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Green corrosion inhibitors from leaves, bark and root extracts of *Piliostigma reticulatum* (*p-reticulatum*) and their performance on steel reinforcement in concrete subjected to acidic and chloride environment

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Abstract: Corrosion inhibition of the samples treated with P-*reticulatum* plant in acid medium was measured; the samples containing leaf bark and root extracts have the potential values of -150mV to -250mV, -100mV to -200mV and -100mV to -250mV, respectively. The samples treated with leaves, bark and root extracts in chloride medium, the potential difference obtained is within the range of -200mV to -300mV, -50mV to -200mV and -100mV to -200mV, respectively. The control samples containing no inhibitor from the two plants have the value of -400mV to -500mV in acid medium and -350mV to -450mV in chloride medium which is an indication of severe corrosion condition. Generally, all the samples treated with plant extracts were found to have inhibition performance but the efficiency varies. Scanning electron microscopy (SEM) results obtained show that all the protected samples were found to have low corrosion attack while the unprotected or control samples show severe corrosion attack. For the concrete resistivity measurement, the result was achieved by using Wenner four probes method which was done by placing the four probes in contact with the concrete directly above the reinforcing steel bar. All the samples treated with chloride showed negligible corrosion risk.

Key words: Corrosion, concrete, reinforcement, inhibitors, *Piliostigma Reticulatum* (P-*reticulatum*)

1. Introduction

It is globally known that structures exposed to seawater and areas that are vulnerable to acid rain are highly prone to chloride and acid attack which accelerate corrosion initiation in reinforcing steel, which is the main anguish behind the present anxiety regarding concrete durability. The cost of renovating or replacing deteriorated structures is enormous (Bertolini *et al.,* 2004). Therefore, there is a need to combat this problem with environment friendly and cost-effective corrosion-inhibiting substances that will prevent or reduce corrosion rate, and consequently reduce the cost of maintenance and improve durability of structures exposed to marine environments (Salta *et al.,* 2012).

Corrosion of steel embedded in concrete is not a challenge unless the normal passive state in the alkaline medium is influenced by external agents or corrosive species in the environment. Over the years, various methodologies have been examined to increase the time of deterioration of structures due to corrosion (Sun *et al.,* 2012; Angst, 2018). Efforts have been made in environmental modifications, good proper engineering designs, such as increased concrete cover over reinforcing steel, and also the use of specialised systems for corrosion prevention (Brown, 1999; ElHamahmy, 2025).

To retard corrosion, soluble organic or inorganic inhibitors are added to corrosive media to adsorb on the metal surface, and therefore, create a barrier against the arrival of aggressive species as H^+, OH^- or dissolved O_2 (Ajmal *et al.,* 2002; Mihit *et al.,* 2006; Ahmad *et al.,* 2010; Zarrouk *et al.,* 2012; Zehra *et al.,* 2020; Loukili *et al.*, 2022; Salim *et al.,* 2024; Zamindar *et al.,* 2024). Researchers proposed natural plant extracts, called green inhibitors, rich in various compounds containing aromatic rings, double bonds and/or heteroatoms (O, N, S..), readily adsorbed on the metal surface (Saleh *et al.,* 1982; Hammouti *et al.*, 1995; Abboud *et al.,* 2009; Verma *et al.,* 2018). Some authors postulated that the inhibitory effect is due to the synergistic intermolecular action of the various components (Benali *et al.,* 2013; Khadom *et al.,* 2022; Lrhoul *et al.,* 2023; Sunjay *et al.,* 2024).

The objective of this study is to test leaves, bark and root extracts of *Piliostigma reticulatum* (p-reticulatum) and their performance on steel reinforcement in concrete subjected to acidic and chloride environment.

2. Materials and methods

2.1 Materials

Steel rods, Measuring cylinder, Filter paper, Separating funnel, 500 ml Beakers, Digital multi-meter (Model: DT 9205A), Cement (Portland cement), Sand, Aggregate, Water and Organic inhibitors (*Piliostigma Reticulatum*), **Photo 1**.

Photo 1: Image of leaves, bark and root of *Piliostigma Reticulatum*

2.2 Chemicals

Methanol, sulphuric acid, calcium chloride, and copper sulphate $CuSO_4$

2.3 Methods

2.3.1 Sampling

Three different parts, namely, the root, bark, and root of *Piliostigma Reticulatum*, were collected from various locations in the Zaria local government to ensure sample representativeness. The samples were separated based on the part of each plant and were taken to the laboratory for pre-treatment.

2.3.2 Pre-treatment of the samples

The samples were washed and dried and then ground into fine powder using a pestle and mortar.

2.3.3 Preparation of the Standard Solutions

1.0M calcium chloride ($CaCl_2$)

One molar solution of $CaCl_2$ was prepared by measuring 111.00g of calcium chloride using a weighing balance. The measured $CaCl_2$ was carefully poured into 1L volumetric flask, a small amount of distilled water was added and swirled until the calcium chloride was completely dissolved, and then the flask was filled to the mark (Ayeni *et al.,* 2012).

1.0M sulphuric acid (H_2SO_4)

One molar H_2SO_4 was prepared by measuring 55.55 ml of concentrated H_2SO_4 and pouring it into a volumetric flask containing 500 cm^3 distilled water. The flask was filled with distilled water up to the mark (Ayeni et al., 2012).

1.0M copper sulphate

One molar solution of $CuSO_4$ was prepared by weighing 159.60 g of copper sulfate using a weighing balance. The measured $CuSO_4$ was carefully poured into 1L volumetric flask, and a small amount of distilled water was added and swirled until the copper sulfate was completely dissolved to the mark. This solution was used as a copper-copper reference electrode in the half-cell container (Kumar *et al.,* 2013).

2.3.4 Extraction of Plant Samples

One hundred grams of the powdered leaves were measured and dissolved in 1000 ml methanol at room temperature for 24 h, and they were filtered using filter paper. The extracts were recovered by filtration and stored at 40 ^{0}C in a rotary vacuum evaporator. The same process was performed for the pre-treated (powdered) bark and root samples (Loto *et al.,* 2011). 15 g of each organic extract was used as an inhibitor in each concrete prism produced (Elsener *et al.,* 2003).

2.3.5 Preparation of Reinforced Concrete Prisms

Reinforced concrete prisms of size 200×70×70mm were prepared using cement, sand, and aggregates in a ratio of 1:2:3. A reinforcement bar of diameter 8 mm and length 200 mm was embedded while casing at a distance of 40 mm from the prism and allowed to dry before testing began. In addition, 15 g of each inhibitor was added to each concrete prism during mixing. Seven concrete prisms were prepared, six of which contained 15 g of the inhibitor each from the root, leaves, and bark of *Piliostigma Reticulatum* and immersed in acid and chloride solution separately, while the remaining solution served as a control (Jorge *et al.,* 2012).

2.4 Characterization

2.4.1 Potential Difference Measurement

The corrosion potential of the reinforced prisms was measured using a half-cell potentiometer at an interval of 2 weeks up to 12 weeks (Morris, 2002). The electrical potential of a point on the surface of the steel reinforcing bar was measured by comparing its potential with that of the copper– copper sulfate reference electrode on the surface (Reilly *et al.,* 2013). In practice, this was achieved by connecting a wire from one terminal of a voltmeter to the reinforcement and another wire to the copper sulfate reference electrode (Eyu *et al.,* 2013).

2.4.2 Concrete Electrical Resistivity Measurement

Electrical resistivity of concrete is an effective parameter to assess the risk of corrosion in reinforcing steel embedded in concrete, especially for chloride-induced corrosion. In this study, the Wenner four probes method was used, which was done by placing the four probes in contact with the concrete directly above the reinforcing steel bar. Different readings were taken at different locations at the surface of the concrete. The mean values of the readings were recorded as the final readings of the resistivity (Mahdi *et al.,* 2015).

2.4.3 Scanning Electron Microscopy Analysis

Surface analysis of the reinforcing bars by scanning electrode microscopy was carried out on Phenom X MVE 0987612. The surface morphology of unprotected (i.e.. control samples) and protected (i.e. treated samples) reinforcement bars was analysed after removal from the concrete prism using a crushing machine. The scanning was done at magnifications 1000 to have clearer images and operated at a voltage of 15 kV (Jabeera *et al.,* 2006)

3. Results and Discussion

3.1 Inhibition Performance of *Piliostigma reticulatum* (*P-reticulatum*) In Acidic Medium

Visual inspection confirmed no pitting corrosion occurred for the samples treated with bark extracts of (*P-reticulatum*) because of the absence of pits on the surface of the reinforcement while pitting corrosion occurred for the samples treated with leaves and root extracts. This is confirmed by the presence of small pits in the surface the concrete reinforcements. The magnitude of the potential difference measured for the samples treated with (*P-reticulatum*) leaves and root extracts in acid medium is up to -250mV which is considered intermediate risk of corrosion i.e. moderately safe (Figures 1 and 3), while for the sample treated with bark extracts, the magnitude of the measured potential obtained is within the range of -100mV to -200mV (Figure 2) which is considered to be 10% corrosion risk (i.e. safe) according to corrosion engineers (Akshatha *et al.,* 2015).

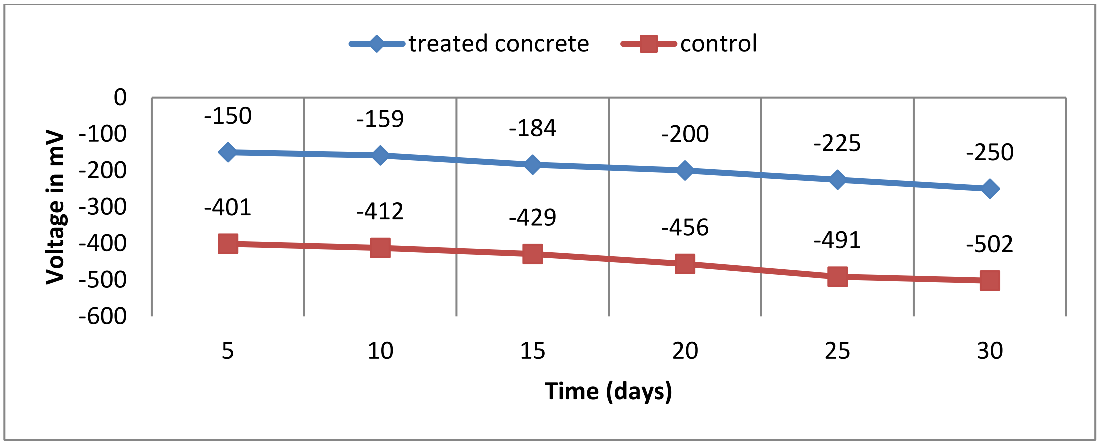


Fig.1: Potential Difference Vs Time (days) for Concrete Treated with *P-reticulatum* Leaves Extracts in Acidic Medium.

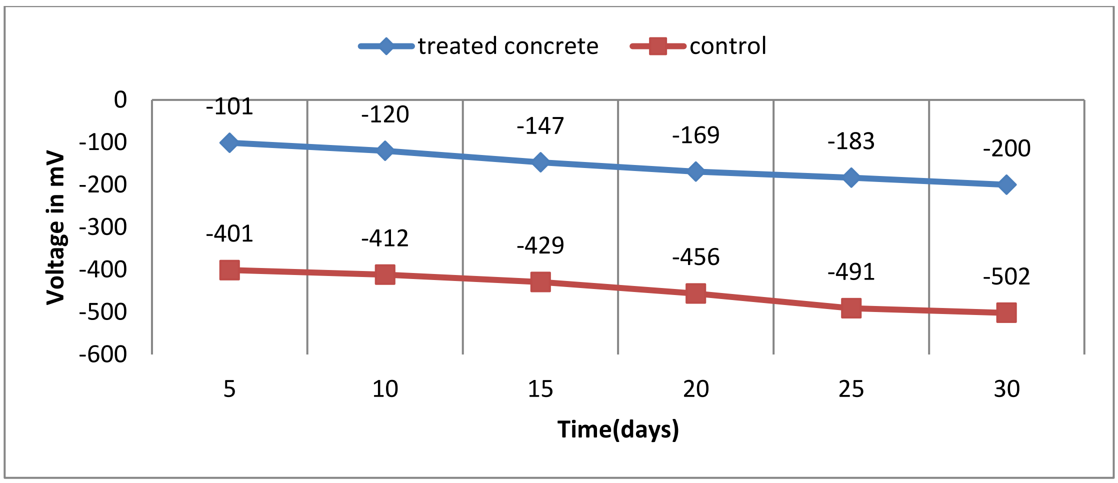


Fig.2: Potential Difference Vs Time (days) for Concrete Treated with *P-reticulatum* Bark Extract in Acidic Medium.

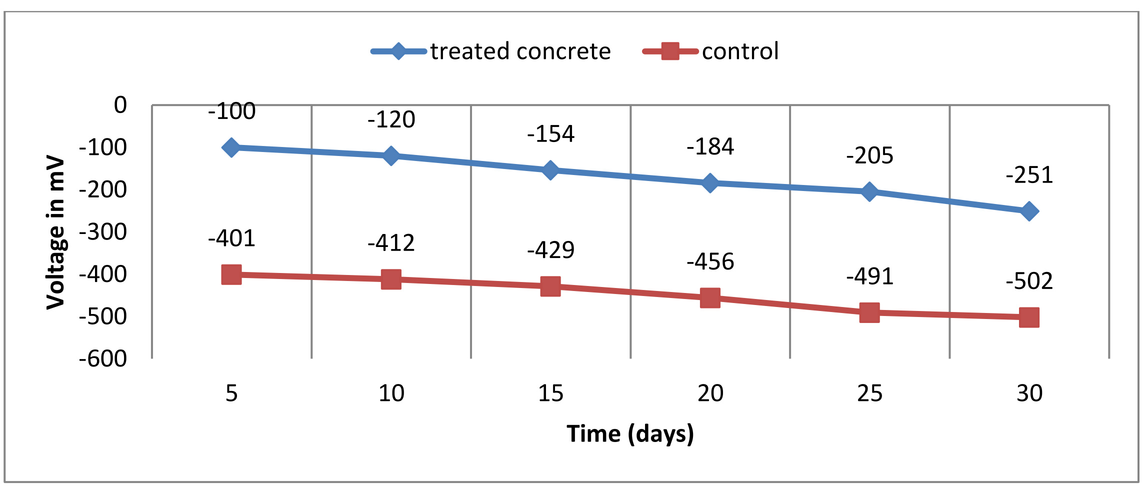


Fig.3: Potential Difference Vs Time (days) for Concrete Treated with P-*reticulatum* Root Extracts in Acidic Medium.

3.2 Inhibition Performance of *Piliostigma reticulatum* (P-*reticulatum*) In Chloride Medium

For all samples treated with leaves, barks, and root extracts in chloride medium, visual inspection showed no pitting corrosion on the reinforcement surface. The magnitude of the measured potential difference showed for the samples treated with bark and root extract of *P-reticulatum* indicate more corrosion inhibition efficiency in chloride medium as the values obtained are greater than -200mV (Fig 5 and Fig 6) which is considered safe i.e. 10% corrosion risk while for the samples treated with leaves extract, the magnitude of the potential difference measured is less than -200mV but greater than -300mV (fig 4) which is moderately safe (i.e. intermediate corrosion risk) (Elsener *et al*., 2003).

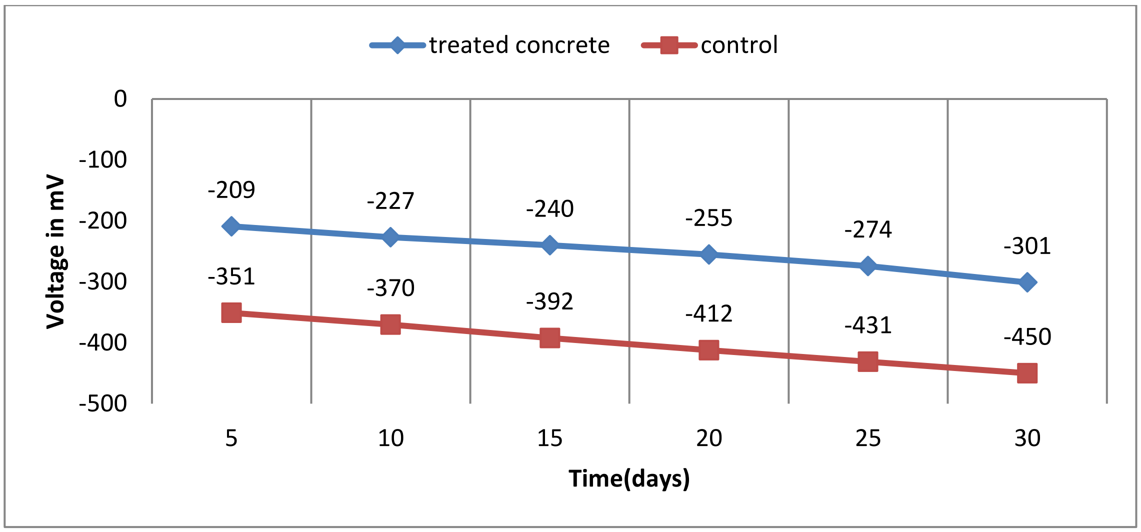


Fig.4: Potential Difference Vs Time (days) for Concrete Treated with P-*reticulatum* Leaves Extracts in Chloride Medium.

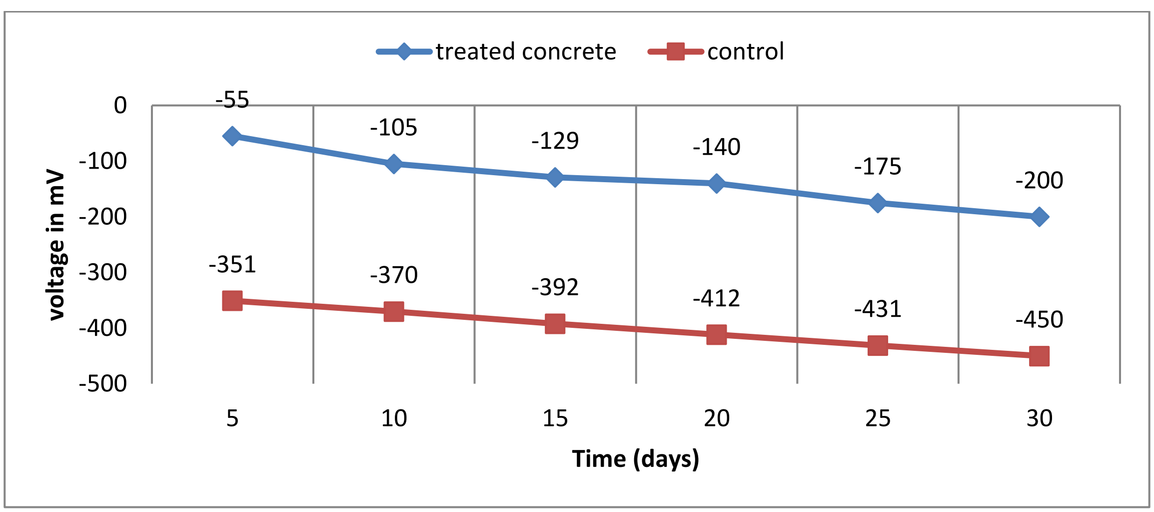


Fig.5: Potential Difference Vs Time (days) for Concrete Treated with P-*reticulatum* Bark Extracts in Chloride Medium

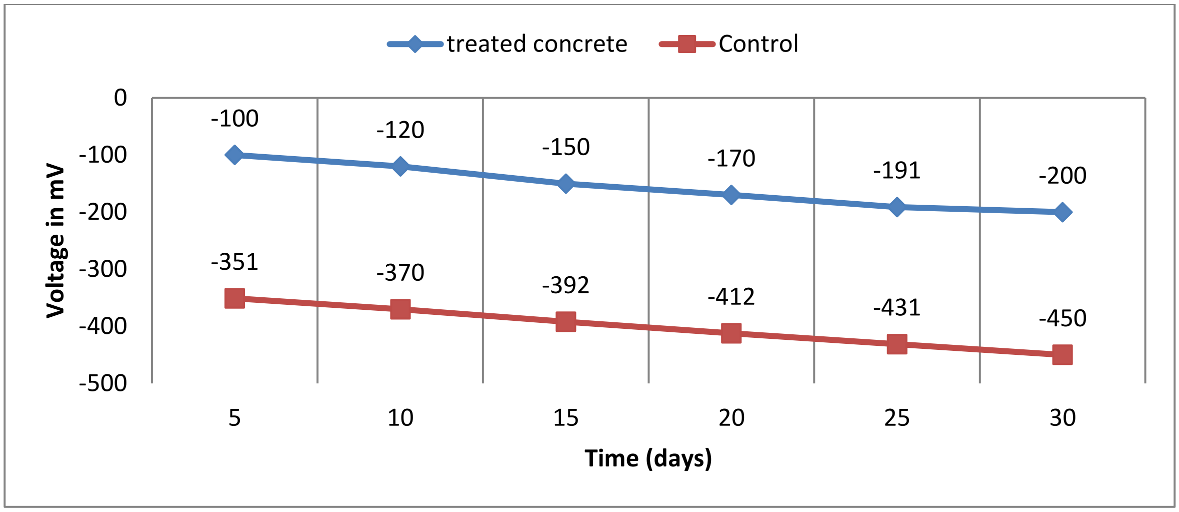


Fig.6: Potential Difference Vs Time (days) for Concrete Treated with P-*reticulatum* Root Extracts in Chloride Medium.

3.3 Concrete Electrical Resistivity Measurement

Electrical resistivity of concrete is an effective parameter to assess the risk of corrosion in reinforcing steel embedded in concrete, especially for chloride-induced corrosion. Concrete resistivity (ρ_c) of the samples immersed in chloride solutions was measured and the results obtained were plotted as a function of immersion duration in days. According to Morris *et al.,* (2002), reinforcing steels are in active corrosion risk when ρ_c is below 10kΩcm and attain passivity at resistivity greater than 30kΩcm. And as previously illustrated in Table 2.3 (Song and Saraswathy, 2007) in the literature review, resistivity above 20kΩcm shows negligible corrosion risk while between 10kΩcm to 20kΩcm suggests low corrosion risk. Therefore, all the samples treated with inhibitors were found to have negligible corrosion risk while the control samples have low risk of corrosion as indicated in (Fig 7).

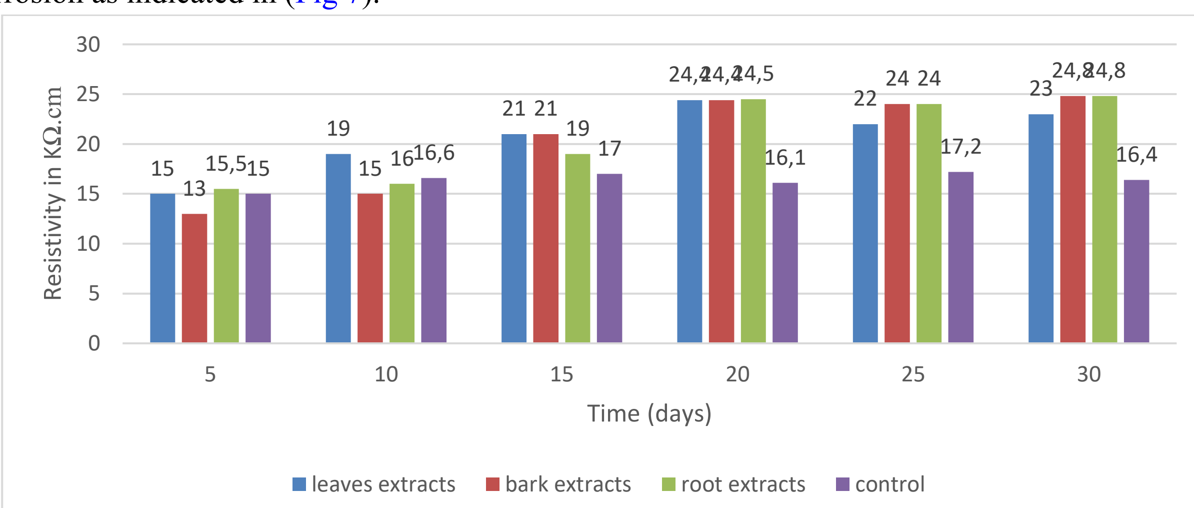


Fig 7: Concrete resistivity readings versus immersion time for samples treated with P-*reticulatum* Plants immersed in chloride solution

3.4 Surface Analysis

Surface analysis of reinforcing bars by scanning electrode microscopy was carried out on Phenom X MVE 0987612. The surface morphology of unprotected and protected reinforcement bars were analysed at magnification of 1000X and operated at a voltage of 15KV. Scanning electron microscopy revealed that the plant extract adsorbed on the surface of the steel reinforcing bars increased its smoothness thereby decreasing the corrosion attack (Plate (c) and (d)). Scanning electron microscopic picture of unprotected or control reinforcing bar in acid and in chloride shows severe corrosion attack (plate (a) and (b)) according to the findings of (Nossoni *et al.,* 2011) and (Singh and Singh 2012).

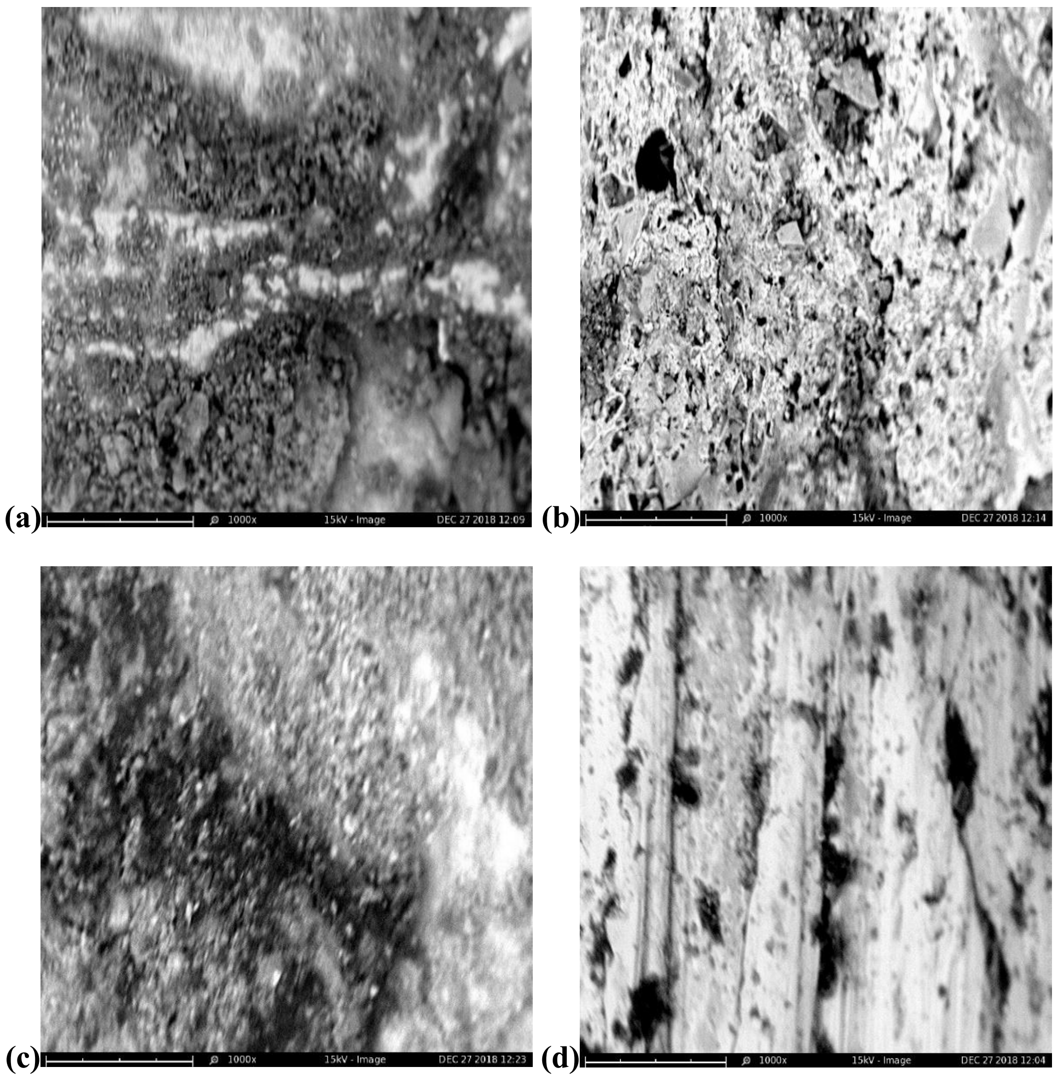
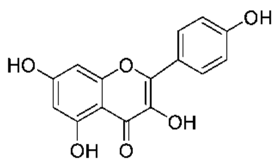
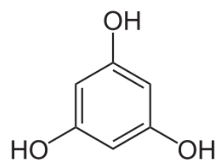


Fig 7: Surface analysis by scanning electrode microscopy

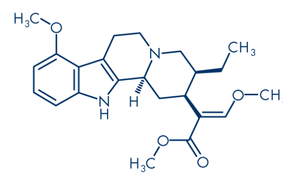
Corrosion inhibition may be explained by the presence of bioactive molecules as reported in literature (Boualam *et al.*, 2021; Sharma *et al.,* 2023; Lrhoul *et al.*, 2023). Phytochemical analysis indicated that the content of methanol extract of *Piliostigma reticulatum* leaves yielded the presence of alkaloids, glycosides, steroids, tannins and saponins. Only Flavonoids, saponins, and tannins were quantified, and their concentrations in the sample using methanol extraction were 3.70 µg /ml, 5.29 µg /ml and 2.90 µg /ml, respectively (Sani *et al.,* 2024). The presence of molecular structures of flavonoids, saponins, tannins … rich in aromatic rings and heteroatoms that facilitate adsorption on the metal surface reinforces the synergistic intermolecular effect, ensuring corrosion protection of metallic materials (Popoola *et al*., 2023).



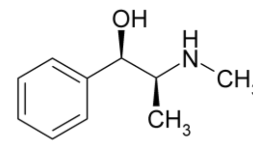
Flavonoids



Tannins



Saponins



Alkaloids

Conclusion

i. All the concrete treated with leaves, bark and root extracts of P-*reticulatum* were found to have relatively same inhibition performance in acid medium for their corrosion condition was found to be an intermediate corrosion risk.
ii. Corrosion condition of concrete reinforcement treated with bark and root extracts of P-*reticulatum* in chloride medium was also an intermediate corrosion risk while high corrosion risk was observed on the concrete treated with leaves extracts of P-*reticulatum*.
iii. Highly negative potential difference was observed in all the control samples and their corrosion condition was severe corrosion risk when compared with standard corrosion condition.
iv. Scanning electron microscopy shows low corrosion attack on the concrete treated with inhibitor and high corrosion attack was observed on the unprotected samples.
v. Concrete electrical resistivity shows negligible corrosion risk for concrete treated with inhibitor immersed in chloride solution.

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