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Research Article

Fabrication of Chia oil encapsulated biobased poly (Urea-formaldehyde) microcapsules for corrosion-resistant coatings

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ABSTRACT

In the present study, Chia oil (CO) was successfully encapsulated within poly(urea-formaldehyde) (PUF) shells via in situ polymerization, and the resulting microcapsules were incorporated into epoxy to develop sustainable microcapsule-based anticorrosive coatings. The fabricated microcapsules were characterized by optical microscopy, Fourier-transform infrared spectroscopy (FTIR), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and particle size analysis to evaluate morphology, chemical composition, thermal stability, and size distribution. Successful encapsulation of chia oil within the PUF shell was confirmed by FTIR spectral analysis, while satisfactory thermal stability of the developed microcapsules under elevated temperatures was demonstrated by thermal analyses. The formation of both nano- and micro-sized capsules with relatively uniform size distribution was revealed by particle size analysis. The anticorrosive performance of microcapsule-loaded epoxy coatings was investigated through long-term immersion tests conducted in 3.5 wt % NaCl and 0.2 M HCl media using scratched mild steel panels. Improved corrosion resistance and delayed degradation behavior were exhibited by microcapsule-containing coatings compared with neat epoxy coatings. Under saline conditions, comparatively superior resistance against corrosion progression over prolonged immersion periods was demonstrated by the 3.0 wt% microcapsule-loaded coating, whereas under acidic conditions, enhanced stability and delayed coating failure were exhibited by the 7.0 wt% formulation. The improved protective behavior has been attributed to the combined effects of enhanced coating barrier properties and the potential release of encapsulated chia oil at damaged regions, which may contribute to the formation of a secondary protective layer through oxidative polymerization. However, while enhanced corrosion resistance was indicated by immersion studies, direct experimental evidence of autonomous self-healing behavior was not established in the present work; the observed performance should therefore be interpreted as improved corrosion resistance with potential healing functionality. The developed microcapsule-based coatings are demonstrated to hold promising potential as environmentally sustainable, corrosion-resistant protective coatings for metallic substrates exposed to aggressive saline and acidic environments.

1. Introduction

Among the important advancements made in the field of materials science, the successful development of self-healing anticorrosive coatings designed to reduce the corrosion on a various substrate, especially metallic surfaces gained significant industrial appreciation [1–4]. These anticorrosive coatings emerged as wonder materials for several industries, including automotive, aeronautics, oil and gas, and marine engineering which faced some serious challenges of corrosion over a long period [2]. Fundamentally, when the surface of the Iron substrate

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or a metal deteriorates due to chemical or electrochemical reaction with the surrounding environment, this phenomenon is known as corrosion. The corrosion of Iron surface is often categorised as oxidative corrosion, electrochemical corrosion and carbon dioxide corrosion [5–7]. The term oxidative corrosion in the present context is used to describe the direct corrosion of the iron substrate with the surrounding media, resulting into an oxidative chemical reaction. Since oxidation accounts for the majority of this corrosion, a layer of loose oxide forms on the metal surface. Under dry conditions, the corrosion of the metal substrate relatively proceeds slowly because the thin layer of oxidised iron (FeO) formed on the surface of the metal prevents further steel corrosion as a protective film at room temperature. Enhanced oxidative corrosion has been observed in the environments with higher temperatures and relatively higher humidity levels. The losses associated with damages caused due to corrosion affect all types of domestic, government and industrial infrastructure(s) which are exposed directly to the water and air [8–11]. The most affected infrastructure is associated with the renewable energy generation due to its direct deployment and contact with the air, water and soil thus, alarming us on the sustainability and life cycle claims [12,13]. The estimated global losses due to corrosion, which surpasses 2.5 trillion USD annually, can be minimized by the incorporation of anticorrosive coatings [9,14–16].

It has been observed that mild steel is comprised of iron, carbon, and various other elements, and owing to their differing electrode potentials, numerous microcells are created. Under favorable conditions, within the anode region, iron undergoes oxidation, transforming into Fe^{2+} ions within the aqueous film over iron, while in the cathode region, dissolved oxygen is reduced to form OH^- ions. These ions then combine to form $Fe(OH)_2$, which then further oxidized to form $Fe(OH)_3$ appearing as a reddish-brown, loose and flaky rusting layers shown by the chemical equations during corrosion process [17,18]. In some conditions, where there is high probability of carbon dioxide, under ambient conditions its negative impact on metals has not been observed, however transported supercritical carbon dioxide (sCO_2) is reported to damage steel in the aqueous medium. The localised corrosion mechanisms of steel by CO_2 consist of flow-induced local corrosion, immersion corrosion, and pitting corrosion [19].

1.1. Anti-corrosion chemical treatment

A chemical agent is applied to a surface of metal to create a protective layer or altered structure that greatly increases the resistance of metal surface towards the corrosion [20]. Coating [21], anodising, passivation [22] and phosphating are few examples of common chemical treatments to inhibit the corrosion. In severe conditions, these chemical treatments can successfully lower the rate of metal corrosion, increase the dependability of metals and including the service life [22–24]. In recent years, attention has been drawn by protective coatings among researchers worldwide, and promising anticorrosive coatings have been developed [25–31]. Traditional protective coatings are commonly required to undergo manual repair after being damaged, which in turn leads to increased maintenance costs and downtime in the production process [32,33]. In the development of self-healing coatings, sophisticated chemical engineering and functional materials were used to make the system engineers capable of autonomously repairing minor damages [34]. Immense emphasis has been given towards the design and development of smart coating materials consisting of different types of stimuli responsive coatings which inhibit the corrosion or slow down the mechanism of degradation responsible for the loss of materials properties [35]. Among the self-healing coatings which usually require synthetic and natural materials, the coatings and materials derived from renewable resources or from bio-based sources gained much attention due to sustainability factors and comparative synthesis advantages [15, 36,37]. The functional coatings are recovered automatically upon mechanical damage by microcracks, cut, stress and wear [7,38]. To achieve these coatings, intrinsic self-healing mechanisms, which are activated spontaneously, as well as extrinsic methods, which require the addition of supplementary materials, have been found to be implementable [39, 40].

1.2. Plant based oils as alternate biomaterials

At present, global attention has been directed toward renewable resources, bio-based, green and low-cost materials which can be produced on a mass scale and processed accordingly for industrial applications [41–44]. Towards the development of sustainable coatings, have been recognized for their desired properties to provide the suitable advantage during chemical process and applications [45]. The vegetable oils are classified as drying, semi-drying and non-drying oils [46]. The drying oils are composed of different triglycerides derived from one molecule of glycerol and three molecules of unsaturated fatty acids [47]. The higher percentage composition of oleic acid, linolenic acid, linoleic acid and eleosteric acid structurally contain the desired unsaturation which is responsible for the drying rate of vegetable oils [46]. The drying oils have been known to be applied as protective coatings of woods, domestic structures and metallic utilities [48,49].

The term "microcapsule-based potential healing coatings" was introduced for the first time by White et al. [34] and coworkers, by whom a polymeric functional material containing microcapsules was developed, and upon penetration of cracks, the microencapsulated healing agents were released. Upon contact with an embedded catalyst, the healing agent was found to be polymerized, which eventually triggered the healing process [50,51]. Often, the healing process requires the catalyst (Grubbs' catalyst) to trigger the healing, however there are certain notable healing materials have been identified in which no catalyst is needed to initiate healing processes, which is exactly featured by the vegetable oils [52,53].

Microcapsule-based anticorrosive coatings have been derived from drying oils and have been reported to possess potential healing functionality, contributing to the development of smart functional coatings [54]. Very high commercial importance has been attributed to anticorrosive coatings, which have been recognized for their support of decarbonization and their value toward UNSDGs, sustainability, climate change mitigation, and environmental protection; consequently, significant growth and interest have been observed in this field [15,53, 55–58]. The drying oils are encapsulated into microcapsules/ nano capsules which upon embedding into epoxy are coated on the metal, wood and the desired surfaces. It has been observed that upon any physical damage in these coatings, the microcapsules containing healing agent(s) i.e., oils flow into the cracks and get solidified due to the auto-oxidation of oils in the presence of atmospheric oxygen and moisture [45,59–61].

1.3. Self-healing polymers and anticorrosive properties

The developed self-healing polymer matrix containing the Dicyclopentadiene (DCPD) as healing agent and Grubb's catalyst for Ring opening metathesis polymerization (ROMP), which led to the ROMP of DCPD polymer matrix was automatically self-healed i.e., at room temperature [62]. The LO (Linseed oil) encapsulated Polyurea-formaldehyde (PUF) microcapsules were developed by C. Suryanarayana et. al. and the paint containing these microcapsules showed effective anticorrosive property [53]. The Tung oil (TO) encapsulated microcapsules also demonstrated the satisfactory anticorrosive properties [52]. The dual core i.e., healing agent and corrosion inhibitor containing microcapsules were developed by [18–25], whereby better corrosion protection was exhibited by coatings incorporating these microcapsules compared to control panels, i.e., epoxy coatings without microcapsules i.e., epoxy coating without microcapsules [63–69]. The anticorrosive property of the healing agents is improved by the Urea formaldehyde (UF) microcapsules, which are supplied with linseed oil and mercaptobenzothiazole (MBT) as core material [63]. The anti-corrosive performance of multi-functional polyurethane

coatings has been improved by the development of multicore microcapsules that contain the two commercially available inhibitors, 2-mercaptobenzimidazole (2-MBI) and 2-mercaptobenzothiozole (2-MBT) where, superior performance was demonstrated by 2-MBT [70]. Developed microcapsules containing soybean oil were found to function as self-healing agents in paints and anticorrosive coatings [71]. A self-healable polyurethane coating was developed through the incorporation of the Diels-Alder cycloaddition healing agents in hydroxyl-functionalized polyester resin and uretdione crosslinker[72]. A Polyurethane (PU) coating embedded with microcapsules containing quinoline corrosion inhibitor and xylene core solvent was evaluated for its anticorrosive performance in acidic environments. Microcapsule-loaded PU coatings with a 4.0 wt% microcapsule content showed significant corrosion resistance, with a corrosion rate of 0.65 mm/yr [73]. The reported PU coatings have been substantially improved in terms of self-healing and anticorrosive properties by the incorporation of polyamido-amine (PAMAM) based polyurea microcapsules and Neem oil-based polyester amide microcapsules [74]. The self-healing and self-lubricating coating were developed by integrating microcapsules into epoxy coatings, resulting in coatings with dual-functional properties. As determined by electrochemical impedance spectroscopy, an impedance modulus four orders of magnitude higher than that of pure epoxy in NaCl solution was recorded for coatings with 10 wt% microcapsules, and this performance was sustained after 30 days of immersion [75]. An epoxy ester derived from tall oil-fatty acids was proposed for use as a healing agent for anticorrosive smart self-healing coatings. An artificial microcrack was observed to heal and covered by a new layer as a result of the self-healing function via auto-oxidation and self-healing and crosslinking reaction of the released epoxy ester [76].

Vegetable oil-encapsulated microcapsule-based anticorrosive coatings with potential healing functionality, including linseed oil, tung oil, soybean oil, and other bio-derived oils, have been extensively investigated for development of environmentally sustainable enhanced corrosion-resistant coatings. Among these, Chia oil (CO) has been attracting attention because of its high unsaturated fatty acid content and enhanced oxidative drying behavior, which may improve autonomous healing performance. In previous studies, CO-loaded microcapsules have mainly been evaluated for self-healing capability, anticorrosive performance, and drying kinetics relative to conventional oils such as linseed oil [77]. However, despite these developments, a comprehensive understanding of the structural integrity, encapsulation behavior, thermal stability, long-term stability of microcapsules, and their influence on coating performance remains limited.

Therefore, the present work was aimed to address some of the significant research gaps through the systematic fabrication and integrated characterization of CO-loaded poly(urea-formaldehyde) (PUF) microcapsules. Microcapsule formation and stability during fabrication were evaluated by optical microscopy, and encapsulation efficiency, particle size distribution, surface morphology, thermal degradation behavior (TGA), thermal transitions (DSC), and structural characteristics were also assessed whereas most of them were not included in the previous report. Furthermore, the fabricated microcapsules were incorporated into epoxy coatings and were evaluated under saline and aggressive corrosive environments to establish the relationship between microcapsule properties and anticorrosive performance. Through this integrated approach, deeper insight into the design of thermally stable, bio-based microcapsules was provided, and current understanding was extended beyond previously reported CO-based systems, thereby contributing to the development of sustainable anticorrosive coating technologies.

2. Experimental

2.1. Materials

Urea, ammonium chloride, resorcinol, polyvinyl alcohol, sodium chloride, and hydrochloric acid were purchased from SD Fine Chemicals (India). Formaldehyde was obtained from Fisher Scientific. Sodium lauryl sulfate (SLS) was procured from Finar Chemicals (India). Chia seed oil was supplied by Vintage Aroma, Jaipur, India, while epoxy resin and hardener were obtained from Shree Surya Coatings, Nashik, India.

Urea and formaldehyde were used as precursors for synthesis of the poly(urea-formaldehyde) (PUF) shell material, which forms the encapsulating wall around the healing agent. Resorcinol was employed as a crosslinking modifier to enhance shell stability and polymerization efficiency, whereas ammonium chloride acted as a catalyst and buffering agent during in situ polymerization. Polyvinyl alcohol (PVA) and sodium lauryl sulfate (SLS) were utilized as stabilizing and emulsifying agents to promote emulsion stability and facilitate uniform microcapsule formation. Ammonia solution and hydrochloric acid (HCl) were used to regulate emulsion pH during synthesis, as polymerization and shell formation are strongly influenced by reaction pH. Chia seed oil (CO) was employed as the core (healing) material owing to its high unsaturated fatty acid content, which contributes to self-healing functionality and anticorrosive performance. Epoxy resin was used as the coating matrix, while the hardener served as a curing agent to promote crosslinking of the epoxy network. For immersion studies, sodium chloride (NaCl) and hydrochloric acid (HCl) solutions were used as saline and aggressive corrosive media, respectively, to evaluate coating performance under corrosive environments.

2.2. Fabrication procedure of Chia oil microcapsules

PUF microcapsules were synthesized using the oil-in-water (O/W) emulsion method, which is among the several synthesis techniques reported for encapsulation, as shown in Fig. 1. In addition to its many advantages, this method is simple, cost-effective, and can be easily scaled up for industrial requirements [78].

A pre-prepared 25.0 ml of 5.0 wt% aqueous polyvinyl alcohol (PVA) solution, 5.0 ml of 5.0 wt% aqueous sodium lauryl sulphate (SLS) solution, and 75.0 ml of deionized water were agitated at 750.0 RPM using a mechanical stirrer in a 500.0 ml three-neck flask. Subsequently, 6.76 g of urea and 0.5 g of ammonium chloride were added, and the pH of the emulsion was adjusted to 9.0 using ammonia solution. Then, 25.0 ml of chia oil was added dropwise, and the emulsion was stabilized for 30.0 min. Subsequently, 17.40 ml of 37.0 wt% formaldehyde was introduced into the stabilized emulsion, and the mixture was slowly heated to 66.0 ℃ at 780 RPM, while the pH was adjusted to 2.0–3.0 using diluted hydrochloric acid. Thereafter, 1.0 g of resorcinol was added as a crosslinking agent, and the reaction conditions were maintained for 3.5 h. During this period, the stability of the microcapsules was observed under an optical microscope at specific time intervals. Finally, the fabricated microcapsules were recovered using test sieves and dried at room temperature.

2.3. Analysis of Chia oil

The analysis of the Chia Oil was carried out by Fourier Transform Infrared Spectroscopy (Alpha II, Bruker) at 20.0 ℃.

2.4. Optical microscope images

The primary stability of the synthesized microcapsules was observed in an Olympus optical microscope (CX21i-EPL−3) under 40–100 X magnification at different time interval in deionised water as immersed media.

2.5. Surface morphology

The surface morphology of microcapsules was studied by the Field Emission Scanning Electron Microscopy (FESEM) model FEI Nova NanoSEM450.

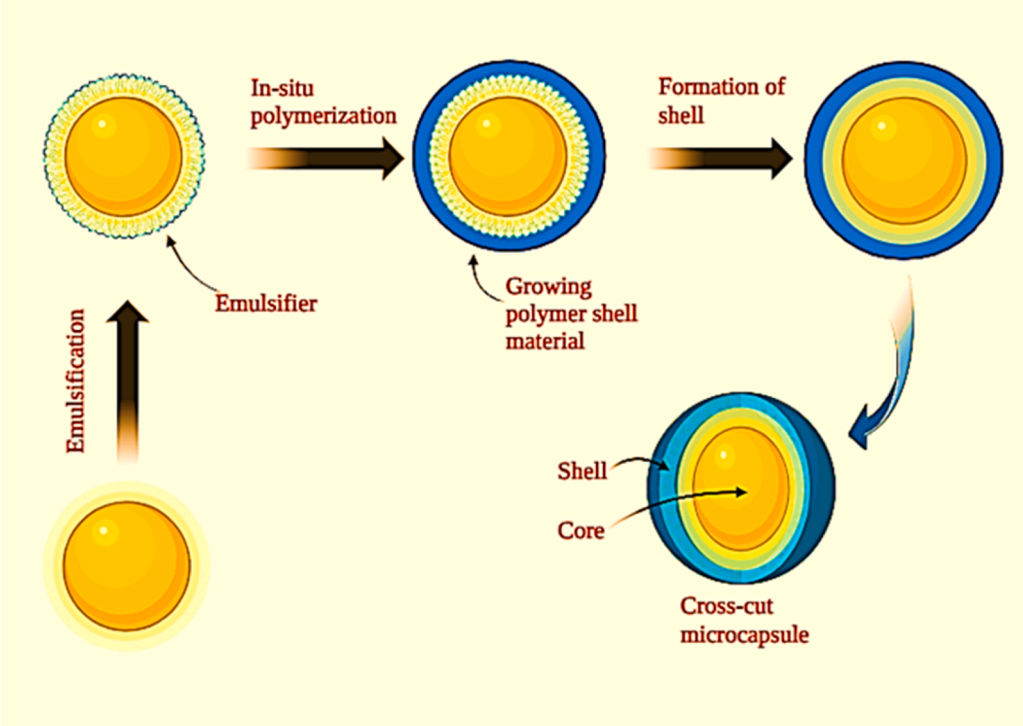


Fig. 1. Flow diagram showing the steps involved in the process of fabrication of CO microcapsules from Chia Oil obtained by oil in water emulsion method.

2.6. Thermal stability

The thermal stability of CO microcapsules was analysed by thermogravimetric analyser (TGA 50: Shimadzu) by heating the CO microcapsules, CO and PUF resins up to 600.0 ℃ by 10.0 ℃ min^{-1}.

2.7. Differential scanning calorimetry

Differential scanning calorimetry (DSC) was performed using a Hitachi NEXTA, Japan, Model: DSC 200, equipped with a Polyscience Nitra cooler. The samples were analysed from 30 ℃ to 300 ℃ at a heating rate of 10 ℃ per minute, in a nitrogen environment.

2.8. Particle size analysis

Particle size analysis of CO microcapsules was performed using a NanoPlus 3 Particle Size Analyzer based on the Dynamic Light Scattering (DLS) technique, which measures the hydrodynamic diameter of particles dispersed in a medium.

2.9. X-ray diffraction

X-ray diffraction (XRD) analysis of the fabricated materials was performed using a Rigaku D/MAX 2500 diffractometer for wide-angle X-ray diffraction (WAXD) measurements employing Cu Kα radiation. The instrument was operated at an accelerating voltage of 40 kV and a current of 150 mA. The samples were analyzed over a diffraction angle (2θ) range of 10°–80°, with a scanning rate of 4° min^{-1} to investigate the crystalline characteristics and structural features of the fabricated microcapsules.

2.10. Soxhlet extraction

The amount of core material encapsulated was determined using the Soxhlet extraction method, employing xylene as the extraction solvent. The extraction process enabled extract encapsulated Chia oil from the microcapsules, while the remaining dried shell material was subsequently weighed. The amount of core encapsulated was calculated according to the following equation,

$$\textit{Amount of core content in} \quad (\%) = \frac{W_m - W_s}{W_m} \times 100$$

where W_m is the initial weight of microcapsules, and W_s is the weight of the residual dried shell material after extraction. The calculated amount of core content represents the proportion of encapsulated core material relative to the total microcapsule mass.

2.11. Preparation of epoxy coating containing microcapsules

For immersion study we have prepared mild steel (MS) panels having dimension of 3.15 cm to 6.50 cm. Polished MS panels rubbed with emery paper to remove the any form of oil, metal dust particles and impurities present on metal panels. Then panels were washed with acetone and alcohol to avoid any form of microbial growth on metal panel surface.

The enhanced corrosion-resistant epoxy coatings containing different concentrations of microcapsules were prepared by incorporating 3–7 wt% microcapsules into the epoxy matrix through mechanical stirring to achieve homogeneous dispersion. The formulations were then applied onto pretreated mild steel substrates using a brush coating technique and cured at room temperature for 24 h. The coated samples were cured under room temperature to obtain fully crosslinked coatings. Efforts were made to maintain uniform coating deposition across all formulations during application. However, slight variations in coating thickness were observed due to changes in viscosity and the increasing concentration of microcapsules within the epoxy matrix.

The measured coating thickness ranged from approximately 490 μm to 670 μm, depending on the microcapsule content. The control epoxy coating (without microcapsules) exhibited an average thickness of approximately 490 μm, whereas coatings containing increasing microcapsule loadings showed progressively higher thickness values, reaching approximately 670 μm for the formulation containing 7 wt%

microcapsules, as summarized in Table 1. The increase in coating thickness with higher microcapsule content is attributed to the greater volume fraction of microcapsules incorporated within the epoxy matrix, resulting in increased coating build-up.

The coating thickness varied between 490 μm and 670 μm depending on microcapsule loading, with higher microcapsule concentrations resulting in increased coating build-up. The influence of thickness variation on corrosion performance was considered during interpretation of anticorrosion behavior, as coating thickness contributes to barrier protection.

Subsequently, they were subjected to mechanical impact, specifically make an artificial crack using a Tapariya blade. The procedure was performed to evaluate the corrosion resistance and possible protective response of microcapsule-containing coatings under mechanically damaged conditions. The scratched metal panels were cured for one day under the normal conditions. Subsequently, the samples were monitored for a duration of 24.0 h to detect any minor changes and images were taken of all metal panels before immersion in both medias for further record. Thereafter, all metal the metal panels were immersed in respective medias to conduct cyclic immersion studies. Images of all immersed panels from salt (3.5 wt% NaCl) and acid media (0.2 M HCl) were captured at 24.0 h time intervals to monitor the anticorrosive properties and alterations resulting from microcapsule-loaded epoxy coatings.

3. Result and discussion

3.1. Analysis of Chia oil

The structural analysis of Chia Oil has been done with Fourier Transform Infra-Red (FTIR) Spectroscopy shows the following absorption bands and the corresponding functional groups are assigned as shown in Table 2.

3.2. Fourier transform infrared spectroscopy

The FTIR spectra of pure Chia oil (CO), UF resin, and fabricated microcapsules are shown in Fig. 2. Pure CO exhibited a characteristic ester carbonyl (C═O) stretching vibration at 1747 cm^{-1}, corresponding to triglyceride ester groups, together with CH_2 stretching frequencies at 2923 cm^{-1} and 2856 cm^{-1}, characteristic of fatty acid chains. The UF resin displayed a broad absorption band at 3334 cm^{-1}, attributed to N–H/O–H stretching vibrations, while peaks at 1630 cm^{-1} and 1525 cm^{-1} corresponded to amide-related vibrations.

The microcapsule spectrum retained characteristic absorption bands associated with both CO and UF resin, indicating successful incorporation of the core material within the shell structure. Notably, the ester carbonyl peak shifted from 1747 cm^{-1} (CO) to 1722 cm^{-1} (microcapsules), suggesting interaction between the encapsulated oil and UF shell matrix. Additionally, the broad N–H/O–H stretching band shifted from 3334 cm^{-1} (UF resin) to 3301 cm^{-1} (microcapsules), indicating possible hydrogen bonding or intermolecular interactions during encapsulation.

The persistence of CH_2 stretching peaks at 2923 cm^{-1} and 2856 cm^{-1} further confirms retention of chia oil within the microcapsules. The observed peak shifts, together with retention of characteristic absorption bands from both shell and core materials, provide evidence for successful encapsulation of chia oil within the UF resin matrix.

Table 1
The formulation of epoxy containing microcapsules.

Sl. No.	Formulations	Epoxy (g)	Hardner (g)	Microcapsules (g)	Coating Thickness (μm)
1	Control Panels	3.80	1.20	0.0	490
2	3.0 wt%	3.70	1.15	0.15	490
3	5.0 wt%	3.61	1.14	0.25	620
4	7.0 wt%	3.53	1.11	0.35	670

3.3. Morphology

The synthesized microcapsules observed under optical microscope showed that circular and stable microcapsules dispersed in deionized water having variation in sizes as shown in Fig. 3.

The FESEM (Field Emission Scanning Electron Microscope) was employed to analyze the surface morphology and the size distribution of the microcapsules. The dried microcapsules showed a smooth and non-porous surface at different magnifications as shown in Fig. 4. The microcapsules exhibited a sphere-shaped morphology and a diverse size distribution, with a diameter of 1200.0 μm to about 1800.0 μm. The particle size affects the overall quality of the coatings and the amount of encapsulated oil, which subsequently impacts its therapeutic efficacy [79,80].The nano-capsules adhered on the microcapsules surface make it rough and change its spherical shapes [81].

3.4. Particle size analysis

The particle size distribution shown in Fig. 5 revealed a predominant nanoscale population with a primary peak centred at 276.1 nm, indicating successful formation of nanosized capsules. The cumulants analysis yielded a characteristic particle diameter of 362.4 nm, whereas the overall average diameter was 690.2 nm. In addition, a secondary broader population was observed around 3004.8 nm (~3.0 μm), corresponding to a minor fraction of larger capsules formed during synthesis. The obtained polydispersity index (PDI = 0.240) suggests moderate size distribution of the fabricated capsules.

These results confirm the successful formation of predominantly nanoscale capsules suitable for incorporation into epoxy coatings, while the limited presence of larger capsules indicates some degree of aggregation or broader particle distribution during encapsulation. The variation in size is affected due to various parameters i.e. stirring rate, shape of stirring blade, temperature, and pH [60]. The suspension containing microcapsules in the vicinity of stirring blade or away from the blade also plays crucial role for the size of microcapsules. The microcapsules in the vicinity of blade are smaller in size while the away from the blade are bigger in size [71].

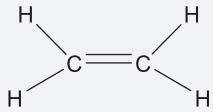
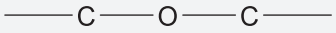
3.5. Thermogravimetric analysis

The thermogravimetric analysis (TGA) of pure Chia oil (CO), UF resin, and microcapsules is presented in Fig. 6. The microcapsules exhibited a multistep thermal degradation profile, indicating contributions from both shell and core materials. An initial minor weight loss below 100 ℃ was attributed to evaporation of physically adsorbed moisture and volatile compounds.

The first major degradation stage, observed between approximately 220 and 330 ℃, corresponded to decomposition of the poly(urea-formaldehyde) (PUF) shell material, resulting in approximately 18 wt % mass loss. This degradation is associated with cleavage of polymeric UF chains and breakdown of the crosslinked shell structure. The second degradation stage, occurring between approximately 335 and 500 ℃, was attributed to thermal decomposition of encapsulated Chia oil (CO), primarily associated with degradation of triglycerides and long-chain fatty acids. This stage contributed approximately 68 wt% mass loss, indicating substantial incorporation of chia oil within the microcapsules. Therefore, the TGA profile suggests a core material loading of approximately 68 wt%, reflecting efficient encapsulation of the corrosion-resistant material.

Furthermore, the degradation temperatures of microcapsules were shifted relative to pure components, indicating enhanced thermal stability due to encapsulation effects. The correspondence between decomposition regions of pure UF resin, pure CO, and fabricated

Table 2
Designation of absorption bands observed under FTIR spectroscopy analysis of Chia Oil.

Sl. No.	FTIR Absorption bands (cm^{-1})	Corresponding Functional Groups
1.	1458, 3015	 (Non-conjugated, unsaturated linkage)
2.	1747	 (Corresponding alkyl esters)
3.	1159	 (functional group of esters)
4.	2923, 2856	 (additional weak band around 2960 cm^{-1} corresponding terminal methyl groups)
5.	719	 (sequences of the aliphatic chain of fatty acids)

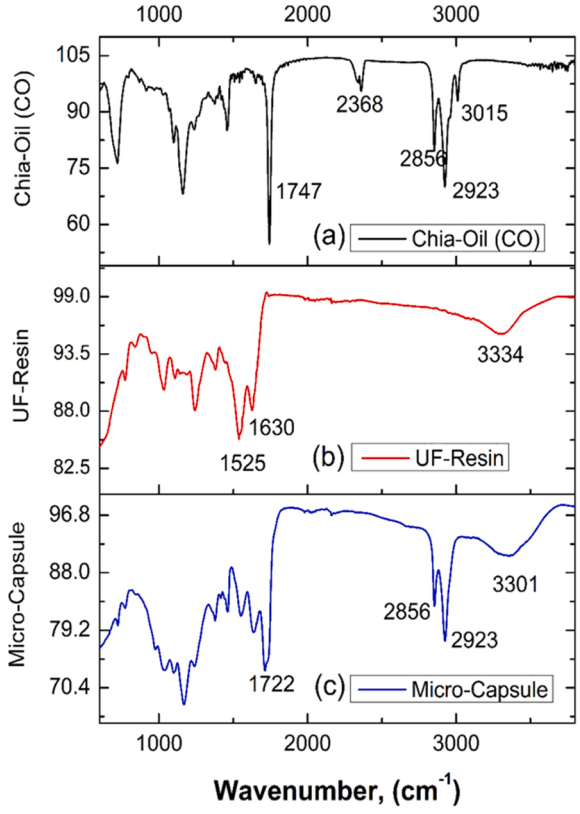


Fig. 2. FTIR spectrum observed under transmission mode during the comparative analysis of a) Chia Oil; b) UF resin; and c) CO Microcapsules.

microcapsules confirms successful formation of CO-loaded PUF microcapsules with improved thermal resistance compared with individual components.

3.6. *Differential scanning calorimetry (DSC)*

The stability of microcapsules is evaluated by differential scanning calorimetry (DSC). The DSC thermograms of microcapsules exhibited an initial endothermic peak, as depicted in Fig. 7, occurring between 40 ℃ and 100 ℃, which is ascribed to the evaporation of moisture and free formaldehyde [82]. The secondary endothermic melting (Tm) peak ranges from 150.0 ℃ to 200.0 ℃, indicating the melting of the PUF shell of microcapsules [83]. The microcapsules demonstrate significant stability at ambient temperature. The deterioration of the PUF shell commences at 225.0 ℃, as corroborated by thermogravimetric testing of the microcapsules [15].

3.7. *X-ray crystallography*

The diffraction peaks observed at 2θ 20.0°, 22.5°, and the prominent peaks at 2θ 38.0°, 44.0°, and 65.0° as shown in Fig. 8 indicate the high crystalline structures in UF resins [84,85]. CO Microcapsules demonstrate an increase in peak intensity around 20.0°, suggesting that the shell wall has formed around the oil droplets and has some kind of crystallinity in CO microcapsules as well. Additionally, the decrease in the peak at 2θ 38.0°, 44.0°, and 65.0° indicates a significant amount of oil has been encapsulated within the microcapsules.

3.8. *Soxhlet extraction*

The amount of core material encapsulated in CO-loaded microcapsules was evaluated using Soxhlet extraction with xylene as the extraction solvent. The microcapsules exhibited a maximum amount of core

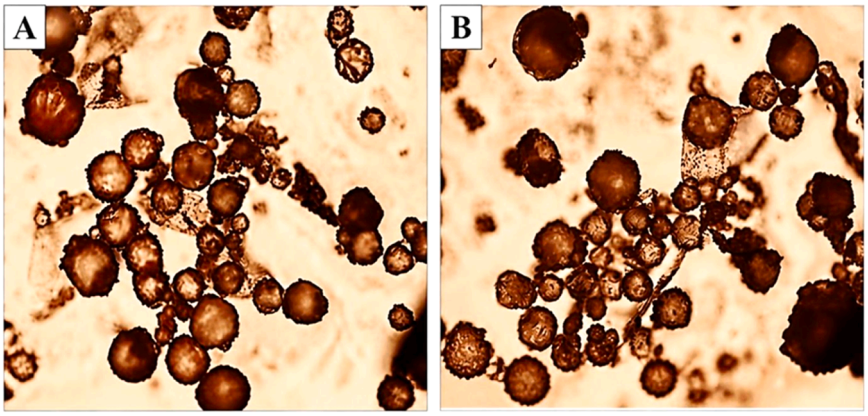


Fig. 3. Images of the fabricated CO microcapsules observed during their preparation under the Optical Microscope at a resolution of 40x; (A) after 2.0 h and; (B) after 3.0 h.

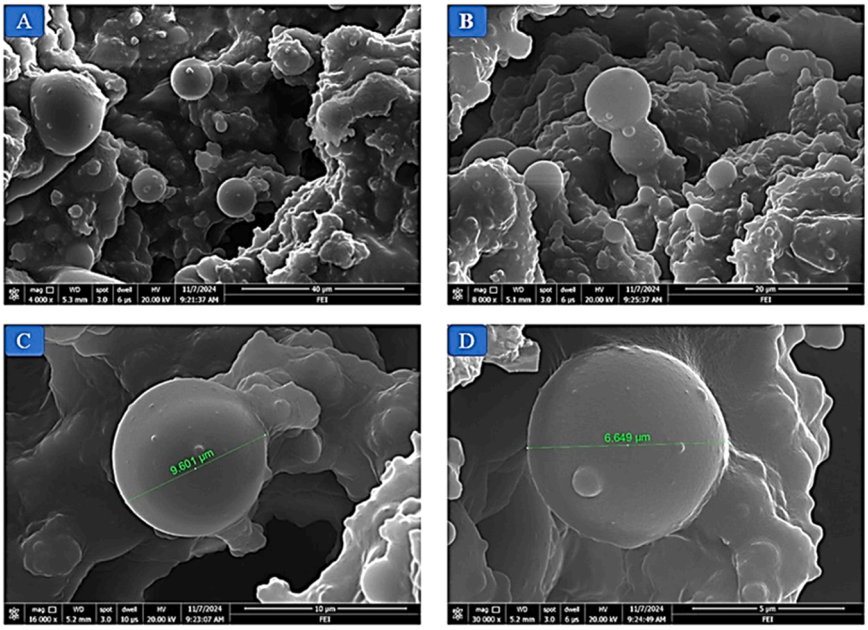


Fig. 4. FESEM images of CO microcapsules captured at different magnifications, and scales. (A) 4000x; 40 µm, (B) 8000x; 20 µm, (C) 16000x; 10 µm and (D) 32000x; 5 µm.

encapsulated is 70.0%, indicating effective incorporation of Chia oil within the poly(urea-formaldehyde) shell structure. The relatively high amount of core suggests successful encapsulation and adequate retention of the CO within the PUF.

3.8.1. Immersion studies in saline (3.5 wt% NaCl) and acidic media (0.2 M HCl)

The immersion performance of coated metal panels was evaluated in 3.5 wt% NaCl and 0.2 M HCl media to investigate the durability and corrosion resistance of epoxy coatings containing different concentrations of (CO) microcapsules. The coatings were intentionally scratched prior to immersion to assess their ability to maintain protection under damaged conditions.

3.8.2. Immersion behavior in 3.5 wt% NaCl solution

Visual observations before and after immersion are presented in Table S1. After 1 month of immersion, the 5.0 wt% formulation exhibited corrosion initiation along the artificial scratch, while the control and 7.0 wt% microcapsule coatings showed noticeable blistering. In contrast, the 3.0 wt% microcapsule coating maintained comparatively lower degradation, indicating improved resistance against saline-induced corrosion. After 2 months, substantial corrosion propagation was observed in the control, 5.0 wt%, and 7.0 wt% formulations, whereas the 3.0 wt% coating remained relatively intact with minimal corrosion traces. This suggests that incorporation of 3.0 wt% microcapsules provided optimal protection against electrolyte penetration and delayed corrosion initiation. Following 5 months of immersion as given in Table 3, corrosion became evident in all formulations; however, the 3.0 wt% microcapsule coating exhibited comparatively lower corrosion severity than the control and other microcapsule loadings. These observations indicate that moderate microcapsule incorporation enhances coating durability and prolongs resistance against corrosive attack.

Corrosion initiates when moisture and oxygen penetrate coating

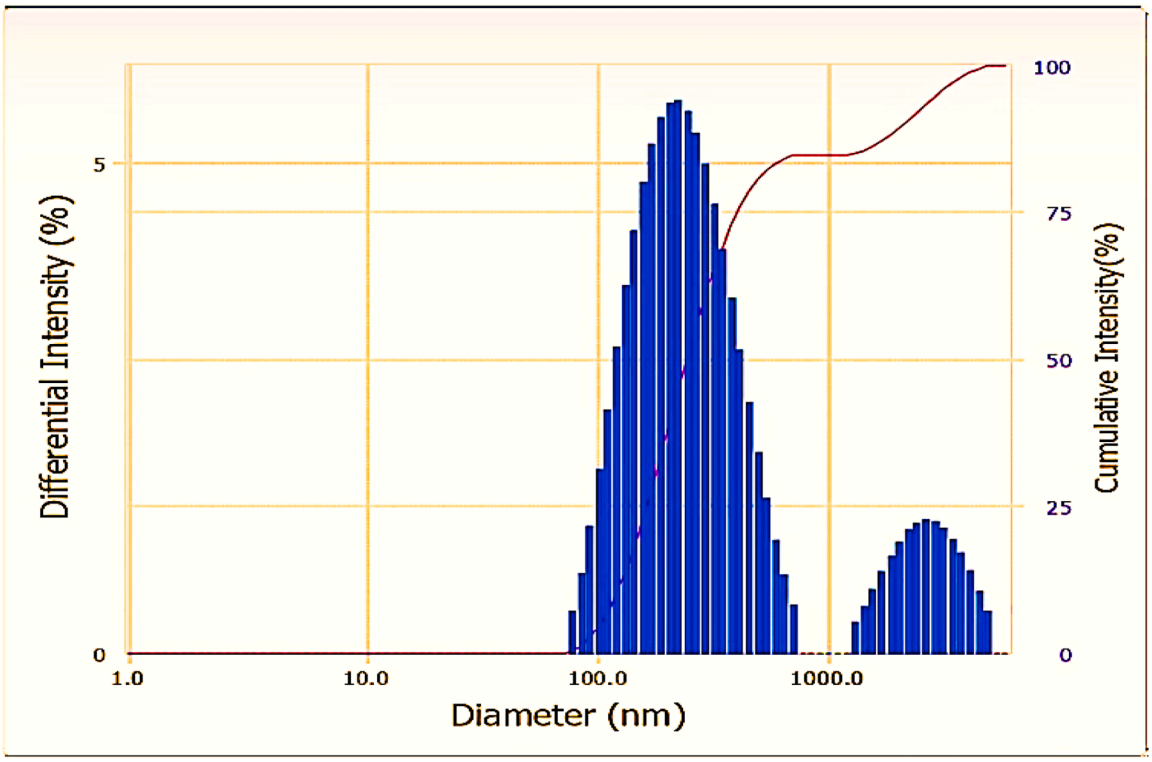


Fig. 5. Particle size distribution of the fabricated CO microcapsules.

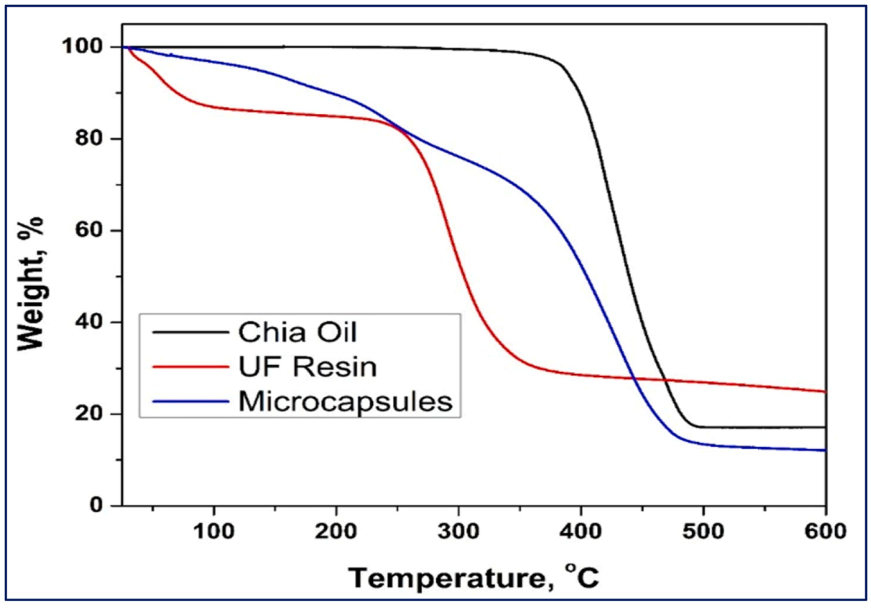


Fig. 6. Thermograms obtained during comparative thermogravimetric analysis of Microcapsules (CO microcapsules), UF resin and Chia Oil.

defects and reach the metal–coating interface, promoting electrochemical reactions and rust formation. In microcapsule-containing coatings, mechanical damage may rupture embedded microcapsules and release encapsulated chia oil into scratched regions. The released oil, containing unsaturated fatty acids, may undergo oxidative polymerization in the presence of oxygen and moisture, potentially forming a secondary protective layer that delays further electrolyte ingress [45]. Therefore, the improved long-term resistance observed for certain formulations may be associated with both barrier protection and potential healing effects, although immersion tests alone cannot

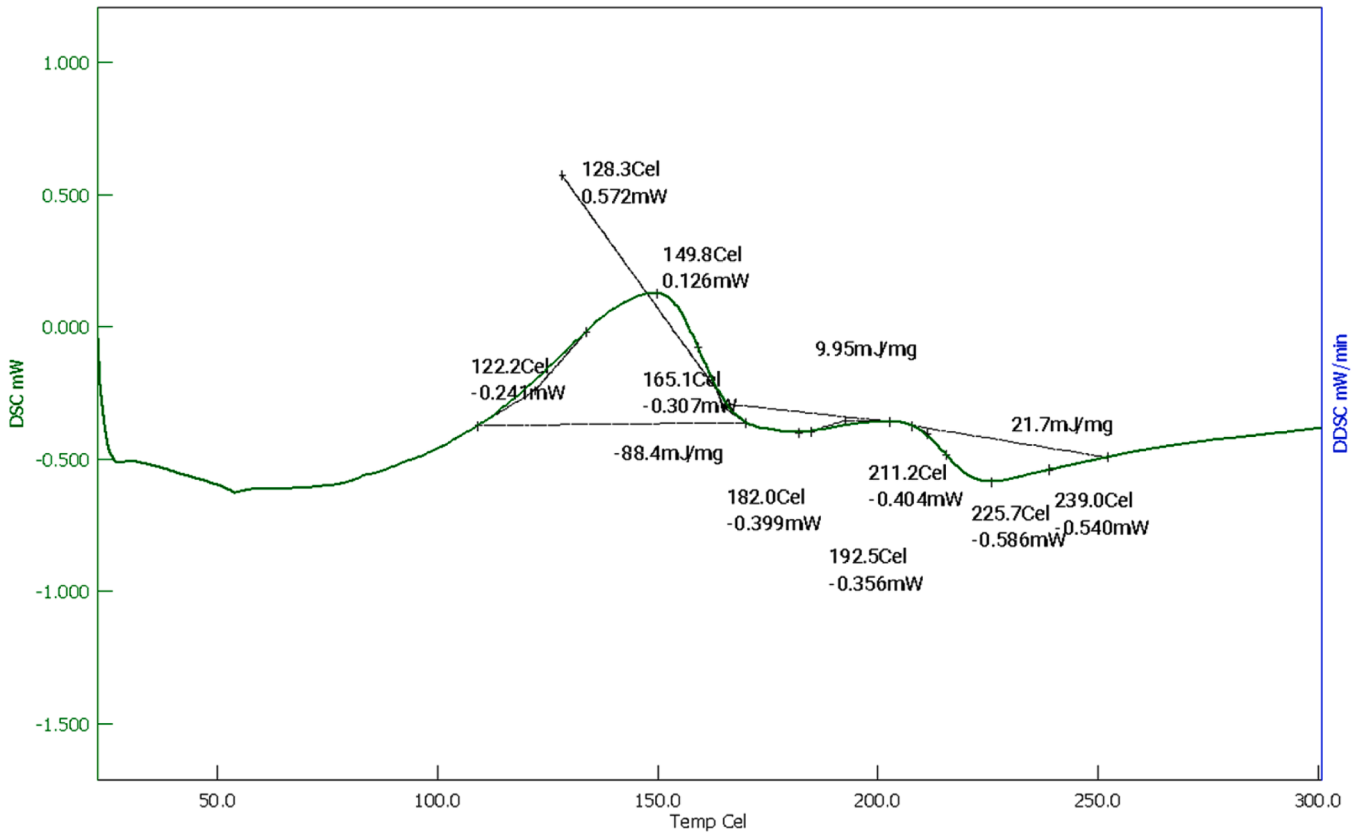


Fig. 7. Differential Scanning Calorimetry (DSC) thermogram of CO microcapsules.

conclusively confirm self-healing capability.

Although improved corrosion resistance and delayed coating degradation were observed in microcapsule-containing formulations, the present immersion studies alone do not provide direct evidence of autonomous self-healing behavior. Additional characterization techniques, including crack closure visualization, electrochemical recovery measurements, or healing efficiency analysis, are required to conclusively demonstrate self-healing functionality.

3.8.3. Immersion behavior in acidic medium (0.2 M HCl)

The coating degradation behavior in 0.2 M HCl was more aggressive due to the highly corrosive acidic environment. Images before and after immersion are shown in Table S2.

After 2 days, the 3.0 wt% coating exhibited pronounced blistering compared with other formulations. By 4 days, this formulation experienced severe adhesion loss, while the 5.0 wt% coating showed initial corrosion signs. Conversely, the 7.0 wt% microcapsule coating maintained structural integrity with minimal corrosion, whereas the control coating exhibited substantial blistering. After 6 days as given in Table 4, extensive corrosion was observed for the control, 3.0 wt%, and 5.0 wt% coatings, while the 7.0 wt% formulation remained comparatively resistant. Following 15 days of immersion, the control and lower microcapsule-loaded coatings experienced significant coating degradation and corrosion, whereas the 7.0 wt% coating showed only blistering with delayed corrosion initiation.

These results suggest that under highly acidic conditions, higher microcapsule loading (7.0 wt%) contributed to prolonged coating stability and enhanced corrosion resistance. The improved performance may be attributed to increased availability of encapsulated healing agents and greater resistance to corrosive media penetration. However, coating thickness variations associated with increasing microcapsule content may also contribute to enhanced barrier properties and should be considered when interpreting corrosion performance.

Although the present study report comparative study of neat epoxy coatings with microcapsule-loaded coatings, a direct comparison with coatings containing non-encapsulated chia oil was not performed. The incorporation of free oil into epoxy systems may negatively influence coating adhesion, curing behavior, and structural integrity, whereas encapsulation improves storage stability and enables controlled release of the healing agent. Future studies should include non-encapsulated oil systems to further quantify the specific advantages of encapsulation.

4. Conclusion

In the present study, CO-loaded PUF microcapsules were successfully fabricated via in-situ polymerization and incorporated into epoxy to develop environmentally sustainable corrosion-resistant coatings. Spherical morphology with effective encapsulation of CO was exhibited by the fabricated microcapsules, while successful microcapsule formation, morphological integrity, and higher thermal stability were confirmed by FTIR, SEM, TGA, and particle size analyses.

The coating performance under aggressive environments was significantly influenced by the incorporation of CO microcapsules into epoxy. Improved corrosion resistance and delayed degradation behavior were demonstrated by microcapsule-containing coatings compared with neat epoxy coatings, as evidenced by immersion studies conducted in 3.5 wt% NaCl and 0.2 M HCl media. Among the investigated formulations, prolonged resistance against corrosion progression was exhibited

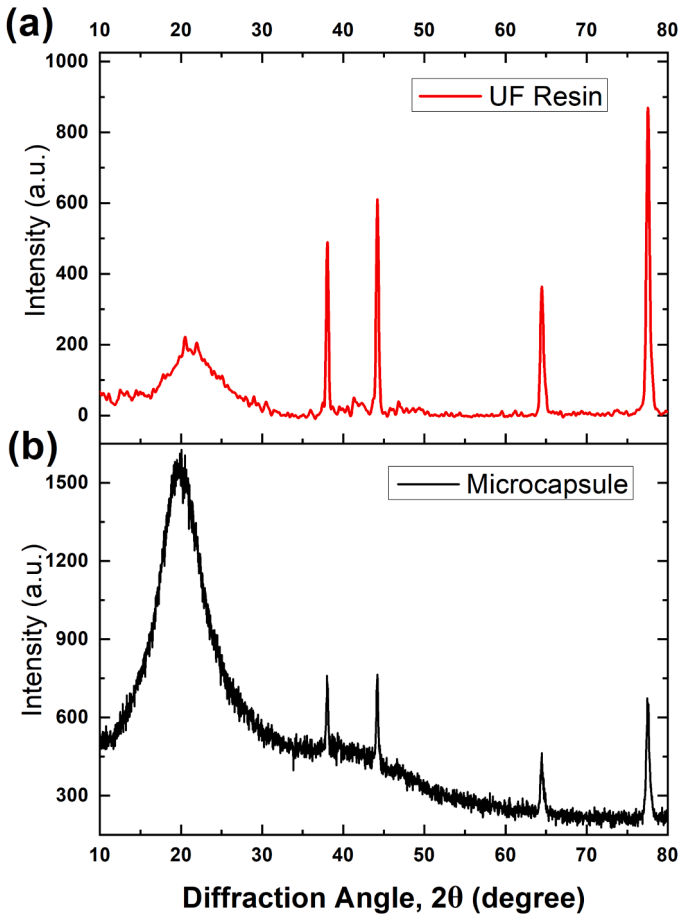


Fig. 8. X-ray Diffraction patterns observed during the comparative analysis of (a) UF resin; and (b) CO microcapsules.

Table 3
Summary of the output images observed at various stages during the salt immersion studies (before and after) of control panel and microcapsules containing coated panels (3.0 wt%, 5.0 wt% and 7.0 wt%) in 3.5 wt% NaCl solution. The microcapsules coating has been applied directly on the MS panels during the salt immersion studies. All the panels were coated without applying any primer.

Time Interval	Control Panel	3.0 wt%	5.0 wt%	7.0 wt%
Before Immersion				
After 5 Months				

by coatings containing optimized microcapsule concentrations, indicating enhanced protective performance. The improved behavior has been attributed to the combined effects of increased barrier properties and the potential release of encapsulated chia oil at damaged regions, through which the formation of a secondary protective layer may be contributed via oxidative polymerization. However, although enhanced corrosion resistance and delayed coating deterioration were observed, direct experimental evidence confirming autonomous self-healing behavior, such as crack closure visualization, electrochemical recovery analysis, or quantitative healing efficiency measurements, was not provided in the present study. Therefore, the observed performance should primarily be interpreted as improved corrosion resistance with

Table 4
Summary of the output images observed at various stages during acid immersion studies (before and after) of control panel and microcapsules containing coated panels (3.0 wt%, 5.0 wt% and 7.0 wt%) in 0.2 M HCl solution. The microcapsules coating has been applied directly on the MS panels during the acid immersion studies. All the panels were coated without applying any primer.

Time Interval	Control Panel	3.0 wt%	5.0 wt%	7.0 wt%
Before Immersion				
After 6 Days				

potential healing functionality, rather than as conclusive self-healing capability.

Overall, promise has been demonstrated by the developed CO-loaded microcapsule-based epoxy coatings as sustainable corrosion-resistant protective coatings for metallic substrates exposed to saline and acidic environments. Future investigations involving electrochemical impedance spectroscopy (EIS), crack closure monitoring, healing efficiency quantification, and long-term durability studies are recommended to be undertaken in order to establish and quantify the healing behavior of these systems.

List of abbreviations

Abbreviations	Description / Full form
CO	Chia oil [Salvia hispanica (Lamiaceae)]
DCPD	Dicyclopentadiene
ROMP.	Ring opening metathesis polymerization
FTIR	Fourier Transform Infrared Spectroscopy
HCl	Hydrochloric (Acid)
LO	Linseed oil [Linum usitatissimum L. (Linaceae)]
MS	Mild Steel
MBT	Mercaptobenzothiazole
MBI	Mercaptobenzimidazole
PUF	Poly(urea-formaldehyde)
RPM	Revolutions Per Minute / Rounds Per Minute
SEM	Scanning Electron Microscope
NaCl	Sodium Chloride
SBO	Soya Bean Oil [Glycine max (L.)]
SLS	Sodium Lauryl Sulphate
TGA	Thermogravimetric Analysis
TO	Tung Oil
PU	Polyurethane
PAMAM	polyamido-amine
O/W	Oil in Water

In memoriam

The authors wish to dedicate this work to the memory of Mr. Vikas C. Patil and his Father Mr. Chudaman Patil. Mr. Vikas made meaningful contributions to this work, including the preparation of graphical representations and figures. His dedication and creative effort are reflected in the visual presentation of this research. He is deeply missed by all who had the privilege of working alongside him.

CRediT authorship contribution statement

Chetan B. Patil: Writing – review & editing, Writing – original draft, Supervision, Project administration, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Priyanka S. Shisode:** Writing – review & editing, Resources, Project administration, Investigation, Data curation. **Pradip S. Patil:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Methodology, Formal analysis. **Omkar S. Kushwaha:** Writing – review & editing, Writing – original draft, Validation, Supervision, Software, Data curation. **Pramod P. Mahulikar:** Writing – review & editing, Supervision, Project administration. **Akash N. Ghoti:** Software, Resources, Data curation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.nxmate.2026.102849.

Data availability

The original data are available from corresponding authors upon reasonable request.

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