

Dual-Layer Self-Healing Coatings for Carbon Steel: A Sustainable Approach to Enhanced Performance and Durability

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Abstract

The industrial sectors are continually searching for durable materials with high resistance to wear. While traditional coatings are commonly used, they tend to deteriorate over time, resulting in costly repairs and negative environmental impact. Therefore, this study investigates the development of single and double-layer smart coating systems designed to enhance both durability and sustainability for carbon steel, particularly in harsh environments. The double-layer smart coating (DL-SC) incorporates benzotriazole and boiled linseed oil within an epoxy matrix. The DL-SC is applied in two layers, while the single-layer smart coating (SL-SC) with the same composition is used for comparison. Characterization techniques, including Fourier transform infrared (FTIR) spectroscopy and field emission scanning electron microscopy (FE-SEM), confirm the successful incorporation of the self-healing agents and corrosion inhibitors into the coatings. Additionally, the adhesion strength of DL-SC retained 68.93% of its adhesion strength after immersion in a 3.5 wt% NaCl solution in contrast to 61.05% for SL-SC and 47.92% for traditional epoxy coatings, which highlights the enhanced durability and long-term sustainability of the double-layer system. The enhanced performance of DL-SC is due to the efficient release of BTA and BLO in response to external stimuli, providing extended protection. In conclusion, the double-layer smart coating provides superior durability and long-term protection for carbon steel compared to single-layer.

1. Introduction

The increasing demand for sustainable and durable materials in industrial sectors such as oil and gas has led to a growing focus on developing advanced protective technologies. As industries strive to reduce their carbon footprint and operational costs, there is a pressing need for innovative solutions that provide long-term corrosion protection while minimizing the environmental and economic consequences of material degradation. Smart coatings, with their self-healing and corrosion-resistant properties, represent a promising approach to addressing these challenges.

In the oil and gas industry, corrosion is a major challenge, especially for carbon steel pipelines, which are prone to degradation in highly corrosive environments [1], [2], [3], [4]. Metal corrosion is the loss of material resulting from chemical or electrochemical reactions with the surrounding environment [5], not only compromises the integrity of infrastructure but also leads to costly equipment repairs, maintenance, and unplanned production downtime. Furthermore, these disruptions significantly impact productivity and result in substantial economic losses, as well as increased environmental costs due to the frequent need for maintenance and material replacement [6], [7]. In high-temperature and high-pressure environments, corrosion rates increase, making effective corrosion management essential for maintaining infrastructure integrity and ensuring operational safety.

Traditional protective coatings have been employed to reduce corrosion; however, these coatings often degrade under harsh conditions such as those found in coastal or industrial environments, leading to frequent recoating and maintenance [7], [8], [9], [10]. This results in the need for frequent maintenance and recoating, which not only increases operational costs but also contributes to material waste and environmental impact.

In response to these limitations, recent advancements conducted by Habib *et al.*, have introduced polymeric smart coatings [11]. These coatings have the unique ability to self-repair and respond to external stimuli like temperature, humidity, and pH changes [10] as shown in **Fig. 1**. The unique properties of polymers enable the formation of protective layers that can self-heal, providing an effective defence against corrosion on metal surfaces [10]. Recent studies have shown that these coatings are highly effective due to their incorporation of micro- or nanoscale containers that release corrosion inhibitors when damage is detected, activating a self-repair mechanism [11], [12], [13], [14].

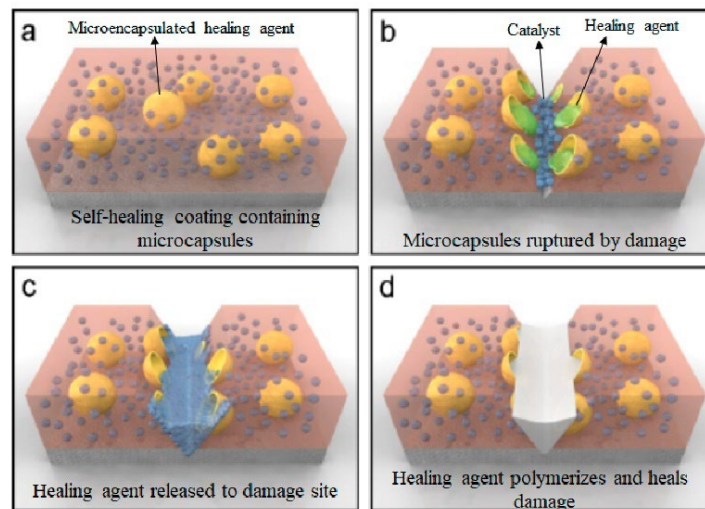


Fig. 1 Self-repair mechanism of self-healing coating [12]

The integration of advanced functionalities into coatings represents an innovative solution to corrosion. Nanocontainers, embedded within the polymer matrix, hold corrosion inhibitors that are released when the coating detects surface damage. This not only repairs the damaged areas but also neutralizes corrosive agents that have penetrated the protective layer, thus providing extended corrosion resistance. However, further research is required to optimize the release mechanisms and to evaluate the performance of these coatings across a wider range of materials and environmental conditions [15]. A study conducted by Hassanein *et al.*, incorporates corrosion inhibitors such as benzotriazole (BTA) and self-healing agents like boiled linseed oil (BLO) which help address these challenges [16].

Benzotriazole (BTA) is well-known for its excellent performance as a corrosion inhibitor, largely due to its ability to create a stable protective layer on metal surfaces, effectively blocking corrosive agents from causing damage [17]. Numerous studies highlight the effectiveness of BTA across diverse conditions. For instance, Youssef *et al.*, reported that BTA achieved corrosion inhibition rates as high as a protective film on the metal [18]. Similarly, research conducted by Petrunin *et al.*, found that BTA as a coating additive significantly enhances the metal's corrosion resistance [19].

Other than that, microencapsulation of healing agents like boiled linseed oil (BLO) further boosts the protective capabilities of smart coatings. BLO is advantageous because of its ability to polymerize and form a protective film upon exposure to air. Encapsulating BLO in urea-formaldehyde microcapsules ensures that it is only released when mechanical damage occurs, facilitating localized repair and enhancing the coating's durability

[20]. Combining BTA and BLO provides both chemical and physical protection, significantly improving the overall performance of smart coatings, as demonstrated in Fig. 2 [11], [12], [21].

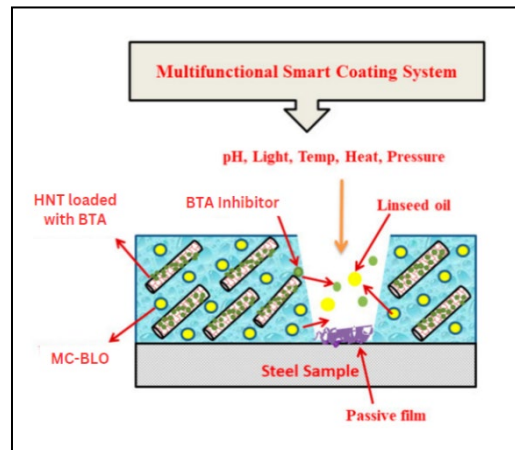


Fig. 2 Smart coating contained BTA-HNT and MC-BLO [22]

Although smart coatings exhibit superior properties compared to conventional coatings, their effectiveness may gradually diminish after prolonged exposure to harsh environmental conditions, particularly single-layer smart coating [16]. Addressing these research gaps is crucial for advancing the development and implementation of effective, sustainable smart coatings for corrosion protection in the oil and gas industry by developing smart coating with self-healing capabilities with coating additives of BTA and BLO. Thus, this paper aims to investigate the effectiveness of double-layer smart coating systems for corrosion protection and sustainability for carbon steel.

2. Methodology

2.1 Materials

To develop the smart coating, BTA-HNT and MC-BLO are required. The synthesis of BTA-HNT involves using halloysite nanotube particles (HNT) and benzotriazole (BTA), both supplied by Sigma-Aldrich (M) Sdn. Bhd. (Malaysia), as nanocontainer and corrosion inhibitor, respectively, with acetone as the solvent for encapsulation. For the fabrication of microcapsules containing boiled linseed oil (MC-BLO), materials such as polyvinyl alcohol (PVA), urea, 37 wt% formaldehyde, resorcinol, ammonium chloride (NH₄Cl), hydrochloric acid (HCl), and 1 wt% PVA are necessary, along with boiled linseed oil (BLO) supplied by Easy Fix Malaysia Sdn. Bhd., and xylene, with BLO chosen for its fast-drying characteristics. The smart coating is formulated using EPIKOTE 828 epoxy resin and LSD308 hardener, while Araldite glue is utilized for adhesion testing. A carbon steel plate, measuring 100 mm × 100 mm × 2.5 mm, serves as the substrate, and the surface preparation is completed using silicon carbide (SiC) paper for cleaning and polishing.

2.2 Coating Additive Preparation

The coating additives were prepared through a series of steps to ensure proper encapsulation and functionality of the active components. To synthesize BTA-HNT, 3 g of dry halloysite nanotube particles were mixed with 37.5 mL of a saturated benzotriazole (BTA) solution at 60 mg/mL in acetone. This mixture was placed in a vacuum jar, and a vacuum pump was used to remove the suspension, followed by restoring air pressure. This cycle was repeated three times to maximize the loading of BTA into the nanotubes. The BTA-HNTs were then centrifuged at 5000 rpm for 5 minutes, rinsed three times with ultrapure water to remove unbound BTA and impurities, and dried at 60°C for 1 hour. Finally, the BTA-HNTs were immersed in a 0.08 M CuSO₄ solution for 60 seconds to form copper-BTA complexes, which controlled the release of BTA from the nanotubes, following the method outlined by Li et al. (2021) [17]. Fig. 3 shows the BTA-HNT preparation process.

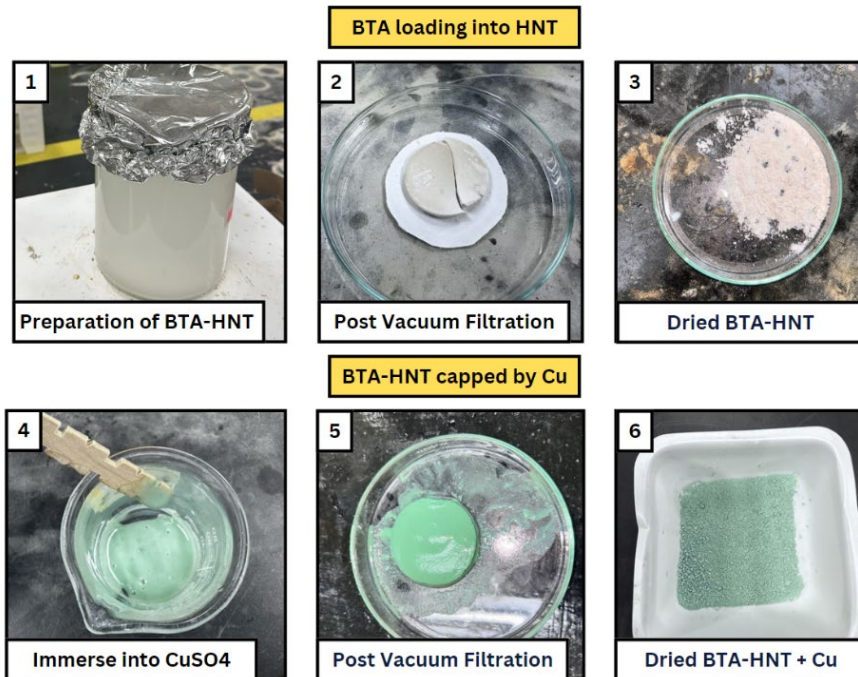


Fig. 3 Preparation stage of BTA-HNT

The preparation of microcapsules containing boiled linseed oil (MC-BLO) followed the method by J. Li et al. (2020). For the synthesis, 3 g of urea, 0.3 g of resorcinol, and 0.3 g of ammonium chloride were mixed with 1000 mL of a 1 wt% polyvinyl alcohol (PVA) solution. The solution's pH was adjusted to around 3.0 using hydrochloric acid (HCl). Boiled linseed oil was gradually added to the solution while stirring at 1000 rpm to form an emulsion. Then, 9 g of 37 wt% formaldehyde was added dropwise, and the mixture was heated to 55°C with continuous stirring for 10 hours. The resulting microcapsules were collected by vacuum filtration, washed with xylene to remove residual substances, and dried at 150°C. Fig. 4 illustrates the preparation of MC-BLO.

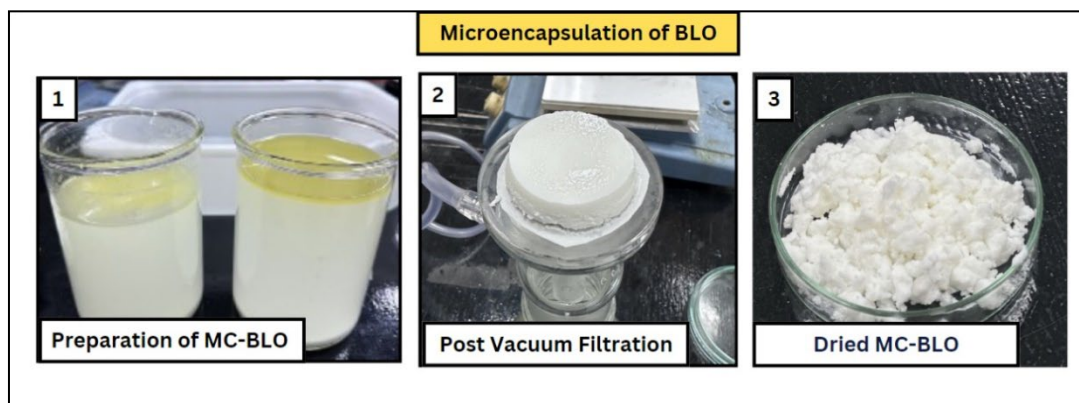


Fig. 4 Preparation stage of additive MC-BLO

2.3 Smart Coating Preparation

Three types of coatings were prepared: pure epoxy coating (EP), single-layer smart coating (SL-SC), and double-layer smart coating (DL-SC), as detailed in Table 1. To create the EP, 6 g of EPIKOTE 828 epoxy resin was combined with 3 g of LSD308 hardener and mixed for 10 minutes. For the fabrication of SL-SC and DL-SC, 5.0 wt% of BTA-HNT and 5.0 wt% of MC-BLO were added to 5.0 g of EPIKOTE 828 epoxy resin, followed by stirring for 30 minutes. Subsequently, 1.25 g of LSD308 hardener was incorporated, and the mixture was stirred for an additional 10 minutes.

For SL-SC, the coating mixture was applied to a cleaned and polished carbon steel substrate using a paintbrush and allowed to cure for one week until it was dry and hardened, reaching a final thickness of about 780 μm . For

DL-SC, the first layer was applied with a wet thickness of 390 μm and cured for 48 hours at 25°C. After curing, a second layer of the same thickness was applied on top, resulting in a total thickness of approximately 780 μm . The application process followed ASTM D823 standards for coating application.

Table 1 Formulation of EP, SL-SC and DL-SC

No of Samples	Formulation	Sample Coding
1	Epoxy + Hardener	EP
2	Single layer of 15 wt% of BTA-HNT and MC-BLO + Epoxy + Hardener	SL-SC
3	Double layer of 15 wt% of BTA-HNT and MC-BLO + Epoxy + Hardener	DL-SC

2.4 Characterization of Smart Coating

To achieve the study's objectives, FTIR was employed to verify the successful encapsulation of BTA within HNT and BLO within microcapsules. Additionally, FE-SEM provided high-resolution images of the surface morphology, while EDX facilitated elemental analysis and mapping to evaluate the microstructural features and composition. These techniques ensured the coatings' effectiveness and durability, supporting the development of smart coatings with improved corrosion protection and self-healing capabilities.

2.5 Adhesion Properties of Smart Coating

The pull-off adhesion test was conducted to assess the adhesion strength between the coating and the substrate, following ASTM D4541 standards. This test offered crucial insights into the coatings' bonding ability with the substrate, which is essential for long-term durability and performance. This test measures the force required to detach the coating from the substrate, offering a reliable indication of the coating's adhesive properties.

3. Results and Discussion

3.1 Characterization of Morphological Structure of Developed Smart Coating

3.1.1 FTIR Spectra

The FTIR results for BTA, HNT, and BTA-HNT are illustrated in Fig. 5 whereas BLO and MC-BLO are shown in Fig. 6. The FTIR spectrum of HNT exhibited characteristic peaks at 3621 cm^{-1} and 3693 cm^{-1} corresponding to O-H stretching vibrations, which are essential to the HNT structure [16]. In the BTA spectrum, distinct peaks were observed for N-H stretching at 3070 cm^{-1} and 2991 cm^{-1} , C-N stretching at 1268 cm^{-1} , and C-H in-plane bending at 738 cm^{-1} and 751 cm^{-1} . In the BTA-HNT spectrum, the presence of Al-OH bending at 907 cm^{-1} [16] and the absence of key BTA peaks suggested that BTA was predominantly encapsulated within the HNT lumen, with minimal amounts present on the external surface [17].

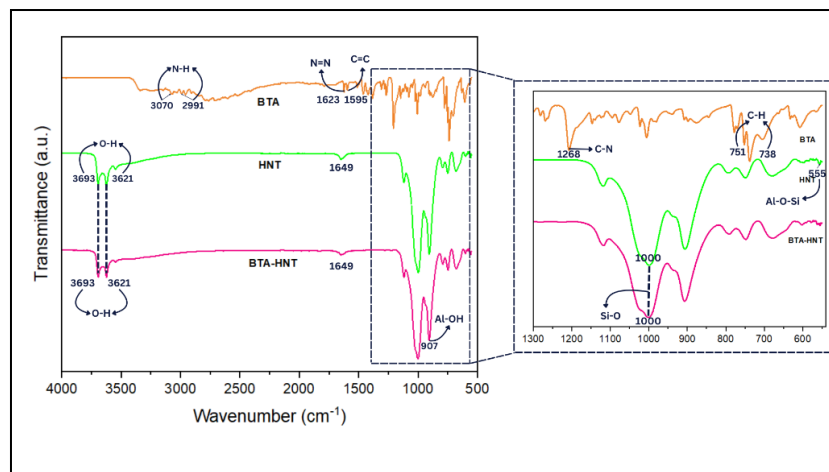


Fig. 5 FTIR spectra of BTA, HNT and BTA-HNT

Similarly, the FTIR spectra of BLO and MC-BLO, shown in Fig. 6, confirmed successful encapsulation, as evidenced by characteristic peaks. Notable peaks included 3010 cm^{-1} for =C-H, 2924 cm^{-1} and 2854 cm^{-1} for -C-H stretching vibrations, and 1743 cm^{-1} for ester carbonyl groups. Peaks at 1460 cm^{-1} (C-H bending) and 720 cm^{-1} (=C-H) were also observed, consistent with the findings of Hassanein et al., [16]. The presence of additional peaks at 1423 cm^{-1} and 844 cm^{-1} further verified the successful encapsulation of BLO. These results confirm that the encapsulation process was effective in maintaining structural integrity, consistent with prior research on HNT encapsulation capabilities.

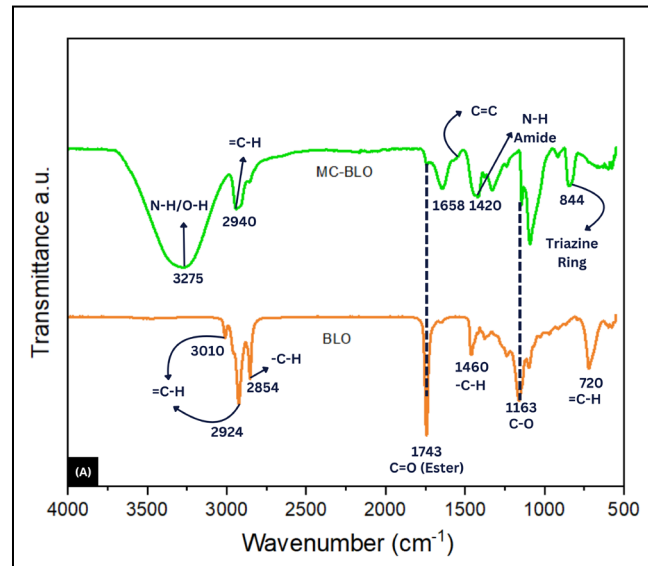


Fig. 6 (A) FTIR spectra of BLO and MC-BLO

3.1.2 FESEM Analysis of Coating

FE-SEM analysis was conducted to study the surface morphology of MC-BLO, BTA-HNT, and EP coatings. For MC-BLO, Fig. 7B (50kX) shows well-formed spherical microcapsules with a rough surface, confirming successful encapsulation and structural integrity which align with the findings of Hassanein et al., [16]. However, Fig. 7C reveals some fragmented microcapsules, indicating inconsistencies in the encapsulation process. These observations emphasize the need to optimize encapsulation parameters to produce uniform and structurally sound microcapsules, which are essential for effective self-healing functionality.

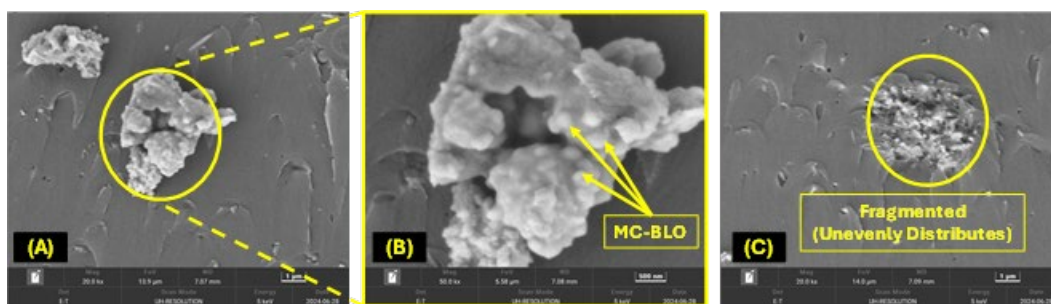


Fig. 7 FE-SEM of (a) MC-BLO with 20kX magnification; (b) MC-BLO with 50kX magnification (c) Fragmented MC-BLO

For BTA-HNT, Fig. 8B (30kX) illustrates a detailed view, showing individual, intact nanotubes, while Fig. 8C (50kX) confirms uniform encapsulation of BTA, despite the natural variation in the size and shape of the nanotubes. The successful loading and structural preservation of BTA within the HNTs is essential for ensuring the self-healing capabilities and long-term corrosion resistance of the smart coatings aligning with the findings of Habib et al., and Hassanein et al., [11], [16].

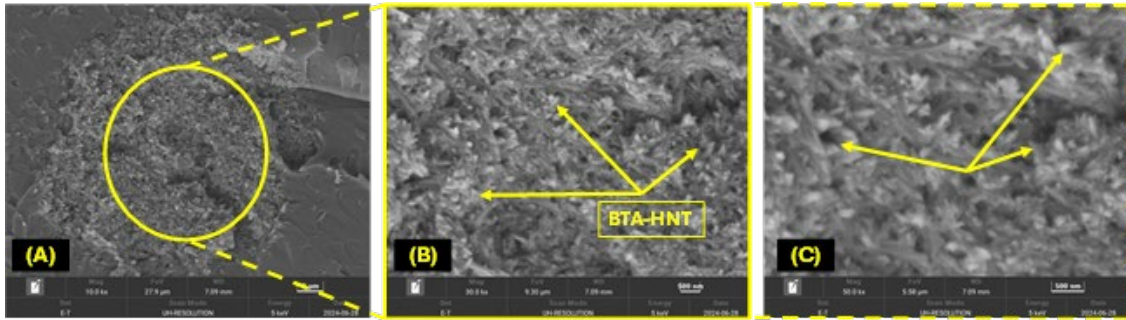


Fig. 8 FE-SEM of BTA-HNT at (a)10kX magnification; (b) 30kX magnification; (c) 50kX magnification

The FESEM morphological analysis of EP coatings at varying magnifications demonstrates a uniform and smooth surface. As shown in Fig. 9A (10.0 kX magnification) and Fig. 9B (20.0 kX magnification), the surface texture remains consistent, with no detectable inclusions or encapsulated materials.

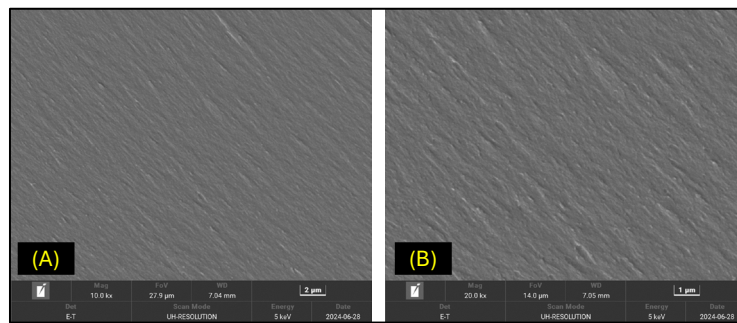


Fig. 9 FE-SEM of EP at (a)10kX magnification; (b) 20kX magnification

Overall, the FE-SEM analysis of MC-BLO, BTA-HNT, and EP coatings highlights the importance of optimized encapsulation processes and stringent control to ensure the performance and integrity of smart coatings.

3.2 Adhesion Properties of Smart Coating

3.2.1 Scratch Test

Fig. 10 presents the scratch test results for the EP, SL-SC, and DL-SC coatings after 14 days in a 3.5 wt% NaCl solution. On day 0, the EP coating showed no rust, but by day 7, rust began to develop, and it worsened by day 14, indicating its poor long-term protection. The SL-SC coating initially showed no rust, with visible microcapsules of MC-BLO and BTA-HNT. By day 7, a protective oxide layer had formed, suggesting partial self-healing. Although some corrosion was observed by day 14, the oxide layer remained, providing better protection than EP. DL-SC outperformed both coatings, showing no rust on day 0, a protective oxide layer by day 7, and only minimal corrosion by day 14, demonstrating the effectiveness of its multilayer design.

A comparison indicates that EP provided insufficient long-term protection, with rust emerging by day 7 and worsening by day 14. Although SL-SC exhibited partial self-healing, corrosion signs were visible by day 14, underscoring its limitations. In contrast, DL-SC showed the best performance, exhibiting minimal rust and corrosion by day 14. The self-healing in both SL-SC and DL-SC was facilitated by the release of encapsulated corrosion inhibitors, such as MC-BLO and BTA-HNT, which formed protective barriers to prevent further damage. BTA-HNT contributed both structural reinforcement and chemical protection by passivating the metal surface with benzotriazole [23]. This study highlights the significance of optimizing encapsulation processes to improve the performance of smart coatings, aligning with the findings of Hassanein et al., [16].

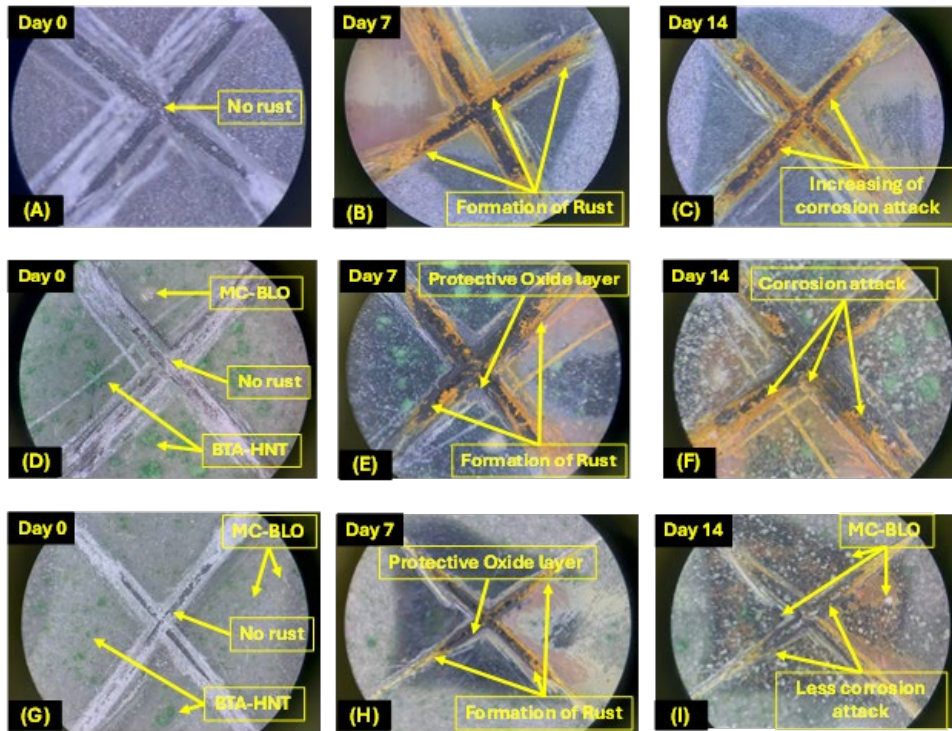


Fig. 10 (a, b, c) EP coating; (d, e, f) SL-SC; (g, h, i) DL-SC after immersion

3.2.2 Adhesion Strength

Fig. 11 presents the adhesion strength test results for EP, SL-SC, and DL-SC coatings, both before and after immersion in a 3.5 wt% NaCl solution. Initially, the adhesion strengths were recorded at 1.92 MPa for EP, 1.72 MPa for SL-SC, and 1.77 MPa for DL-SC. Following immersion, these values decreased to 0.92 MPa for EP, 1.05 MPa for SL-SC, and 1.22 MPa for DL-SC. EP coating initially exhibited the highest adhesion due to its pure matrix which lacks self-healing properties resulting in a significant decrease in adhesion strength.

SL-SC, containing self-healing agents, demonstrated improved corrosion resistance; however, its single-layer structure still led to a 38.95% reduction, indicating that a single layer is inadequate for long-term durability. Similar findings were reported by Hassanein et al., who noted a gradual decrease in the effectiveness of SL-SC under harsh conditions [17]. DL-SC performed the best, with the lowest reduction of 31.07%, due to its double-layer structure and self-healing agents in both layers, which created stronger protection against corrosion compared to EP and SL-SC.

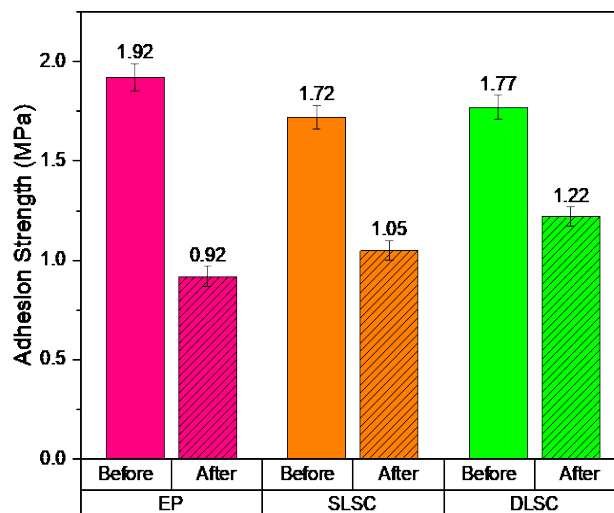


Fig. 11 Adhesion strength of EP, SL-SC and DL-SC

In summary, the adhesion strength of DL-SC retained 68.93% of its adhesion strength after immersion in a 3.5 wt% NaCl solution in contrast to 61.05% for SL-SC and 47.92% for traditional epoxy demonstrating its exceptional long-term corrosion protection and durability. These findings highlight the benefits and sustainability of multilayer smart coatings over single-layer and pure epoxy coatings [16].

4. Conclusion

In conclusion, this study assessed the corrosion protection offered by single-layer and double-layer smart coatings (SL-SC and DL-SC) that incorporate encapsulated BTA-HNT and MC-BLO. Comprehensive characterization using FTIR and FE-SEM confirmed the successful encapsulation and integration of self-healing agents and corrosion inhibitors. Performance evaluations, including scratch and adhesion tests, revealed that DL-SC outperformed SL-SC, exhibiting superior corrosion resistance. The retained 68.93% of its adhesion strength after immersion in a 3.5 wt% NaCl solution highlights a robust self-healing mechanism that successfully repaired the damage, ensuring coating integrity over time. These results support the goal of this study by highlighting DL-SC's significant improvement over traditional coatings and SL-SC, making it a promising solution for industrial applications where long-term durability and reduced maintenance are critical. Additionally, DL-SC aligns with sustainable practices, providing an innovative approach to corrosion protection. Future research should focus on scaling these coatings for large-scale industrial use and testing their performance in diverse real-world conditions, such as varying temperatures, to ensure practical applicability.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

Study conception and design: Sitti Shalyza Qasidah Binti Salleh, Puteri Sri Melor Megat Yusoff; **Data collection:** Sitti Shalyza Qasidah Binti Salleh; **Analysis and interpretation of results:** Sitti Shalyza Qasidah Binti Salleh, Nur Fahanis Che Lah, Muhammad Yasir; **Draft manuscript preparation:** Sitti Shalyza Qasidah Binti Salleh, Muhammad Yasir. All authors reviewed the results and approved the final version of the manuscript.

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