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Preparation of Linseed Oil-Filled Urea–Formaldehyde Microcapsules and Anti-Corrosion Performance of Self-Healing Epoxy Coatings on Low Carbon Steel Substrate

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ABSTRACT

This investigation utilised the in-situ polymerisation method to generate urea-formaldehyde (UF) microcapsules that were filled with linseed oil. Microcapsules with a mean diameter of 20-200 µm were obtained. The self-healing coating was constructed by incorporating 7.5wt. % microcapsules into an epoxy matrix and subsequently applying it to the low carbon steel substrate. The impact of microcapsules on the coating properties was examined through an immersion test in a 3.5wt. % NaCl medium for 7, 14, 21, 28, and 35 days. Weight loss and corrosion rate measurements were taken to investigate the effect of embedded microcapsules on the anticorrosive properties of the self-healing coating. Based on visual inspection, mass loss, and corrosion rate results, the complete self-healing coating demonstrated superior anticorrosive properties to the epoxy coating without microcapsules. The scratched self-healing coating's failure was investigated using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). Overall, the results and tests showed that the self-healing coating is more corrosion-resistant than the coating without microcapsules.

Keywords: *Linseed oil, Microcapsule, Self-healing coating, Corrosion protection*

1. INTRODUCTION

Low carbon steels are used in a wide range of industrial applications due to their mechanical properties, ease of welding, availability, and low cost [1]. Low carbon steels are widely used in the marine and offshore industries, including shale gas energy, for ships, plants, and pipe construction. However, drawbacks such as low hardness and poor corrosion resistance limit their application potential, particularly in the oil and gas application [1, 2]. The effectiveness of “smart” or “trendly” coating for corrosion protection relies on the specific formulation or secret recipe of the coating. This allows for the controlled release of active substances when needed, or a combination of different mechanisms to repair cracks and restore the coating without any external interference. These responses are triggered by environmental stimuli when corrosion processes take place.

The idea of smart coatings has progressed from modified coatings with specific functions to coatings containing inert ingredients that provide distinct properties (not present in conventional coatings), and subsequently to coatings that can detect and react to environmental changes reliably and predictably [3–5].

Self-healing coatings are a type of smart coatings that can protect metal substrates from corrosion and repair themselves after sustaining damage. This allows the coating to regain its original properties to a significant extent. The mechanism of coatings can repair physical damage or restore practical performance with minimal or no interference [4]. “Self-healing coatings” are distinguished from conservative non-healing coatings, which are primarily designed to passivate metal substrates against corrosive environments, by their ability to restore coating properties. In recent years, the term “self-healing coatings” is commonly used to refer to coatings that have the capacity to repair a defect or imperfection. However, the term “active anticorrosion coatings” is used to describe coatings that contain preloaded energetic ingredients, such as corrosion inhibitors, that autonomously safeguard the metal surface from corrosion issues [5, 6]. Investigators view both classes as “self-healing coatings”, which can provide a range of benefits and multiple ways to achieve protective coatings with various mechanisms, as illustrated in Figure 1.

Plentoful vegetable oils are one of the most prevalent alternative "green" methods. Drying oils, including linseed and tung oil, can be used to create self-healing coatings. These oils cross-link with the oxygen in the atmosphere,

forming a protective layer between subtract and the environment [6-9]. Expensive catalysts are unnecessary for the drying of oils. They are common and inexpensive active agents for self-healing polymer coatings. The primary method of oil microencapsulation involves the in-situ polymerisation technique, which guarantees an impenetrable protective wall, superior mechanical properties, high stability, and moisture resistance [10-11]. Linseed and tung oil are the two most frequently employed drying oils.

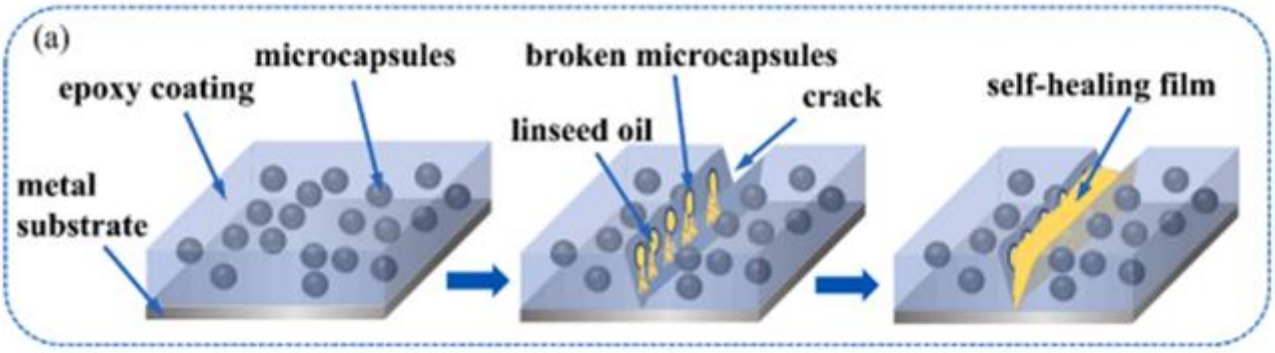


Figure 1. Schematic diagram of self-healing and anti-corrosion mechanism of epoxy coating containing microcapsules [3].

Linseed oil is a type of oil that dries and forms a solid film when exposed to air. This reaction occurs due to the presence of atmospheric oxygen [6]. Linseed oil has gained significant interest as a healing agent for microcapsule-type self-healing coatings. Its excellent film-forming ability, environmentally friendly properties, and affordability have made it a popular choice. The majority of self-healing protective coatings that are based on linseed-oil-loaded microcapsules have been developed for metal protection thus far [7-10, 12, 22]. Linseed-oil-based microcapsule-type self-healing coatings for cementitious materials have not been reported, as far as we are aware.

The objective of this study is to create self-healing coatings using microencapsulated linseed oil. This study involved the synthesis of microcapsules using urea-formaldehyde as a shell and linseed oil as a core through in situ polymerisation [3, 9, 15, 16]. The study focused on examining the effectiveness of these microcapsules in repairing cracks or scratches that are present in an epoxy coating. The study examined the corrosion resistance of scratched coatings on a low carbon steel substrate through immersion testing in 3.5wt.% NaCl media. This was done to verify the self-healing property over a period of 7, 14, 21, 28, and 35 days.

2. MATERIAL AND METHODS

The current study focused on a profitable low carbon steel substrate. Upon receipt, the material was in the form of a 3 mm thick plate with a ferrite-pearlite structure. Table 1 displays the composition of the metal substrates utilised in this study, specifically low carbon steel. A sheet of low carbon steel measuring 20 × 20 mm and with a thickness of 3 mm was laser cut, as depicted in Figure 2. The substrate material was coated on one side with the investigated coating, while the other side was coated with a layer of paraffin wax to avoid contact with the medium.

Table 1 Chemical composition (wt.%) of the low carbon steel substrate

Alloy element	C	Si	Mn	P	S	Cr	Ni	Cu	Fe
Base metal	0.16	0.18	0.52	0.037	0.026	0.13	0.087	0.33	Balance

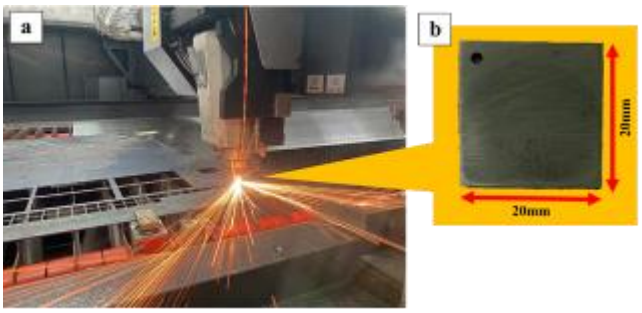


Figure 2. (a) Performing laser cutting to low carbon steel and (b) low carbon steel.

The substrate surface preparation of the experimental coupons for corrosion testing involves abrasion with silicon carbide papers of various grit sizes (80, 100, 300, 500, 800, 1000, and 2000). The coupons are then rinsed with distilled water, subjected to sonicated to remove any remaining particles, soaked in ethanol at room temperature for 20 min, dried, and stored in a desiccator to prevent atmospheric corrosion before use.

Microcapsules were synthesised through in-situ polymerisation within an oil-in-water emulsion. A mixture was prepared by combining 160 ml of deionised water and 10 ml of a 5wt. % aqueous solution of polyvinyl alcohol (PVA) in a 1000 ml beaker at room temperature. While in a state of agitation, a solution was formed by dissolving 5g of urea, 0.5g of ammonium chloride, and 0.5g of resorcinol.

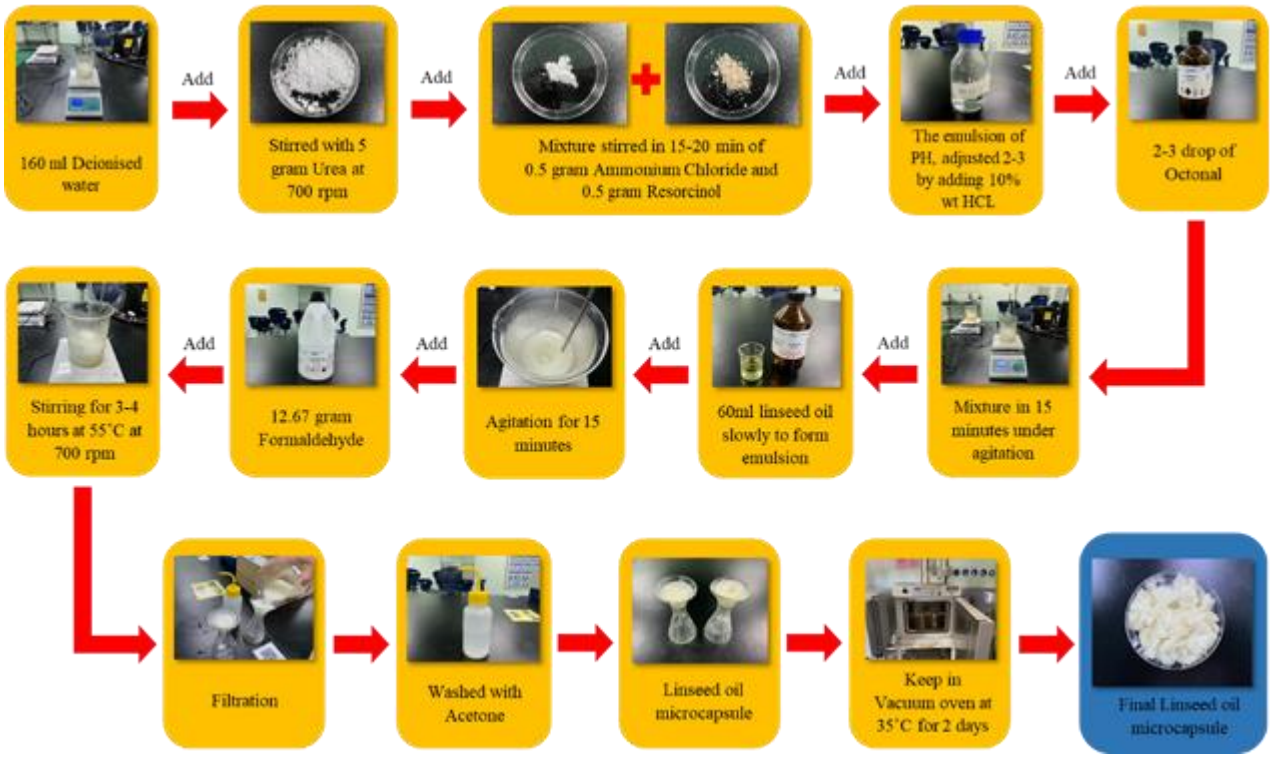


Figure 3. Process of the preparation of microcapsules.

The pH was modified to approximately 3.5 by utilising a 5wt.% solution of hydrochloric acid in deionised water. As an antifoaming agent, one to two drops of octanol were incorporated. A slow addition of 70 ml of linseed oil was made to create an emulsion, which was then allowed to stabilise for 15 min while being agitated. After stabilisation, a 37wt. % aqueous solution of formaldehyde was gradually added at a rate of 12.67 g. After being covered, the emulsion was gradually heated and maintained at 55°C while being stirred at 700 rpm for 4 hours. The contents were cooled at room temperature. Microcapsules were recovered from the suspension using vacuum filtration. To eliminate the suspended oil, the objects were rinsed with water and washed with xylene [6-16]. The capsules were dried using a vacuum oven. Figure 3 shows the overall flow process.

The linseed oil (LO) microcapsule was embedded in the epoxy matrix with a 7.5 wt. % selection. The epoxy and LO microcapsule were mixed at room temperature with gentle agitation. Before being applied to the substrate material, the epoxy-containing the LO microcapsule was continuously agitated for 30 min. The coating materials were applied directly to 20 × 20 mm steel substrates using a brush paint application method. The coated specimens were left undisturbed for 7 to 14 days to dry and cure completely. Before the immersion test, all samples were cross-scratched. The scratches are intended to determine whether the self-healing coating can heal or repair coating damage while also protecting the sample substrate better than a pure epoxy coating.

Immersion testing was performed in accordance with ASTM G1-03. All coated and uncoated specimens were immersed in a container with 5 litres of 3.5wt.% NaCl medium for 7, 14, 21, 28, and 35 days. Following each immersion test, the samples were removed from the 3.5 wt.% NaCl solution and soaked to remove the corrosion products. Photos were taken before and after cleaning as part of a visual inspection study. The samples were then rinsed with distilled water, dried under high-pressure air, and weighed. The corrosion rate was calculated by using Equation 1.

$$\text{Corrosion Rate (CR)} = \frac{K \times W}{A \times T \times D} \quad (1)$$

where CR is the corrosion rate (mm/ year), constant, K = 8.76×10^4 , W is the mass loss (g), A is the surface area (cm^2), exposed to the corrosive media, T is the exposure time (hour), d is the density (g/cm^3). Scanning Electron Microscope (SEM) coupled with Energy Dispersive Spectroscopy (EDS) was used to identify the surface analysis for studying the corrosion behaviour on coated and-self-healing coating.

3. RESULTS AND DISCUSSION

3.1 Mechanical Test on Low Carbon Steel Substrate

An optical microscope and Rockwell hardness test are used to conduct microstructural and hardness analysis. Figure 4 (a-c) displays the microstructure of the substrates following etching with a 2% Nital solution under an optical microscope at varying magnifications. When examined

closely, the etching solution provides a more distinct perspective of the grain boundary. The microstructure of the sample consisted of pearlite and ferrite structures. The darker region is pearlite, while the brighter region is ferrite. Low carbon steels are typically characterised by their low strength and relative softness. Nevertheless, they are highly ductile, which renders them ideal for machining and welding. Additionally, they are cost-effective for oil and gas applications.

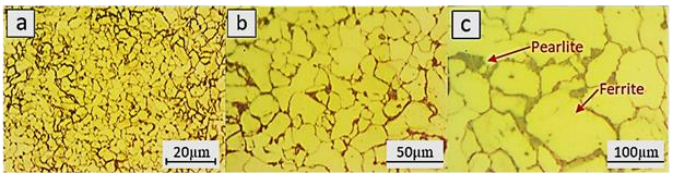


Figure 4. Microstructure of low carbon steel as substrates under optical electron microscope by (a) 20×, (b) 50× and (c) 100× magnification.

The average hardness value is approximately 60 Hv. The value remains within the Rockwell B range for the hardness test when compared to the standard. It is evident that the metal substrates are low carbon steel, as indicated by both results. In general, low carbon steels contain less than 0.25% carbon and cannot be strengthened through heat-treating; instead, they can be strengthened through cold working. The low carbon material is relatively soft and weak, but it possesses exceptional ductility and toughness properties.

3.2 Microcapsule Properties

The size of microcapsules in self-healing coatings affects the amount of healing agent available for delivery to the cracked or scribed area. Figure 5 (a-c) depicts the average diameter of the microcapsules, which ranges from 180 to 200 µm. The amount of microcapsule added was approximately 7.5 wt. % of the total self-healing coating material. According to a previous study by Hatami et al. [19], the optimum concentration of microcapsules is 10 to 20wt. %, because when the concentration of microcapsules is too low, the healing agent delivered to the damaged region is insufficient, whereas when the concentration of microcapsules is too high, the coating porosity increases and density decreases, which promotes to poor water resistance.

SEM micrographs provide additional details about the sub-micron debris and surface features of the microcapsules (Figure 5 (c)). The microcapsules had a mostly round shape, and their outer shell was rough and not porous, similar to the ones studied in [15-19]. The microcapsules' rough morphology allowed for strong mechanical bonding to the coating matrix. The capsules' spherical shape allowed for efficient storage and easy dispersion into the coating. As mentioned by Benzab et al. [16], the appearance of microcapsules is greatly influenced by the properties of the core material (such as viscosity, surface tension, miscibility with the shell material, etc.), the ratio of core to shell, and the process of microencapsulation [9, 17]. Figure 5 (d) shows the EDS spectrum of a linseed oil (LO) microcapsule, which contains carbon, nitrogen, and oxygen due to the composition of linseed oil and poly-urea formaldehyde (PUF) as the shell of the microcapsule. Figure 5 (e) depicts the visual appearance of a microcapsule after one week of filtration and drying.

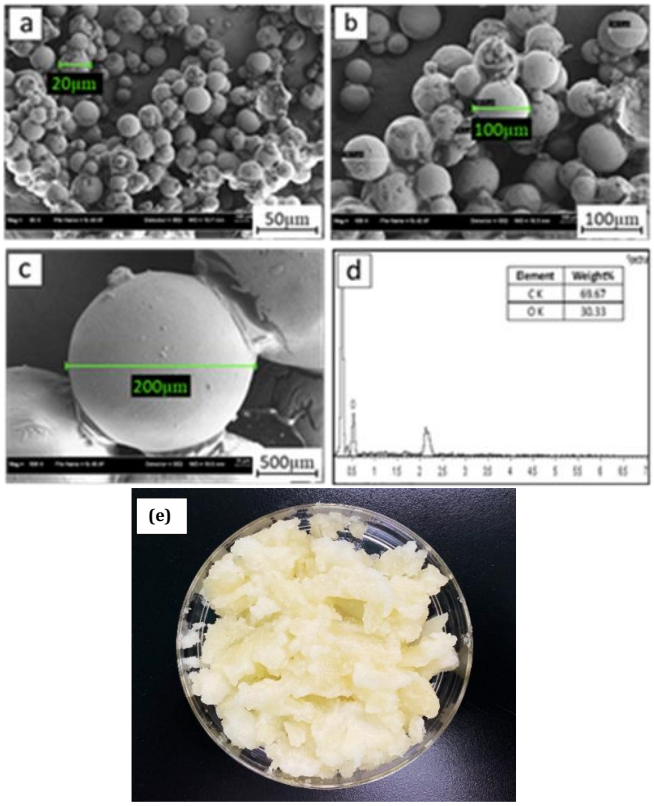


Figure 5. SEM micrograph of microcapsules (a) 50×, (b) 100× and (c) 500× magnification and (d) the element contained in linseed oil microcapcule by EDX analysis (e) The image of linseed oil microcapsules after filtration.

3.3 Self-Healing Performance

In order to achieve optimal healing performance, it is crucial for the microcapsules present in the epoxy coating to promptly rupture and release the healing agent whenever cracks or any form of damage occur on the coating surface. According to the SEM/EDX observation, the surface of the microcapsules' shell varied in texture, with some being rough and others being smooth. The rough surface is expected to facilitate strong bonding with the epoxy coating matrix. Figure 6 (a and b) displays the healing observed in a coating layer recorded under SEM/EDX, both with and without the presence of microcapsule. Based on the observation, it was noted that the crack had an initial maximum open length of ~135.1 µm (Figure 6 (a1)), with no protection or exposure to the environment. The self-healing coating demonstrates exceptional protection, with minimal re-healed area measuring ~ 96.18 µm.

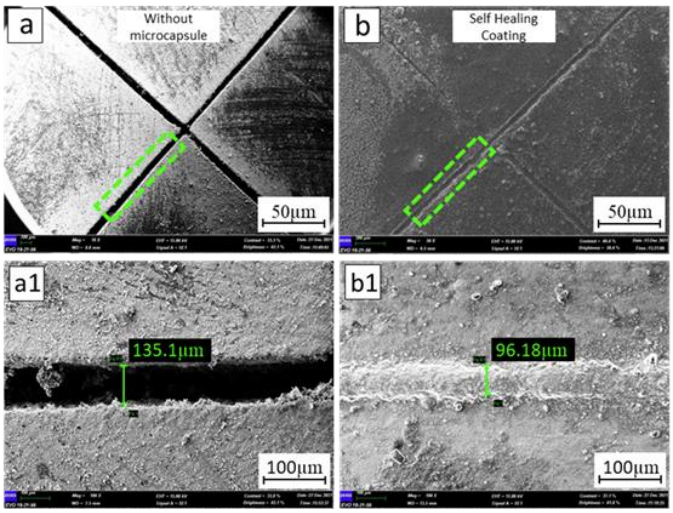


Figure 6. SEM micrograph (a) without microcapsule and (b) self-healing coating, (a1) without microcapsule and (b1) self-healing coating after 35 days exposure in NaCI.

Figure 7 (a and a1) depicts a standard epoxy with no microcapsules, measuring ~ 139.3 µm. The value indicates that without the microcapsule, there is no significant change in thickness variation. The self-healing coating is demonstrated when epoxy is embedded with a 7.5wt. % linseed oil microcapsule particle bond coating, resulting in a thickness of approximately ~ 300 µm on the surface of the low carbon steel, as illustrated in Figure 7 (b and b1). It can also be observed that the epoxy coating layer has good bonding with the low carbon steel substrate due to rough surface from sandblasting preparation. The morphology with uniform arrangement and microcapsule particle distribution is clearly shown. Clearly the presence of microcapsules increased the thickness of coating dramatically.

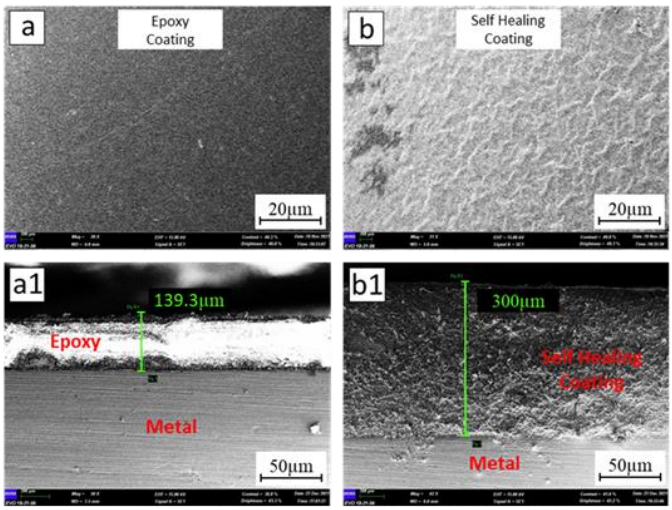


Figure 7. SEM micrograph of top view (a) epoxy coating and (b) self-healing coating, and the thickness cross-section of (a1) epoxy coating and (b1) self-healing coating.

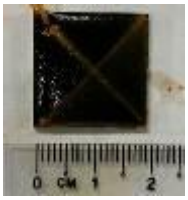
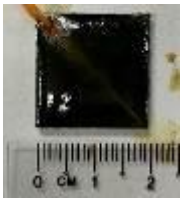
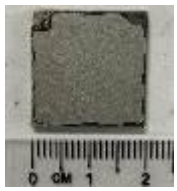
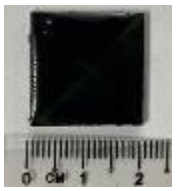
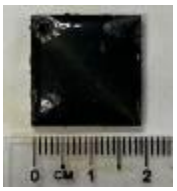
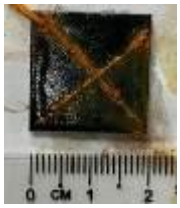
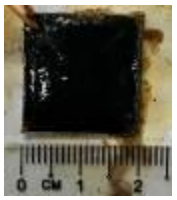
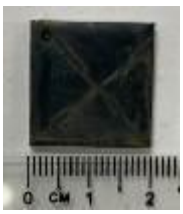
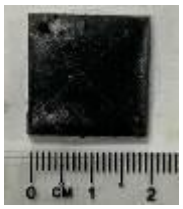
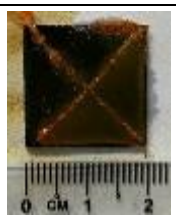
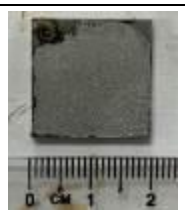
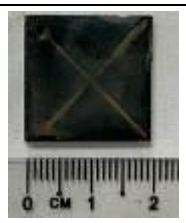
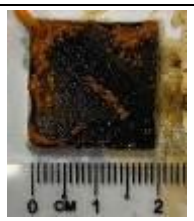
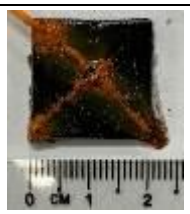
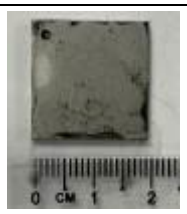
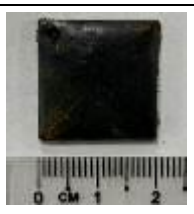
3.4 Visual Inspection After Immersion Test

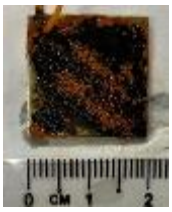
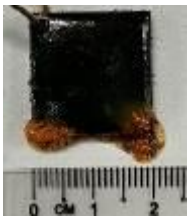
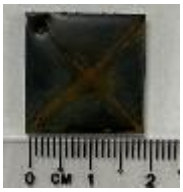
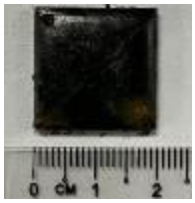
An immersion test was conducted for different durations to study the corrosion behaviour of the self-healing microcapsule embedded in epoxy coated samples. According to Table 2, the uncoated sample exhibits significant corrosion due to the absence of any protective coating, resulting in complete loss of protection. Subsequently, a corrosion product was observed on the epoxy-coated sample, which did not contain a microcapsule. This suggests that the plain epoxy coating was permeable to chloride ions, water, oxygen, and other substances, which led to corrosion of the steel substrate material.

However, due to the healing agent's excellent ability to heal the damaged region, there is almost no corrosion product observed on the self-healing coated sample after 7 days of immersion testing. According to the visual observation results shown in Table 2, the corrosion products were mostly found on the epoxy coated without microcapsule samples. Table 2 shows that no delamination, blisters, peeling, or cracking defects were observed in either coated sample [17-23]. While there was less corrosion product visible on the surface of the LO microcapsule embedded in the epoxy self- healing coated sample.

Based on visual inspection after 14 and 21 days of immersion in 3.5wt. % NaCl medium, it was discovered that the amount of corrosion product on the surface of epoxy coated without microcapsule samples increased when compared to self-healing coated samples. The amount of corrosion product observed on the self-healing coating, on the other hand, indicated that some areas were not fully protected with the healing agent. When compared to the self-healing coated samples, the amount of corrosion product was mostly found on uncoated and epoxy-coated samples after 28 and 35 days of immersion. Corrosion products were also formed on the self-healing coated samples because chloride ions were able to break the unprotected area of the coating after prolonged exposure and the microcapsules were unable to heal all of the coating surfaces.

Table 2 Sample of immersion test in 3.5wt. % of NaCl medium

Duration of Immersion Test (day)		Without Coating	Coat with Epoxy Coating	Coat with Self Healing Coating
7	Before			
	After			
114	Before			
	After			
21	Before			
	After			
28	Before			
	After			

35	Before			
	After			

3.5 Weight Loss and Corrosion Rate Measurement

Figure 8 shows the overall mass loss measurements of the immersion test and corrosion rates. Previous research [9, 16, 19] has demonstrated that the exceptional anticorrosion properties of self-healing coatings on steel substrates are a result of the healing agent that is released from ruptured microcapsules. This agent is capable of automatically sealing and healing the damaged area.
Figure 8 shows that as the immersion period increases, the weight loss for the epoxy coated without microcapsule samples also increases. However, the samples with self-healing coating show an initial increase but start to decrease after 7 days of immersion testing. Ever since the self-healing coated samples started repairing the damaged coating by releasing the LO microcapsule, they have been able to prevent any further corrosion. Even though the amount of weight loss reduces with time, the corrosion product was still visible on the samples after prolonged contact because not all of the microcapsules are released from the coating because it depends on the crack location, crack size, and the enough linseed oil to heal the crack.

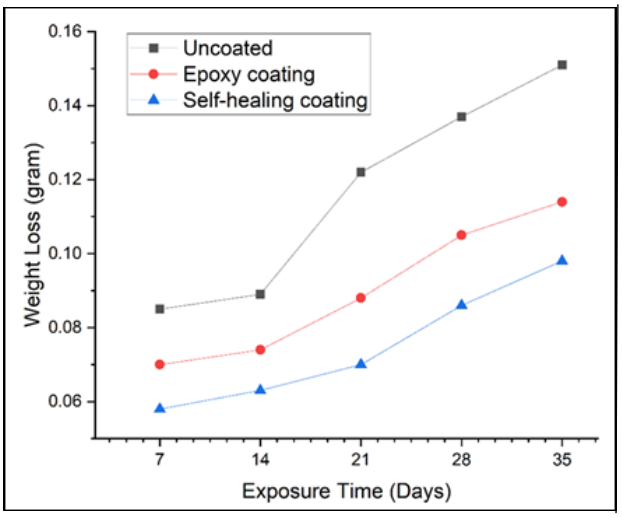


Figure 8. Graph of Weight Loss in 3.5wt. % NaCl Solution.

Figure 9 presents a general decrease in corrosion rate. It is understandable that the chloride ions in the 3.5wt. % NaCl medium would be depleted after a period of time. As a result, the corrosion process would be slowed. Furthermore, the formation of corrosion products on the samples' surfaces acted as a corrosion barrier. The epoxy without microcapsule coated samples has a higher corrosion rate than the self-healing coated samples because the epoxy coating that was attacked by the corrosive solution was not restored, causing the samples to corrode. On the other hand, the corrosion process was significantly reduced in the self-healing coated samples. This was observed in the areas where a layer of LO microcapsules was incorporated into the epoxy coating, especially in the scribed region. When the coating was damaged by the corrosion, the linseed oil was released from the microcapsule. Once the coating is repaired or healed, it will provide protection to the underlying steel substrate, preventing any further corrosion. As a result, the corrosion rate of self-healing coated samples is lower than epoxy samples without microcapsules coated samples. Hatami Boura et al. [19] found that after 1, 7, and 21 days, the corrosion resistance of epoxy coatings in a 3.5wt. % NaCl solution decreased as the immersion time increased.

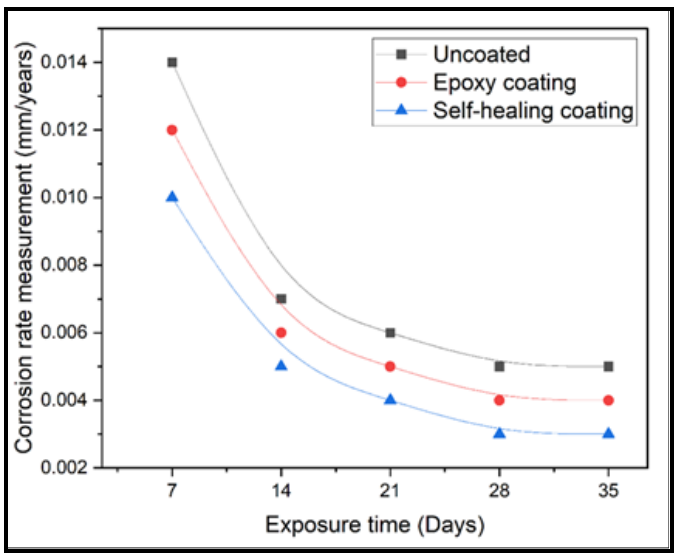


Figure 9. Graph Corrosion Rate in 3.5wt. % NaCl Solution.

3.6 Surface Study by SEM and EDS

Prior to the corrosion test, an initial test was performed to ensure that the self-healing coating worked properly. A cross scratch was made on the self-healing coated sample, which was then placed in a normal environment at room temperature for seven days. Similarly, for control and comparison, epoxy paint samples that had not been microcapsule coated were tested. The samples were then analysed with SEM, and the results are shown in Figures 10 (a–c). The uncoated sample demonstrates corrosion protection because the sample's appearance consists of general and localised corrosion, as shown in Figure 10 (a). Figure 10 (b) depicts an open-scribed region for pure epoxy paint that reveals the steel substrates, as in the [15-17] studies. The open area size of ~ 135.1 µm (Figure 6) promotes corrosion behaviour. While, for self-healing coating, the microcapsules embedded in the epoxy were ruptured or broken and released the healing agent, linseed oil, which healed the scratch area as shown in Figure 10 (c) after 35 days and the size of the scribed area was reduced to ~ 96.18 µm to protect the metal substrates. Figure 11 (c) demonstrates the performance after 55 days of exposure to 3.5 wt. % medium completely protected.

The discussion by Huang M. et al. [20] is based on several assumptions. These assumptions include: (a) the presence of microcapsules with a consistent diameter that are evenly distributed in the epoxy paint coating matrix; (b) each microcapsule contains the same amount of fill content; (c) the shell of the microcapsules is negligible; (d) when a scratch or crack occurs in the coating, all microcapsules located at the scratch plane will rupture; (e) the healing agent contained within the ruptured microcapsules will freely flow into the scratch; (f) the healing species will spread throughout the scratch [23].

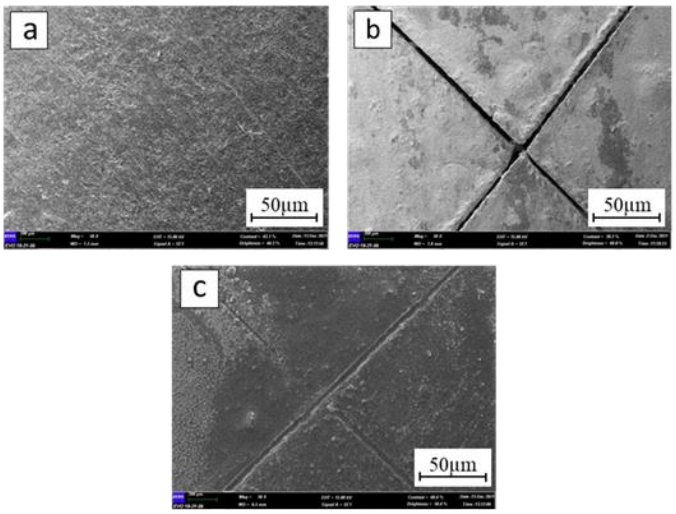
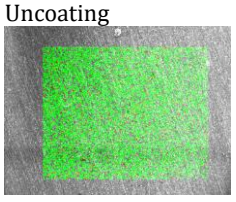
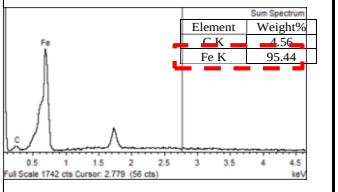
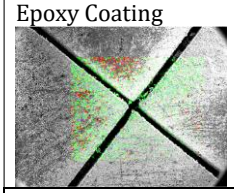
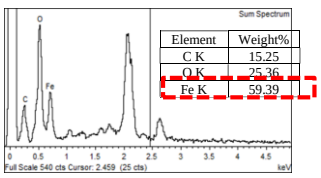
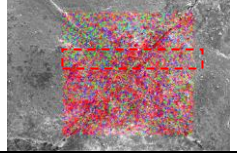
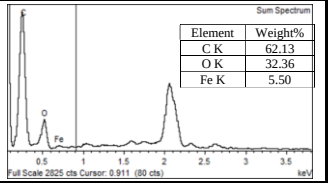


Figure 10. SEM morphology after 35 days immersion test in 3.5 wt.% NaCI of (a) uncoated, (b) epoxy coating and (c) self-healing coating.

To further validate the self-healing coating mechanism for corrosion protection, an EDX analysis was conducted on the scribed area of the epoxy coating without microcapsule and the self-healing coating surface. This analysis aimed to identify the elements present in the newly formed layer at the scribed area. Table 3 shows that only Fe and C elements are present in the open or unprotected area of the steel substrate composition. Table 3 also shows that the epoxy coating itself contains C, O, and Fe elements, indicating that the open area was not covered by any coating element. While the self-healing coating performance shows great re-covering of the healing agent and the amount of composition only shows the C, O and Fe with the small of value ~ 5.50 wt.%.

Table 3 The analysis of samples using SEM/EDX

Days	Sample	Composition
35	Uncoating	  Sum Spectrum: Element / Weight% — C K 4.56; Fe K 95.44
	Epoxy Coating	  Sum Spectrum: Element / Weight% — C K 15.25; O K 25.36; Fe K 59.39
	Self Healing Coating	  Sum Spectrum: Element / Weight% — C K 62.13; O K 32.36; Fe K 5.50

CONCLUSION

The weight loss and corrosion rate demonstrate the corrosion behaviour on the self-healing performance and SEM/ EDS analysis is being used in the current work to study the surface morphology after immersion testing. This paper reveals the significant uses of linseed oil embedded in epoxy coating for anti-corrosion for low carbon steel substrates for various applications, particularly in the oil and gas industry.

1. Self-healing coatings can repair and restore damage, reducing corrosion risk. Linseed oil microcapsules, which are used as a healing agent, were created using in-situ polymerisation. According to the procedure, the self-healing coating was made by combining 7.5 wt.% linseed oil with epoxy paint. The average size of the microcapsules of linseed oil was between 20 and 200 μm, and they were shaped like spheres, as revealed by scanning electron microscopy or SEM. Electron Dispersive X-ray (EDX) and scanning electron microscopy (SEM) have been employed to observe and analyse the performance of self-healing coating. In comparison to epoxy coatings, it was observed that self-healing coatings exhibit superior performance.
2. The results of the weight loss measurement and corrosion rate measurement in this study indicate that the self-healing coating sample outperformed the epoxy coating and uncoated surfaces. Specifically, the weight loss measurement of the self-healing coating was lower than that of the epoxy coating and uncoated surfaces. The average weight loss for the uncoated was 0.115 gram, the epoxy coating was 0.090 gram, and the self-healing coating was 0.075 gram. The corrosion rate of self-healing coatings is also lower than the corrosion rates of epoxy coatings and uncoated. The different values of corrosion rate uncoated, epoxy coating and self-healing coating of 28 and 35 days were the same value which was uncoated 0.005 (mm/years), epoxy coating 0.004 (mm/years) and self-healing coating 0.003 (mm/years).
3. SEM and EDX were used to analyse the surface morphology of uncoated, epoxy coated, and self-healing coating. The results shows that the element Iron, Fe, was present in high composition in uncoated and epoxy coating due to the presence of corrosion activity on the substrate surface. The composition of Iron, Fe in epoxy coatings was 59.39 wt.%, while in self-healing coatings was showed only 5.50 wt.% because of the scribed-area already healing with of linseed oil which is the composition consists of C elements.
4. This study highlights the importance of self-healing mechanisms for corrosion resistance, as evidenced by the coating's superior performance on a low carbon steel substrate. As a result, self-healing coatings can be used to reduce corrosion species attacks while also extending the lifespan of metal components.

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