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

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Abstract. The development of smart coatings with possible for active anticorrosion and self-healing protection of metals is essential for long-term performance of metallic structures in marine environments. In this study, tung oil-filled Poly-Urea-Formaldehyde (PUF) capsules were created utilising in-situ polymerization, and 7.5 wt.% of synthesized microcapsules were combined with commercial epoxy paint. The poly-urea-formaldehyde (PUF) microcapsules with linseed oil encapsulation had a core content of about 80 % and a diameter of 180 μm. Self-healing coatings were realized by the releasing of tung oil from microcapsules under the scarp area. Tung oil has excellent film-forming property when they contact with oxygen, self-healing anti-corrosion properties. The performance of the self-healing coating was examined through immersion testing in 3.5 wt% medium for 7, 14, 21, 28 and 35 days. The microcapsule, self-healing capability, and surface morphology of the substrate following the corrosion test were all characterised using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), weight loss, and corrosion rate methods. The experimental result indicate that the scratched of self-healing coating samples were able to resist corrosion behavior in salt solution thus, the microcapsules can be used to significantly extend the service life of the coatings. Compared to uncoated and epoxy coating, the self-healing coating exhibits superior anticorrosive capabilities after immersion testing. A more profound knowledge of the self-healing process to extend life could result from this research.

Keywords: Tung oil, microsapsule, self-healing coating, weight loss, corrosion rate

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Introduction

Low carbon steel has a distinct advantage over other materials in a variety of industrial applications, including pipeline assembly, off- and on-shore structures, vessels, and tanks [1]. This advantage comes from the low cost, ease of welding, exceptional formability, and accessibility. In environments related to marine activities, such as oil and gas applications, industrial processes, and maintenance, low carbon steel is susceptible to significant corrosion. As a result, industries implement measures to prevent corrosion. Several approaches are available to control corrosion, including selecting suitable materials, designing appropriate equipment, applying coatings for surface protection, adjusting metal electrode voltage, and using corrosion inhibitors [2]. Among these strategies, surface protection, such as coating, is considered one of the most practical and cost-effective ways to safeguard metals against corrosive attacks in marine settings [2-3].

Commercial paint and polymer coatings are frequently used either to alter the look of substrates, for aesthetic reasons or to prevent corrosion [4]. The paint covering experiences mechanical property changes during service and application, which lead to the development of micro cracks, scratches, blasting, and other flaws that spread and expose the metal substrate to air moisture, chloride, seawater, and oxygen. Greater flake generation from the substrate's coating interface and increased disbanding or peeling of the coating are both results of this action [5]. A distinct class of pigments, unique additives, composite materials, and binders can be thought of as polymer coating. Later, the concept of self-healing cracks, which has been reported for composites, can be applied to coatings to provide longer durability. An attempt to heal scratches on oil and gas coatings using secret polymer properties has been reported [6].

The fascinating potential ability of self-healing coatings to restore their structural integrity on their own after the occurrence of micro-cracks, which may occur due to the release of internal stress in a coating or mechanical damage, most likely explains the remarkable interest in these materials. For offshore structures like platforms and pipelines, this kind of functionality is projected to lead to less maintenance and corresponding cost reductions. Tung oil was chosen as a healing agent in this investigation together with driers because of its capacity to produce films through air oxidation. Urea-formaldehyde was employed as the shell and drying oil as the core to make microcapsules via in situ polymerization [8]. The effectiveness of these microcapsules in the healing of cracks in an epoxy coating as well as corrosion protection has been demonstrated.

Materials and Methods

Table 1 shows the composition of low carbon steel using LECO Glow Discharge Atomic Emission Spectrometers (GD-AES), GDS 850A, UK. A laser cut was used to cut the low carbon into dimension of 20 mm width and 3 mm thickness. The examined coating was applied to only one side of the substrate material, and the other side was covered in paraffin wax to keep it from in contact with the medium.

Table 1: Chemical composition (wt.%) of the substrates.

Alloy element	C	Si	Mn	P	S	Cr	Ni	Cu	Fe
Metal Substrates	0.164	0.180	0.612	0.0265	0.0217	0.113	0.0768	0.232	Balance

The experimental coupons surfaces are abraded with silicon carbide papers 80, 100, 300, 500, 800, 1000, and 2000 before being rinsed in distilled water, polished residual particles are removed using a sonic cleaner in an ethanol bath at room temperature for 20 minutes, dried, and then placed in a desiccator to avoid atmospheric corrosion. Microcapsules were created using in-situ polymerization in an oil-in-water emulsion. 160 ml of deionized water and 10 ml of a 5 wt. % aqueous solution of polyvinyl alcohol (PVA) were combined at room temperature in a 1000 ml beaker. 5 g of urea, 0.5 g of ammonium chloride, and 0.5 g of resorcinol were dissolved in the solution while being stirred. By applying a 5 wt. % solution of hydrochloric acid in deionized water, the pH was brought down to roughly 3.5. Octanol was added as an antifoaming agent in the amounts of one to two drops. In order to create an emulsion, about 60 ml of tung oil was gently added. The emulsion was then given 15 minutes to stabilize while being stirred. After stabilization, 12.67 g of a 37 wt.% aqueous formaldehyde solution was gently added. For five hours, the emulsion was slowly heated at a temperature between 55 °C and 60 °C while being stirred at a speed of 700 rpm. At room temperature, the contents were chilled. The suspension was filtered under a vacuum to recover the microcapsules. To remove the suspended oil, the suspension was rinsed with water and washed with xylene [6-11]. The capsules were dried in a vacuum oven.

About 7.5 wt.% of the tung oil (TO) microcapsule was added in the epoxy matrix. The epoxy and TO microcapsule were mixed at room temperature with slow agitation. The epoxy containing the TO microcapsule was constantly stirred for 30 min prior to being applied to the substrate material. The coating components were then directly painted with a brush application method onto 20 × 20 mm steel substrates. For one to two weeks, the coated specimens were left undisturbed to thoroughly dry and cure. Cross-scratching was done on each sample before the immersion test. The goal of the scratching is to compare the self-healing coating to pure epoxy coating in terms of how well it protects the sample substrate and can heal or repair coating damage. The ASTM G1-03 standard was followed when conducting the immersion tests. All coated and uncoated specimens were submerged for 7, 14, 21, 28, and 35 days in a container holding 5 liters of 3.5 weight percent NaCl medium. The samples were taken out of the 3.5 wt. % NaCl solution following each immersion test, and corrosion products were eliminated by soaking the samples. Before and after cleaning, photos were taken for a visual inspection study. The samples were then dried using high-pressure air, washed with distilled water, and weighed. The following formula was used to compute the corrosion rate:

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

where C_R 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). The corrosion behaviour of coated and uncoated self-healing coatings was studied using a scanning electron microscope (SEM) (Model: Zeiss/Evo) 18 and energy dispersive spectroscopy (EDS).

Results and Discussion

Microcapsule Properties and Self-healing Performance

The volume of the healing agent that may be delivered to the scribed or cracked area depends on the size of the microcapsules used in self-healing coatings. The microcapsules average diameter, which spans from 20 μm to 150 μm, is shown in Figure 1. About 7.5 wt.% of the total self-healing coating material was applied as microcapsules. The ideal concentration of microcapsules is between 5 and 20 weight percent, according to a previous study by Hatami et al. [12]. This is because when the concentration of microcapsules is too low, the healing agent delivered to the damaged area is insufficient, and when the concentration of microcapsules is too high, the porosity in coating increases and density decreases, which encourages poor water resistance.

SEM micrographs provide greater details regarding the microcapsules' surface characteristics and sub-micron races (Figure 1). These microcapsules were relatively spherical in shape, with a rough and non-porous exterior shell wall similar to [12-13] studied. The microcapsules' abrasive shape made for strong mechanical adhesion to the coating matrix. The capsules' spherical shape ensured both their ability to store material and their simple dispersion into the covering. The surface morphology of microcapsules is heavily influenced by the qualities of the core material (such as viscosity, surface tension, miscibility with shell material, etc.), core: shell ratio, and microencapsulating technique, as addressed by Benzab et al. [10]. The EDS spectrum of Tung oil (TO) microcapsule, which contains carbon, nitrogen, and oxygen, is shown in Figure 1(c) due to the composition of tung oil and poly-urea formaldehyde (PUF) as the shell of the microcapsule. Figure 1(d) depicts the visual appearance of a microcapsule after one week of filtration and drying.

When cracks or any other damage are created on the coating surface, the microcapsules included in the epoxy coating should break instantly to release the healing agent, ensuring efficient and good healing performance. Figure 2 depicts the healing as it was scribed in a coating layer that was captured using a scanning electron microscope both with and without a microcapsule. Figure 2(a) illustrates the length of the crack that is open, unprotected, and exposed to the environment. While tung oil, which is produced when the microcapsules burst, provides self-healing coating with protection by filling in the cracks (scribed region), further producing a new film by tung oil oxidative polymerization process at atmospheric oxygen at ambient temperature as presented in Figure 2(b).

SEM micrographs in Figure 3(a) shows an epoxy coating with thickness of approximately ~ 100 μm on the surface of the carbon steel. Due to the rough surface created by the sandblasting preparation, it can also be seen that the epoxy coating layer bonds well to the carbon steel substrate. The self-healing coating had a thickness of roughly ~ 150 μm on the surface of the carbon steel, as shown in Figure 3(b). As can be seen at substrate/coating layer interfaces, the self-healing coating layer has a solid connection with the carbon steel substrate. It is obvious that self-healing coating has covered the two main components with uniform distribution of epoxy and linseed oil microcapsules as shown in Figure 3(b).

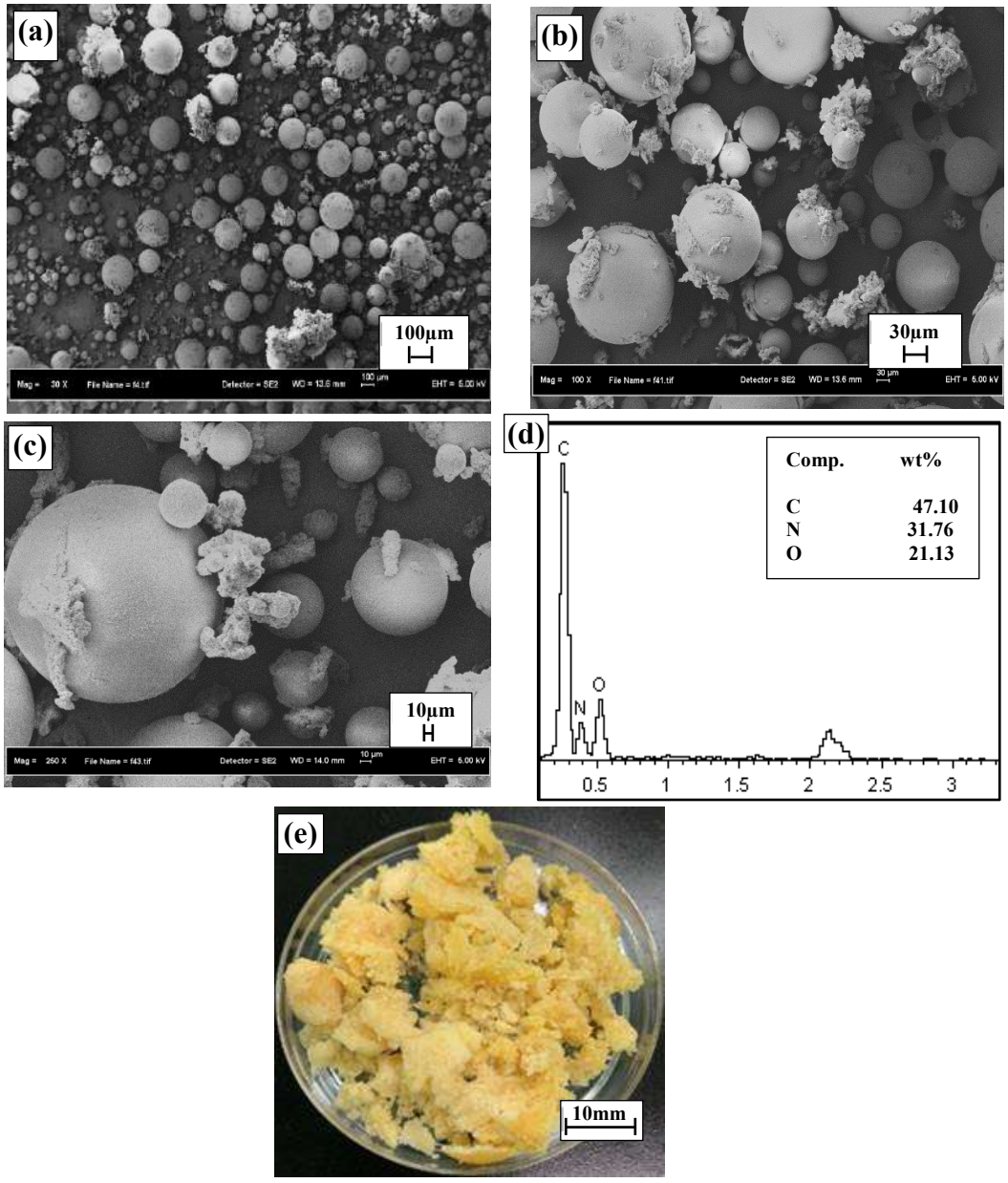


Figure 1: SEM micrographs for (a) microcapsule of Tung oil (TO) after synthesis, (b) final size of microcapsule, (c) single microcapsule spectrum, (d) EDS spectrum of (c) and (e) physical microcapsule after filtration

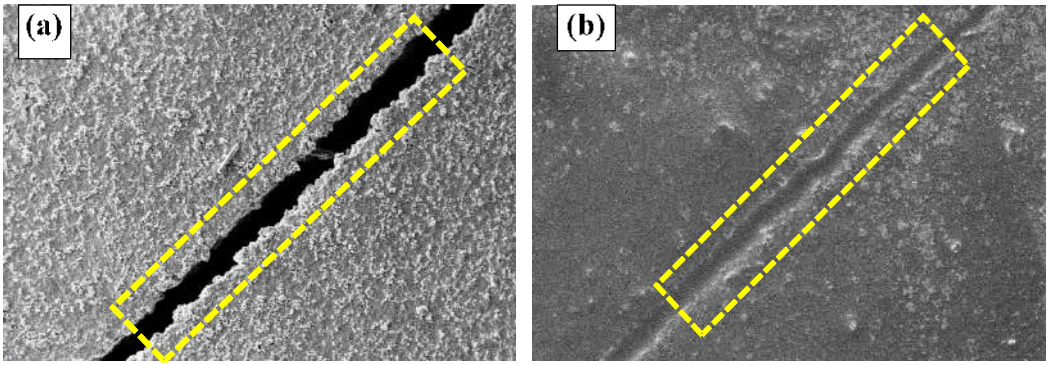


Figure 2: SEM micrographs of comparison coated low carbon steel substrates for (a)eEpoxy coating and (b) self-healing coating respectively

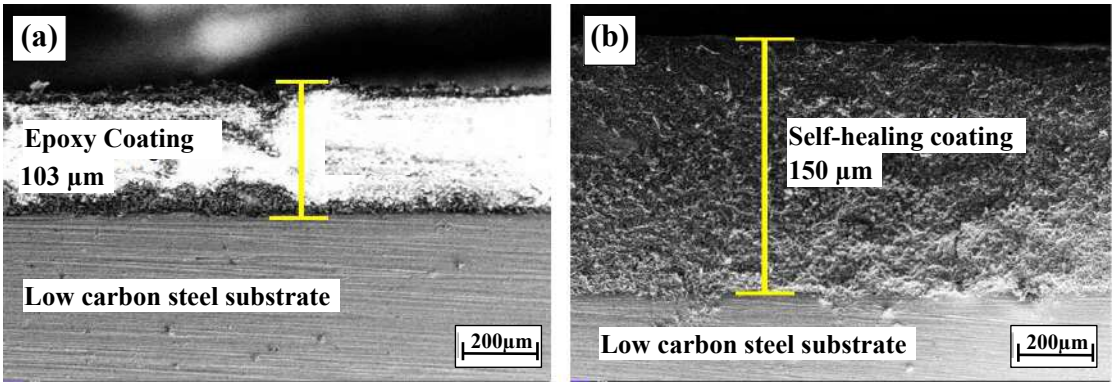


Figure 3: SEM micrographs of coating thickness for coated carbon steel substrates (a) epoxy coating and (b) self-healing coating respectively

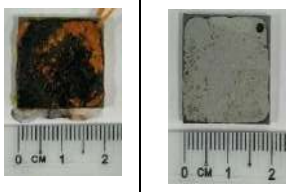
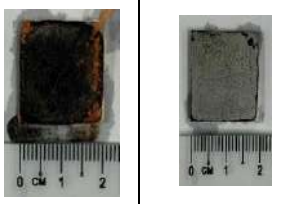
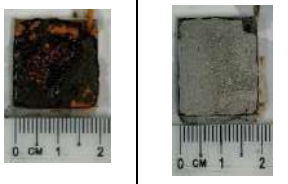
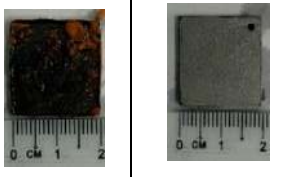
Visual Inspection After Immersion Test

In order to evaluate the corrosion behavior of the self-healing microcapsule placed in epoxy coated samples, the immersion test was run for 7, 14, 21, 28 and 35 days. Table 2 demonstrates that because there is no protection and it is fully lost, the uncoated sample suffers from or has extremely poor corrosion protection. After that, the epoxy covered sample without a microcapsule developed some corrosion product. This suggests that the plain epoxy covering allowed chloride ions, water, and oxygen to get through, leading to corrosion on the steel substrate material.

However, after 7 days of immersion testing, there is hardly any evidence of corrosion products on the sample coated with a self-healing material. This is due to the high efficiency of the healing agent in repairing the damaged region. 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 [10-15]. After the addition of TO microcapsule in the epoxy, self-healing coated sample had less corrosion product evident on its surface.

Based on visual inspection following 7, 14, 21, 28, and 35 days of immersion in 3.5 wt.% NaCl media, it was found that, in comparison to self-healing coated samples, the amount of corrosion product on the surface of epoxy coated without microcapsule samples increased. On the other hand, the amount of corrosion product found on the self-healing coating suggested that some places were not completely protected by 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 24, 21, 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 the coating surfaces.

Table 2: Sample after immersed in 3.5 wt.% of NaCl solution

Duration of immersed (days)	Uncoated		Coated with epoxy coating		Coated with self-healing	
	Before cleaning	After cleaning	Before cleaning	After cleaning	Before cleaning	After cleaning
7						
14						
21						
28						
35						

Mass Loss and Corrosion Rate Measurement

Figure 4 displays the total corrosion rates and mass loss measurements from the immersion test. Previous research [2,9,12] has suggested that the remarkable anticorrosion properties of self-healing coatings on steel substrates are attributed to the release of the healing agent from ruptured microcapsules. This agent has the ability to automatically seal and heal the damaged area.

The weight loss for the epoxy coated without microcapsule samples rises with increasing immersion time. The self-healing coated samples, on the other hand, increase initially but start to decline following the 28-day immersion test. The sample was shielded from additional corrosion because the self-healing coated samples started to repair or heal the damaged coating by releasing the TO microcapsule. 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 is depending on the crack location, crack size, and the enough linseed oil to heal the crack.

The general decrease in corrosion rate is shown in Figure 4(b). The chloride ions in the 3.5 wt.% NaCl medium would become reduced over time. As a result, corrosion process was slowed. Additionally, the corrosion products that formed on the surface of the samples served as a corrosion barrier. The epoxy coating that was attacked by the corrosive solution and was not repaired resulted in the samples corroding at a higher rate than the samples covered with self-healing microcapsules. In the meantime, the corrosion process was halted in the scribed area of self-healing coated samples that were covered by a layer of TO microcapsules embedded in the epoxy coating. The Tung oil was discharged from the microcapsule when the coating ruptured due to the corrosion assault. Once the coating was fixed or healed, the steel substrate sample would be shielded from additional corrosion. As a result, self-healing coated samples have a lower rate of corrosion than epoxy samples that are not covered with microcapsules. Hatami Boura et al. [12] discovered the corrosion resistance of epoxy coatings in a 3.5 wt.% NaCl solution declined with increasing immersion time after 1, 7, and 21 days.

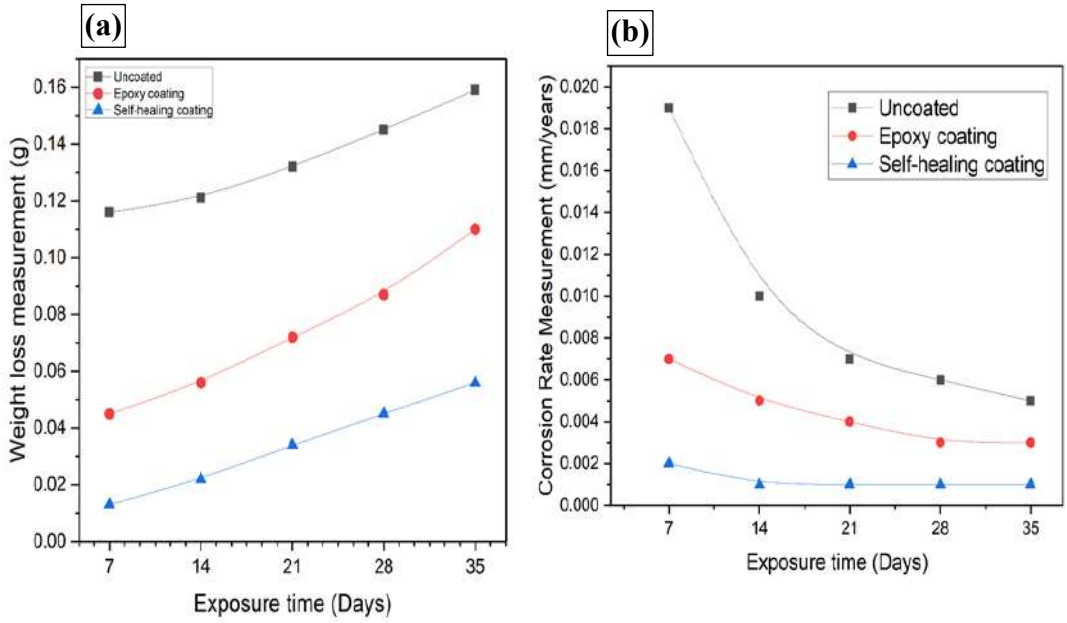


Figure 4: Immersion test results after 7, 14, 21, 28 and 35 days for (a) weight loss and (b) corrosion rate measurement for uncoated, epoxy paint and self-healing coating in 3.5 wt. % NaCl medium

Surface Analysis by SEM/EDS

The first test was conducted to see if the self-healing coating would work adequately before the corrosion test. The epoxy and self-healing coated sample was scratched, and it was then kept in a regular setting at room temperature for seven days. The identical procedure was

also carried out on unfilled epoxy paint samples for comparative and control purposes. Figures 5 presents the outcomes of SEM and EDS analysis performed on the samples (a) to (f). Because the composition of Fe, C, and O is not covered and supported by EDS, Figure 5(a) and (b) clearly displays the severe corrosion behaviour. Figure 5(c) and (d) shows an open scribed zone for the original epoxy paint that exposes the steel substrates as in the [15–20] research. Less Fe, C, and O are present in the open area at the scribed area, as seen in the EDS spectrum. As seen in Figure 5, the self-healing coating was created by breaking or rupturing the microcapsules included in the epoxy, which allowed Tung oil to be released and heal the scratch (Figure 5(e) and (f)). The effectiveness of protection has been demonstrated using the EDS spectrum to include the release of a healing agent or inhibitor into the scratch location after the damage has already occurred, as well as the composition of epoxy paint and linseed oil. The crucial component is Fe, which demonstrates that the epoxy paint without microcapsules has a greater amount to represent an unprotected area at the scribed area, whereas Figure 5(d) displays a reduced amount of Fe initially protected by linseed oil rupture at the scribed area.

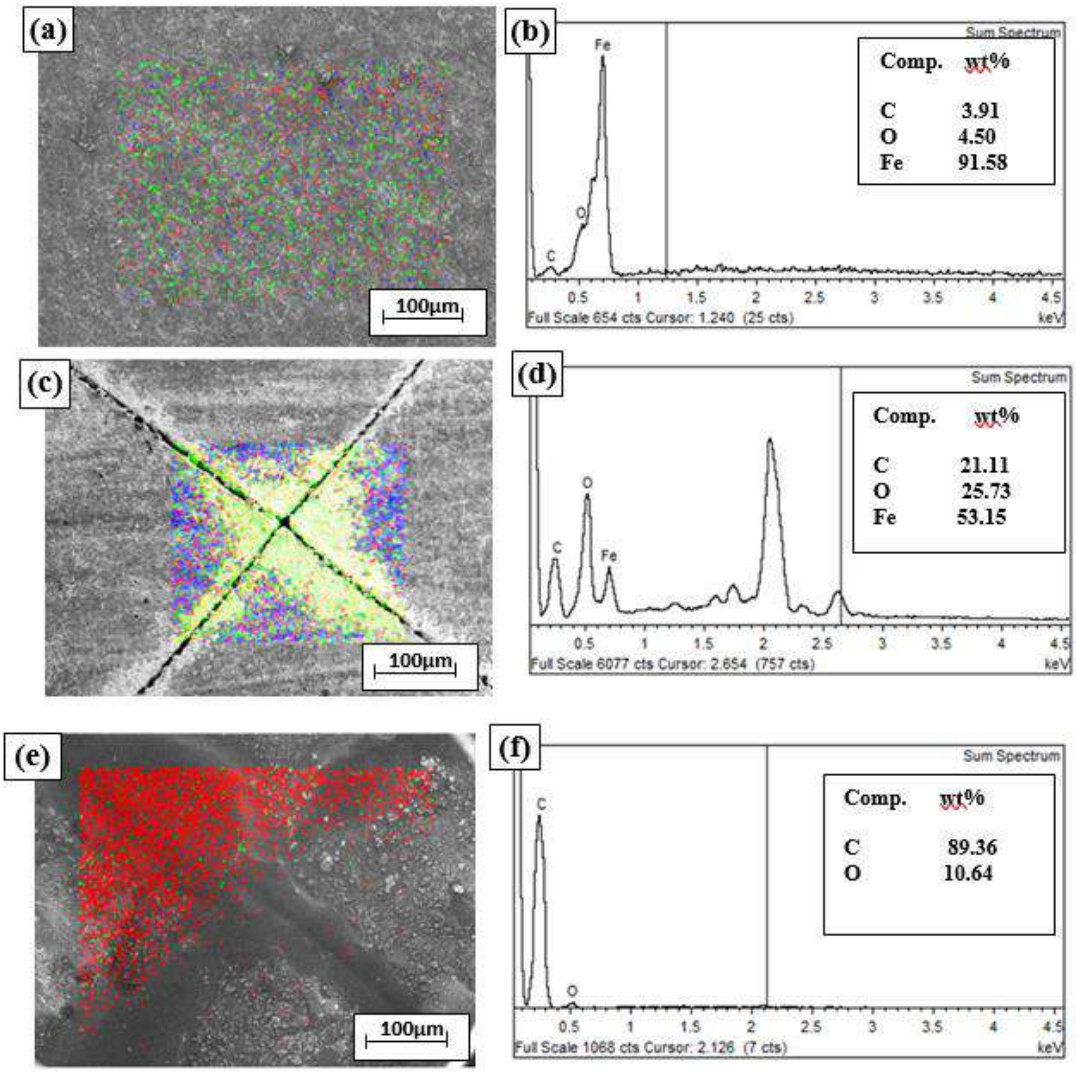


Figure 5: SEM micrographs a EDS composition of (a) uncoated substrates, (b) EDS of (a), (c) samples without microcapsules, (d) EDS of (c), (e) self-healing coating and (f) EDS of (e) after 35 days in 3.5wt.% NaCl

In Huang et al. [13] studied, they found several assumptions that support the finding for this study including: (a) that microcapsules with uniform diameter are evenly distributed in the epoxy paint coating matrix; (b) that the fill content of each LO microcapsule is the same; (c) that the shell of the microcapsules is negligible; and (d) that when a scratch or crack forms in the coating, all of the microcapsules located at the scratch plane are ruptured; (e) all of the encapsulated healing agent of ruptured microcapsules will freely flow into the scratch; (f) the healing species will spread within the scratch [13]. The coating showed good self-healing abilities while also safeguarding the substrate. The successful self-healing coating, according to Suryanarayana et al. [9], happens when the microcapsules integrated into the paint film shatter instantly to release healing material when cracks in the paint film are produced. Additionally, it has been found that microcapsules shell surfaces are quite rough, which will promote strong bonding with the film matrix [9,17-20].

Conclusions

In this study, the in-situ polymerization process was used to successfully create PUF microcapsules containing tung oil. This study used a 7.5 weight percent microcapsule concentration in commercial epoxy paints to show the potential for creating self-healing anticorrosion coatings. SEM images and EDS spectra proved that the tung oil was successfully released from the capsules implanted in the epoxy coating upon mechanical damage. The microcapsules broke and released the linseed oil drying oil substance during the immersion test of the self-healing coating's anticorrosion performance, which effectively mended cracks with satisfactory anticorrosive qualities throughout mechanical damage. Because of this, the coated low carbon steel substrates had much better anti-corrosion performance and slower corrosion rates. By releasing the healing agent from microcapsules incorporated in the epoxy matrix, the self-healing coating effectively repairs damage that occurs on the coating of a steel substrate and seals the exposed area from further corrosion.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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