UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Preparation and characterization of linseed/tung oil-filled urea–formaldehyde microcapsules and their effect on mechanical properties of an epoxy-based coating

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**A thesis submitted
in fulfillment of the requirements for the degree of
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DECLARATION

I declare that this thesis entitled "Preparation and characterization of linseed/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 choose an item has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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APPROVAL

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Manufacturing Engineering Technology (BMMW) with Honours.

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DEDICATION

I dedicate my dissertation work to my family and my supervisors and my co supervisors Madam Nur Aiman Hanis Binti Hasim and Ts. Dr. Mohd Fauzi Bin Mamat. A special feeling of gratitude to my loving parents, Md Akhir Bin Chin and Khoriah Binti Ismail whose words of encouragement and push for tenacity ring in my ears. My brothers and sisters have never left my side and are very special. I also dedicate this dissertation to my many friends and family who have supported me throughout the process. I will always appreciate all they have done. I dedicate this work and give special thanks to my supervisor Madam Nur Aiman Hanis Binti Hasim and my co supervisors Ts. Dr. Mohd Fauzi Bin Mamat for being there for me throughout the entire bachelor program.

ABSTRACT

One of the most formidable foes that metals have to contend with is corrosion. It is a natural occurrence that is brought on by the interaction of metal and the components of the environment. Nowadays, applying a protective coating to metal surfaces is a common method of protecting them against corrosion. In the event of defect such as crack, scratch and other unexpected damage on steel or pipeline, this layer may be rendered useless. A self-healing coating was a new coating method for preserving steel against corrosion. There were self-healing coatings that can fix themselves. Self-healing coating is one of the smart coatings that may mend or restore coating damage in order to prevent additional corrosion. Micro container releases included into the polymer enable active functioning. The self-healing properties of this coating help to save costs. The goal of this research project to developing self-healing coatings from polymeric materials and evaluating their performance and corrosion behavior on steel substrates. In this self-healing coating, Linseed oil have been mixture with Tung oil to get microcapsules and work together to repair the scartch at the low carbon steel. According to this research, the steel have been protected from corrosion by a self-healing coating. The self-healing coating on low carbon steel substrate in a 3.5wt% NaCl solution have been tested. Low carbon steel substrate with dimensions of 20mm length x 20mm width x 2mm thickness have been subjected to a hardness test for mechanical testing. To fabricate microcapsules that contain 50% linseed oil and 50% tung oil as a healing agent, this study had used an epoxy and hardener ratio of 4:1 to make microcapsule shells and have been analyse by using Attenuated total reflection - Fourier-transform infrared (ATR-FTIR). Immersion tests have been carry out by separating the samples into three categories: uncoated, epoxy-coated, and self-healing coated, all in a 3.5 wt% NaCl solution. There were 7, 14, 21, 21, 28 and 35 day intervals in which the immersion test have been done. Material characterizaion have been examined using a Scanning Electron Microscope / Energy Dispersive X-Ray (SEM/EDX). The result on analysis, the scratch on the substrate self healing coating were closed and protect the substrate from corrosion while for uncoated substrate the scratch were not closed and corrosion have been happen. The result after 35 days, the average weight loss for self healing coated was 0.008g, epoxy coated was 0.11g and uncoated was 0.029g and corrosion rate was 0.0039g for self healing coated, 0.0056 for epoxy coated and 0.0142 for uncoated. Based on this two measurements, self-healing coating have been the lowest values compared to uncoating and epoxy coating. The visual inspection when comparing the self-healing coating sample to epoxy coating and uncoating, the visual evaluation of the immersion test have been demonstrate greater corrosion resistance. The EDX result also have been prove that self healing coating were closed the scartch and uncoated were not based on Fe, which are 5.50wt% for self healing sample and 95.44wt% for uncoated sample. As conclusion, corrosion resistance was proved by the sample's ability to completely heal a scratch. The application of this self-healing coating can be use on oil and gas industry.

ABSTRAK

Salah satu musuh paling hebat yang perlu dihadapi oleh logam ialah kakisan. Ia adalah kejadian semula jadi yang dibawa oleh interaksi logam dan komponen alam sekitar. Pada masa kini, menggunakan salutan pelindung pada permukaan logam adalah kaedah biasa untuk melindunginya daripada kakisan. Sekiranya berlaku kecacatan seperti retak, taburan dan kerosakan lain yang tidak dijangka pada keluli atau saluran paip, lapisan ini mungkin menjadi tidak berguna. Salutan penyembuhan sendiri ialah kaedah salutan baharu untuk memelihara keluli daripada kakisan. Terdapat salutan penyembuhan diri yang boleh membaiki sendiri. Salutan penyembuhan sendiri ialah salah satu salutan pintar yang mungkin membaiki atau memulihkan kerosakan salutan untuk mengelakkan kakisan tambahan. Keluaran bekas mikro yang dimasukkan ke dalam polimer membolehkan berfungsi aktif. Ciri-ciri penyembuhan diri salutan ini membantu menjimatkan kos. Matlamat projek penyelidikan ini untuk membangunkan salutan penyembuhan diri daripada bahan polimer dan menilai prestasi dan kelakuan kakisannya pada substrat keluli. Dalam salutan penyembuhan sendiri ini, minyak biji rami telah dicampur dengan minyak tung untuk mendapatkan mikrokapsul dan bekerjasama untuk membaiki parut pada keluli karbon rendah. Menurut penyelidikan ini, keluli telah dilindungi daripada kakisan oleh salutan penyembuhan sendiri. Salutan penyembuhan sendiri pada substrat keluli karbon rendah dalam larutan NaCl 3.5wt% telah diuji. Substrat keluli karbon rendah dengan dimensi 20mm panjang x 20mm lebar x 2mm ketebalan telah tertakluk kepada ujian kekerasan untuk ujian mekanikal. Untuk membuat mikrokapsul yang mengandungi 50% minyak biji rami dan 50% minyak tung sebagai agen penyembuhan, kajian ini telah menggunakan nisbah epoksi dan pengeras 4:1 untuk membuat cangkerang mikrokapsul dan telah dianalisis dengan menggunakan Refleksi total yang dilemahkan - Fourier-transformasi inframerah (ATR-FTIR). Ujian rendaman telah dijalankan dengan mengasingkan sampel kepada tiga kategori: tidak bersalut, bersalut epoksi, dan bersalut penyembuhan sendiri, semuanya dalam larutan NaCl 3.5 wt%. Terdapat selang 7, 14, 21, 21, 28 dan 35 hari di mana ujian rendaman telah dilakukan. Pencirian bahan telah diperiksa menggunakan Mikroskop Elektron Pengimbasan / X-Ray Penyebaran Tenaga (SEM/EDX). Hasil analisis, calar pada salutan penyembuhan sendiri substrat telah ditutup dan melindungi substrat daripada kakisan manakala untuk substrat yang tidak bersalut parut tidak ditutup dan kakisan telah berlaku. Hasilnya selepas 35 hari, purata penurunan berat badan untuk bersalut penyembuhan sendiri ialah 0.008g, bersalut epoksi ialah 0.11g dan tidak bersalut ialah 0.029g dan kadar kakisan ialah 0.0039g untuk bersalut penyembuhan sendiri, 0.0056 untuk bersalut epoksi dan 0.0142 untuk tidak bersalut. Berdasarkan kedua-dua ukuran ini, salutan penyembuhan diri adalah nilai yang paling rendah berbanding salutan tanpa salutan dan epoksi. Pemeriksaan visual apabila membandingkan sampel salutan penyembuhan diri kepada salutan epoksi dan salutan, penilaian visual ujian rendaman telah menunjukkan rintangan kakisan yang lebih besar. Keputusan EDX telah membuktikan bahawa salutan penyembuhan sendiri telah ditutup parut dan tidak bersalut berdasarkan Fe, iaitu 5.50wt% untuk sampel penyembuhan sendiri dan 95.44wt% untuk sampel tidak bersalut. Kesimpulan, rintangan kakisan telah dibuktikan oleh keupayaan sampel untuk menyembuhkan sepenuhnya calar. Penggunaan salutan penyembuhan diri ini boleh digunakan pada industri minyak dan gas.

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LIST OF SYMBOLS AND ABBREVIATIONS

CO_2	-	Carbon dioxide
NaCl	-	Sodium chloride
NaOH	-	Sodium hydroxide
wt%	-	Weight percentages
SEM	-	Scanning Electron Microscope
EDX	-	Energy Dispersive X-Ray
pH	-	Potential Of Hydrogen
S	-	Sulphur
P	-	Phosphorus
Mn	-	manganese
Cu	-	Cuprum
Al	-	Aluminium
C	-	Carbon
mm	-	Milimiter
ml	-	Mililiter
AISI / SAE	-	American Iron and Steel Institute / Society of Automotive Engineers
ASTM	-	American Society for Testing and Materials
HSLA	-	High-strength low-alloy steel
ASM	-	American Society for Metals
CRAs	-	corrosion-resistant alloys
DCPD	-	Dicyclopentadiene
UF	-	Urea formaldeyde
LACs	-	Low alloy stars
PVA	-	Polyvinyl alcohol
CeO_2	-	Cerium(IV) oxide
$CaCO_3$	-	Calcium carbonate
ATR-FTIR	-	Attenuated total reflection - Fourier-transform infrared

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Corrosion is commonly defined as the nearly unavoidable decomposition of a substance due to a chemical reaction with its surroundings that is determined by thermodynamics. Corrosion that goes unchecked wastes resources and infrastructure, and if left unattended, can result in unexpected infrastructure damage, injury, and death. An electrochemical reaction requires four ingredients for corrosion to occur which are the presence of an anode (where material is lost due to oxidation), the presence of a cathode (which is typically the driving element for electrochemical reactions to occur at the anode and is a reduction reaction), an electrical conductor to allow charge to flow via electron flow, and an electrolyte to allow charge to flow via ion flow. Any change to one (or more) of these four factors will have an impact on the total corrosion process (Ben DuBose, 2020).

Any change to one or more of these four factors will have an impact on the total corrosion process. Corrosion can be localised or widespread (general). Both of these basic kinds are undesirable and result in waste and cost. Localized corrosion, on the other hand, is more insidious than general corrosion because it causes a large amount of metal loss in a limited area and is frequently not observed as quickly as general corrosion, leading to catastrophic failures that occur without any warning (Trethewey, Chamberlain, 1988).

However, corrosion genarally can be controlled by take the step to protected metal. Various methods can be taken to control corrosion on steel such as barrier coating, corrosion inhibitor, material selection (aloy steel), cathodic protection and sacrificial protection. Steel can also be protected with protective coatings from external causes such as the harmful effects of gases in the atmosphere. Epoxy, powder, or sel-healing coatings are some of the options for this type of surface treatment (Sauvant-Moynot et al., 2008).

With the advancement of technology in recent years, the need for smart materials has increased greatly. Self-healing materials are one of the newest smart materials to hit the market, and they have some impressive properties in polymeric coatings. During the service life of a coating or composite material, it is common to undergo unexpected and unwanted defects, cracks, and failure. Mechanical damage can occur during the production process in rare circumstances. Thus, strengthening the coating's ability to withstand unwanted mechanical loads is a top issue for the development of long-lasting coating materials (E. Brown et al., 2003).

Self-healing polymeric coatings on low carbon steel usually used in the oil and gas industry, were first tested for their anticorrosion capabilities. Combining electrical field-sensitive film shapers with protective coatings and a pH close to the default layer structure resulted in self-healing metal structures (Sauvant-Moynot et al., 2008). In addition to mechanical performance, self-healing features like as electrical, optical, and physicochemical qualities are rapidly being added. In reality, maintaining the key materials' functionality may be less expensive than completely fixing them (Sauvant-Moynot et al., 2008).

1.2 Problem statement

Corrosion is one of the most harmful processes, especially for the automotive, marine, aerospace industries and oil and gas, which suffer greatly from it. There has been a concerted effort taken to reduce losses due to metal corrosion in sustainable resources and material selection. If the corrosive cannot be avoided and the repair procedure is lengthy, the cost of repair will be significant. Coating is the one of ways to protect iron from corrosion. It is because coating will give a cover to the metal as protection. While, when the coating barrier on a low carbon steel surface is disrupted, typically by a microcrack or a scratch, corrosion will begin to occur. In recent years, we've needed to develop a new approach or smart method of coating technology that allows for automatic and rapid replication of the action against mechanical damage caused by the external environment. The new coating design will act as an effective barrier, preserving mechanical qualities. Self-healing properties may aid in preventing corrosion and extending the life of the low carbon steel. The purpose of this research is to create a self-repairing layer that can protect low carbon steel from rust.

1.3 Objective of study

The main purpose of this study is to prepare and characterise of linseed mix tung oil urea–formaldehyde microcapsules and know the effect on mechanical properties of an epoxy-based coating. Specifically, the objectives are as follows:

i. To prepare self healing coating produce by linsed oil mix tung oil filled urea formaldeyde.
ii. To study the impact of microcapsule loading and size on the mechanical properties and healing ability of an epoxy-based coating was investigated.

iii. To investigate the effect of self healing in corrosion between uncoated, coating and coated low carbon steel substrate in 3.5 wt% NaCl medium.

1.4 Scope of Study

The scope of this research are as follows:

i. The purpose of this study is to identify the best healing agent composition for corrosion protection performance of self-healing coatings on low carbon steel substrates employed in the oil and gas industries.

ii. Cuting the material with dimension 20 x 20 mm size and 2 mm thickness by using laser cut machine.

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

iv. To perform self-healing on the base metal that has been coated with a healing agent-containing coating.

v. To conduct the immersion test in a 3.5wt% NaCl solution to investigate corrosion behaviour. The exposure time which are 7, 14, 21, 28, and 35 days, all samples were submerged in a NaCl solution containing 3.5 wt% NaCl.

vi. The sample have been divided into three groups: uncoated, epoxy coating, and self-healing coating, and immersed in 3.5wt% NaCl for immersion testing.

vii. To study the weight loss and corrosion rate of each substrate after immersion test.

viii. To study the corrosion behaviour on the substrate surface by using the Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray (EDX).

1.5 Significant of study

The study is to find shed light on the significance of the research. This section also discusses the analysis' utility and potential benefits. The aim of this study is to develop a self-healing microcapsule that uses linseed oil mix tung oil as both a curative and a binding agent. These microcapsule-filled healing agents have been researched for their usefulness in treating fractures produced by paints or coatings. Encapsulating functional materials in hollow microspheres is a promising method for preserving and preserving such compounds until they are needed for a specific use. The purpose is to prevent corrosion of platform coatings in oil and gas industries. Findings from this study hopefully aid oil and gas companies in to reduce corrosion on onshore and offshore platforms and pipelines.

1.6 Organization of thesis

This study is actually divided into five chapters, but for PSM 1 just need to do three chapters which are introduction, literature review and methodology. For PSM 2, this thesis is continue another two chapters which are result and conclusion. The contents of which are summarised as describe:

i. Chapter 1. Introduction. The study's background, research problems, objectives, scope, contributions, and significance are presented in this chapter.

ii. Chapter 2. Literature review. This chapter is briefing about introduction of corbon steel and more spicified to low carbon steel. Corrosion then occurs on low carbon steel. An overview coating is discussed later in this chapter. Additionally, a brief description of self-healing is included. Following that, a brief introduction to linseed oil and tung oil.

iii. Chapter 3. Methodology. This chapter discusses the process for estimating the formula of a healing agent, as well as the strategy employed in this investigation. Additionally, it had a methods section.

iv. Chapter 4. Result and Discussion. This chapter consists of a review of the study's results as well as an examination of the substrate, which is low-carbon steel, microstructural testing, hardness testing, the performance of self-healing coating, SEM/EDX testing, and immersion testing(corrosion rate and weight loss).

v. Chapter 5. Conclusion and Recommendation. This chapter is devoted to the conclusion and recommendations, which include a general conclusion regarding this study, recommendations, and the possibilities for further research on this topic.