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

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DECLARATION

I declare that this thesis entitled "Preparation and Characterization of Linseed 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 work is dedicated gratefully to all my beloved possessions.

I can write this dedication to my dear parents who have supported me from the beginning till today.

To my fellow wings, Nur Nadirah bt Dolah, who is always with me, if necessary.

I also appreciate my siblings, sisters, close friends, and classmates who have always supported me and motivated me to finish my studies with lovely words.

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ABSTRACT

Nowadays, one popular method for protecting metals against corrosion is to cover their surfaces with a suitable coating material. Corrosion is the degradation of a material's properties as a result of interactions with its environment, and corrosion of the most of metals was inevitable. Self-healing coating is considered one of the smart coatings since it has the potential to heal or repair coating damage in order to avoid additional corrosion. This coating aids in cost reduction and itcan re-self healing form corrosion occurs. The aim of this study is to create self-healing coatings out of polymeric materials and analyse their performance and corrosion behaviour when coated on steel substrates. The self-healing coating that consists of linseed oil microcapsules as healing agent. As this investigation, the self-healing coating is a barrier to protect the steel from corrosion attack. A self-healing coating has recently evolved as one of the smart coating methods used to protect steel from corrosion. Self-healing coating might repair spontaneously by itself. The capacity to self-heal may assist to avoid corrosion and guarantee a long life. The performance of self-healing coating on sample which was we utilised low carbon steel been assessed in a 3.5 wt.% NaCl solution. The mechanical testing has been done on low carbon steel sheet dimensions of 20 mm length x 20 mm width x 2 mm thickness by applying a hardness test. In this work, the fabrication of self-healing coating by in-situ polymerization of urea-formahdehyde to build microcapsules, shell that contain linseed oil as healing agent of 7.5 wt.% been created with ratio 4:1 epoxy and hardener. The immersion test had been performed by immersing the samples in a 3.5 wt.% NaCl solution and separating into three groups which was uncoating, epoxy coating, and self-healing coating in distinct containers. Each container has six samples. The immersion test has been done in 7, 14, 21, 28, and 35 days. The sample has been studied using Scanning Electron Microscope / Energy Dispersive X-Ray (SEM/EDX). The visual inspection of day 35 revealed that the weight loss measurement and corrosion rate measurement of self-healing coating had the lowest value of weight loss and corrosion rate compared to uncoating and epoxy coating. The amount of weight loss increased with each sample, with the range of weight loss varying between 0.058 to 0.151 gram rising for each sample through time. The total average of uncoating was 0.115 gram and epoxy coating was 0.090 gram, while for self healing coating was 0.075 gram. Furthermore, the different value of corrosion rate in day 7 was 0.002 (mm/years). Meanwhile the value of corrosion rate for 14, 21, 28, and 35 days shown the uncoating, epoxy coating and self healing coating was been the same value which was 0.001 (mm/years). As the conclusion, from the visual examination of the immersion test had demonstrated that the self-healing coating sample has good corrosion resistance compared to the epoxy coating and uncoating. The effectiveness of self-healing coating as corrosion resistance was shown when the scratch region on sample completely healed.

ABSTRAK

Pada masa kini, satu kaedah popular untuk melindungi logam daripada kakisan adalah dengan menutup permukaannya dengan bahan salutan yang sesuai. Hakisan ialah kemerosotan sifat bahan akibat daripada interaksi dengan persekitarannya, dan kakisan kebanyakan logam tidak dapat dielakkan. Salutan penyembuhan sendiri dianggap sebagai salah satu salutan pintar kerana ia berpotensi untuk menyembuhkan atau membaiki kerosakan salutan untuk mengelakkan kakisan tambahan. Salutan ini membantu dalam pengurangan kos dan ia boleh menyembuhkan sendiri semula bentuk kakisan berlaku. Matlamat kajian ini adalah untuk mencipta salutan penyembuhan sendiri daripada bahan polimer dan menganalisis prestasi dan kelakuan kakisannya apabila disalut pada substrat keluli. Salutan penyembuhan diri yang terdiri daripada mikrokapsul minyak biji rami sebagai agen penyembuhan. Sebagai penyiasatan ini, salutan penyembuhan diri adalah penghalang untuk melindungi keluli daripada serangan kakisan. Salutan penyembuhan sendiri baru-baru ini telah berkembang sebagai salah satu kaedah salutan pintar yang digunakan untuk melindungi keluli daripada kakisan. Salutan penyembuhan diri mungkin membaiki secara spontan dengan sendirinya. Keupayaan untuk menyembuhkan diri boleh membantu mengelakkan kakisan dan menjamin hayat yang panjang. Prestasi salutan penyembuhan diri pada sampel yang kami gunakan keluli karbon rendah telah dinilai dalam larutan NaCl 3.5 wt.% berat. Ujian mekanikal telah dilakukan pada dimensi kepingan keluli karbon rendah 20 mm panjang x 20 mm lebar x 2 mm ketebalan dengan menggunakan ujian kekerasan. Dalam kerja ini, fabrikasi salutan penyembuhan sendiri dengan pempolimeran in-situ urea-formahdehid untuk membina mikrokapsul, cangkerang yang mengandungi minyak biji rami sebagai agen penyembuhan 7.5 wt.% telah dicipta dengan nisbah 4:1 epoksi dan pengeras. Ujian rendaman telah dilakukan dengan merendam sampel dalam larutan NaCl 3.5 wt.% dan mengasingkan kepada tiga kumpulan iaitu tidak bersalut, salutan epoksi, dan salutan penyembuhan sendiri dalam bekas yang berbeza. Setiap bekas mempunyai enam sampel. Ujian rendaman telah dilakukan dalam 7, 14, 21, 28, dan 35 hari. Sampel telah dikaji menggunakan Mikroskop Elektoron Pengimbas / X-Ray Penyebaran Tenaga (SEM/EDX). Pemeriksaan visual pada hari ke-35 mendedahkan bahawa ukuran penurunan berat badan dan ukuran kadar kakisan salutan penyembuhan sendiri mempunyai nilai penurunan berat badan dan kadar kakisan yang paling rendah berbanding dengan salutan tidak salutan dan epoksi. Jumlah penurunan berat badan meningkat dengan setiap sampel, dengan julat penurunan berat badan berbeza-beza antara 0.058 hingga 0.151 gram meningkat untuk setiap sampel mengikut masa. Purata jumlah pembuka salutan ialah 0.115 gram dan salutan epoksi ialah 0.090 gram, manakala salutan penyembuhan sendiri ialah 0.075 gram. Tambahan pula, nilai kadar kakisan yang berbeza pada hari ke-7 ialah 0.002 (mm/tahun). Manakala nilai kadar kakisan untuk 14, 21, 28, dan 35 hari menunjukkan salutan tidak salutan, salutan epoksi dan salutan penyembuhan sendiri adalah nilai yang sama iaitu 0.001 (mm/tahun). Sebagai kesimpulan, daripada pemeriksaan visual ujian rendaman telah menunjukkan bahawa sampel salutan penyembuhan sendiri mempunyai rintangan kakisan yang baik berbanding dengan salutan epoksi dan tidak salutan. Keberkesanan salutan penyembuhan diri sebagai rintangan kakisan ditunjukkan apabila kawasan calar pada sampel sembuh sepenuhnya.

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LIST OF SYMBOLS AND ABBREVIATIONS

NaCl	-	Sodium chloride
NaOH	-	Sodium hydroxide
CO_2	-	Carbon dioxide
$CaCO_3$	-	Calcium carbonate
CeO_2	-	Cerium(IV) oxide
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	-	Mililiter
g	-	Gram
cm	-	Centimeter
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

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Corrosion is usually produced by corrosive agents behaving as waste metals. On the other hand, the deterioration of a substance caused by contact and its environmental effect is a wider definition. Corrosion is a natural process that occurs in non-metallic materials including such concrete and polymers, and also during crushing operations (Schofield, 2002). The most frequent types of corrosion are electrochemical processes. When the majority or all of the atoms on a metal surface oxidise, the whole surface deteriorates. The majority of metals seem to lose electrons to oxygen and other molecules when exposed to air or water. This results in the formation of a metal oxide, whereas oxygen (which generates electrons) is decreased. When various metal kinds come into touch with one other, the process is known as galvanic corrosion. Between two electrical contacts with applied electric tension, water or other moisture produces electrolytic corrosion, which is particularly prevalent in electronic equipment. It would result in the formation of an electrolyte cell through mistake. Many regularly used kinds of corrosion prevention exist, such as physical shielding or careful monitoring of the corrosion reaction, but regrettably, very few options are suited or practicable in oral circumstances. In fact, once corrosion has started, it seems to be self-perpetuating (Schofield, 2002).

To build a thick barrier against corrosive species, the metal surface is often strengthened with polymer coating solutions. In addition to coatings, cathodic protection is employed in a number of applications to prevent the metal structure from corrosion in the event of coating damage (Sauvant-Moynot *et al.*, 2008). Self-healing technology is a new generation of technology that has the potential to substantially enhance product efficiency, including reliability and lifetime. The scientific community's interest in self-healing polymers and polymer composites has grown. Numerous repairing methods have been developed. The study first focused on the anticorrosion effectiveness of self-healing polymeric coatings on low-carbon steel intended for use in the oil and gas industry. By integrating electrical field-sensitive film shapers with protective coatings and a pH near to the default layer structure, self-healing metal structures were produced (Sauvant-Moynot et al.2008). Self-healing benefits are rapidly being extended beyond mechanical performance to include electrical, optical, and physicochemical properties, for example. In reality, restoring the main materials' functioning may be less costly than repairing the materials entirely.With the recent growth of self-healing science and technology, research on the transition of useable materials and associated characterisation approaches has begun. For example, the automobile industry has developed self-healing exterior painting that removes defects on low carbon steel surfaces as a matter of aesthetic need through the viscoelasticity-driven plastic deformation of basic polymers (Sauvant-Moynot *et al*.2008).

1.2 Problem Statement of Study

Corrosion always happens when the coating barrier of a low carbon steel surface is mechanically destroyed as a result of a microcrack or scratch on the surface. When the damage occurs, the corrosive species will penetrate to the surface of the low carbon steel substrate, and the coating will need a specific feature that has the potential to self-heal in order for the damage to be healed automatically. If the corrosive can not be avoided and the mending procedure takes time, the cost of fixing the damage will be considerable. In recent years, we have needed to build a new technique or smart means of coating technology so that the action against mechanical damage caused by the external environment may be reproduced automatically and quickly. The new coating design will serve as a good mechanical property protecting barrier and will have the capacity to preserve mechanical properties. The capacity to self-heal may help to avoid corrosion and extend the life of low carbon steel. The goal of this study is to create a smart self-healing coating that can prevent corrosion on low carbon steel.

1.3 Objective of Study

The main aim of this study shown as below:

i. To develop self healing coating consist of linseed oil as healing agent.

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

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

1.4 Scope of Study

The scope of this study consists:

i. To study the best formulation of healing agent to perform as anti-corrosion performance of self-healing polymeric coatings on low carbon steel substrate for oil and gas industries application.

ii. Using laser wire cut machines to cut the material into the size with 20 x 20 mm size and 2 mm thickness.

iii. To perform mechanical testing on the substrate by using hardness test and optical microscope.

iv. To perform the self healing performance at the base metal that was coated by the coating that consist the healing agent.

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

vi. Will be divide the sample into three group which is uncoated, epoxy coating and self-healing coating will be immersed in 3.5wt% NaCl for immersion test.

vii. All samples were immersed in the NaCl medium of 3.5wt% for 7, 14, 21, 28 and 35 days.

viii. Using Scanning Electron Microscope (SEM) and EDX to study the corrosion behavior on the substract after corrosion test.

1.5 Significant of Study

The study findings offer insight on the study's significance. This section also addresses the analysis's relevance and possible advantages. The purpose of this research is to create a self-healing coating utilising linseed oil as both a cure agent. The effectiveness of these microcapsule-filled healing agents in the treatment of cracks caused by paints or coatings has been studied. Encapsulating functional materials in hollow microspheres is a desired technique to keep and keep such compounds from being kept until they are required for suitable application. The purpose is to assist industrial oil and gas companies to prevent corrosion on platform coatings. This study's findings seek to assist the oil and gas sectors in reducing corrosion on platforms, pipelines, onshore or offshore.

1.6 Organization of Thesis

Based on the previously stated goals and the methodology suggested, this thesis is divided into three (3) chapters for PSM 1, the contents of which are summarised as follows:

1. Chapter 1. Introduction. This chapter discusses the study's background, research problems, objectives, scope, contributions, and significance.

2. Chapter 2. Literature review. This chapter about begin with a brief introduction to low carbon steel. Then, corrosion on low carbon steel occur. Later in this chapter, an overview coating is described. Additionally, a short overview on self-healing. After that, a brief overview of linseed oil.
3. Chapter 3. Methodology. This chapter provide the methodology used to estimate the formula of a healing agent, which includes a description of the technique used in this analysis. Also, it detailed the research methodology.
4. Chapter 4. Expected results. This chapter about result and discussion of this study, included study of substrate which is low carbon steel, microstruture test, hardness test, performance of self healing coating and immersion test.
5. Chapter 5. Conclusion and recommendation. This chapter is about conclusion and recommendation which is overall conclusion about this study, recommendation and the potential to develop more on this topic of study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of Carbon Steel

Carbon steel is among the most frequently utilized materials in contemporary civilization. It is critical to define carbon steel in both the general sense and the specific of this research. Steel is often used to refer to an iron-based alloy having less than 2% carbon (Gandy, 2007). Carbon steels (occasionally referred to as plain carbon steels, ordinary steels, or straight carbon steels) are steels that contain no elements other than carbon in residual quantities, except for those added for deoxidation (such as silicon and aluminium) and those added to mitigate the detrimental effects of residual sulphur (such as manganese and cerium). As a result, it would be pure iron in the absence of carbon. By carbonizing steel, it becomes stronger and more durable. For this reason, many companies prefer or choose conventional steel over historical iron. However, not all metals in all goods have the same carbon to iron ratio, since certain steels have a higher carbon to iron ratio than others. The table below categorizes steels into three groups based on their carbon content: low carbon steel, medium carbon steel, and high carbon steel (Callister, William D., 2014). The following table compares their carbon content, microstructure, and characteristics:

Table 2.1 Carbon content, microstructure and characteristics of carbon steel (Gandy, 2007).

Steel Type	Carbon Content (wt%)	Microstructure	Characteristics
Low carbon steel	$0.04 \leq$ Carbon < 0.25 Sulphur - 0.055% Phosphorus - 0.055%	Ferrite and pearlite	Low hardness and cost. High ductility, toughness, machinibility and weldability