PREPARATION AND CHARACTERIZATION OF TUNG OIL-FILLED UREA-FORMALDEHYDE MICROCAPSULES AND THEIR EFFECT ON MECHANICAL PROPERTIES OF AN EPOXY-BASED COATING

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2022

DECLARATION

I declare that this thesis entitled " Preparation and characterization of Tung Oil-Filled Urea-Formaldehyde Microcapsules and their Effect on Mechanical Properties of an Epoxy-Based coating" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Manufacturing Engineering Technology with Honours.

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DEDICATION

This thesis is dedicated to my beloved parents, who have supported me through the ups and downs of my bachelor's degree journey at UTeM.

Apart from that, not to forget my sisters, brothers, close friends and classmates that had always kept supporting and encouraging me with good words to finish this study.

Moreover, the most important person, Dr. Mohd Fauzi bin Mamat, guided and helped me with advice to successfully write this thesis.

Finally, this thesis is dedicated to all people that know me and keep supporting me through thick and thin as a student.

ABSTRACT

Nowadays, corrosion is a issue that causes substantial harm to metal properties such as carbon steel. It's the deterioration of a material's properties due to interactions with its surroundings, and the corrosion of most metal was unavoidable. Corrosion always occurs when the coating on the steel surface mechanically damages the surface area because of a microcrack or scratch. Furthermore, the cost of repairing the damage is high, and the process is time-consuming. The aim of this study was developing self-healing coating that consists of tung oil microcapsules as healing agent. The self-healing coating is a barrier to protect the steel from corrosion attack. A self-healing coating has recently emerged as one of the smart coating methods used to protect steel from corrosion. Self-healing coating could heal automatically by itself. The ability to self-heal can help to prevent corrosion and ensure a long life. The performance of self-healing coating on low carbon steel as substrate been evaluated in a 3.5 wt% Nacl solution. The mechanical testing has been done on low carbon steel size of 20 mm x 20 mm x 2 mm by microstructure and hardness test. In this study, the preparation by in-situ polymerization of urea-formahdehyde to create microcapsules shell that contain tung oil as healing agent. The microcapsules of tung oil were manually added with epoxy and harderner in a proportion of 7.5 wt% with a ratio of 4:1. The immersion test had been performed by immersing the samples in a 3.5 wt% NaCl solution and dividing them into three groups: uncoating, epoxy coating, and self-healing coating in different containers. Each container has six samples. The immersion test has been done in 7, 14, 21, 28, and 35 days. The sample had been observed using Scanning Electron Microscope / Energy Dispersive X-Ray (SEM/EDX). The visual inspection of day 35 shown that the weight loss measurement and corrosion rate measurement of self-healing coating had the lowest value which is 0.01 gram of weight loss and 0.001 (mm/years) corrosion rate compared to epoxy coating and uncoating. So that, from the visual inspection of the immersion test had shown that the self-healing coating sample has excellent corrosion resistance compared to the epoxy coating and uncoating. The effectiveness of self-healing coating as corrosion resistance was proved when the scratch region on sample fully healed. Thus, self-healing coating can be one as an alternative to help the industries of oil and gas to reduce corrosion attack on platform, onshore or offshore especially for open system.

ABSTRAK

Pada masa kini, kakisan adalah isu yang menyebabkan kemudaratan besar kepada sifat logam seperti keluli karbon. Kemerosotan sifat bahan disebabkan oleh interaksi dengan persekitarannya, dan kakisan kebanyakan logam dan bahan lain tidak dapat dielakkan. Kakisan selalu berlaku apabila lapisan pada permukaan keluli rosak secara mekanikal pada permukaan kerana mikrackack atau calar. Tambahan pula, kos untuk memperbaiki kerosakan adalah tinggi, dan prosesnya memakan masa. Tujuan kajian ini menghasilkan salutan swa sembuh yang mengandungi mikrokapsul minyak tung sebagai agen penyembuhan. Salutan swa sembuh adalah sebagai penghalang untuk melindungi keluli daripada serangan kakisan. Salutan swa sembuh baru-baru ini muncul sebagai salah satu kaedah salutan pintar yang digunakan untuk melindungi keluli daripada kakisan. Salutan swa sembuh boleh sembuh secara automatik dengan sendirinya. Keupayaan untuk menyembuhkan diri boleh membantu untuk mengelakkan kakisan dan memastikan hayat yang panjang. Prestasi salutan swa sembuh pada keluli karbon rendah sebagai substrat telah dinilai dalam larutan Nacl 3.5 wt%. Ujian mekanikal telah dilakukan pada keluli karbon rendah saiz 20 mm x 20 mm x 2 mm dengan ujian struktur mikro dan kekerasan. Dalam kajian ini, penyediaan secara in-situ pempolimeran urea-formahdehid untuk menghasilkan cengkerang mikrokapsul yang mengandungi minyak tung sebagai agen penyembuhan. Mikrokapsul minyak tung telah ditambah secara manual dengan epoksi dan lebih keras dalam perkadaran 7.5% berat dengan nisbah 4:1. Ujian rendaman telah dilakukan dengan merendam sampel dalam larutan NaCl 3.5 wt% dan membahagikannya kepada tiga kumpulan: tidak disaluti, disaluti epoxi, dan disaluti swa sembuh didalam bekas yang berbeza. Setiap bekas mempunyai enam sampel. Ujian rendaman telah dilakukan selama 7, 14, 21, 28, dan 35 hari. Sampel telah diperhatikan dengan menggunakan Mikroskop Elektoron Pengimbas / X-Ray Penyebaran Tenaga (SEM/EDX). Pemeriksaan visual hari ke-35 menunjukkan bahawa ukuran penurunan berat dan ukuran kadar kakisan salutan swa sembuh mempunyai nilai terendah iaitu 0.01 gram penurunan berat dan kadar kakisan 0.001 (mm/tahun) berbanding salutan epoksi dan tiada salutan. Oleh itu, daripada pemerhatian visual ujian rendaman telah menunjukkan bahawa sampel salutan swa sembuh mempunyai rintangan kakisan yang sangat baik berbanding dengan salutan epoksi dan tiada salutan. Keberkesanan salutan swa sembuh sebagai rintangan kakisan dibuktikan apabila kawasan calar pada sampel sembuh sepenuhnya. Oleh itu, salutan swa sembuh boleh menjadi salah satu alternatif untuk membantu industri minyak dan gas untuk mengurangkan serangan kakisan pada platform, darat atau luar pesisir terutamanya untuk sistem terbuka.

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LIST OF SYMBOLS AND ABBREVIATIONS

NaCl	-	Sodium chloride
NaOH	-	Sodium hydroxide
HCl	-	Hydrochloric acid
wt%	-	Weight percentages
SEM	-	Scanning Electron Microscope
EDX	-	Energy Dispersive X-Ray
FTIR	-	Fourier Transform Infrared Spectroscopy
rpm	-	Revolutions per minute
pH	-	Potential of hydrogen
mm	-	Millimeter
mL	-	Milliliter
g	-	Gram
L	-	Liter
AISI / SAE	-	American Iron and Steel Institute / Society of Automotive Engineers
ASM	-	American Society for Metals
ASTM	-	American Society for Testing and Materials
HSLA	-	High-strength low-alloy steel
C	-	Carbon
O	-	Oxygen
Fe	-	Iron
Si	-	Silicon
Ni	-	Nickel
S	-	Sulfur
P	-	Phosphorus
Mn	-	Manganese

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

In nowadays, corrosion is a issue that causes substantial harm to metal properties, such as carbon steel, as well as added the costs. Carbon steel is an iron-based alloy that contain carbon about less than 2% (Gandy, 2007). Corrosion protection can be achieved by coating with organic materials. Corrosion is the degradation of a material's characteristics as a result of interactions with its surroundings, and corrosion of most metals (and many other materials) is unavoidable (Shaw and Kelly, 2006). It always happens when the surface exposed then react between electrochemical and its environment (Seal, 2017). When the oxidation occurs, It has a proclivity for losing electrons to oxygen and other chemicals in water and air. The metal surface oxide when the oxygen reduced.

Apart from that, the self-healing coating technology is used to prevent corrosion on carbon steel. This self-healing coating have the ability that automatically repaired any defect that happened and will provide good recovery from corrosion which is in smart mechanism characteristic defence by incorporating drying oil microcapsules, such as tung oil, with epoxy resin and coating the substrate surface (Li *et al.*, 2021).

Next, tung oil-filled urea formaldehyde microcapsules as a self-healing mechanism to act as a protective barrier against corrosive substances that attack the surface of the substrate materials. Tung-oil is a plant-based oil from tung tree. The seed of the tung tree, which is native to Eastern Asia, is pressed to make tung oil (Farabaugh, 2019). Urea formaldehyde is also known as urea-methanal is a type of thermosetting polymer resin and the basic characteristics of these as a capsule are strong responsiveness, quick cure, water soluble, non-flammability, abrasion resistance and excellent thermal properties (Katoueizadeh *et al.*, 2019). The characteristic of tung oil is containing a large glyceride of Eleostearic acid, each Eleostearic acid molecule has three carbon-carbon double bonds, making it easier for the oil to dry in the atmosphere (Li *et al.*, 2021).

Furthermore, an epoxy-based coating is a coating made up of two components: an epoxy resin and a polyamine hardener, often known as a catalyst. The resin and hardener mixed then it will be engaging as the element cures, a chemical process occurs that causes cross-linking. An example of nowadays that use self-healing mechanism in industrial are in automotive, aerospace, building and construction, and oil and gas sector. In this study, used self-healing coating method to provide a good protection from corrosion for the oil and gas industrial for their platform and pipeline at offshore (Olajire, 2018). The benefit from self-healing mechanism for this industry is long term reliability (Zhang *et al.*, 2018). By applying this mechanism of self-healing, the corrosion can be avoidable and can has longer lifespan. It also can reduce the total of cost to repair of the material. In this study, we used urea-formaldehyde microcapsules that contain tung oil as a self-healing agent.

1.2 Problem Statement

Corrosion always occurs and many effort had been made to avoid the corrosion attack of metal in oil and gas platform, automative and marine industries by applying coating as corrosion protection. But by applying polymer as a coating protection its tend to microcrack and scratch. When the coating barrier of low carbon steel surface mechanically damaged because of the microcrack or scratch on the surface area, the corrosive species will penetrate to the surface of low carbon steel substrate, and the coating will need a special characteristic that have ability self-heal so that the damage will be repaired automatically. The cost of repairing the damage will be high cost if the corrosive cannot be avoidable and it take time of repairing process. In this recent year we need to design a new method or smart way of technology coating so that the action towards mechanical damage cause by external environment can be reproduce automatically fast. The new design of coating will be as a good of protective barrier of the mechanical properties and be a potential for sustain the mechanical properties. The ability of self-heal can prevent the corrosion and providing a long life for the low carbon steel. The aim for this study is to design a smart self-healing coating that can prevent the corrosion on the low carbon steel.

1.3 Objective of Study

The research objective of this study shown as below:

i. To develop self-healing coating consist of tung oil microcapsules as healing agent.

ii. To evaluate the performance of self-healing coating on low carbon steel substrate

iii. To determine the corrosion behaviour of self-healing coating, uncoated and coated low carbon steel substrate in 3.5wt% NaCl.

1.4 Scope of Study

The scope of research of this study consists:

i. The scope of this study is to find the best healing agent formula for preventing corrosion in self-healing polymeric coatings on low carbon steel substrates for application in the oil and gas industries.

ii. Laser cut equipment used to cut the material into 20 × 20 mm squares with a 2 mm thickness.

iii. Use a hardness test and an optical microscope to do mechanical testing on the substrate.

iv. Perform the self-healing performance at the base of low carbon steel that coated by the coating consist of the healing agent.

v. To study the corrosion behaviour via immersion test in 3.5 wt% NaCl solution.

vi. The six samples immersed in a 3.5 wt% NaCI solution for immersion testing.

vii. The 3.5 wt% NaCl solution was used to immerse all of the samples for 7, 14, 21, 28, and 35 days.

viii. After the corrosion test, the corrosion behaviour on the substrate been studied using a scanning electron microscope (SEM).

1.5 Significant of Study

The result of this study finding provided an information about the self-healing mechanism to avoid the corrosion itself. The focus of study about an epoxy-based coating that encapsulated with tung oil filled urea formaldehyde microcapsules to reduce the coating failure from corrosion species attack and extend lifespan of metal substrate. This finding of study aims to help the industries of oil and gas to reduce the corrosion on the platform, pipeline, onshore or offshore.

1.6 Organization of Thesis

This thesis is made up of five (5) chapters. The summarize of content for each chapter are as following:

1. Chapter 1. This chapter is about introduction, included background of study, problem statement, objective of study, scope of study, significant of study and organization of thesis.
2. Chapter 2. This chapter is about a literature review. Literature review about low carbon steel and the type of corrosion species that attack on the steel. The finding of how other researcher prevent the corrosion species from attack the steel by using self-healing coating method which is epoxy based-coating.
3. Chapter 3. This chapter about research methodology on how the process and procedure of the study preparation of self-healing coating been performed via in-situ polymerization urea-formahdehyed that consists tung oil as healing agent. The flowchart had been shown in this chapter step by step to complete the study.
4. Chapter 4. This chapter about result and discussion of microstructure, hardness test, immersion test in 3.5 wt% Nacl of sample for 7, 14, 21, 28, and 35 days. The visual inspection, weight loss measurement and corrosion rate after the immersion test. The performance self-healing coating on substrate compared with uncoating and epoxy coating.
5. Chapter 5. This chapter is about conclusion and recommendation which is overall conclusion about self-healing coating study to prevent corrosion attack on low carbon steel, recommendation for future study of self-healing coating and the project potential to been implement in industries.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of Carbon Steel

Carbon steel as a substrate. Carbon steel is a steel with a higher percentage of carbon than other steels. In addition, carbon steel is divided into five categories: ultra low carbon steel with a carbon content of less than 0.015%, ultra low carbon steel with a carbon content of 0.015 % to 0.05%, low carbon steel with a carbon content of 0.05% to 0.19%, medium carbon steel with a carbon content of 0.2% to 0.49% and high carbon steel with a carbon content greater than 0.5% (Masteel, 2018). Carbon steel is widely used in a variety of industries, particularly the oil & gas, and petrochemical industries, due to its remarkable flexibility (Masteel, 2018).

2.1.1 Type of Carbon Steel

Carbon steels categorize into three main type which is low carbon steel (mild carbon steel), medium carbon steel and high carbon steel. Each type of carbon steel has a different carbon content, microstructure and properties. Table 2.1 shown type of carbon steel with their properties and example.

Table 2.1 Type of carbon steel and their properties (Matmatch Gmbh, 2020).

Type of carbon steel	Carbon content (wt.%)	Microstructure	Properties	Example
Low carbon steel	< 0.25	Ferrite, pearlite	Low hardness and cost. High ductility, toughness, machinability, and weldability	AISI 304, ASTM A815, AISI 316L
Medium carbon steel	0.25 – 0.60	Martensite	Low hardenability, medium strength, ductility, and toughness	AISI 409, ASTM A29, SCM435
High carbon steel	0.60 – 1.25	Pearlite	High hardness, strength, low ductility	AISI 440C, EN 10088-3

2.1.2 Low Carbon Steel

The term "low carbon steel" refers to an iron or carbon alloy having a low carbon content ranging from 0.05% to 0.30% (Delich, 2021). Low carbon steel is the most prevalent steel type because it is inexpensive and has material properties that are suitable for a large number of applications. Due to its low carbon content, it is neither brittle nor ductile on the outside, malleable and has a lower tensile strength (Finkel'shtein *et al.*, 2008). Low-carbon steels are also known as high-strength, low-alloy steels (HSLA), though they frequently contain other elements including copper, nickel, vanadium, and molybdenum (Matmatch Gmbh, 2020). Table 2.2 shown example of properties and application of low carbon steel. In a micrograph of low-carbon AISI/SAE 1010 steel, Figure 2.1 shown a matrix of ferrite grains (white etching component) and pearlite (dark etching constituent). Marshall's reagent was employed first, followed by a 2% nital solution (ASM, 2002).