Reed Leaves Extract as Corrosion Inhibitor for Reinforcing Steel in Concrete

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Abstract. The effect of reed leaves extract (RLE) as an eco-friendly corrosion inhibitor for reinforcing steel in concrete immersed in 3.5% NaCl solution was investigated. The work was carried out using Linear polarization resistance, Potentiodynamic polarization, cyclic polarization, photographic examination, impressed voltage and compressive strength tests. Results obtained from Tafel polarization curves after 180 days immersion showed that corrosion rate was decreased and efficiency was increased with increasing of concentration of RLE inhibitor. The minimum corrosion rate was 2.259 μmpy with maximum efficiency of 76.98% at 0.5% RLE concentration (by cement weight). Also, the photographic study results indicated that inhibited surfaces are more smooth compared to control surface (without inhibitor). Cyclic Voltammetry curves showed that steel samples with RLE inhibitor have low tendency to pitting corrosion compared with control samples. On the otherhand, compressive strength test indicated that addition of RLE inhibitor to concrete mix increased compressive strength by 7.16 % at 0.5% concentration. Impressed voltage test showed that corrosion of reinforced steel bar initiated on the seventh day of experiment for control sample while on the eighteenth day for RLE sample at 0.5% concentration. The results of this study revealed that RLE acts as a good eco-friendly corrosion inhibitor.

Keywords. reinforcing steel, corrosion, reed leaves extract, inhibitor, tafel polarization, cyclic polarization, impressed voltage, compressive strength.

1. Introduction
The corrosion of rebar steel is the major factor in deteriorating reinforced concrete structures, especially when they are exposed to corrosive environments or chloride attacks [1-6]. Corrosion inhibitors are widely used as a practical and low cost solution to inhibit rebar corrosion in concrete [7]. Due to the toxicity of present corrosion inhibitors of reinforcing steel in concrete and their hazards to the environment, the researchers in recent time performed research which works to develop eco-friendly, inexpensive, biodegradable, and non-toxic corrosion inhibitors [8-14]. Using plant extract as an eco-friendly corrosion inhibitor for concrete reinforcing steel has been reported by several researchers [15-21].

Plant extract contains heteroatoms such as P, N, S, and O. The protective films are formed due to the coordination of these atoms with corroded metal atoms. These inhibitors were used to protect different types of metals in different mediums. Also, plant extract inhibitors can be used either alone or in combination with other inhibitors. Different parts of plants were used to extract as green corrosion inhibitors such as leaves, fruits, flowers, seeds, and barks and extract operation can be done by different types of solutions such as alcohol, acid, and water [16]. Reed, scientists named as phragmites australis, found widely in the middle and south regions of Iraq. Reed was found to contain lignans, alkaloids, flavonoids, and O-substituted aromatic amines. [22] Few works have been performed using reed leaves extract as a corrosion inhibitor of mild steel. [22] investigated the impact of reed leaves extract inhibitor on mild steel in 1.0 M HCl and 0.5 M H₂SO₄ by potentiodynamic polarization methods and weight loss approaches. Results showed that inhibition efficiency was increased with the increase of RLE inhibitor concentration and RLE is mixed type inhibitor in HCl solution but cathodic type inhibitor in H₂SO₄ solution. The best efficiency was
achieved in 0.5 M H$_2$SO$_4$ solutions. While [23] studied the effect of reed leaves extract inhibitor at concentrations of 2, 4, 6, 8 and 10% on low carbon steel immersed in 3.5% NaCl using weight loss methods. The results showed that the increase of inhibitor concentration decrease weight loss amount. This work aims to study the inhibition of reed leaves extract (RLE) on the corrosion of reinforcing steel in cement mortar using linear polarization resistance, Tafel polarization, cyclic voltammetry, and impressed voltage tests.

2. Experimental Work

2.1. Inhibitor Preparation
Reed leaves from Iraqi marshlands and swamps in Hillah were used as inhibitor in this work. It contains vehicles aldehydes, ketone, amines, polyamides and alcohols or compounds of aromatic or phenolic which have inhibition properties [23]. The active compounds in powder of the leaves extract were detected using FTIR test as shown in Figure (1) [22]. Reed leaves were dried first to be grinded with an electric grinder. The result of this operation is a powder of reed leaves, this powder was weighted to the required weight (0.009, 0.09, 0.9 or 4.5 grams) and soaked in the required amount of boiling water (495 ml) for 30 minutes then filtrated with qualitative filter paper. The resulting inhibitor amount was measured again to be used. This preparation method depended on the method that used in [22] with a little suitable changes.

![Figure 1. FTIR test for reed leaves extract.](image)

2.2. Steel Bar Preparation
Ukrainian steel grade HRB with a 10 mm diameter from local suppliers was used in this research. Its chemical composition is shown in Table (1). Steel bar preparation started with cutting it to the required length, cleaning started by immersing the steel bar in concentrated hydrochloric acid for 2-3 minutes then rinsed with water, the bar was immersed in sodium hydroxide (5M) for neutralization then rinsed with water, after that the bar was dried and scrubbed with wire brushing machine to remove any rust product and pigments. 8cm in length of the bar was chosen as exposure area, the rest 8 cm of the bar was painted with three layers epoxy paint (Don Construction Products made by British DCP company) to prevent from atmospheric corrosion.

| Material | Percentage (%) |
|----------|----------------|
| C        | 0.25           |
| Si       | 0.8            |
| Mn       | 1.6            |
| P        | 0.045          |
| S        | 0.045          |
| V        | 0.00008        |
| Fe       | 97.25992       |
2.3. Preparation of Cylindrical Mortar Specimens
Cylindrical mortars were used for Electrochemical tests and impressed voltage test, the compositions of the paste of blank (control) samples were mixed together, the ratios of these compositions are listed in Table (2) below. The mortar paste was enough to mold three cylindrical samples.

| Material                     | Ratio |
|------------------------------|-------|
| Cement/Sand (C/S)            | 1/3   |
| Water/Cement (W/C)           | 0.55  |
| Salt % Cement                | 1.5%  |

The preparation of inhibited mortars was done with the same components shown above with adding reed leaves inhibitor in the required concentration as showing in Table (3) below. The inhibited mortar paste was also enough to mold three cylindrical samples.

| Ratio (% cement) |
|------------------|
| RLE₁             | 0.001 |
| RLE₂             | 0.01  |
| RLE₃             | 0.1   |
| RLE₄             | 0.5   |

Casting process was done with three layers, each layer was tamped around 25 times with a rod, vibrated for 60 seconds to eliminate the hollow spaces and cavities in the mortar. One steel bar was embedded in the center of the cylinder. The mortar specimens were kept in molds for 24 hours and then de-molded and cured in tap water for 7 days at room temperature, after that the top of the mortar was painted twice with acrylic paint and let to be dried for 24 hours after each painting process, then partially cured (at 7 cm height) in 3.5% NaCl solution for 180 days. The NaCl solution was kept at the same level for the curing period and it was changed every 14 days to refresh the solution.

2.4. Preparation of Cubic Concrete Specimens
These specimens were used for compressive strength test, the compositions of the concrete paste at a ratio of 1:1.5:3 (Cement:Sand:Coarse aggregate) and 0.45 W/C were mixed together in laboratory concrete mixer [26], each paste was enough for three samples, two pastes were made, one for the blank and the other one with adding RLE₄ (0.5%). The casting operation was done with 15 x 15 x 15 cm steel molds, casting three layers, each layer was tamped around 30 times with a cylindrical rod and then the top side was finished with a trowel. The samples were kept at molds to dry for 24 hours at room temperature, after 24 hours they were de-molded and cured in tap water for 28 days.

2.5. Electrochemical Measurements
Linear polarization resistance, potentiodynamic polarization, and cyclic voltammetry tests were used to investigate the RLE inhibition effect on reinforcing steel in concrete in 3.5% NaCl solution at room temperature. This test was done using the program of Wenking MLab Potentiostat Galvanostat instrument (GERMAN origin).
2.5.1. Polarization Tests
This part contains two tests which were done on the same specimen. The cell contains reinforcing steel in mortar as working electrode, Ag/AgCl as reference electrode and platinum rode as counter electrode which were immersed partially in 3.5% NaCl solution (up to 7 cm height) in a suitable container. These tests were done after 180 days of exposure with three samples for each concentration.

- Linear Polarization Resistance test
  The potential of the reinforcing steel electrode was swept at a scan rate of 0.167 mV/s with a potential range of -20 mV to +20 mV from the open circuit potential. The polarization resistance \( R_p \) of embedded steel in cement mortar was determined from the resulting slope according to equation (1) below [17]:
  \[
  R_p = \frac{\Delta E}{\Delta I}
  \]
  Where \( \Delta I \) is the current difference and \( \Delta E \) is the potential difference.

- Potentiodynamic test
  Tafel polarization method was used to determine the corrosion parameters of reinforcing steel bar embedded in cement mortar. The potential of the reinforcing steel electrode was swept at a scan rate of 0.167 mV/s and a potential range of -200 mV to +200 mV from the open circuit potential. Wenking MLab Potentiostat Galvanostat instrument (German origin) under potentiodynamic conditions was used to carry out the tests. The program of this instrument was used to evaluate the corrosion parameters such as corrosion current \( (I_{corr}) \), corrosion potential \( (E_{corr}) \), anodic Tafel slope \( (b_a) \) and cathodic Tafel slope \( (b_c) \) from the conducted polarization curves. All the experiments were carried out at room temperature. Corrosion inhibition efficiency \( (IE \%) \) and corrosion rate \( (\mu m/y) \) was calculated according to the measured corrosion current density by using equations (2) and (3) below [18]:
  \[
  IE\% = \frac{I_{corr} - I}{I_{corr}} \times 100
  \]
  Where \( I_n \) and \( I \) are corrosion current density without and with inhibitor respectively.
  \[
  \text{Corrosion Rate (} \mu \text{m/y)} = 3.27 \frac{I_{corr} \cdot E.W}{d}
  \]
  Where \( d \) is the density of iron (7.874 g/cm\(^3\)) [27], \( I_{corr} \) is the corrosion current density (\( \mu A/cm^2 \)), \( E.W \) is the equivalent weight of iron (27.93 gm).

2.5.2. Cyclic Voltammetry Test
This test was done after 70 days of immersion in 3.5% NaCl solution using a conventional three-electrode cell mentioned above. Pine Wave Driver 10 Potentiostat/Galvanostat (American origin) instrument was used for cyclic voltammetry measurements. The cyclic potentiodynamic curves were obtained from scanning the potential in forward direction from \(-1.1 \text{ V to 0.6 V and then reversed in the backward direction to } -1.1 \text{ V, vs. Ag-AgCl with a scan rate of 10 mV/s. Immersion was done in aerated test solution at room temperature and atmospheric pressure.}

2.5.3. Photographic Test
Reinforcing steel samples were photoed after each polarization test.

2.5.4. Compressive Strength Test
The compressive strength test was carried out after 28 days of curing in tap water, (as ASTM C-39) using a digital compression machine of 2000 KN capacity. Three specimens for control sample (without inhibitor) and three samples for 0.5% RLE\(_4\) as inhibitor were tested and average values were recorded.
2.5.5. Impressed Voltage Test
This method is one of several methods used for accelerating corrosion damage which gives results in a relatively short time [19]. Cylindrical reinforced steel mortars with no inhibitor and with RLE\textsubscript{4} (0.5\%) inhibitor were used in this test after 7 days of curing. The used cell contained reinforced steel in mortar as working electrode, platinum rod as counter electrode, power supply (VICTOR/VC8054- II type) to apply the required voltage and a multimeter to record current with time, the two electrodes immersed in 3.5\% NaCl solution for height up to 7 cm. The NaCl solution was changed every 4 days. 10 V DC was applied to reinforced steel mortar and the current was recorded every day for 28 days.

3. Results and Discussion

3.1. Polarization Measurements

3.1.1. LPR Polarization Measurement
Figure (2) shows the LPR curves for reinforcing steel in concrete immersing in 3.5\% NaCl solution for 180 days with and without different concentrations of RLE inhibitor. The values of \( R_p \) that calculated from these curves are recorded in Table (4). According to Table (4), the polarization resistance (\( R_p \)) increases with increasing RLE inhibitor concentration compared to the control sample. The lowest \( R_p \) value for control sample means there is a damage in the passive film on steel surface due to Cl\textsuperscript{−} ions attack [9,20]. The increase in \( R_p \) values with the addition of RLE inhibitor may be due to the blocking of pore network by the formation of solid compound from the RLE inhibitor and the pore solution components which slow the propagation of oxygen and Cl\textsuperscript{−} ions through the pores and as a result the corrosion current passing through the pore solution decreases, or maybe the RLE inhibitor forming dense structure protective film on steel surface [9].

![LPR curves for reinforcing steel in concrete immersing in 3.5\% NaCl solution for 180 days with and without different concentrations of RLE inhibitor.](image)

**Figure 2.** LPR curves for reinforcing steel in concrete immersing in 3.5\% NaCl solution for 180 days with and without different concentrations of RLE inhibitor.
Table 4. \( R_p \) for reinforcing steel in concrete immersing in 3.5% NaCl solution for 180 days with and without different concentrations of RLE inhibitor.

|                | \( R_p \) (\( \Omega \)) |
|----------------|--------------------------|
| Control (0.0%) | 685.68                   |
| RLE\(_1\) (0.001%) | 1314.2                   |
| RLE\(_2\) (0.01%) | 1367.2                   |
| RLE\(_3\) (0.1%) | 2469.4                   |
| RLE\(_4\) (0.5%) | 2669.2                   |

3.1.2. Potentiodynamic Polarization Measurements

Figure (3) presents Tafel polarization curves of reinforced steel embedded in mortar samples without and with 0.001%, 0.01%, 0.1% and 0.5% from cement weight RLE inhibitor after 180 days of immersion in 3.5% NaCl solution. Corrosion parameters’ values, including corrosion current density \( (I_{corr}) \), corrosion potential \( (E_{corr}) \), anodic and cathodic Tafel slopes \( (b_a \) and \( b_c ) \), inhibition efficiency (IE) and the corrosion rate (CR) are recorded in Table (5).

As shown in Figure (3), the presence of RLE in mortar mix samples affects both cathodic and anodic branches of Tafel polarization curves as compared with the control sample. There is a large shift of anodic and cathodic curves towards the low current density side with increasing inhibitor concentration, indicating that RLE inhibitor suppressed both the anodic and cathodic reactions through adsorption on the corroded steel surface [22].

Inspection of the results in Table (5) reveals that the corrosion current density \( (I_{corr}) \) and the corrosion rate (CR) of reinforcing steel in mortar samples decreased and the inhibition efficiency increased with the addition of RLE inhibitor. The corrosion current density of control sample is 0.848 \( \mu A/cm^2 \) which indicates that the passive film formed on steel surface due to the alkalinity of cement did not withstand the attack of chloride ions, but with the addition of 0.001% by weight of cement RLE inhibitor, the \( I_{corr} \) of reinforcing steel decreased to 0.649 \( \mu A/cm^2 \), showing inhibition efficiency of 23.45%. Increasing the inhibitor concentration to 0.5% decrease \( I_{corr} \) to 0.195 \( \mu A/cm^2 \), exhibiting inhibition efficiency of 76.98%. This behavior associated with resistance polarization \( (R_p) \) increase which indicates that a protective film was formed on the metal surface [2,10,21].

Also, it can be seen from Table (5) that the presence of the RLE inhibitor shifts corrosion potential \( (E_{corr}) \) slightly with irregular manner in the negative direction and positive direction compared with the control sample (blank). According to the literature when the shifting in \( E_{corr} \) is less than \( \pm 85 \) mV with respect to the \( E_{corr} \) of the blank, so the inhibitor can be classified as mixed type [11]. In this research, the largest shift was -73.6 mV, which is lower than -85 mV, this indicates that RLE is mixed-type inhibitor. Also, the values of \( b_a \) and \( b_c \) do not increase or decrease in a regular manner which again proved the mixed mode inhibition of this inhibitor [12].

Table (5) shows that increasing the inhibitor concentration causing a significant decrease in the current density \( (I_{corr}) \) values and slight changes in values of \( b_a \) and \( b_c \) and corrosion potential \( (E_{corr}) \) compared with blank sample. This suggests that the RLE molecules are adsorbed on the surface of rebar steel and blocking the corrosion of active areas or in other word do not alter the mechanism of corrosion reaction of rebar surface [13].
Figure 3. Polarization curves of reinforcing steel in mortar without and with different concentrations of RLE inhibitor after 180 days of curing in 3.5% NaCl solution.

Table 5. Corrosion parameters obtained from polarization curves of reinforcing steel in mortar with no inhibitor and with varying RLE inhibitor concentrations after 180 days of curing in 3.5% NaCl solution.

|        | $E_{\text{corr}}$ (mV) | $i_{\text{corr}}$ (µA/cm$^2$) | $b_a$ (mV/Dec) | $b_c$ (mV/Dec) | CR (µm/yr) | IE%   |
|--------|------------------------|-------------------------------|----------------|----------------|------------|-------|
| Control | -480.6                 | 0.848                         | 132.5          | 141.4          | 9.825      |       |
| RLE1 (0.001%) | -512.4                | 0.649                         | 174.5          | 191.1          | 7.519      | 23.45 |
| RLE2 (0.01%)  | -554.2                 | 0.427                         | 134.5          | 143.9          | 4.947      | 49.68 |
| RLE3 (0.1%)   | -438.7                 | 0.212                         | 140.8          | 141.1          | 2.456      | 75.05 |
| RLE4 (0.5%)   | -442.4                 | 0.195                         | 128.3          | 137.4          | 2.259      | 76.98 |

3.2. Cyclic Voltammetry Measurements
Cyclic polarization test in this paper was used to study the effect of RLE inhibitor on the protective film stability and the tendency of rebar steel embedded in concrete to pitting corrosion. It is a relatively fast electrochemical technique. Many researchers [3,4,28,29] reported that the size of the hysteresis loop is an indication of the tendency of metals to pit; the smaller loop, the lesser tendency to pit and easier to re-passivate the pit if it occurs and the vice versa.

Figure 4 shows the cyclic polarization curves of reinforcing steel in mortar without and with different concentrations of RLE inhibitor after 70 days of curing in 3.5% NaCl solution. It is clear from the figure that the control sample had the largest hysteresis loop, this means its tendency to pit is the most. Hysteresis loops of inhibited samples were smaller than the control sample for all inhibitor concentrations, this indicates that the pitting tendency of reinforced steel decreased and became easier to re-passivate the damaged passive film. The size of the hysteresis loop was decreased with increasing inhibitor concentration and it is the lowest at 0.5% RLE$_4$ concentration.
3.3. Photographic Examination
Observations the photos of reinforcing steel in mortar samples immersed in 3.5% sodium chloride solution without inhibitor and with varying RLE inhibitor concentrations for 180 days after tafel polarization test are shown in Figure (5). It can be seen from figures that the inhibited samples are cleanest compared to control sample and the increase in inhibitor concentration increase the cleanness of the samples. It is evident from the figures that the reinforcing steel in mortar containing 0.5% RLE inhibitor is the cleanest one.

Figure 5. Photos after Tafel polarization test of reinforcing steel in mortar samples immersed in 3.5% sodium chloride solution for 180 days without and with RLE inhibitor as a percent of cement weight: (A) 0.0% (control), (B) 0.001% (RLE1), (C) 0.01% (RLE2), (D) 0.1% (RLE3), (E) 0.5% (RLE4).

3.4. Compression Strength Test
The compressive strength values of concrete cubes without and with 0.5% RLE inhibitor after 28 days of curing in tap water were given in Table (6). The results showed that the additions of RLE inhibitor to the concrete mix did not have any adverse effect on the mechanical properties of concrete, but there is an increase in compressive strength about 7.16%. This increase may be due to block the pores of the
concrete structure by RLE inhibitor [5]. As a result, it can be concluded that RLE inhibitor combined a good inhibition efficiency with compressive strength improvement advantages.

Table 6. Compression strength results of concrete with and without inhibitor after 28 days of curing in tap water.

|                  | Compression Strength (Mpa) |
|------------------|----------------------------|
| Control          | 34.593                     |
| RLE4             | 37.263                     |

3.5. Impressed Voltage Test

This method was used to assess the initiation time of corrosion under accelerated test conditions. The initiation time of corrosion can be identified by a sharp rise in current [6,14]. Figure (6) shows the current variations in constant voltage impressed test of 28 days period for reinforced steel in mortar of blank sample and in mortar contains 0.5% RLE immersed in 3.5% NaCl solution. It can be seen from the figure that initially there is a small amplitude variation in the current but after that initiation time, corrosion started with a large increase in current on the seventh day for the control sample and on the eighteen day for the inhibited sample. Also, it is noted that the maximum current obtained for the control sample is 50.7 mA on the thirteenth day and for the inhibited sample is 37.95 mA on the twenty-fifth day. This means that the RLE inhibitor has long corrosion initiation time and long failure time compared with blank mortar sample due to the protective film formed on reinforcing steel by the RLE inhibitor. [14]

Figure (7) shows photos after impressed voltage test of reinforced steel in mortar samples immersed in 3.5% sodium chloride solution for 28 days without and with 0.5% RLE inhibitor. This photo explains that the corrosion area in control sample is larger than inhibited sample.

**Figure 6.** Impressed voltage test results for control and 0.5% RLE inhibitor (RLE4) samples after 28 days.

**Figure 7.** Photos after impressed voltage test of reinforced steel in mortar samples immersed in 3.5% sodium chloride solution for 28 days without and with 0.5% RLE inhibitor (RLE4).
4. Conclusion

- The maximum IE for 180 days was 76.98% at RLE\textsubscript{4} (0.5%) with corrosion rate of one quarter the value in the case of no inhibitor.
- The Potentiodynamic polarization curves showed that RLE inhibitor acts as a mixed-type inhibitor.
- Cyclic Voltammetry test results showed that the tendency of reinforcing steel in mortar to pitting corrosion decreased with the increase of RLE inhibitor concentration, the best result was at 0.5% RLE concentration.
- Compression strength test results showed that RLE inhibitor increased the compression strength by 7.16% at 0.5% RLE.
- Impressed voltage test showed that the corrosion of reinforced steel bar initiated on the seventh day of experiment for the control sample while on eighteenth day of the experiment for RLE sample at 0.5% concentration.
- RLE inhibitor is a good eco-friendly corrosion inhibitor for concrete reinforcing steel, results showed that IE increased even at low inhibitor concentrations.

5. References

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