONFIRMATION BY PANEL OF EXAMINERS</b>	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENTS	V
TABLE OF CONTENT	vi
LIST OF TABLES	ix
LIST OF FIGURES	xii
ABBREVIATIONS	xiv
NOTATIONS	XV
CHAPTER ONE: INTRODUCTION	1
1.1 Background of Study	1
1.2 Problem Statement	4
1.3 Research Objectives	5
1.4 Scope and Limitation of Study	6
1.5 Significance of Study	7
CHAPTER TWO: LITERATURE REVIEW	8
2.1 Corrosion in Oil and Gas Platform Coatings	8
2.2 Primer Coatings	9
2.3 Unsaturated Polyester (UPE) Primer Coatings	12
2.4 The Incorporation of UPE with GR and Other Substances	13
2.5 The Mixing Techniques and Mixing Times	17
2.6 The Incorporation of Silane Coupling Agent	18
2.7 Corrosive Properties of Coatings	21
2.8 Mechanical Properties of Coatings	22
CHAPTER THREE: RESEARCH METHODOLOGY	25
3.1 Reagents and Chemicals	25
3.2 Conceptual Framework	25
3.3 Instruments	27