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Performance of pomegranate peel extract coated rebars in concrete

Authors:



Assist.Prof. Jeyanth Baskaran Department of Civil Engineering Bannari Amman Institute of Technology Erode, Tamilnadu, India jeyanthbaskaran5890@gmail.com Corresponding author



Prof. Sureshkumar Paramasivam, PhD. CE Department of Civil Engineering University College of Engineering Panruti, Tamilnadu, India erpsuresh@rediffmail.com

Jeyanth Baskaran, Sureshkumar Paramasivam

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This paper aimed to develop an affordable, eco-friendly, and accessible natural inhibitor derived from the peel of the pomegranate fruit. An extract was prepared from the pomegranate peel and utilised as a corrosion-resistant coating known as pomegranate peel extract (PPE) applied to the steel rebar. The corrosion resistance of the PPE-coated steel was evaluated using accelerated carbonation, impressed voltage, half-cell potential, weight loss, and accelerated corrosion tests. The carbonation test revealed that the uncoated steel rebar displayed patches, indicating carbonation, whereas the PPE-coated rebar was not affected, demonstrating an uncarbonated zone. Moreover, the half-cell potential test clearly indicated that the PPE-coated steel rod exhibited a moderate risk of corrosion with a potential of -247 mV. Particularly, the weight loss of the uncoated conventional steel was 40 % greater than that experienced by the PPE-coated rod. In the accelerated corrosion test, the PPE-coated rods took longer to develop initial cracks in the concrete, with durations of 182, 206, and 230 h for samples 1, 2, and 3, respectively, which were comparatively higher than those of conventional concrete. Thus, the test results of the PPE-coated steel were compared with those of the uncoated conventional steel, and the performance of the PPE-coated rod in terms of corrosion resistance was evaluated.

Key words:

corrosion, coatings, organic inhibitor, accelerated corrosion test, inhibition efficiency, carbonation, chlorination

Prethodno priopćenje

Jeyanth Baskaran, Sureshkumar Paramasivam

Učinkovitost armature premazane ekstraktom kore nara u betonu pri zaštiti od korozije

Cilj je ovog rada razviti troškovno pristupačan, ekološki prihvatljiv i lako dostupan prirodni inhibitor dobiven iz kore ploda nara. Ekstrakt je pripremljen od kore nara i upotrijebljen kao premaz otporan na koroziju, poznat kao ekstrakt kore nara (PPE), a nanosi se na čeličnu armaturu. Otpornost čelika premazanog PPE-om na koroziju ocijenjena je pomoću ubrzane karbonatizacije, utisnutog napona (engl. impressed voltage), potencijala polućelije, gubitka mase i ubrzanih korozijskih ispitivanja. Ispitivanje karbonatizacije pokazalo je da nepremazana čelična rebrasta armatura ima mrlje, što upućuje na karbonatizaciju, dok armatura premazana PPE-om nije pokazala promjene, demonstrirajući nekarbonatizirano područje. Nadalje, ispitivanje potencijala polućelije jasno je pokazalo da čelična šipka premazana PPE-om pokazuje umjereni rizik od korozije s potencijalom od -247 mV. Naime, gubitak mase nepremazanog običnog čelika bio je 40 % veći nego u slučaju šipke premazane PPE-om. Pri ubrzanom korozijskom ispitivanju, šipkama obloženim PPE-om trebalo je više vremena da razviju početne pukotine u betonu, s trajanjem od 182, 206 i 230 sati za uzorke 1, 2 i 3, što je dulje nego u slučaju običnog betona. Stoga su rezultati ispitivanja čelika obloženog PPE-om uspoređeni s rezultatima običnog čelika bez premaza te je ocijenjena učinkovitost šipke obložene PPE-om u pogledu otpornosti na koroziju.

Ključne riječi:

korozija, premazi, organski inhibitor, ubrzano korozijsko ispitivanje, djelotvornost inhibicije, karbonatizacija, djelovanje klorida

1. Introduction

Steel reinforcement enhances the ductile and flexural strengths of concrete. However, corrosion of steel rebars severely degrades the strength and durability of concrete. Corrosion, a major structural challenge, manifest as cracks, fractures, and spalling in rebars, leading to the deterioration of concrete structures. The corrosion process in concrete occurs due to the internal reaction of its constituent elements with high alkalinity. This enhances rust formation in steel, thereby increasing the volume of the rebar and modifying its structural and mechanical properties. [1, 2, 24]. This process is destructive in nature, as it results in material stress and deterioration. Table 1 lists the constitutive elements of steel. he two main compounds that initiate corrosion in rebars are chlorides and CO₂. Thus, carbonation and chlorination are the two major mechanisms through which steel is attacked, leading to corrosion [3]. Carbonation occurs when CO, reacts with concrete to form carbonic acid. This process leads to the formation of calcium carbonate, thereby reducing the pH level of the concrete to less than 8 and creating a corrosive environment. [4]. The carbonation process is illustrated by Equations (1) and (2).

$$CO_2 + H_2O \rightarrow H_2CO_3 \tag{1}$$

$$H_2CO_3 + Ca(OH)_2 \rightarrow CaCO_3 + 2H_2O$$
(2)

Corrosion in concrete is also induced by chlorination, in which chloride reacts with Fe²⁺ ions which are readily available in steel, thereby forming a chloride ion complex which further reacts with the hydroxide present in the concrete to form ferrous hydroxide and expels excess chloride ions. Notably, the presence of oxygen is necessary for chloride ions to initiate corrosion of steel. Consequently, oxidation is a significant component of the corrosion process, and the presence of FeO (rust) leads to major distress in concrete owing to the initiation of corrosion on the rebar. FeO.OH is more stable than Fe²⁺ and decreases the pH of concrete to less than 11 [2].

$$Fe^{2+} + Cl^{-} \rightarrow [FeCl Complex]^{+}$$
(3)

$$Fe^{2+} + 2(OH)^{-} \rightarrow Fe(OH)_{2} \tag{4}$$

$$4Fe(OH)_{2} + O_{2} + 2H_{2}O \rightarrow 4Fe(OH)_{3}$$
(5)

$$2Fe (OH)_{3} \rightarrow Fe_{2}O_{3} + 3H_{2}O \tag{6}$$

Hydrated Ferric oxide (Rust)

A decrease in the pH of concrete results in the initiation of the corrosion process. Figure 1 displays a pictorial representation of chloride-attack induced corrosion in concrete [1].



Figure 1. Pictorial representation of corrosion formation in chloride attack [1, 2]

In general, corrosion occurs in two phases. The initial phase of corrosion is called the inception stage, and when an abnormal internal stress develops and damages the steel, it is known as the propagation stage. The process of corrosion due to chlorination is described by Equations (7), (8), and (9).

$$3Fe + 4H_2O \rightarrow Fe_3O_4 + 8H^+ + 8e^-$$
(7)

$$2Fe + 3H_2O \rightarrow Fe_2O_3 + 6H^+ + 6e^-$$
(8)

$$Fe + 2H_2O \rightarrow HFeO_2 + 3H^+ + 3e^-$$
(9)

Hence, to extend the lifespan of rebars in concrete and protect it against corrosion, researchers have developed different methods. Commonly used corrosion protection methods include coating reinforcement, electrochemical methods, alternative reinforcements, and corrosion inhibitors. Corrosion Inhibitors are chemical substances that decrease the corrosion rate when present in a corrosion system at a suitable concentration. Since the 1970s, these inhibitors have played a vital role in corrosion protection methods [4]. The different types of inhibitors are shown in Figure 2.



Figure 2. Types of inhibitors

Table 1. Chemical Composition of steel

Element	С	Si	Mn	Р	S	Cr	Mo	Ni	Со	Cu	Sn	Fe
Weight [%]	0.260	0.280	0.920	0.020	0.008	0.050	0.016	0.003	0.013	0.0.020	0.028	97.40

For decades, researchers have focused on discovering natural inhibitors that are affordable, accessible, and eco-friendly [4, 6, 7]. Thus, plant extracts have been used as natural green inhibitors since the 1990s. Several plants such as palm fruit, orange peel, and novel sludge have been utilised as inhibitors of corrosion. Pomegranate fruit was used in this study due to its antioxidant properties [8, 9, 23]. The peel of the pomegranate is rich in fibres and consists of hydroxyl, carbonyl, and aromatic groups, including gallo tannic acid, as shown in F3. These components can counteract the oxidation processes that lead to corrosion [10, 11].



Figure 4. Preparation of pomegranate peel extract (PPE)

This research aims to investigate the corrosion resistance of steel coated with PPE and to assess the performance of the PPE-coated rebar in concrete protection against corrosion.

Figure 3. Chemical structure of gallo tannic acid



2. Materials

2.1. Preparation of pomegranate peel extract (PPE)

The peel of the pomegranate fruit was collected from various sources. The collected peel was cleaned with distilled water and dried in a dark room for seven days at room temperature. The dried samples were cut into small pieces and ground to a fine powder, and the peel powder was sieved through a 75 m sieve. Finally, the powder that passed through the sieve was tested. The pomegranate peel powder was replaced with an extract to coat the steel. The peel powder was boiled for 100 °C and the hot solution was cooled and filtered several times to acquire the extract [10, 12]. The prepared extract was coated onto steel by manual brushing and tested in a corrosive environment. Figure 4 depicts the entire preparation process of PPE utilised to coat the steel.

2.2. PPE Coated steel rod

The sample used in this study was a mild steel rod because it is utilised in concrete as rebar. A steel rod with a diameter of 10 mm was used, and the length of the bar used in the test was 15 cm. The

selected bars were cleaned, dried, and wrapped in an emery sheet. The cleaned sample was dried before being utilised for coating. Subsequently, the extract was coated onto the specimens by brushing. The extract should be completely coated over the steel sample, ensuring that it has been completed without missing even a small portion of the steel, as it will become a highly destructive zone. Finally, the PPE-coated steel rods were prepared for testing.

3. Methodology

The work plan for this study is shown in Figure 5; this image clearly explains the flow of the research which begins with the collection of materials for the experimental test results.



Figure 5. Methodology

The flow chart is outlined in series, along with the collection of materials and process of extract preparation The test was planned to assess the corrosion resistance of the PPE-coated steel rebar against corrosion.

4. Experimentation

4.1. Accelerated carbonation test method (ES 12390-12:2020)

A basic chemical test was performed by spraying phenolphthalein on concrete. Carbonation is a major defect in concrete which leads to its corrosion and deterioration [13]. Carbonation occurs when environmental carbon dioxide reacts with the calcium hydroxide present in the cement matrix. To perform the carbonation test, two concrete cubes measuring 10 cm \times 10 cm were cast along with the rebars. The cast cubes were then cured for 28 d. The specimens were preconditioned for 14 d under controlled exposure conditions with an increased level of carbon dioxide, as per the recommendation given in ES 12390-12:2020. After 90 d, the concrete cubes were cleaned, dried, and broken. Phenolphthalein was sprayed onto the newly broken concrete to track the carbonation depth. Thus, the carbonated zone of the concrete could be visualised.

4.2. Half cell potential measurement

To measure the probable rate of corrosion of steel rebar, a potentiostatic test was performed using a single reference electrode. The specimen selected for corrosion rate assessment was prepared with one-fourth of the steel was immersed in concrete, whereas the remaining portion was left exposed and connected to the electrode through a voltmeter. As per the standard ASTM C-876, the electrode and specimen were connected in series. The probable corrosion rate of the steel was then calculated based on the potential difference between the rebar in concrete and the electrode. Figure 6 illustrates the half-cell potential measurement setup used to measure the corrosion rate.



Figure 6. Half-cell potential measurement test set up

The performance of concrete rebars can be evaluated by measuring their potential. Although this test does not provide the specific corrosion rate of concrete, the probability of corrosion on the steel rebar in concrete can be obtained [14].

4.3. Weight loss measurement

The rate of corrosion of the steel rebar was obtained using the weight loss method. The test was performed using a steel sample with a length of 15 cm and a diameter of 10 mm. The steel samples were cleaned, dried, and rubbed prior to testing. The PPE coated and uncoated rebars were immersed in 250 ml of 3 % NaCl solution, and the initial weights of both the rods were recorded before they were submerged in the solution [15-17]. The setup was left undisturbed for 90 d at room temperature, and the readings were recorded weekly. The samples were removed from the solution, cleaned, and dried before weighing. The samples were removed from the solution and then cleaned and dried before weighing.

$$W = (w1 - w2)/A$$
 (10)

where W is the weight loss (mg/cm²), w1 and w2 are the weights of the sample before and after the corrosion (mg/cm²), respectively, and A is the surface area of the specimen (cm²). The inhibitor efficiency (η) was determined weekly using the general efficiency equation.

$$\eta = \frac{W_{uncoated} - W_{coated}}{W_{uncoated}} \cdot 100\%$$
(11)

4.4. Impressed voltage test

The impressed voltage–current method is a laboratory test method used to accelerate the corrosion of steel rebars in concrete and identify the rate of corrosion. To conduct the test, concrete cylinders were cast with a diameter of 75 mm and height of 200 mm. A steel bar with a length of 150 mm and diameter of 12 mm was reinforced in the concrete such that 100 mm of the bar was left exposed [18-20].



Figure 7. Schematic representation of the impressed voltage test

The concrete specimen with the rebar was immersed in a 3 % NaCl solution; this solution served as the electrolyte for the electrochemical test. The rebar in the concrete acted as the anode, while a stainless-steel bar served as the cathode. A schematic of the test arrangement is shown in Figure 7.

The anode and cathode were connected to a DC power source, providing a consistent power of 4 v to the circuit. The applied voltage was kept constant, and the variation in the current with time was determined. The current and time required for the initial crack in the concrete were recorded. The current flow increased as cracks developed and the steel surface was exposed to the electrolyte.

4.5. Morphological study

Morphological characterisation of the sample is used to determine the presence of chemical compounds formed during the corrosion process. Thus, microscopic analysis was performed on the specimen using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). The rods were immersed in a NaCl solution for corrosion. The samples were cut into 5-mm thick pieces, and then the samples were cleaned and completely dried before undergoing morphological analyses. The uncoated steel and PPE-covered steel rods are shown in Figures 8.a and 8.b.



Figure 8. a) Uncoated rod; b) PPE coated rod

5. Results & discussion

5.1. Accelerated carbonation test method (ES 12390-12:2020)

Carbonation is the process through which atmospheric CO_2 enters concrete. Subsequently, the CO_2 reacts with calcium hydroxide in the hydrated cement to form calcium carbonate. When carbonation reaches the steel, it breaks down the naturally present passivating film on the steel and initiates the process of corrosion. This can be tested by simply spraying a phenolphthalein indicator onto a specimen. The pink colour of the sample indicates the uncarbonated state

of the concrete specimen. The absence of a colour change in the sample indicates the occurrence of carbonation in the concrete.

Figure 10.a shows the concrete with the PPE-coated steel rebar. Figure 10.b shows the concrete with an uncoated ordinary steel rebar. The concrete with the uncoated rebar is completely carbonated, as the carbonated zone covers the steel. Although carbonation influences the specimen with PPE-coated steel, the PPE coating protected the passivating film, thereby preventing corrosion. Thus, the PPE-coated rebar protects the passivating film on the steel against corrosion.



Figure 9. Concrete specimen with PPE-coated rebar



Figure 10. a) PPE coated rebar; b) Uncoated rebar

5.2. Half cell potential measurement

The half-cell potential measurement of the rebar against an AgCl reference electrode was performed according to the ASTM C 876 – 1992. The experimental results are listed in Table 2. Among various methods for measuring the corrosion rate, half-cell potential measurement is an optimal non-destructive testing method. Table 2 presents the potential values for uncoated steel from -74 mV to -367 mV and for the coated rebar from -61 mV to -247 mV, determined using potentio-static measurement over a period of 12 weeks. The severity of corrosion conditions for concrete with an AgCl electrode according to the ASTM C 876 standard is listed in Table 3. The potential value of the PPE-coated reaches -247 mV which is below the threshold of 90 % corrosion susceptibility. Hence, it is evident that the PPE-coated rods exhibit only an intermediate corrosion risk.

Time in weeks	Uncoated rebar [mV]	PPE coated rebar [mV]
1	-74	-61
2	-91	-77
3	-123	-91
4	-142	-110
5	-155	-131
6	-174	-152
7	-190	-170
8	-212	-186
9	-232	-197
10	-270	-217
11	-311	-231
12	-367	-247

Table 2. Half cell potential measurement

Table 3, ASTM C876-91	criteria for	corrosion	condition	of steel	concrete	versus AgCI
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Potential of steel Vs AgCl electrode [mV]	Corrosion condition		
> - 100	Low (10 %) risk of corrosion		
- 100 do -250	Intermediate corrosion risk		
< - 250	High (90 % risk of corrosion)		
< - 400	Severe corrosion		

The half-cell potential values were plotted against time (weeks), as shown in Figure 11. The graph shows that the potential of the uncoated conventional steel bar is relatively higher, reaching approximately 367 mV. This value is much lower for the PPE-coated rebar. The value of the uncoated steel rod indicates a serious state of corrosion, whereas that of the PPE-covered rebar indicates intermediate corrosion. Thus, the concrete specimen with the PPE-covered rebar exhibited better performance against corrosion.



Figure 11. Variation of potential over time (weeks) for the concrete specimen with PPE-coated and uncoated rebars

The results obtained from the half-cell potential measurement were compared to the corrosion condition limits specified in the ASTM C 876-91 standard (Table 3). These limits provide guidance on assessing the corrosion risk of steel rebars in concrete using AgCl electrodes. This standard states that potential values greater than -100 mV indicate an exceptionally low risk of corrosion, whereas potential values lower than -400 mV indicate that the risk of corrosion is extremely high.

5.3. Weight loss measurement test

Table 4 reports the results of weight loss measurement for the PPE-coated and uncoated conventional rebars. In addition to loss in weight in the specimens, the efficiency of the PPEcoated steel against the corrosion was obtained by utilizing Eq. (10) [11, 16, 21]. The loss of weight in the specimen due to corrosion was observed and noted for the different time intervals. The percentage of weight loss for the uncoated steel rebar was 89.68 % for 84 days of immersion in 3 % HCl, whereas the percentage of weight loss for the specimen coated with PPE was recorded as 47.19 %. These results indicate that the weight loss in the PPE-coated rebar was lower compared to the uncoated rebar. Hence, the PPE coating over the steel offered considerable resistance against corrosion. Based on Equation (11), the efficiency of the PPE-coated rebar against corrosion was determined and presented along with the weight loss measurement. The results of the sample were recorded for 3

Time	Weight loss in uncoated rebar [mg]	Weight loss in coated rebar [mg]	Inhibitor efficiency η [%]
7	0.002990	0.00101	66.22
14	0.003460	0.00118	65.90
21	0.004180	0.00129	69.14
28	0.004850	0.00139	71.34
35	0.005910	0.00168	71.57
42	0.006830	0.00190	72.18
49	0.007910	0.00206	73.96
56	0.008520	0.00219	74.30
63	0.008990	0.00231	74.30
70	0.014400	0.00310	78.47
77	0.021000	0.00428	79.62
84	0.029000	0.00510	82.41

Table 4. Weight loss of PPE Coated and Uncoated rebars with inhibitor efficiency

months. The efficiency of the steel was 66.22 % after 7 days, and the efficiency percentage increased to 82.41 % after 84 days. The antioxidant property in the PPE due to the presence of the Gallo tannic acid opposes the ingression of oxygen into the steel. Thus, the chloride in the NaCl solution would not affect the steel as the possibility of corrosion is extremely low in the absence of oxygen.

A graph of the PPE efficiency versus time for the PPE-coated rebar is plotted in Figure 11. The efficiency of the PPE-coated steel against the corrosion is 82.41 %, indicating low risk of corrosion to steel. Hence, the PPE coating on steel enhances the life of steel and improves its durability in concrete.

5.4. Impressed voltage test

This test was performed in the laboratory as an accelerated corrosion test to determine the rate of corrosion. To obtain more accurate results, the test was performed using three specimens. Concrete with rebar was cast. After curing, the concrete was placed in a 3 % sodium chloride solution. A steady voltage was applied using a voltmeter connected to a steel rod and a stainless-steel cathode [20, 22]. The current induced by the applied potential was recorded every 12 h. Table 5 illustrates the time required by the concrete for its initial cracking at the most extreme anodic current. The first crack in the conventional concrete is observed at 134 h, and the current recorded at the corresponding time is 68 mA. However, for the concrete with the PPE-coated rebar, the test was performed using three samples to determine the corrosion rate. The tabulated results clearly show that the time taken for the initial crack in concrete with the PPE-coated rebar is 182, 206, and 230 h for samples 2, 3, and 4, respectively, and the corresponding anodic currents are 47, 44, and 41 mA. These results validate that the capacity of the PPE-coated rebar under corrosive conditions was 40 % higher than that of conventional rod.

The current record of the anode versus time for conventional and PPE-coated steels is plotted in Figure 12. The graph shows that the current recorded on the conventional steel is higher, indicating highly corrosion-prone conditions, whereas the PPEcoated rebar exhibits a low probability of corrosion. The time required for the concrete sample to reach the initial crack is illustrated in Figure 13.



Figure 12. Anode current versus time

5.5. Morphological study

Along with the electrochemical measurements and accelerated corrosion tests, the steel samples were tested for morphological characterisation to study the surface of the corroded specimens at the microlevel. Two steel bars with a length of 10 cm and a diameter of 12 mm were used for observation. One rod was coated with PPE, and the other was kept uncoated. The prepared samples were immersed in 3.5 % NaCl solution for 30 days. The arrangement was left undisturbed for a predefined time. After 30 days, the steel bars were removed from the cleaned

Current [mA]

	No.	Samples	Time [hours]	
		Conventional steel rod	24	
			48	
	1		72	
			96	
			120	

Table 5. Impressed voltage-current test results with induced current and time

1		24	64
	Convertional starling d	48	66
		72	59
	Conventional steel rou	96	65
		120	54
		134	68
		24	39
	PPE coated rod (Sample 1)	48	43
		72	41
		96	50
_		120	45
2		134	42
		158	49
		182	49
		206	51
		230	47
	DDE costod rad (Samala 2)	24	41
		48	52
		72	49
		96	49
2		120	42
	FFE coated fod (Sample 2)	134	39
		158	47
		182	41
		206	44
		230	44
		24	38
		48	44
		72	40
		96	49
	DDE costod rod (Comple 2)	120	46
4		134	41
		158	41
		182	47
		206	39
			42

and dried arrangements. The steel rods were examined using SEM to study the external structure of the corroded specimens. Figure 13 (a) and (b) show SEM images of the PPE-coated and uncoated steel rods, respectively. The image of the uncoated rod reveals the formation of rust over the rod, whereas the picture of the PPE-coated rod shows less rust formation, and the effect of corrosion over the coated steel bar is extremely low. The high rate of corrosion of the uncoated steel rod is attributed to the presence of chloride particles in NaCl. Because PPE is an antioxidant, the chlorination of steel in the presence of chloride ions is not initiated rapidly.



Figure 13. a) SEM image of uncoated rod b) SEM image of PPE-coated rod



Figure 14. EDX image of PPE Coated and uncoated rebar samples

The scanned samples were subjected to Energy Dispersive X-Ray Spectroscopy (EDS) to identify the chemical composition of the steel rod. Figure 14 Shows the presence of chemical components on the coated steel bar due to the response of the NaCl, to the scale of 1 μ m. The results show the maximum presence of oxygen and iron.

6. Conclusion

The assessment of corrosion inhibition of PPE coated steel rebar illustrates the following conclusion:

- The carbonation of concrete with ordinary steel was influenced completely and formed rust patches on the steel, whereas the concrete with PPE-coated steel showed an uncarbonated zone which demonstrates the capacity of the coated steel against carbon.
- Half-cell potential test demonstrated that the capability of uncoated steel arrives at a limit of - 367 mV and the equivalent for the steel coated with PPE arrives at the value limit of -231 mV. According to the ASTM C 876-91 code, the uncoated steel rod displays a high risk of corrosion, and the coated rod presents a distinctly moderate risk, as the value of the potential is not exactly -250 mV.

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- Weight loss measurements of the samples indicated that the loss in weight of the uncoated steel rod was 40 % higher than the PPE-coated steel rod. This indicated that the decreased weight of the coated steel was attributed to passivation framed on the steel which created an ideal opportunity for the rod to become corroded.
- The laboratory test of accelerated corrosion revealed that the time taken for the initial crack in the concrete with ordinary rebar was only 134 h, whereas the time required for the PPE-coated steel was 182, 206, and 230 h for Tests 1, 2, and 3, respectively. This explains the performance of the concrete sample with the PPEcoated rebar against the corrosive components.
- The SEM and EDS images showed that the regular steel had rust fixes and arrived at the last spread period of corrosion. Moreover, the coated rod was not impacted by the corrosion-leading components.

Thus, the experimental study proved that PPE exhibited satisfactory inhibition against corrosion. Nonetheless, this study aimed to enhance the performance of rebars with different coating thicknesses.

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