



**COMPARATIVE STUDIES OF TUNG AND LINSEED OIL  
MICROCAPSULE AS SELF-HEALING COATING FOR  
CORROSION PROTECTION OF LOW CARBON STEEL  
SUBSTRATES**

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**BACHELOR OF ENGINEERING TECHNOLOGY  
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**Faculty of Mechanical and Manufacturing Engineering  
Technology**



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CARBON STEEL SUBSTRATES**

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**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**2023**

## DECLARATION

I declare that this thesis entitled “Comparative Studies of Tung and Linseed Oil Microcapsule as Self-Healing Coating for Corrosion Protection of Low Carbon Steel Substrates ” 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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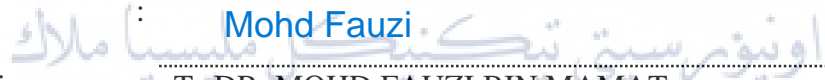
## APPROVAL

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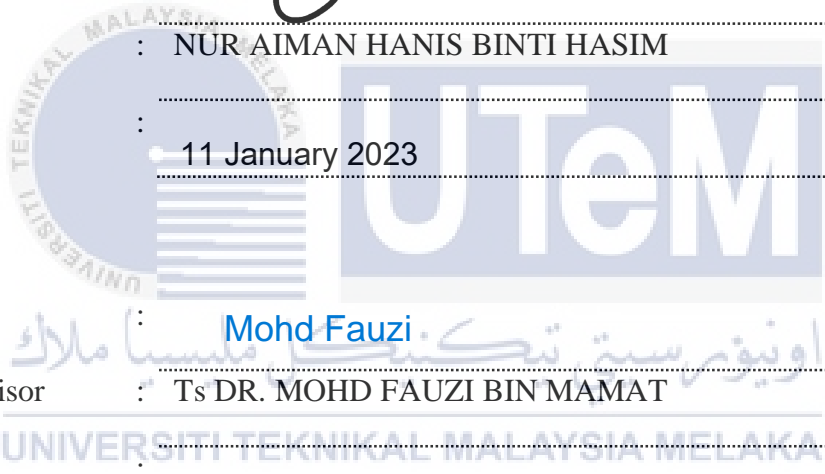
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## **DEDICATION**

*Any difficult activity necessitates both self-effort and the guidance of elders, particularly those dear to my heart.*

*My grateful heart and small effort are dedicated to my great and loving family, particularly my mother, father, and all my siblings.*

*Whose love, which is a constant source of inspiration, encouragement, and prayers, has enabled me to reach such glory and success.*

*Including my extensive training and admiration for my supervisor and co-supervisor, Mdm.*

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*Not to mention my supportive friends and colleagues who shared their knowledge and provided me with fantastic guidance on how to conduct this research.*

*Million Thanks to all.*

## ABSTRACT

Corrosion, the gradual breakdown of a substance due to its contact with its environment, can happen at any stage of the refining process for petroleum and natural gas. Polymer coatings have traditionally served as a barrier against corrosion species, shielding the surface of metallic materials from corrosive attack. In fact, this concept is almost always used in reference to metal alloys. However, the corrosion process cannot be avoided if the barrier layer is mechanically broken, and corrosive species can penetrate the metal surface. One of the most significant properties required of the coatings is the ability to self-heal, which means that a chemical component of the coating will naturally restore the damaged surface. Self-healing protective coatings, which can automatically cure damage, have recently been actively explored to lengthen material lifetime, reduce maintenance costs, and improve public safety. In this study, the main objective is to develop a self-healing coating using tung and linseed oil as a healing agent and analyse the corrosion behaviour of uncoated and coated low-carbon steel substrates in 3.5 wt.% sodium chloride (NaCl). The second objective is to investigate the effectiveness between tung oil and linseed oil as self-healing agent coating on a low carbon steel substrate. Mechanical testing, namely a hardness test, has been performed on a low carbon steel substrate of 20 mm in length, 20 mm in width, and 2 mm in thickness. After that, the microstructure study of low carbon steel substrate has been analyse using a microscope. The microcapsule was prepared by in-situ polymerization of urea-formaldehyde (UF) to construct a microcapsules shell containing linseed and tung oil in self-healing coating formulation on 7.5 wt.% of microcapsule which has been added in the coating. The samples have been submerged in a 3.5 wt.% NaCl solution and sorted into four containers namely uncoated, epoxy coated, linseed oil self-healing, and tung oil self-healing for 7, 14, 21, 28, and 35 days of immersion test. The Attenuated total reflectance - Fourier-transform infrared spectroscopy (ATR-FTIR) process is used to detect structural changes in biomolecules for epoxy, linseed oil, and tung oil. Scanning electron microscopy and energy dispersive X-ray spectroscopy (SEM/EDX) analysis has been performed for surface morphology study after immersion test. The results demonstrated that when comparing uncoating and epoxy coating, linseed oil self-healing coating had the lowest values for weight loss and corrosion rate. By measuring the weight loss and corrosion rate, we were able to determine which healing agent is more efficient. After 35 days of the immersion test, linseed oil shows a 0.018g of weight loss average while tung oil is 0.020g. The EDX analysis after 35 days of immersion reveals that 83.14 wt.% of iron (Fe) exists in the uncoated sample, 53.15 wt.% in the epoxy coating, 18.79 wt.% in the self-healing coating tung oil, and zero occurrence of iron (Fe) in the self-healing coating linseed oil. Based on the value, linseed oil self-healing coating was more effective at repairing the scratched area of the material. After conducting the research, it was revealed that linseed oil is more effective than Tung oil as a self-healing agent, and the corrosion behaviours of uncoated and coated low-carbon steel substrates were identified after immersion in 3.5 wt.% NaCl. The application on this study is suitable in oil and gas industries.

## ABSTRAK

Hakisan, pecahan beransur-ansur bahan akibat sentuhan dengan persekitarannya, boleh berlaku pada mana-mana peringkat proses penapisan untuk petroleum dan gas asli. Salutan polimer secara tradisinya berfungsi sebagai penghalang terhadap spesies kakisan, melindungi permukaan bahan logam daripada serangan menghakis. Konsep ini selalu digunakan merujuk kepada aloi logam. Walau bagaimanapun, proses kakisan tidak dapat dielakkan jika lapisan penghalang dipecahkan secara mekanikal, dan spesies menghakis boleh menembusi permukaan logam. Salah satu sifat paling penting yang diperlukan untuk salutan ialah keupayaan untuk menyembuhkan sendiri, yang bermaksud komponen kimia salutan akan memulihkan permukaan yang rosak secara semula jadi. Salutan swa sembuh boleh menyembuhkan kerosakan secara automatik, baru-baru ini telah diterokai secara aktif untuk memanjangkan hayat bahan, mengurangkan kos penyelenggaraan dan meningkatkan keselamatan awam. Dalam kajian ini, objektif utama adalah untuk membangunkan salutan swa sembuh menggunakan tung dan minyak biji rami sebagai agen penyembuhan dan menganalisis tindak balas kakisan substrat keluli karbon rendah yang tidak bersalut dan bersalut dalam 3.5 wt.% natrium klorida (NaCl). Objektif kedua, untuk menyiasat keberkesanan antara minyak tung dan minyak biji rami sebagai salutan agen penyembuhan sendiri pada substrat keluli karbon rendah. Ujian mekanikal dalam bentuk ujian kekerasan, dilakukan pada substrat keluli karbon rendah dengan panjang 20 mm, lebar 20 mm, dan ketebalan 2 mm. Kajian struktur mikro substrat keluli karbon rendah dianalisis menggunakan mikroskop. Mikrokapsul disediakan melalui pempolimeran in-situ urea-formaldehid (UF) untuk membina cangkerang mikrokapsul yang mengandungi minyak biji rami dan tung dalam perumusan salutan swa sembuh pada 7.5 wt.% mikrokapsul yang ditambah dalam salutan. Sampel direndam dalam larutan NaCl 3.5 wt.% dan diasingkan ke dalam empat bekas selama 7, 14, 21, 28, dan 35 hari rendaman ujian. Proses Attenuated total reflection - Fourier-transform infrared spectroscopy (ATR-FTIR) digunakan untuk mengesan perubahan struktur dalam biomolekul untuk epoksi, minyak biji rami dan minyak tung. Pengimbasan mikroskop elektron dan analisis spektroskopi sinar-X (SEM/EDX) penyebaran tenaga dilakukan untuk kajian morfologi permukaan selepas ujian rendaman. Keputusan menunjukkan apabila membandingkan sampel tidak bersalut dan salutan epoksi, salutan swa sembuh minyak biji rami mempunyai nilai terendah untuk penurunan berat dan kadar kakisan. Dengan mengukur penurunan berat dan kadar kakisan, kami dapat menentukan agen penyembuhan yang lebih berkesan. Selepas 35 hari ujian rendaman, minyak biji rami menunjukkan purata penurunan berat sebanyak 0.018g manakala minyak tung ialah 0.020g. Analisis EDX selepas 35 hari rendaman mendedahkan bahawa 83.14 wt.% besi (Fe) wujud dalam sampel tidak bersalut, 53.15 wt.% dalam salutan epoksi, 18.79 wt.% dalam minyak tung salutan swa sembuh, dan kejadian sifar besi (Fe) dalam minyak biji rami salutan swa sembuh. Berdasarkan nilainya, salutan swa sembuh minyak biji rami adalah lebih berkesan untuk membaiki kawasan bahan yang tercalar. Selepas menjalankan penyelidikan, didapati bahawa minyak biji rami lebih berkesan daripada minyak Tung sebagai agen penyembuhan diri, dan tingkah laku kakisan substrat keluli karbon rendah yang tidak bersalut dan bersalut dikenal pasti selepas rendaman dalam 3.5 wt.% NaCl. Kajian ini sesuai penggunaannya dalam industri petroleum dan gas asli.



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## LIST OF SYMBOLS AND ABBREVIATIONS

NaCl	-	Sodium chloride
NaOH	-	Sodium hydroxide
CO <sub>2</sub>	-	Carbon dioxide
CaCO <sub>3</sub>	-	Calcium carbonate
CeO <sub>2</sub>	-	Cerium (IV) oxide
HCl	-	Hydrochloric acid
wt. %	-	Weight percentages
SEM	-	Scanning Electron Microscope
EDX	-	Energy Dispersive X-Ray
rpm	-	Revolutions per minute
pH	-	Measurement level of acidity
mm	-	Millimetre
ml	-	Millilitre
g	-	Gram
cm	-	Centimetre
L	-	Litre
AISI / SAE	-	American Iron and Steel Institute / Society of Automotive Engineers
ASTM	-	American Society for Testing and Materials
HSLA	-	High-strength low-alloy steel
SEM	-	Scanning Electron Microscope
EDX	-	Energy Dispersive X-Ray
ATR-FTIR	-	Attenuated total reflectance - Fourier-transform infrared spectroscopy

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of study

Metal corrosion is slowly becoming a focal point of worldwide industrial development. Corrosion is a hazardous and expensive condition. It has the potential to cause the collapse of buildings and bridges, the rupture of oil pipelines, the leakage of chemical facilities, and the flooding of toilets. Corroded electrical contacts can create fires and other issues, rusted medical implants could affect blood poisoning, while air pollution has rusted works of art across the world (Landolfo et al., 2010). There is no one-size-fits-all approach for preventing metal parts from corroding. Manufacturers must utilise a variety of strategies to prevent and control corrosion in different metals because there are so many different types of metal and so many diverse applications. Electrochemical corrosion is accelerated by the high humidity and high salt content of the marine atmosphere, such as an offshore oil production platform, which produces a thin film of salt on metal surfaces (H. Zhang et al., 2021).

There are five techniques to manage corrosion, one of the techniques is cathodic protection. The shielded metal is protected by being attached to a more easily corroded "sacrificial metal," which causes corrosion more readily (as the anode) while protecting the more valuable metal object in concern (as the cathode) (Krystal Nanan, 2020). The next step is to choose a material that is environmentally friendly and has strong corrosion resistance. Then, to manage corrosion, design selection also is one of the methods to prevent a corrosion.

The engineering stage is where corrosion control begins. Manufacturers should design items considering corrosion resistance in mind if it will be used in corrosive environments. Furthermore, Corrosion control method involving the application of a thin coating of a metal with a lower (than the protected metal) electrode potential (higher position in the table of Electrochemical series). In addition, is a corrosion inhibitor, which is the process of preventing corrosion by adding compounds that, when added in small amounts, can dramatically retard corrosion. Internally, inhibition is employed as a cost-effective control option for stainless steels and alloys, as well as coatings on nonmetallic components (Ahmad, 2006).

The coating is the major means of corrosion control for ships and other offshore installations. The coating is a layer of material deposited onto a substrate to enhance the surface properties for corrosion and wear protection. Metal plating and organic coating are the two most used ways of preventing air corrosion at the moment. Because metal platings are not limited by the shape of materials and the procedure is easy, it was frequently employed. However, because salt spray in the maritime environment rapidly enters the porous nature of plantings, causing metal corrosion, the usage in the marine environment is limited. The organic coating effectively prevents water, oxygen, and corrosive media from coming into contact with metal components, preventing the formation of an electrical circuit for electrochemical corrosion of the metal beneath the coating and inhibiting corrosion, and thus becoming the primary method of marine atmospheric corrosion protection (H. Zhang et al., 2021). Scientists have devised a model of self-healing materials based on the notion of organisms self-healing after being harmed.

There are two types of self-healing coatings: intrinsic and extrinsic. Intrinsic-type coatings have a latent self-healing capability, which means the coating matrixes can mend themselves. The microencapsulated is healing agent is incorporated with the coating matrix in extrinsic-type self-healing coatings (Yang & Huang, 2015). Rupture of the microcapsules results in the release of a healing agent, which then fills the injured area and initiates the healing response. When compared to an intrinsic technique without microcapsules, a microcapsule-based coating system has a significant benefit.

In this study, a two types of organic coating material, which is Linseed oil and Tung oil is used as an agent for self-healing coating on low-carbon steel substrate in order to make a comparative study between these two types of organic material. Linseed oil is also known as drying oil, which means it hardens to a solid film following exposure to air: in the presence of atmospheric oxygen, linseed oil undergoes a crosslinking reaction. Linseed oil has received a lot of interest as a healing agent for microcapsule-type self-healing coatings because of its good film-forming ability, environmental friendliness, and inexpensive cost. Tung oil is distinguished by the presence of a high proportion of eleostearic acid glycerides. Each eleostearic acid molecule has three carbon-carbon double bonds, making it easier for the oil to dry in the atmosphere (Kim et al., 2018). Thus, tung oil is an ideal core material for its self-drying with oxygen with no need for catalysts.

## 1.2 Problem statement

Metal corrosion is primarily caused by two factors: the material and its surroundings. Corrosion, in particular, is described as the degradation of a material, usually a metal, as a result of a reaction with its surroundings. Coatings designed to resist rust and corrosion keep your component safe from the dirt and chemicals that shorten its useful life. Coatings are useful because they protect against the elements, make products last longer, and improve their aesthetics and functionality. In service, corrosion occurs when the coating barrier of a low carbon steel surface is mechanically eroded as a result of a surface microcrack or scratch. When the damage occurs, the corrosive species will penetrate to the low carbon steel substrate's surface, necessitating the use of a coating with the ability to self-heal in order for the damage to be automatically repaired. The cost of repairing the harm will be significant if the corrosive cannot be avoided and the repairing operation takes time. The development of a new technique or smart ways of coating technology in recent years takes the action against mechanical and the new coating design will act as a mechanical property barrier and will have the ability to maintain mechanical properties. The ability to self-heal may aid in the prevention of corrosion and the extension of the life of low-carbon steel. The purpose of this study is to develop a smart self-healing coating using Tung and Linseed oil as an healing agent that can protect low carbon steel from corrosion damage caused by the external environment can be replicated automatically and make a comparison of the effectiveness between tung oil and linseed oil as a self-healing agent.

### 1.3 Objective of study

The main objective of this studies is:

- i. To prepare a self-healing coating consist of Linseed and Tung oil as healing agent.
- ii. To analyse the corrosion behaviour of uncoated and coated low carbon steel substrates in Sodium chloride (NaCl).
- iii. To investigate the effectiveness between Tung oil and Linseed oil as a self-healing agent coating on low carbon steel substrate.

### 1.4 Scope of study

The scope of this research are as follows:

- i. To determine the best healing agent formulation for performance of anti-corrosion in self-healing polymeric coatings on low carbon steel substrates for use in the oil and gas sectors.
- ii. Laser cut machine were used to cut the material into  $20 \times 20$  mm squares with a 2 mm thickness.
- iii. An immersion test in 3.5wt% NaCl solution was used to investigate corrosion behavior. For 7, 14, 21, 28, 35 days, all samples were submerged in a 3.5wt % NaCl medium.
- iv. Uncoated, epoxy coating, and self-healing coating (Tung and Linseed oil as a healing agent) samples will be divided into four groups and immersed in 3.5wt % NaCl for immersion testing.
- v. For 7, 14, 21, 28, 35 days, all samples were submerged in a 3.5wt % NaCl medium.