Evaluation of Greener Corrosion-Inhibiting Admixtures for Steel Reinforcements in Concrete

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Abstract
In this study, corrosion of thermo-mechanically treated (TMT) rebars with and without 3.5% NaCl for various concentrations (0.1%, 0.2%, 0.3% and 0.4%) of green corrosion inhibitors, namely glucosamine and stevioside, was systematically studied in simulated concrete pore solution (SCPS). Open-circuit potential measurements revealed that inhibited system showed passive behaviour compared with control system in SCPS. Potentiodynamic polarization studies revealed that inhibitor stevioside offers more than 88% inhibition efficiency at 0.3% concentration in SCPS containing 3.5% NaCl. Similarly, linear polarization resistance and impedance measurements showed higher polarization resistance and charge transfer resistance values, respectively, at 0.3% concentration for inhibited systems when compared with control (without inhibitor).

In the second part, the optimized concentration of glucosamine and stevioside showed better performance against corrosion of rebar in the solution study admixed into concrete. The corrosion of TMT rebar embedded in concrete with and without inhibitors is subjected to simulated marine exposure for a period of 180 days and evaluated the corrosion inhibition. Gravimetric weight loss and electrochemical techniques were employed to study the efficacy of inhibitors in SCPS and concrete medium. It is found that the time taken for corrosion-induced cracking in concrete admixed with stevioside inhibitor is approximately 20 times that of control concrete and that stevioside inhibitor is relatively better corrosion inhibitor than other inhibitor reported.

Keywords Reinforcement corrosion · Green corrosion inhibitor · Concrete · Electrochemical techniques · Marine exposure

1 Introduction
Reinforced concrete is utilized in most structures such as buildings and bridges, because of its excellent service and easy maintenance. The durability of reinforced concrete structures is decreased due to several processes such as alkali-aggregate attack, carbonation, chloride attack and corrosion. Of the above-mentioned processes, corrosion is the dominant process affecting the durability of reinforced concrete structures, especially those exposed to marine and industrial environments. Corrosion is the chemical or electrochemical interaction of the metal with the corrosive environments, where the nature of the metal in an environment plays a vital role. The corrosive agents are either directly chloride ions or indirectly carbon dioxide. The solid layer of corrosion products formed on metal is termed as rust. This rust occupies up to six times of the initial volume of metal that is formed in the process of dissolution of metal ions into metallic oxide. The concrete undergoes crack, disintegration and debonding of rebar as a consequence of corrosion in rebar. There are certain methods to control the occurrence of corrosion, such as coating rebar, cathodic protection and corrosion inhibitors. The corrosion inhibitor has gained popularity because of its ease to handle, mix into concrete and cost-effectiveness of the available methods. Moreover, the addition of inhibitors has proven to mitigate the corrosion efficiently [1–5].

Several authors have evaluated the performance of inorganic corrosion inhibitors in solutions [6–15]. Calcium nitrate and nitrite inhibitors are widely used in concrete as corrosion inhibitors worldwide in spite of their drawbacks.
like an irritation to skin and eyes because of their toxicity [16]. There have been reviews published on the most commonly used organic corrosion inhibitors and their possible mechanism [17–21]. Organic corrosion inhibitors are probable source to retard corrosion initiation by admixing it in concrete [22] due to its readily soluble in water and compatibility with protected materials. The inclusion of synthesized organic corrosion inhibitor and leaf extract improves the mechanical properties and corrosion inhibition [23]. The organic corrosion inhibitors may be natural or synthesized [23–25]. The green corrosion inhibitors synthesized from various natural products and their inhibition efficiency are reported [26–35]. The study on the adoption of green corrosion inhibitors in concrete is fewer although the greener corrosion inhibitors have reported.

The inhibitors used in this study are heterocyclic compounds that possess higher basicity and electron density, thereby controlling the corrosion at a relatively lower rate [36]. The better concentration of inhibitors, namely glucosamine ($G_{inh}$) and stevioside ($S_{inh}$), to yield maximum inhibition efficiency is evaluated in SCPS and also in concrete through various non-electrochemical and electrochemical methods. The objective of the present investigation was to study the performance of greener corrosion inhibiting admixture towards the corrosion of TMT rebar in marine environments. Hence, this study covers inhibitors in both simulated concrete pore solution and concrete. The corrosion inhibition efficiency is evaluated for an exposure period of 180 days.

## 2 Materials and Methods

### 2.1 Materials

The chemical name, chemical formula, IUPAC (International Union of Pure and Applied Chemistry) name and chemical structure of inhibitors employed in the present study are given in Table 1. Ordinary Portland cement (OPC) of 43 grade was used for concrete specimen preparation. Manufactured sand (M-sand) was used as fine aggregate with a specific gravity of 2.73 and conforms to zone II as per IS 456:2000. The coarse aggregate of maximum size 20 mm with a specific gravity of 2.85 was used. M20 grade of concrete was designed as per IS 10262:2009. The concrete ingredients proportioned for M20 grade concrete consist of OPC 43 grade—300 kg/m$^3$, fine aggregates—626 kg/m$^3$, coarse aggregates—946 kg/m$^3$ and water—132 L/m$^3$.

### 2.2 Steel Rebar Preparation

The chemical composition of thermo-mechanically treated (TMT) steel rebar used is given in Table 2. The steel rebar of...
Table 2 Chemical composition of TMT steel rebar

| Type of Steel | Chemical composition: IS-1786:2008 wt. (%) |  |
|--------------|------------------------------------------|---|
|              | Carbon | Sulphur | Phosphorus | Sulphur + phosphorus (S + P) | Carbon equivalent (CE) | Fe |
| Fe 500D      | 0.25   | 0.038   | 0.042      | 0.075                          | 0.42                     | 99.175 |

dimensions 12 mm diameter and 50 mm length was used for solution and concrete studies. The steel rebar was cleaned by pickling solution and washed with distilled water followed by drying in an air-dryer. The electrical wire was connected with steel rebar and sealed with epoxy.

2.3 Preparation of SCPS and Rebar in SCPS medium

The SCPS was used as an electrolyte and prepared by adding 3.36 g of KOH and 7.6 g of NaOH dissolved in 1 L of saturated calcium hydroxide solution [37]. The solution was filtered through Wattman filter paper to remove undissolved CaO precipitate, and 35 g of NaCl was dissolved in it. The pH of this chloride-contaminated SCPS was 12.80. The prepared steel bar was immersed in a beaker containing the above electrolyte. The various concentrations of inhibitors, namely glucosamine and stevioside, in the order of 0.1%, 0.2%, 0.3% and 0.4% are added to the electrolyte in each beaker separately and monitored for a period of 180 days.

2.4 Studies in Concrete Medium

2.4.1 Casting of Concrete Specimens for Electrochemical Studies

Reinforced cylindrical concrete specimens of size 30 mm in diameter and 60 mm in height were cast with centrally placed rebar of size 12 mm in diameter and 50 mm in length. Specimens were cast with 0.3% (optimized from solution studies) of inhibitors GS inh and SV inh admixed into concrete by weight of cement. The concrete specimens were subjected to a curing period of 28 days in distilled water. After curing, the concrete specimens were taken out and dried at room temperature. The top and bottom portions of concrete specimens were sealed with epoxy to ensure the flow of chloride ions is only from the surrounding surface of cylindrical concrete. The concrete specimens were subjected to alternate wet and dry cycles in 3.5% NaCl solution for an exposure period of 180 days. The corrosion resistance properties of inhibitor admixed in concrete were evaluated through electrochemical techniques and compared with control concrete (specimens without inhibitor).

2.5 Electrochemical Test Methods

2.5.1 OCP Measurements

The potential-time behaviour of steel immersed in SCPS containing 3.5% NaCl in the absence and presence of GS inh and SV inh inhibitors was periodically monitored using a high-impedance voltmeter (20 M.Ohm) for an exposure period of 180 days. The OCP was measured as per the ASTM C876-09 standard. Similarly, the open-circuit potential of the steel rebar embedded in concrete with and without inhibitors was recorded with time upon exposure to 3.5% NaCl solution (considered as simulated marine environments) for a period of 180 days.

2.5.2 Potentiodynamic Polarization

Potentiodynamic polarization studies were carried out for steel rebar embedded in concrete without and with inhibitors that are subjected to alternate wet and dry cycles of 3.5% NaCl for the exposure period of 180 days. Figure 1 shows the experimental set-up for polarization studies. The steel rebar serves as a working electrode and the saturated calomel electrode (SCE) as a reference electrode for both SCPS and concrete medium, whereas the counter electrode is the platinum foil and stainless steel for SCPS and concrete medium, respectively. The potentiodynamic polarization of the rebar was performed over the potential range of ±200 mV (vs. SCE) from OCP. The sweep rate was 0.1 mV/s. The above-mentioned potentiodynamic conditions were adopted in common for both SCPS and concrete medium. Both cathodic and anodic polarization curves were recorded using an electrochemical analyser. This instrument itself has provision to calculate the corrosion kinetic parameters such as corrosion potential (E corr), corrosion current (I corr), anodic (b a) and cathodic Tafel slopes (b c).

2.5.3 Linear Polarization Resistance (LPR)

The polarization condition corresponds to ±20 mV vs. SCE. The sweep rate is 0.1 mV/s. The plots of potential versus current were recorded for the rebar both in SCPS and concrete medium. The linear polarization resistance parameters were calculated by using the in-built software of the instrument.
2.5.4 Electrochemical Impedance Spectroscopy

The same three-electrode cell assembly was used for impedance studies in both SCPS and concrete medium. The Nyquist plot of the real part ($Z'$) and the imaginary part ($-Z''$) of the cell impedance was measured in the frequency range from 30,000 to 0.1 Hz.

2.6 Gravimetric Weight Loss Method

The prepared clean steel rebar of known weight was immersed in chloride-contaminated SCPS for an exposure period of 180 days. The rebar was removed out of SCPS at the end of the exposure period, and then, the formation of rusts was removed by using pickling solution. The air-dried rebar without any rust was weighed as the final weight of rebar, and the corrosion rate of the steel rebar is calculated by using Eq. 1 [38]:

$$C.R = \frac{87.6(w_a - w_b)}{DA \tau}$$

where C.R represents the corrosion rate and its unit is millimetre per year (mmpy), $w_a$ and $w_b$ are an initial and final weight of the rebar (mg), respectively, ‘$D$’ is the density of the steel rebar (g/cm$^3$), ‘$A$’ is the exposure area of the steel (cm$^2$) and ‘$\tau$’ is the exposure time (h).

In the concrete medium, at the end of the exposure period, the concrete specimens were split open and the steel rebar was taken out. The formation of rust products was identified. Then, the corrosion rate of steel rebar in concrete is calculated by the above-said procedure.

2.7 Cement Mortar and Concrete Specimens Cast

The cement mortar cubes of dimensions 7.06 cm × 7.06 cm × 7.06 cm with and without inhibitors were cast in accordance with IS:4031(part6)-1988. The concrete cube of dimension 15 cm × 15 cm × 15 cm was cast with and without the inhibitors admixed in concrete. All cubes were subjected to a curing period of 28 days in potable water. After respective curing periods, all the cast specimens were subjected to compression testing as per IS 516-2006.

3 Results and Discussion

3.1 Electrochemical Studies of Rebar in SCPS and Concrete Medium Without and with Inhibitor

3.1.1 OCP Measurements

The electrochemical corrosion potential of steel rebar in SCPS containing 3.5% NaCl with various percentages as 0%, 0.1%, 0.2%, 0.3% & 0.4% of inhibitors GS$_{inh}$ and SV$_{inh}$ with respect to SCE over an exposure period of 180 days is as shown in Fig. 2(a–b). Figure 2(a) shows the potential-time behaviour of steel rebar in SCPS containing 3.5% NaCl with 0%, 0.1%, 0.2%, 0.3% and 0.4% of GS$_{inh}$. The OCP values were $-555 \pm 5$ mV, $-525 \pm 5$ mV, $-440 \pm 5$ mV, $-390 \pm 5$ mV and $-540 \pm 5$ mV vs. SCE, respectively.

The OCP of steel rebar in GS$_{inh}$ inhibited solution shifted towards a more positive direction when compared with control. Moreover, the potential shifted towards positive direction up to 0.3% concentration and turned towards a negative shift at 0.4% concentration. It is observed from Fig. 2 (b) that the same trend is noticed in SCPS containing 3.5%
NaCl with 0%, 0.1%, 0.2%, 0.3% and 0.4% of SV inh. In comparison with the control system, both the inhibitors shifted the potential towards the more positive direction. During the OCP measurements, the fluctuation is observed up to 6 days of exposure in both the systems including inhibited solutions. It indicates minimum 6-day incubation period is required to stabilize the potential of the steel rebar. Thereafter, the OCP values of steel rebar in inhibited systems shifted towards a positive direction indicating the formation of a passive film of inhibitor molecules on the steel rebar interfacial region which mitigates the attack of aggressive chloride ions [39]. At the end of exposure, both the inhibited system showed more positive potential when compared with control system (absence of inhibitor). OCP values indicate that the probability of corrosion occurrence is less in corrosion-inhibited system at steel rebar/concrete interface.

3.1.2 Potentiodynamic Polarization

The potentiodynamic polarization curves of steel rebar immersed in SCPS with 3.5% NaCl and various percentages of GS inh and SV inh are shown in Fig. 4(a-b), and their corresponding corrosion kinetic parameters are given in Table 3. It is observed from Fig. 4(a) and Fig. 4(b) that the E corr values of GS inh and SV inh systems were gradually shifted to positive directions (up to 0.3%) when compared with a control system (without inhibitor) and the corrosion potential (E corr) values of steel rebar in SCPS with 3.5% NaCl and various percentages of 0%, 0.1%, 0.2%, 0.3% and 0.4% of GS inh and SV inh with respect to SCE were −615 mV, −600 mV, −504 mV, −450 mV, −612 mV, −615 mV, −557 mV, −4704 mV, −452 mV, −614 mV, respectively. The E corr, I corr and corrosion rate values of steel rebar in SCPS with 3.5% NaCl and various percentages of 0%, 0.1%, 0.2%, 0.3% and
Table 3 | Potentiodynamic polarization parameters for steel rebar in SCPS containing 3.5% NaCl with and without inhibitors

| System                  | Corrosion potential ($E_{corr}$) in mV vs. SCE | Corrosion current density ($I_{corr}$) in mA/cm$^2 \times 10^{-3}$ | Corrosion rate in mmpy $\times 10^{-2}$ | Inhibitor efficiency ($\varphi$) in % |
|-------------------------|-----------------------------------------------|--------------------------------------------------------------------|----------------------------------------|--------------------------------------|
| Control                 |                                               |                                                                    |                                        |                                      |
| SCPS + 3.5% NaCl        | - 615                                         | 2.701                                                               | 3.130                                   | -                                    |
| Glucosamine (GS$_{inh}$) |                                               |                                                                    |                                        |                                      |
| SCPS + 3.5% NaCl + 0.1%GS$_{inh}$ | - 600                                         | 2.093                                                               | 2.194                                   | 22.51                                |
| SCPS + 3.5% NaCl + 0.2%GS$_{inh}$ | - 504                                         | 1.011                                                               | 1.172                                   | 62.57                                |
| SCPS + 3.5% NaCl + 0.3%GS$_{inh}$ | - 450                                         | 0.719                                                               | 0.833                                   | 73.38                                |
| SCPS + 3.5% NaCl + 0.4%GS$_{inh}$ | - 612                                         | 2.313                                                               | 2.681                                   | 14.37                                |
| Stevioside (SV$_{inh}$) |                                               |                                                                    |                                        |                                      |
| SCPS + 3.5% NaCl + 0.1%SV$_{inh}$ | - 557                                         | 1.015                                                               | 1.176                                   | 62.42                                |
| SCPS + 3.5% NaCl + 0.2%SV$_{inh}$ | - 470                                         | 0.327                                                               | 0.379                                   | 87.89                                |
| SCPS + 3.5% NaCl + 0.3%SV$_{inh}$ | - 452                                         | 0.309                                                               | 0.358                                   | 88.56                                |
| SCPS + 3.5% NaCl + 0.4%SV$_{inh}$ | - 614                                         | 2.473                                                               | 2.866                                   | 08.44                                |

0.4% of GS$_{inh}$ and SV$_{inh}$ are given in Table 3. The corrosion current density ($I_{corr}$), corrosion rate (C.R) values of 0.3% GS$_{inh}$ and SV$_{inh}$ system were $0.719 \times 10^{-3}$ mA/cm$^2$, $0.833 \times 10^{-2}$ mmpy and $0.309 \times 10^{-3}$ mA/cm$^2$, $0.358 \times 10^{-2}$ mmpy, respectively.

From the potentiodynamic polarization studies, it was understood that both the inhibitors showed lower corrosion current density and corrosion rate values when compared with the control system. Out of the systems studied, the 0.3% SV$_{inh}$ system showed the best performance and ability to protect the steel rebar immersed in SCPS with 3.5% NaCl when compared with GS$_{inh}$ and control systems. The efficiency of inhibitor ($\varphi$) was calculated from $i_{corr}$ values using Eq. (2) and is given in Table 3.

\[
\text{Inhibitor efficiency (}\varphi\text{)} \% = \frac{i_{corr} - i_{corr(inh)}}{i_{corr}} \times 100 \quad (2)
\]

where $i_{corr}$ and $i_{corr(inh)}$ are the corrosion current density of steel rebar in SCPS containing 3.5% NaCl in the absence and presence of inhibitors, respectively.

It can be observed from Table 3 that the inhibition efficiency obtained in the presence of GS$_{inh}$ and SV$_{inh}$ at 0.3% is 73.38% and 88.56%, respectively. The inhibitor efficiency
Table 4 Potentiodynamic polarization parameters for steel rebar in concrete with and without inhibitors at 1st day and 180th day of marine exposure

| System                                | Corrosion potential (E_{corr}) in mV vs SCE | Corrosion current density (I_{corr}) in mA/cm^2 × 10^{-3} | Corrosion rate in mmpy × 10^{-3} | Inhibitor efficiency (ϕ) in % |
|----------------------------------------|---------------------------------------------|-------------------------------------------------------------|----------------------------------|-------------------------------|
| Control concrete without inhibitor    | −471 ± 5 mV                                 | 2.10 ± 0.05                                                | 2.433 × 10^{-3}                  | −                             |
| Glucosamine (GS_{inh})                | −246 ± 5 mV                                 | 0.506 ± 0.05                                               | 0.587 × 10^{-3}                  | 67.23                         |
| Concrete with inhibitor GS_{inh}      | −207 ± 5 mV                                 | 0.140 ± 0.01                                               | 0.162 × 10^{-3}                  | 93.34                         |
| Concrete with inhibitor SV_{inh}      | −651 ± 5 mV                                 | 7.985 ± 0.07                                               | 9.254 × 10^{-3}                  | 80.13                         |

gets more pronounced with 0.3% of inhibitor concentration and reduces at higher concentration (0.4%). It may be because the desorption of the inhibitor at higher concentrations is the probable reason for the low inhibition efficiency. The maximum inhibition efficiency acquired at 0.3% of SV_{inh} with the lowest I_{corr} values is due to the formation of the protective film over the steel rebar surface [42]. This protective film acts as a barrier for the mass charge transfer process and thereby mitigates the corrosion reaction. Here, it is found that the inhibitor stevioside offers more than 88% inhibition efficiency at 0.3% concentration in SCPS containing higher amounts of chlorides.

The potentiodynamic polarization of steel rebar embedded in concrete without inhibitor and with 0.3% of inhibitors GS_{inh} and SV_{inh} exposed during initial days and end of 180 days of sea water exposure is shown in Fig. 5(a–b), and their corresponding corrosion kinetics parameters are given in Table 4. From Fig. 5(a–b), the E_{corr} values of steel rebar in concrete without inhibitor (control specimen), concrete admixed with 0.3% of inhibitors GS_{inh} and SV_{inh} with respect to SCE exposed during the initial days and 180 days of sea water exposure are -471 ± 5 mV, -246 ± 5 mV, -207 ± 5 mV and -651 ± 5 mV, -580 ± 5 mV and -560 ± 5 mV, respectively. The E_{corr} values of SV_{inh} inhibitors used system were gradually shifted to positive directions when compared with control system (concrete without inhibitors) [43].

The corrosion rate of steel rebar embedded in concrete without inhibitor, with 0.3% of inhibitors GS_{inh} and SV_{inh} is 2.433 × 10^{-3} mmpy, 0.587 × 10^{-3} mmpy, 0.162 × 10^{-3} mmpy and 28.24 × 10^{-3} mmpy, 9.254 × 10^{-3} mmpy and 5.611 × 10^{-3} mmpy, respectively, at the beginning and end of 180-day sea water exposure. From these results, it con-
Table 5  LPR parameters for steel rebar in SCPS containing 3.5% NaCl with and without inhibitors

| System                        | Corrosion potential ($E_{corr}$) in mV vs SCE | Linear polarization resistance ($R_{p}$) in $\Omega \cdot \text{cm}^2 \times 10^3$ | Corrosion current density ($I_{corr}$) in mA/cm$^2 \times 10^{-3}$ | Corrosion rate in mmpy $\times 10^{-2}$ | Inhibitor efficiency ($\phi$) in % |
|-------------------------------|-----------------------------------------------|----------------------------------------------------------------------------------|---------------------------------------------------------------------|--------------------------------------|----------------------------------|
| Control System                |                                               |                                                                                  |                                                                     |                                      |                                  |
| SCPS + 3.5% NaCl              | − 555                                         | 7.814                                                                            | 3.238                                                              | 3.753                                | −                                |
| Glucosamine (GSinh)           |                                               |                                                                                  |                                                                     |                                      |                                  |
| SCPS + 3.5% NaCl + 0.1% GSinh | − 522                                         | 13.365                                                                           | 1.906                                                              | 2.209                                | 41.53                            |
| SCPS + 3.5% NaCl + 0.2% GSinh | − 439                                         | 15.678                                                                           | 1.631                                                              | 1.890                                | 50.16                            |
| SCPS + 3.5% NaCl + 0.3% GSinh | − 388                                         | 31.128                                                                           | 0.795                                                              | 0.921                                | 74.90                            |
| SCPS + 3.5% NaCl + 0.4% GSinh | − 538                                         | 10.924                                                                           | 2.495                                                              | 2.892                                | 28.47                            |
| Stevioside (SVinh)            |                                               |                                                                                  |                                                                     |                                      |                                  |
| SCPS + 3.5% NaCl + 0.1% SVinh | − 495                                         | 15.260                                                                           | 1.658                                                              | 1.922                                | 48.81                            |
| SCPS + 3.5% NaCl + 0.2% SVinh | − 413                                         | 31.940                                                                           | 0.617                                                              | 0.715                                | 75.54                            |
| SCPS + 3.5% NaCl + 0.3% SVinh | − 383                                         | 34.110                                                                           | 0.591                                                              | 0.685                                | 77.09                            |
| SCPS + 3.5% NaCl + 0.4% SVinh | − 539                                         | 8.290                                                                            | 2.888                                                              | 3.338                                | 5.753                            |

firms that 0.3% of inhibitor SVinh inhibits corrosion better compared with concrete without inhibitor and with inhibitor GSinh both at initial days and at the end of 180 days of sea water exposure.

From the potentiodynamic studies, both the inhibitors GSinh and SVinh are lesser in corrosion current and corrosion rate values when compared with control system. But, 0.3% of inhibitor SVinh admixed into concrete can able to protect the steel rebar embedded in concrete and exposed to sea water condition up to 180 days than the inhibitor GSinh and also corrosion progress of inhibitor SVinh is 15 times lesser than control system. Further, the efficiency of inhibitor ($\phi$%) was calculated from $i_{corr}$ values by Eq. 2 and is given in Table 4. It can be observed from Table 4 that the inhibition efficiency obtained in the presence of 0.3% inhibitors GSinh and SVinh during initial and end of 180-day sea water exposure is 67.23%, 80.13% and 75.87%, 93.34%, respectively. The inhibition efficiency of inhibitor SVinh is 23.02% higher than
that of inhibitor GS$_{inh}$. Thereby, the inhibitor SV$_{inh}$ inhibits corrosion better compared with another inhibitor.

### 3.1.3 Linear Polarization Resistance (LPR)

The LPR curves for steel rebar immersed in SCPS containing 3.5% NaCl with various percentages of inhibitors GS$_{inh}$ and SV$_{inh}$ are shown in Fig. 6(a–b), and their corresponding corrosion kinetics parameters are given in Table 5. It is observed from Fig. 6(a–b) that the polarization resistance ($R_p$) values of steel rebar in SCPS with 3.5% NaCl and various percentages of 0%, 0.1%, 0.2%, 0.3% and 0.4% of GS$_{inh}$ and SV$_{inh}$ are $7.814 \times 10^3$ $\Omega$.cm$^2$, $13.365 \times 10^3$ $\Omega$.cm$^2$, $15.678 \times 10^3$ $\Omega$.cm$^2$, $31.128 \times 10^3$ $\Omega$.cm$^2$, $10.924 \times 10^3$ $\Omega$.cm$^2$ and $7.814 \times 10^3$ $\Omega$.cm$^2$, respectively. On the other hand, the corrosion current density values of GS$_{inh}$ and SV$_{inh}$ at 0.3% were $0.795 \times 10^{-3}$ mA/cm$^2$ and $0.591 \times 10^{-3}$ mA/cm$^2$, respectively. The results of LPR experiments confirm that the addition of inhibitor in SCPS containing 3.5% NaCl increased the $R_p$ value and reduced the rate of corrosion reaction on steel rebar surface. It is due to the adsorption of inhibitor molecule, and it forms a protective layer over the steel rebar surface.

The efficiency of inhibitor ($\phi$%) was calculated from $R_p$ values by using Eq. (3) [44] and is given in Table 5.

\[
\text{Inhibitor Efficiency (}\phi\text{)}\% = \frac{R_{P}^{(inh)} - R_P}{R_{P}^{(inh)}} \times 100 \tag{3}
\]

where $R_p$ and $R_{P}^{(inh)}$ are linear polarization resistance of steel rebar in SCPS containing 3.5% NaCl in the absence and presence of inhibitors, respectively. It is observed from Table 5 that the inhibition efficiency obtained in the presence of inhibitors GS$_{inh}$ and SV$_{inh}$ at 0.3% was 74.90% and 77.09%, respectively. The maximum inhibition efficiency acquired was 77.09% for the system 0.3% SV$_{inh}$. LPR studies also confirm that the rate of corrosion reaction of steel rebar was controlled in the presence of inhibitor in SCPS containing 3.5% NaCl solution, which may be due to the formation of passive layer on steel rebar surface, that reduces the mass charge transfer process and mitigates the corrosion reaction [45].

The LPR curve for steel rebar embedded in concrete without inhibitor with 0.3% of inhibitors GS$_{inh}$ and SV$_{inh}$ during initial days and end of 180 days of marine exposure is shown in Fig. 7(a–b), and the corresponding corrosion kinetics parameters are given in Table 6. It is observed from Fig. 7(a–b) and Table 6 that the LPR values of steel rebar in concrete without inhibitor (control specimen) and concrete admixed with 0.3% of inhibitors GS$_{inh}$ and SV$_{inh}$ exposed during initial and 180 days of sea water exposure are $34.81 \times 10^3$ $\Omega$.cm$^2$, $136.83 \times 10^3$ $\Omega$.cm$^2$, $207.21 \times 10^3$ $\Omega$.cm$^2$ and $16.321 \times 10^3$ $\Omega$.cm$^2$, $34.435 \times 10^3$ $\Omega$.cm$^2$, $7.556 \times 10^3$ $\Omega$.cm$^2$ and $16.321 \times 10^3$ $\Omega$.cm$^2$, respectively. From these results, at both initial and end of 180-day exposure the addition of 0.3% inhibitor SV$_{inh}$ shows higher LPR value in comparison with without inhibitor system (control) sample and other inhibitor [46].

Further, the corrosion current density ($I_{corr}$) values of steel rebar in concrete without inhibitor and 0.3% inhibitors GS$_{inh}$ and SV$_{inh}$ at the end of 180 days of sea water exposure condition were $25.880 \times 10^{-4}$ mA/cm$^2$, $10.26 \times 10^{-4}$ mA/cm$^2$ and $6.311 \times 10^{-4}$ mA/cm$^2$, respectively. These results also confirm that the addition of inhibitor SV$_{inh}$ in concrete increases the LPR value and reduces the rate of corrosion reaction on steel rebar surface. It is due to adsorption of inhibitor molecule, and it forms a protective layer on steel rebar surface.

Likewise, the efficiency of inhibitor ($\phi$%) was calculated from LPR values by Eq. 3 [40] and is given in Table 6. It is obvious from Table 6 that the inhibition efficiency obtained by adding 0.3% of inhibitors GS$_{inh}$ and SV$_{inh}$ was...
60.35% and 75.61%, respectively. The inhibitor efficiency gets more pronounced with 0.3% of inhibitor SV inh. The maximum inhibition efficiency acquired was 75.61% in 0.3% of inhibitor SV inh with higher shift in LPR values towards higher direction. This study too confirms the rate of corrosion reaction of steel rebar was controlled in the presence of inhibitor SV inh admixed concrete exposed to sea water condition, which may be due to the formation of passive layer on steel rebar surface and reduction in the mass charge transfer process and rate of corrosion reaction [47].

### 3.1.4 Electrochemical Impedance Spectroscopy

Figure 8 shows the Nyquist plot for steel rebar immersed in SCPS containing 3.5% NaCl with various percentages of inhibitors GS inh and SV inh. The corresponding impedance parameters are given in Table 7. Figure 8(a) shows the Nyquist plot for steel rebar immersed in SCPS containing 3.5% NaCl with various percentages of GS inh. The charge transfer resistance (R ct) values of steel rebar immersed in SCPS containing 3.5% of NaCl with 0%, 0.1%, 0.2%, 0.3% and 0.4% of GS inh are $1.419 \times 10^3$ Ω.cm², $6.976 \times 10^3$ Ω.cm², $10.200 \times 10^3$ Ω.cm², $12.010 \times 10^3$ Ω.cm² and $5.948 \times 10^3$ Ω.cm², respectively. The R ct values are significantly increased by the addition of GS inh up to 0.3%, which indicates the formation of a passive film of inhibitor molecules on the steel rebar surface. Hence, the resistance is created between steel rebar and solution interface and thereby reduces the rate of corrosion.

On the other hand, the Nyquist plot of steel in SV inh system is shown in Fig. 8(b). The R ct value of SV inh system at 0.3% was found to be $13.83 \times 10^3$ Ω.cm², and it is the highest R ct value observed when compared with other counter inhibitors. The capacitance double-layer (C dl) values also gradually decreases, which is due to gradual replacement of the water molecules through adsorption of inhibitor on rebar–solution interface, and it reduces the steel rebar corrosion reaction [44, 45].

Figure 9(a–b) shows the Nyquist plot of steel rebar embedded in concrete without inhibitor, with 0.3% of inhibitors GS inh and SV inh during initial days and end of 180 days of marine exposure. From Table 8, the charge transfer resistance (R ct) values of steel rebar in concrete without inhibitor (control specimen) and concrete admixed with 0.3% of inhibitors GS inh and SV inh with respect to SCE during initial days of marine exposure were $4.417 \times 10^4$ Ω.cm², $8.345 \times 10^4$ Ω.cm² and $18.570 \times 10^4$ Ω.cm², respectively. The Nyquist
Table 7  Impedance parameters for steel rebar in SCPS containing 3.5% NaCl with and without inhibitors

| System                  | Solution resistance ($R_s$) in $\Omega$.cm$^2$ | Charge transfer resistance ($R_{ct}$) in $\Omega$.cm$^2 \times 10^3$ | Double layer capacitance ($C_{dl}$) in F/cm$^2 \times 10^{-5}$ | Corrosion current density ($I_{corr}$) in mA/cm$^2 \times 10^{-3}$ | Corrosion rate in mmpy $\times 10^{-2}$ |
|-------------------------|-----------------------------------------------|---------------------------------------------------------------|-------------------------------------------------|-------------------------------------------------|----------------------------------|
| Control                 |                                               |                                                              |                                                |                                                |                                  |
| SCPS + 3.5% NaCl        | 13.760                                        | 1.419                                                        | 5.154                                          | 18.380                                          | 21.310                           |
| Glucosamine             |                                               |                                                              |                                                |                                                |                                  |
| SCPS + 3.5% NaCl + 0.1%GSinh | 7.606                                         | 6.976                                                        | 2.156                                          | 3.739                                          | 4.334                            |
| SCPS + 3.5% NaCl + 0.2%GSinh | 9.260                                         | 10.200                                                       | 1.545                                          | 2.558                                          | 2.965                            |
| SCPS + 3.5% NaCl + 0.3%GSinh | 6.605                                         | 12.010                                                       | 1.304                                          | 2.171                                          | 2.516                            |
| SCPS + 3.5% NaCl + 0.4%GSinh | 8.929                                         | 5.948                                                        | 1.817                                          | 4.386                                          | 5.083                            |
| Stevioside              |                                               |                                                              |                                                |                                                |                                  |
| SCPS + 3.5% NaCl + 0.1%SVinh | 4.509                                         | 7.868                                                        | 1.430                                          | 3.316                                          | 3.843                            |
| SCPS + 3.5% NaCl + 0.2%SVinh | 8.021                                         | 12.320                                                       | 1.207                                          | 2.117                                          | 2.454                            |
| SCPS + 3.5% NaCl + 0.3%SVinh | 4.699                                         | 13.830                                                       | 1.251                                          | 1.886                                          | 2.186                            |
| SCPS + 3.5% NaCl + 0.4%SVinh | 5.884                                         | 4.647                                                        | 2.868                                          | 5.614                                          | 6.507                            |

Table 8  Impedance parameters for steel rebar in concrete with and without inhibitors at 1st day and 180th day of marine exposure

| Solution resistance ($R_s$) in $\Omega$.cm$^2 \times 10^4$ | Charge transfer resistance ($R_{ct}$) in $\Omega$.cm$^2 \times 10^4$ | Double layer capacitance ($C_{dl}$) in F/cm$^2 \times 10^{-5}$ | Corrosion current density ($I_{corr}$) in mA/cm$^2 \times 10^{-3}$ | Corrosion rate in mmpy $\times 10^{-2}$ |
|----------------|----------------|-----------------------------|-----------------------------|-----------------------------|
| 1st day | 180th day | 1st day | 180th day | 1st day | 180th day | 1st day | 180th day | 1st day | 180th day | 1st day | 180th day |
| Control concrete | 1.519 | 0.233 | 4.417 | 0.101 | 4.952 | 63.97 | 0.591 | 25.83 | 0.685 | 29.94 |
| Glucosamine (GSinh) | 2.932 | 0.340 | 8.345 | 0.469 | 2.392 | 13.78 | 0.313 | 5.568 | 0.362 | 6.454 |
| Concrete with inhibitor GSinh | 2.350 | 0.288 | 18.570 | 1.013 | 4.870 | 4.090 | 0.141 | 2.575 | 0.163 | 2.985 |
| Stevioside (SVinh) | 2.005 | 0.340 | 8.345 | 0.469 | 2.392 | 13.78 | 0.313 | 5.568 | 0.362 | 6.454 |
| Concrete with inhibitor SVinh | 1.250 | 0.288 | 18.570 | 1.013 | 4.870 | 4.090 | 0.141 | 2.575 | 0.163 | 2.985 |

plot indicates the diameter of the semicircle which is greater for concrete with inhibitor SVinh in comparison with concrete with and without inhibitor GSinh. This is due to the higher adsorption capacity of inhibitor SVinh molecule on the steel reinforcement and thereby increases the thickness of the protecting layer at steel concrete interface [48–50]. Moreover, the order of reduction in capacitance double-layer values follows control concrete, concrete with inhibitors GSinh and SVinh to confirm the increase in thickness of protective layer. Similar trend of behaviour is observed in concrete without, with inhibitors GSinh and SVinh at the end of 180-day marine exposure, and their values are reported in Table 8. It indicates the characteristic feature of the inhibitor SVinh does not change with passage of time.

3.1.5 Inhibition Efficiency by Various Electrochemical Techniques

Figure 10 and Table 9 show the inhibition efficiency of inhibitors GSinh and SVinh by various techniques in SCPS and concrete medium. It is evident that inhibition efficiency of inhibitor SVinh admixed in concrete is better compared
with corrosion inhibitor $G_{S inh}$. The inhibition efficiency of inhibitor $S_{V inh}$ admixed in concrete during initial and at the end of 180 days in sea water exposure by potentiodynamic, linear polarization resistance and electrochemical impedance spectroscopy is 80.13%, 75.61%, 76.20% and 93.34%, 86.58% and 90.03%, respectively. It is evident from Table 9 that the corrosion inhibitor $S_{V inh}$ exhibited good corrosion protection at the end of 180 days of marine exposure.

### 3.1.6 Gravimetric Weight Loss and Extended Service Life by Inhibitor Addition

The role of inhibitor admixed in concrete to improve the service life of rebar embedded in concrete is examined with service life prediction model [51]. The time taken for cracking of concrete due to corrosion of embedded rebar is obtained from Eq. 4 [51].

$$T_{cr} = \left( \frac{7117.5 \cdot (D + 2\delta_0) \cdot (1 + \theta + \varphi)}{iE_{eff}} \right) \left( \frac{2 \cdot C \cdot f_{ct}}{D} + \frac{2\delta_0 E_{eff}}{(1 + \theta + \varphi)(D + 2\delta_0)} \right).$$

The effect of addition of corrosion inhibitors $G_{S inh}$ and $S_{V inh}$ into the concrete to improve the time taken for cracking is evaluated using the above equation, and the values are reported in Table 10. It indicates the addition of corrosion inhibitor extends the time taken for cracking. The addition of corrosion inhibitors $G_{S inh}$ and $S_{V inh}$ increases the time taken for corrosion-induced cracking by 2.72 and 20.4 times that of concrete without inhibitor, respectively. It also clearly indicates that the corrosion inhibitor $S_{V inh}$ performed better than $G_{S inh}$.

### 3.2 Hardened Properties of Concrete and Cement Mortar

Table 11 provides the compressive strength of concrete without inhibitor, with corrosion inhibitors $G_{S inh}$ and $S_{V inh}$ at the end of 7 days and 28 days. The compressive strength results confirm the addition of inhibitors $G_{S inh}$ and $S_{V inh}$ does not have any adverse effect on earlier strength and later strength development. It is observed from Table 11 that the compressive strength of concrete admixed with inhibitors $G_{S inh}$ and $S_{V inh}$ is higher than strength of concrete without inhibitor. Table 12 shows the physical properties of cement and mechanical properties of cement mortar with and without inhibitor. It is observed from Table 12 that the addition of inhibitor does not have adverse effect on the physical properties of cement, earlier strength and later strength development of cement mortar.
### Table 9 Comparison of inhibition efficiency by various techniques

| S. no. | System particulars | Electrochemical technique | EIS | LPR | TAFEL |
|--------|-------------------|----------------------------|-----|-----|-------|
|        |                   | Exposure period in days    | 1   | 180 | 1     |
| 1      | Inhibitor glucosamine in SCPS medium | – | 88.19 | – | 74.90 |
| 2      | Inhibitor Glucosamine in concrete medium | 29.05 | 73.36 | 79.49 | 60.35 |
| 3      | Inhibitor stevioside in SCPS medium | – | 89.73 | – | 77.09 |
| 4      | Inhibitor stevioside in concrete medium | 76.20 | 90.03 | 86.58 | 75.61 |

### Table 10 Gravimetric weight loss data and time taken for corrosion cracking of concrete with and without inhibitors

| System | Initial weight of specimen (gm) | Final weight of specimen (gm) | Mass loss (gm) | Corrosion rate (mmpy) × 10⁻⁴ | Corrosion current density (μA/cm²) | Time taken for cracking in days | Time taken for cracking (Hrs) |
|--------|---------------------------------|-------------------------------|----------------|--------------------------------|-----------------------------------|---------------------------------|------------------------------|
| Control specimen | 36.921                          | 36.146                        | 0.775          | 1.512                          | 6.483                             | 55.02                           | 1320.48                      |
| Concrete with inhibitor GS$_{inh}$ | 40.518                          | 40.234                        | 0.283          | 0.553                          | 2.370                             | 150.47                          | 3611.31                      |
| Concrete with inhibitor SV$_{inh}$ | 43.094                          | 43.056                        | 0.038          | 0.007                          | 0.317                             | 1122.99                         | 26,951.83                    |

### Table 11 Compressive strength of concrete with and without inhibitor

| S.No | System                  | Load in tonne | Compressive strength (N/mm²) |
|------|-------------------------|---------------|------------------------------|
|      |                         | 7th day       | 28th day                     |
| 1    | Control Concrete        | 42            | 18.31                        |
| 2    | 40                      | 17.44          | 25.29                        |
| 3    | 37                      | 16.13          | 22.24                        |
| 4    | Concrete with inhibitor GS$_{inh}$ | 49           | 21.36                        |
| 5    | 43                      | 18.75          | 29.21                        |
| 6    | 40                      | 17.44          | 25.29                        |
| 7    | Concrete with inhibitor SV$_{inh}$ | 36           | 15.70                        |
| 8    | 40                      | 17.44          | 22.67                        |
| 9    | 37                      | 16.13          | 21.80                        |

### 4 Conclusions

In the present investigation, the performance of green corrosion inhibitors in SCPS and concrete medium by electrochemical and non-electrochemical techniques is evaluated. The mechanical and physical properties of cement and concrete with and without green corrosion inhibitor are studied.

The following conclusions were drawn from the present investigation:

- OCP measurements revealed that the inhibitors GS$_{inh}$ and SV$_{inh}$ in both simulated concrete pore solution and concrete medium showed a passive behaviour when compared with control system and control concrete.
- Potentiodynamic polarization studies in SCPS medium suggest optimizing 0.3% concentration of inhibitors GS$_{inh}$ and SV$_{inh}$ to be admixed into concrete. The study also revealed that the inhibition efficiency of inhibitors GS$_{inh}$ and SV$_{inh}$ admixed in concrete increases by 12.85% and...
Table 12 Test results on physical properties of cement and mechanical properties of cement mortar with and without inhibitor

| Sample particulars | Sample no | Consistency (%) | Initial setting time (min) | Final setting time (min) | Compressive strength (N/mm²) | Finess (%) | Soundness (mm) |
|--------------------|-----------|-----------------|---------------------------|--------------------------|-----------------------------|------------|---------------|
|                    |           |                 |                           |                          | 3rd day                     | 7th day    | 28th day      |
| OPC 43 Grade Cement| 1         | 31.0            | 88                        | 144                      | 31                          | 37.5       | 44.5          | 2             | 1.50          |
|                    | 2         | 31.5            | 107                       | 324                      | 25                          | 37         | 43.0          | 1             | 1.83          |
|                    | 3         | 31.0            | 92                        | 157                      | 31                          | 37.5       | 44.5          | 2             | 1.50          |
| OPC 43 Grade Cement with Inhibitor GSinh | 4         | 31.5            | 117                       | 361                      | 24                          | 30.5       | 59.5          | 1             | 1.83          |
|                    | 5         | 31.5            | 107                       | 324                      | 25                          | 37         | 43.0          | 1             | 1.83          |
|                    | 6         | 32.0            | 99                        | 330                      | 26                          | 33         | 44.0          | 2             | 1.00          |
| OPC 43 Grade Cement with Inhibitor SVinh | 7         | 32.5            | 93                        | 242                      | 27                          | 44.5       | 49.5          | 2             | 1.42          |
|                    | 8         | 32.0            | 113                       | 292                      | 26                          | 39         | 47.5          | 2             | 1.83          |
|                    | 9         | 31.0            | 88                        | 144                      | 31                          | 37.5       | 44.5          | 2             | 1.50          |

16.48% in comparison with initial periods of marine exposure. Moreover, the inhibition efficiency of concrete admixed with inhibitor SVinh is 23.02% greater than other concrete.

- LPR measurements showed higher polarization resistance values for concrete admixed with inhibitor SVinh both during initial days and end of 180-day marine exposure.
- Impedance data reveal that higher Rct value for concrete admixed with corrosion inhibitor SVinh is due to higher adsorption capacity of inhibitor SVinh molecule on steel reinforcement and thereby increases the thickness of protective layer at steel concrete interface.

- Comparison of inhibition efficiency of inhibitors GSinh and SVinh admixed in concrete by various techniques indicated that concrete admixed with inhibitor SVinh shows higher inhibition efficiency at the end of 180 days of marine exposure. The inhibition efficiency of concrete admixed with corrosion inhibitor SVinh by potentiodynamic, linear polarization resistance and electrochemical impedance spectroscopy is 93.34%, 86.58% and 90.03%, respectively.
- The addition of corrosion inhibitor SVinh in concrete increases the time taken for concrete cracking due to corrosion by 20.4 times than that of concrete without inhibitor.
- The inclusion of inhibitors GSinh and SVinh in concrete and cement does not show any adverse effect on strength of concrete and cement. The physical properties of cement also were not affected.

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