Utilization of bark extract of *Phyllanthus emblica* as a sustainable corrosion inhibitor to reinforced concrete infrastructures in aggressive environments

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Abstract. Reinforced concrete (ReC) infrastructures are used widely in construction due to their durability and strength. However, the premature corrosion of reinforced concrete (ReC) infrastructures poses a significant global problem. Sustainable corrosion inhibitors from plant extracts offer a promising solution for mitigating reinforced concrete corrosion (ReCC). This study examines the anti-corrosive effects of bark extract of the *Phyllanthus emblica* (BEPE) tree as a concrete additive in aggressive environments. Fifteen rectangular ReC beams were exposed in simulated industrial and marine environments and ambient atmospheric conditions using concrete composite (CC) with and without different concentrations of BEPE (500 ppm, 1000 ppm, 2000 ppm, and 4000 ppm). The electrochemical analysis of the reinforced-mild steel (ReMS) rod involved measuring electrochemical corrosion potential (ϕ_{Ecor}) and electrochemical resistivity (R_E), according to ASTM C876-91 standards for seven months. The phytochemical screening, UV-visible, and FT-IR spectroscopic analyses confirmed the presence of secondary metabolites in BEPE, which create a protective layer on the steel surface, reducing corrosion of the ReMS rod. Higher concentrations of BEPE demonstrated better corrosion inhibition in all environments, with shifts of ϕ_{Ecor} to more positive than -126 mV and R_E to more than 20 KΩ.cm value, indicating reduced corrosion of the ReMS rod in CC exposing in three different environments (i.e., normal, industrial and marine). Optical image analysis of the ReMS supported the electrochemical findings. This research could pave the way for developing new, environmentally friendly corrosion inhibitors for concrete additives.

1 Introduction

Reinforced concrete (ReC) is made by fixing metal rods, mesh, or plates of mild steel or stainless steel in a homogeneous aggregate of cement, gravel, and sand paste [1]. It boasts high flexibility, compressive, and mechanical strength, allowing it to be molded into various shapes, sizes, and qualities, as demanded. These versatile properties of the ReC have led to widespread use in engineering aspects for constructing highways, flyovers, bridges, airports, buildings, dams, etc. [2]. Concrete effectively handles large compressive loads due to its dense network of granular contacts. However, its porosity, brittleness, and moisture-covered interfaces weaken it under tensile stress, causing it to break suddenly when its elastic limit is reached [3]. This inherent weakness in tensile strength has led to the extensive use of the ReC. Steel or stainless steel rods, commonly called rebar, are strategically incorporated into ReC infrastructures to augment their tensile strength [4].

The early degradation of engineering ReC structures is due to the corrosion of internal reinforcements [5]. This corrosion causes the metal to expand, which leads to cracks, spalling of the concrete cover, and a decrease in thickness [6]. Factors such as harsh environmental conditions, concrete composition, water-to-cement ratio, and the types and quality of cement used all influence the corrosion of reinforcements [7, 8]. It reported that the high alkalinity of cement, with a pH between 11.8 and 12 during the initial stages of hardening, is reduced to a pH of 9.5 to 8.5 with the introduction of atmospheric pollutant gases [9]. Initially, the reinforcement is shielded from premature decay by a protective layer of iron oxide (mostly Fe2O3) formed due to the alkaline properties of the concrete mixture. However, this protective layer becomes compromised when aggressive environmental pollutants, such as gases, ions, and compounds, infiltrate, lowering the pH to below 10. As a result, the reinforcement becomes vulnerable to corrosion [10].

Reinforcement concrete corrosion can be prevented by using low or high-alloyed corrosion-resistant metals, applying protective metallic or organic coatings to the steel rebar, employing cathodic or sacrificial electrochemical protection techniques, modifying the concrete mix, and using inhibitors [11]. Inorganic and organic corrosion inhibitors have been highly effective in managing ReC corrosion in past

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years [12, 13]. They establish a protective anti-corrosive film on the metal, thus retarding the corrosion process by supplementing the polarization behavior at the anode or cathode, decreasing the migration of metallic ions from the metal surface, and enhancing the electrical resistance of the metal surface [14].

Historically, inorganic or synthetic organic inhibitors were widely employed to prevent ReC corrosion effectively. However, due to their significant environmental hazards, there is a growing trend of imposing restrictions on the use of these inhibitors. An alternative to traditional corrosion inhibitors is sustainable options derived from various parts of the plants. These plant-based inhibitors are widely accessible in Nepal and favored for use with reinforced steel in concrete. They meet all the criteria of green inhibitors, being low-cost, compatible, non-toxic, biodegradable, and suitable for industrial and marine construction applications [15].

Leaf-based extracts from plants native to Nepal, such as Vitex negundo [16], Catharanthus roseus [16], Mangifera indica [17], and Psidium guajava [18], have been used in the past five years as inhibitors to control ReC corrosion in the ambient atmosphere. As the author knows, the bark extract of the Phyllanthus emblica plant (BEPE) is applied as an efficacious corrosion inhibitor in ReC first. Also, the anti-corrosive action of BEPE investigated the ReC in different aggressive environments. Hence, this research focused on studying the comparative anti-corrosive behavior of BEPE in normal atmospheric conditions and simulated industrial and marine environments with the determination of ϕEcor and RE, using ASTM protocol [19]. Surface analysis is appertained to corroborate the electrochemical findings. This research endeavors to focus on formulating environmentally sustainable corrosion inhibitors for the commercial production of concrete additives.

2 Experimental methods

Bark weighing approximately three kilograms from the gooseberry tree (known as AMALA in Nepali) was collected from the botanical garden of Siddarthanagar Municipality-8, Rupendehi district (Fig. 1a). Initially, a herbarium specimen of the plant species was prepared for identifying the collected plant specimen. A Botanist of the National Herbarium and Plant Laboratory of Godabari, Lalitpur identified the collected plant as *Phyllanthus emblica* L using the prepared herbarium specimen of the gooseberry plant and the herbarium voucher deposited in the same office for record. Then, the collected bark was dried in a shaded area until completely dry (Fig. 1b). Once the bark was completely dried, it was ground into a powder using an electric grinder. Then the *Phyllanthus emblica* L powder was dipped in absolute methanol (1:2.5 ratio) and left for two weeks or more with regular stirring (see Fig. 1c). The supernatant was separated by filtration process using Whatman® Grade-1 filter paper [20]. The resulting filtrate was concentrated using a rotary evaporator to produce a crude extract free of methanol (Fig. 1d). This extract, referred to as BEPE, was stored in a refrigerator at 4°C [21] until characterization and used as a concrete additive, as described elsewhere.

A mild steel-reinforced concrete beam specimen, each measuring 40 cm long and featuring a cross-section area of (8×8) cm², was created using different sets of concrete mixes without (control) and with the bark extract of *Phyllanthus emblica* (BEPE) / 0.5M NaCl / 0.5M Na_2SO_4 solution. The concrete mix consisted of gravel, sand, and OPC cement in a 3:2:1 ratio, with distilled water to maintain a 0.45 water-cement ratio, following the ASTM C1582/C1582M-11 protocol [22]. Fifteen reinforced concrete beams were created using rust-free mild steel rods positioned in the center of a rectangular molding box with the concrete mixtures as earlier outlined [18]. To this purpose, 15 concrete mixture sets were used, as summarized in Table 1.

We conducted the electrochemical measurements for recording the ϕ_{Ecor} and R_E to assess the corrosion conditions of the ReMS rod using a two-electrode system, as illustrated in Fig. 2. The part of the ReMS rod that was exposed outside 2 cm of the beam and a saturated calomel electrode (SCE) was used as the working electrode and reference electrode, respectively. We used a multi-meter (OWON®XDM 1241) to measure two electrochemical properties, ϕ_{Ecor} and R_E, at four different surface points of the ReC beam. It did for identifying the three corrosion conditions of the ReMS in concrete beam based on the ASTM C876-15 protocol [19], as summarized in Table 2.

Table 1: Composition details of the fifteen sets of the concrete mix (CM).

S. No.	Sample Name	Composition of the concrete mix (CM)
1	Control	CM without (BEPE / 0.5M NaCl / 0.5M Na_2SO_4)
2	SiM	CM with 0.5M NaCl (simulated marine condition)
3	SiI	CM with 0.5M Na_2SO_4 (simulated industrial condition)
4	500 ppm BEPE	CM with 500 ppm BEPE
5	1000 ppm BEPE	CM with 1000 ppm BEPE
6	2000 ppm BEPE	CM with 2000 ppm BEPE
7	4000 ppm BEPE	CM with 4000 ppm BEPE
8	500 ppm BEPE-SiM	CM with 500 ppm BEPE +0.5M NaCl
9	1000 ppm BEPE-SiM	CM with 1000 ppm BEPE +0.5M NaCl
10	2000 ppm BEPE-SiM	CM with 2000 ppm BEPE +0.5M NaCl
11	4000 ppm BEPE-SiM	CM with 4000 ppm BEPE +0.5M NaCl
12	500 ppm BEPE-SiI	CM with 500 ppm BEPE +0.5M Na_2SO_4
13	1000 ppm BEPE-SiI	CM with 1000 ppm BEPE +0.5M Na_2SO_4
14	2000 ppm BEPE-SiI	CM with 2000 ppm BEPE +0.5M Na_2SO_4
15	4000 ppm BEPE-SiI	CM with 4000 ppm BEPE +0.5M Na_2SO_4

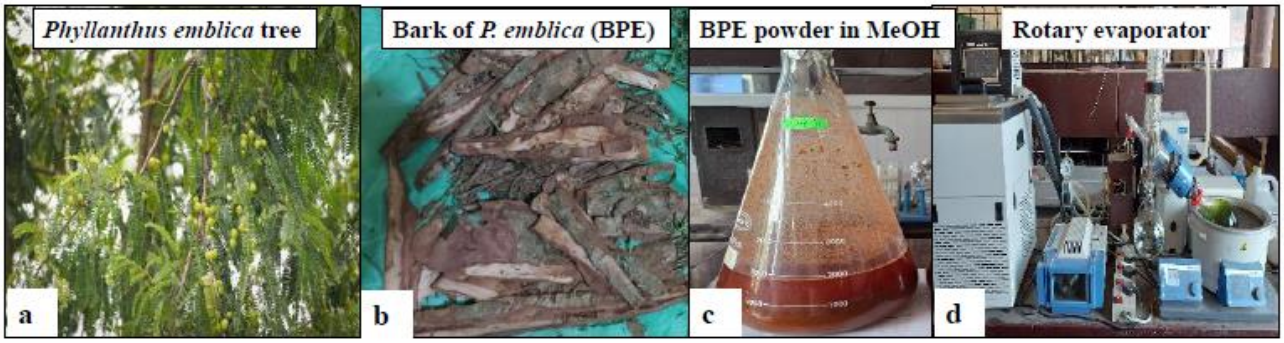

Fig. 1. Steps for the preparation of crude BEPE: (a) *Phyllanthus emblica* L tree, (b) dried bark pieces of *Phyllanthus emblica* L for making powder, (c) soaking bark powder in methanol, and (d) concentrating the filtrate using a rotary evaporator.

Table 2: Qualitative prediction the corrosion conditions of the ReMS, based on ϕ_{Ecor} and R_E.

ϕ_{Ecor} (mV vs SCE)	R_E (K**Ω**.cm)	Corrosion Conditions of ReMS in ReC Beam
> -125	> 20	Minimum corrosion (MnC < 10%)
-125 > ϕ_{Ecor} > -275	20 > R_E > 10	Intermediate corrosion (InC 10-90%)
< -275	< 10	Maximum corrosion (MxC > 90%)

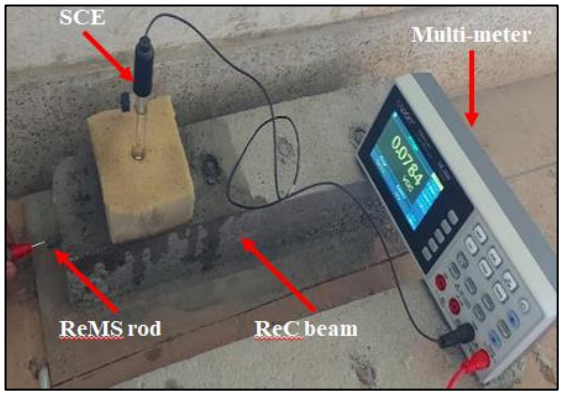

Fig. 2. Set up the 2-electrodes system for ϕ_{Ecor} and E_R measurements, using a digital multi-meter.

Functional groups in BEPE were characterized using UV-visible (SPECORD®200PLUS, Germany) and FT-IR spectroscopy (IR Affinity-1S, SHIMADZU CORP). It also conducted phyto-screening tests on the BEPE to find its secondary metabolites. The surface analysis of the ReMS rod before and after reinforcement in concrete mixes was performed using optical microscopy (Olympus SZ 61) at 6.7X magnification.

3 Results and discussion

Different secondary metabolites in BEPE have been identified through qualitative chemical screening tests, using various reagents and procedures, and the results are listed in Table 3. These secondary metabolites (phenols, alkaloids, flavonoids, glycosides, tannins, terpenoids, steroids, and carbohydrates, etc., containing hetero-atoms or unsaturated electron or aromatic ring compounds) are capable of forming a layer on the surface of reinforcing steel through adsorption. This layer acts as an anti-corrosive or passive layer, preventing corrosion of the reinforced metal [23]. Results from the chemical screening tests are in line with the existing literature [24, 25]. The secondary metabolites of the BEPE should be highly effective in forming an anti-corrosive diffusion layer on the surface of ReMS in concrete beam, thereby effectively preventing corrosion, as described in other studies [26].

The BEPE exhibited distinctive peaks for various secondary metabolites when subjected to UV-visible spectroscopy, as depicted in Fig. 3(a). The depicted compounds contain σ-bonds, lone pairs of electrons, chromophores, and aromatic ring compounds [27]. The UV-visible spectrum revealed absorption peaks at approximately 374 nm and 664 nm [Fig. 3(a)], signifying the presence of heteroatoms (N, O, *etc.*) and unsaturated ring compounds, per existing literature [28]. The FTIR spectra presented in Fig. 3(b) illustrate the identification of functional groups in BEPE. These functional groups are characterized by specific peaks, at around 3200-3400 cm^{-1} for O–H stretching vibration in aromatic compounds [29], 1608 cm^{-1} for aromatic C=C stretching [30], 1457 cm^{-1} for C–H stretching [31], 1206 cm^{-1} for –COOH group [32], C–H stretching at 1038 cm^{-1} for O–H or C–H bending of aromatics [33]. Similarly, the presence of secondary or tertiary amino in the BEPE is confirmed by two peaks in the 1100-1200 cm^{-1} range, indicating the C-N stretching vibration [34].

After performing qualitative chemical tests and spectroscopic analysis on the BEPE, we disclosed that the methanolic BEPE extract effectively prevents corrosion because it contains heteroatoms (O or N atoms), unsaturated bonds, or aromatic ring constituting secondary metabolites, as indicated by UV-Vis and FTIR spectroscopic analysis. These secondary metabolites adhere to the surface, blocking the entry of concrete corrosion-causing pollutants [35].

To assess the efficacy of BEPE in corrosion prevention from the early failures of ReMS in concrete composite, non-destructive and *in situ* electrochemical measurements were performed. These approaches evaluate three levels of corrosion probability: minimum corrosion (MnC), intermediate corrosion (InC), and maximum corrosion (MxC) of the ReMS corrosion in ReC. This evaluation is based on the recorded ϕ_{Ecor} and

Table 3. Primary tests of secondary metabolites in BEPE

Metabolites	Chemical tests	Observations	BEPE
Phenols	Two mL BEPE + 1 mL 10% lead acetate	White precipitate formed	+
Alkaloids	Two mL BEPE + 1 mL Dragendroff's reagent + 2 mL H_2SO_4 (warm)	Orange-red precipitate formed	+
Saponins	One mL BEPE with 2 mL distilled water (shaking)	Did not form foam	-
Flavonoids	One mL BEPE + 2 mL of 2% NaOH solution	Intense yellow color appeared	+
Glycosides	Two mL BEPE + 2 mL of each pyridine & sod. nitroprusside + NaOH	Pink color appeared	+
Tannins	One mL BEPE + 3-4 drops of 10% $FeCl_3$ solution (freshly prepared)	Turned the solution green	+
Terpenoids	Two mL BEPE + 2 mL chloroform + conc. H_2SO_4	Reddish-brown color	+
Steroids	Two mL BEPE + 2 mL chloroform + 2 mL acetic acid + conc. H_2SO_4	Violet to blue-green color	+
Carbohydrates	Two mL BEPE + 2 mL Molisch's reagent, shaking with Conc. H_2SO_4	Formation of violet ring	+

('+' sign refers to the presence & '—'sign refers to the absence)

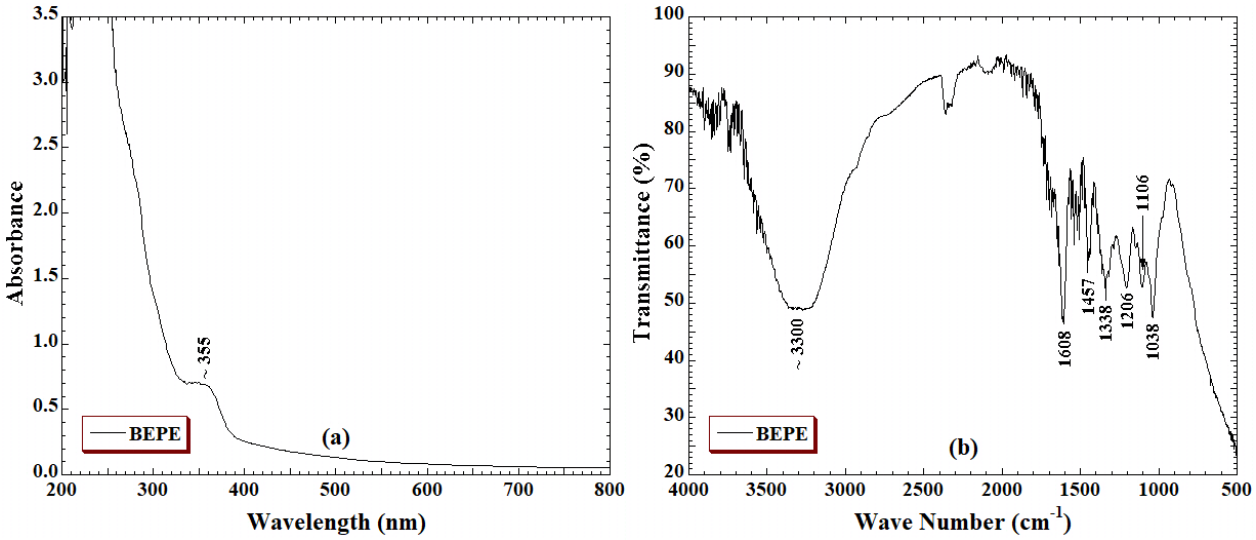

Fig. 3: (a) UV-visible and (b) FTIR spectrum of BEPE.

R_E values. The ongoing research monitors the changes in ϕ_{Ecor} and R_E over exposure intervals of up to 207 days. The investigation aims to assess the corrosion status and the probability of corrosion percentage conditions for the ReMS within the concrete beam.

The research is conducted without (control) and with the additions of 500, 1000, 2000, and 4000 ppm BEPE, BEPE + 0.5M NaCl, or BEPE + 0.5M Na_2SO_4, and the results displayed in Figs. 4, 5, and 6. The corrosion potential of all four points on three types of concrete beams (i.e., control, SiM, SiI) was periodically recorded over seven months. The average ϕ_{Ecor} for each beam specimen was estimated and the results are illustrated in Fig. 4(a). The average corrosion potential shifted from the high corrosion state (MxC) to the less corrosion possibility region (MnC) till 1-2 months of exposure. However, after two months of exposure, the corrosion potentials of the control-, SiM-, and SiI-beams shifted to the InC region, and it attained a steady state potential region till 207 days of exposure. The average ϕ_{Ecor} value of ReMS exposed to a control concrete composite (i.e., control CC) is slightly more positive value than the potential values in the SiM (0.5M NaCl) and SiI (0.5M Na_2SO_4) environment.

During the initial stages, i.e., within the first two months or less, the corrosion potential of these three concrete beams shifted in a positive direction, as illustrated in Fig. 4(a). This phenomenon is attributable to the highly alkaline nature, surpassing a pH of 11 or more, inherent in the concrete mix. As a consequence of exposure for two months or more to aggressive ions in marine and industrial surroundings, the potential transitioned towards the intermediate corrosion state due to shifting pH below 10.

When the concrete beam without BEPE additive (i.e., control beam) is exposed to normal atmospheric condition, the corrosion of the ReMS accelerates between 1-7 months of exposure time. During this period, the recorded average ϕ_{Ecor} value ranges between –126 to –275 mV (SCE) due to the formation of slightly unstable layer of film on the surface of the ReMS, as illustrated in Fig. 4(a). The instability of the thin film, presumably its formation and subsequent rupture on the ReMS surface, arises from the lower alkalinity of the concrete environments. It leads to the intermediate corrosion of the ReMS in CC, as elucidated in previous studies [36].

However, the addition of different concentrations of BEPE (500-4000 ppm) in three different environments - ambient, marine (BEPE + 0.5M NaCl), and industrial (0.5M Na_2SO_4) enhanced in shifting the average ϕ_{Ecor} of the ReMS from the intermediate corrosion zone to a

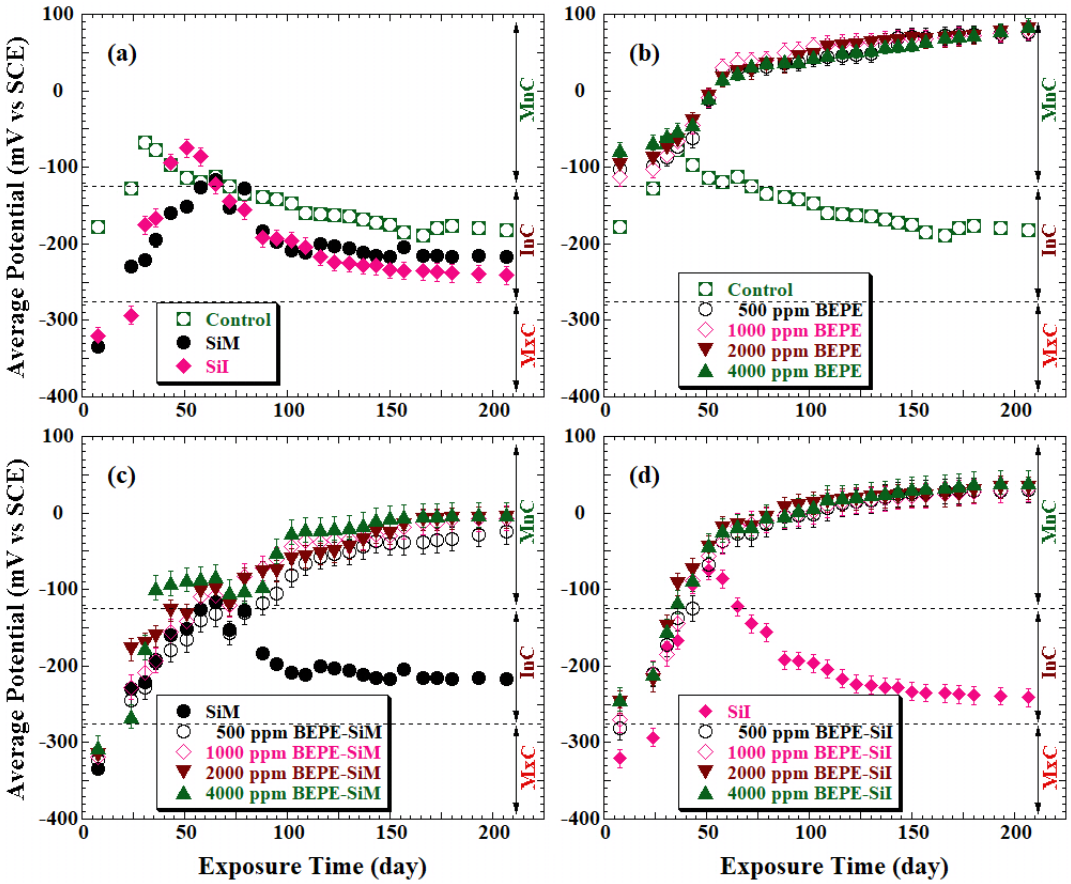

Fig. 4. Variation of average ϕ_{Ecor} of ReMS rod in concrete beam (a) without and with 500 ppm, 1000 ppm, 2000 ppm, and 4000 ppm of BEPE at (b) ambient (normal), (c) marine, and (d) industrial environment with an exposure time of 7 months or more.

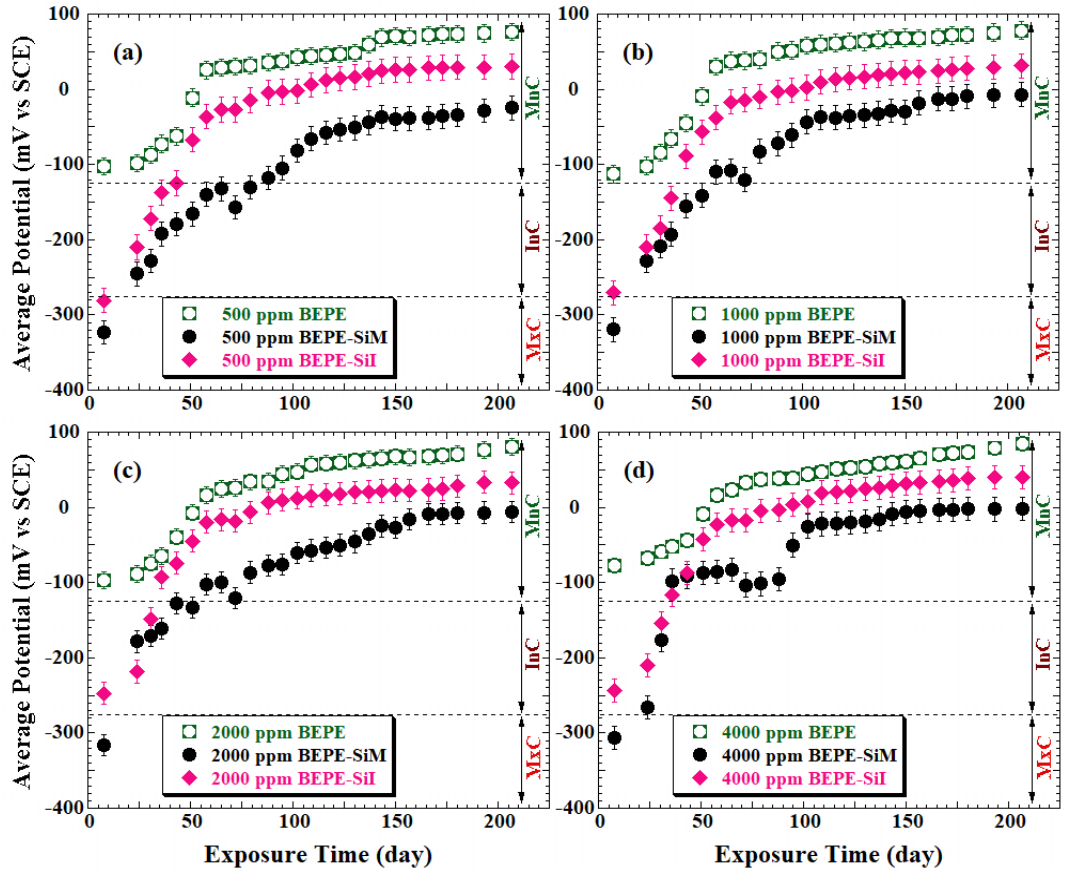

Fig. 5: Variation of average ϕ_{Ecor} of ReMS rod in ReC beams with (a) 500 ppm, (b) 1000 ppm, (c) 2000 ppm, and (d) 4000 ppm of BEPE at different environments with an exposure time of 7 months or more

minimal corrosion zone, as illustrated in Figs. 4(b), 4(c) and 4(d), respectively. However, there are no significant changes in ϕ_{Ecor} with the plant extract' s concentrations between 500- 4000 ppm, which signifies that the additions of 500 ppm or 4000 ppm BEPE do not give meaningful differences. Consequently, 500- 1000 ppm BEPE addition in the concrete mix enhanced the sufficient anti- corrosive properties of the concrete corrosion.

In three different environments - ambient, marine (BEPE + 0.5M NaCl), and industrial (0.5M Na_2SO_4) - the addition of 500, 1000, 2000, and 4000 ppm BEPE resulted in shifting the average ϕ_{Ecor} of the ReMS from the intermediate corrosion zone to a minimal corrosion zone, as shown in Figs. 5(a), 5(b), 5(c), and 5(d), respectively. As noticed in Figs. 5(a)-5(d), the ϕ_{Ecor} continuously increased into the low corrosion zone (ϕ_{Ecor} > -126 mV vs SCE) with exposure periods, with the final corrosion probability presumed to be less than 10% after 207 days of exposure in all three environments. In the ambient environment, the ReMS in concrete beam demonstrated a corrosion probability of less than 10% even at initial exposure time, as illustrated in Fig. 5(a). Conversely, in aggressive marine and industrial environments, the corrosion probability ranged between 10-90% at the initial exposure time till about two months, as demonstrated in Figs. 5(c) and 5(d), respectively. However, the addition of any concentration of BEPE shifted the average ϕ_{Ecor} values in a more positive direction than -126 mV (SCE), indicating a corrosion probability of less than 10%. Furthermore, the average ϕ_{Ecor} value in different concentrations of BEPE is higher in the ambient environment, indicating better anti-corrosive performance compared to the simulated industrial (SiI) or simulated marine (SiM) environment. Figures 5(a)-5(d) indicate that the anti-corrosive efficacy of 500-4000 ppm BEPE is lower in marine environments compared to ambient and industrial environments.

Similarly, the ER of each RCS was measured weekly at four different surface points, and the mean ER of each RCS in various corrosive environments, with or without sustainable corrosion inhibitors, is shown in Fig. 6. The figures indicate that the ER value exceeds 20 KΩ.cm when BEPE is added to the RCS after six weeks of exposure, suggesting minimal corrosion, as supported by ECP results. In contrast, the controlled RCS in both ambient atmosphere and aggressive environments showed almost steady values below 10 KΩ.cm, indicating a higher vulnerability to RCC. Thus, the electrochemical results from ECP and ER are consistent.Furthermore, the shift to more positive and noble ECP and ER values from the initial readings in all twelve RCS with varying BEPE concentrations indicates a reduced corrosion probability for MS rods in concrete exposed to different environments. When the extract is added to the concrete mix, it forms an anti-corrosive passive layer on the MS rod surface. This layer adheres through the formation of coordinate bonds between the metal and secondary metabolites in the extract or via physical attraction between the metal and phyto-molecules. Additionally, this adhered passive layer may block the ingress of pollutants, thereby reducing the corrosion of the reinforced metal rod [37].

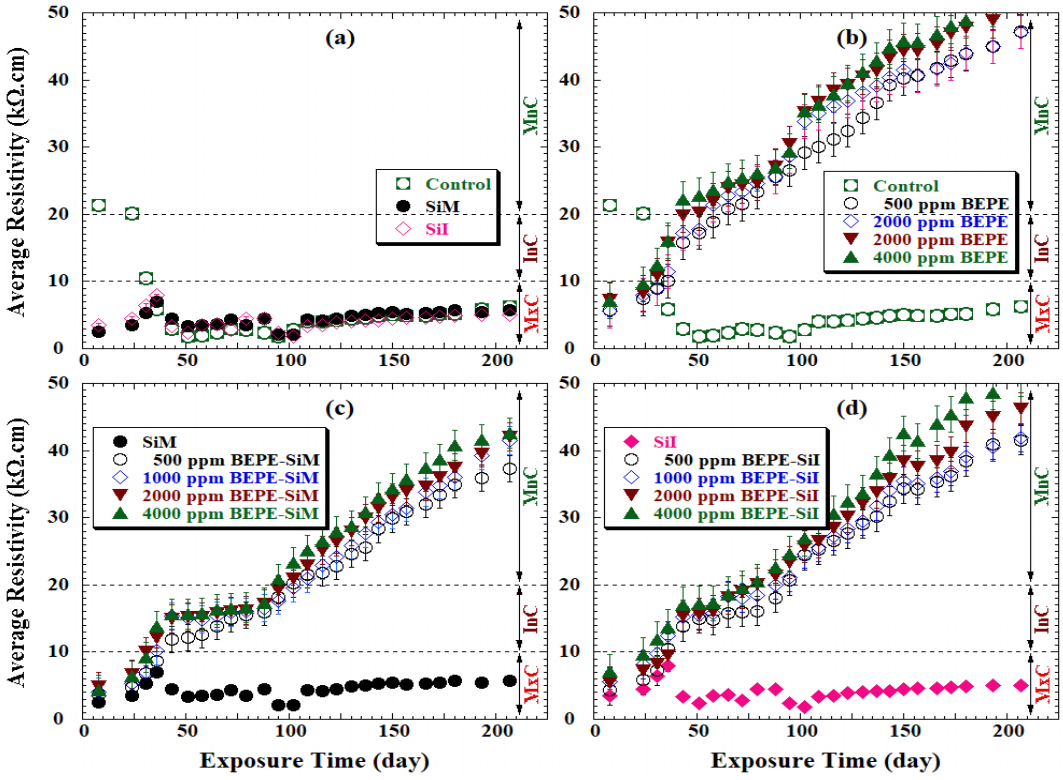

Fig. 6. Variation of the average resistivity (R_E) of ReMS rod in (a) ReC beams without BEPE (control), with 0.5M NaCl (SiM) and 0.5M Na_2SO_4 (SiI), and with different concentrations of BEPE in (b) ambient, (c) industrial, and (d) marine environments after approximately seven months of exposure

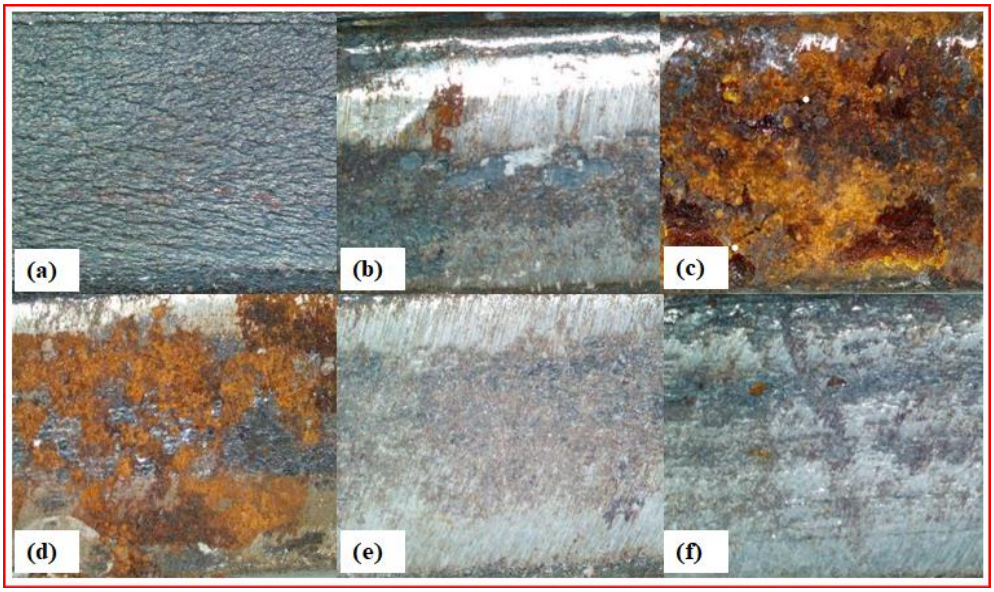

Fig. 7. Optical images of surface layers formed on (a) fresh MS rod and ReMS rod (b) in control concrete at ambient, (c) in industrial environment, (d) in marine environment, (e) in ReC beam with 4000 ppm BEPE at industrial and (f) in ReC beam with 4000 ppm BEPE in marine environment after seven months exposure.

Hence, electrochemical analysis confirms the potency of BEPE as indicated by the characterization of the extract through a phytochemical screening test, spectroscopic analysis, etc.

Surface analysis of MS rods before and after reinforcement in the concrete mix, both in control conditions and with 4000 ppm of BEPE, was conducted using optical microscopy in three different environments. Figure 7(a) shows the optical image of a fresh MS rod before reinforcement, while Figs. 7(b), 7(c), and 7(d) show MS rods reinforced in concrete without bark extract, exposed to normal, industrial, and marine simulated environments for 29 weeks or more. Comparative analysis reveals more corrosion products on the MS rod surfaces exposed to industrial and marine environments for about seven months than in normal conditions. Conversely, when 4000 ppm of bark extract was added to the concrete mix before exposure to industrial and marine environments for seven months, the MS rods showed no significant corrosion, as depicted in Figs. 7(e) and 7(f). These surface analysis results are consistent with those from the electrochemical analysis.

4 Conclusion

The methanolic BEPE extract has outstanding anti-corrosive properties for ReCC in various conditions, including ambient, industrial, and marine environments**.** The extract contains alkaloids, flavonoids, phenols, and steroids, confirmed from the phytochemical analysis using different chemical tests and spectroscopic techniques such as UV-visible and FTIR**.** The compounds with unsaturated bonds, aromatic systems, and heteroatoms like O, N, and S can create a protective film on the RMS surface that helps to prevent corrosion of the RCC**.** The electrochemical analyses conducted to measure ϕ_{Ecor} and R_E revealed a tendency towards positive and noble directions**.**

It suggests the corrosion-mitigating properties of BEPE in concrete**.** Furthermore, higher concentrations of BEPE in the concrete mix augment its anti-corrosion capabilities in ambient and aggressive environments**.** Optical image analysis validated the corrosion prevention effectiveness of BEPE in various conditions**.** This study represents a significant stride toward prolonging the service life of concrete engineering structures by employing innovative and sustainable corrosion inhibitors formulated from the waste parts of plants**.**

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