Corrosion inhibition of green tea extract on steel reinforcing bar embedded in mortar

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Abstract. Polar atoms and π–electron bonds in molecular structures of natural antioxidants make the antioxidants potential mixed-type corrosion inhibitors and ‘green’ alternatives to commercial inorganic corrosion inhibitors. In this paper, the corrosion inhibition efficiency (IE) of green tea extract (GT), being one of the richest sources of natural antioxidants, was compared against commercial calcium nitrite corrosion inhibitor (CI) on steel reinforcing bars (rebars) embedded in mortar. Rebar corrosion was accelerated with impressed current and cyclic wetting-drying, and corrosion development was measured with linear polarization resistance. The IE of CI and GT were compared at same inhibitor volume. Results show that GT had a significantly higher IE than CI (75-80% vs. 14-24%), due to a reduction in iron oxidation rate (βa) and an increase in rebar resistance to polarization potential/current (Rp). Meanwhile, oxygen reduction rate (βc) and rebar open circuit potential (OCP) did not change significantly. The changes in βa and βc with an insignificant change in OCP suggest that GT was a mixed-type corrosion inhibitor, which formed a protective layer on rebar surface and increased Rp of the rebars.

1. Introduction

Corrosion inhibitors are a cost-effective, simple, and practical method to protect steel reinforcing bar (rebar) from corrosion [1]. Therefore, they are a popular alternative to cathodic protection (CP) and stainless steel rebar (SS), which are the most effective corrosion protection methods today [2]. Nonetheless, the current most effective commercial corrosion inhibitors are based on inorganic compounds, with calcium nitrite corrosion inhibitor (CI) being the most established commercially for concrete [3]. As pointed by several authors, use of inorganic compounds arises concerns due to potential health and environment hazards [4-8], Hence, study on less-toxic and more environmentally-friendly (‘green’) organic corrosion inhibitors is encouraged [4-8]. Nevertheless, there are only limited studies on the corrosion inhibition efficiency (IE) of green inhibitors in alkaline pH [5, 7, 9-11]. Thus, this study proposes an investigation on IE of natural antioxidants from green tea at alkaline pH of concrete.

Natural antioxidants are organic compounds which are rich in polar atoms and π–electron bonds [12], making the antioxidants potential mixed-type corrosion inhibitors [9]. Green tea is one of the richest sources of natural antioxidants, and contains potent antioxidants from the flavan-3-ol class [13]. Given the high antioxidant content and potential to be a mixed-type corrosion inhibitor, green tea was chosen as a source of natural antioxidants in this study. It is believed that should green tea fail to prevent rebar corrosion, other sources of natural antioxidants would also likely fail. The green tea was used as a green
tea extract (GT), and was used as an admixed corrosion inhibitor similar to CI, on steel rebar embedded in mortar specimens (i.e. reinforced mortar specimens).

The corrosion inhibition efficiency (IE) of GT was compared with CI, which was incorporated as a positive control. To author’s knowledge, there has been neither a study on IE of green tea in alkaline pH of concrete nor comparison on IE of CI and GT. Both CI and GT were used as liquid corrosion inhibitors, and doses of GT were kept similar to CI doses to prevent an arbitrary selection of GT doses [5, 7, 10, 11, 14] and to allow an indirect adjustment of GT doses based on concentration of aggressive species (i.e. chloride concentration).

2. Experimental studies

2.1 Materials and sample preparation

2.1.1 Preparation of mortar mixture.
A mortar mixture was prepared with Portland Cement CEM II/B-S 42.5N and fine aggregate according to mix design shown in Table 1. During casting of specimens admixed with CI and GT, additional water contributed to mortar mixture by various doses of inhibitors was taken into account, to maintain a consistent water to cement ratio among control, CI, and GT specimens.

Table 1: Constituents of mortar mixture

| Constituent  | Proportion (kg/m³) |
|--------------|--------------------|
| Cement       | 420.00             |
| Water        | 226.51             |
| Fine aggregate | 1084.28           |

Water/cement = 0.54

Dose of CI is commonly expressed as a ratio of chloride concentration to nitrite concentration ([Cl⁻] / [NO₂⁻]) [10]. For an effective IE of CI, manufacturers recommend a range of [Cl⁻] / [NO₂⁻] at 1.2-1.5. Thus, in this study, CI doses were selected at [Cl⁻] / [NO₂⁻] of 0.9, 1.2, 1.5, and 1.8 to encompass the recommended doses, as well as a wider range of doses (Table 2). The ratio at 0.9 was the highest ratio recommended by manufacturer for exposure to an aggressive environment.

The GT was added at same volume with CI (Table 2) to prevent an arbitrary selection of GT doses [14, 15], and 3.5% sodium chloride (NaCl) solution was used as an aggressive environment to simulate seawater. The GT was produced by ultrasonic-assisted extraction of green tea leaves with hot water at 10% (w/w) for 30 minutes, and large particles were removed from the aqueous extract by filtration using a commercial tea strainer. Upon extraction and filtration, concentration of GT was determined by oven-drying method at 100°C, to calculate water contribution of GT to mortar mixture [16]. The concentration of GT ranged from 2.0-3.0% (w/w). For easier results discussion, mortar specimens admixed with different doses of GT and CI were abbreviated according to Table 3.

2.1.2 Preparation of steel rebars.
Steel rebars of 12 mm diameter and 130 mm length were cut from commercial carbon steel rebars [10]. Prior to use, the rebars were sandblasted, degreased with acetone, cleaned with ultra-pure water, and wiped dry [17]. The exterior surface of rebars were painted with high performance epoxy paint [9], except at height of 50 mm (in the middle) and 25 mm (at the top), which remained unpainted for exposure to NaCl solution and electrical connection respectively [18]. The painted steel rebars and alignment at the centre of PVC pipe molds are shown in Figure 1.
Table 2: Volume of calcium nitrite corrosion inhibitor (CI) and the corresponding ratios of chloride concentration to nitrite concentration ([Cl\(^-\)] / [NO\(_2\)-])

| Chloride to nitrite ratio ([Cl\(^-\)] / [NO\(_2\)-]) | Volume of CI (L/m\(^3\) concrete) |
|---------------------------------------------------|-----------------------------------|
| 0.9                                               | 40.14                             |
| 1.2                                               | 30.11                             |
| 1.5                                               | 24.10                             |
| 1.8                                               | 20.08                             |

*The density of CI was 1.22 kg/L and CI concentration was 30% (w/w), being composed of calcium nitrite as the active constituent. The density and concentration of CI were obtained from specification sheet of product used. The corrosive environment was 3.5% (w/v) sodium chloride solution and concrete density was 2,350 kg/m\(^3\).*

Table 3: Nomenclatures of reinforced mortar specimens admixed with different volume of calcium nitrite corrosion inhibitor (CI) or green tea extract (GT)

| Specimen nomenclature | Corrosion inhibitor | Chloride to nitrite ratio ([Cl\(^-\)] / [NO\(_2\)-]) | Volume of inhibitor (L/m\(^3\) concrete) |
|-----------------------|---------------------|------------------------------------------------------|----------------------------------------|
| CI 0.9                | Calcium nitrite     | 0.9                                                  | 40.14                                  |
| CI 1.2                | 1.2                 |                                                      | 30.11                                  |
| CI 1.5                | corrosion inhibitor | 1.5                                                  | 24.10                                  |
| CI 1.8                | 1.8                 |                                                      | 20.08                                  |
| GT 0.9                | Green tea extract   | 0.9                                                  | 40.14                                  |
| GT 1.2                | 1.2                 |                                                      | 30.11                                  |
| GT 1.5                | 1.5                 |                                                      | 24.10                                  |
| GT 1.8                | 1.8                 |                                                      | 20.08                                  |

*Doses of CI are expressed as the ratios of chloride concentration to nitrite concentration ([Cl\(^-\)] / [NO\(_2\)-]), and doses of GT were kept similar to the CI doses. Therefore, GT doses were expressed similarly to expression of CI doses, and the GT was used at the same volume as the CI. The corresponding [Cl\(^-\)] / [NO\(_2\)-] are represented by values after CI and GT annotation.*

2.2 Methodologies

2.2.1 Casting reinforced mortar specimens.

The procedures for mixing and casting reinforced mortar specimens were adopted and modified from Gencel et al. [19] and Karahan and Atis [20] using a laboratory-scale mixer for mortar mixing. Upon mixing, the fresh mortar mixture was filled into PVC pipe molds as shown in Figure 1 in a three-equal layer, with each layer being compacted using a vibrating table for 25 seconds. Three control specimens and three specimens for each dose of CI and GT were prepared. After 24 hours, the hardened mortar specimens were de-molded and cured in saturated limewater for 28 days [20].
2.2.2 Accelerated corrosion.
After 28-day curing, reinforced mortar specimens were subjected to accelerated corrosion, using a combination of impressed current and cyclic wetting-drying exposure. The current density was 100 µA/cm² [22], and the cyclic wetting and drying periods were 4 days wetting and 3 days drying respectively [23]. The setup for accelerated corrosion is shown in Figure 2 and was performed at laboratory temperature (26.4± 1.7°C, 70.3± 7.5% relative humidity). The anode was the steel rebar embedded in mortar and the cathode was an AISI 304 stainless steel plate. On the other hand, the electrolyte was the NaCl solution [24]. The specimens were connected in series to subject all specimens to the same applied current [25]. Since mortar pores dry out during the drying period, mortar resistivity to current flow increases, which increases total potential of connected samples exceeding maximum potential of power supply. Therefore, the impressed current was only applied during the wetting period, and the specimens were allowed to corrode naturally during dry period [26]. The accelerated corrosion study was conducted until the 12th wetting-drying cycle [27].

2.2.3 Corrosion measurements.
Corrosion development, as represented by corrosion kinetic parameters, were measured with linear polarization resistance (LPR) technique at laboratory temperature (26.4± 1.7°C, 70.3± 7.5% relative humidity). The measurement was performed with a Metrohm Autolab potentiostat (model M204) employing a three-electrode setup [27]. The working electrode (WE) was the steel rebar embedded in mortar, and the reference electrode (RE) and counter electrode (CE) were saturated calomel electrode (SCE) and AISI 304 stainless steel plate respectively. The corrosion measurement was performed at the end of a wetting-drying period and prior to start of a new period. Thus, the electrical connection between RE and WE was established using a sponge wetted with diluted detergent [28]. Prior to LPR measurement, open circuit potential (OCP) of the rebar was measured. When the OCP had reached a stable value (fluctuation of less than 10 mV) after 10 minutes, OCP was determined and the rebar was polarized with a scan potential of ±20 mV from OCP and a scan rate of 0.166 mV/s [9]. The following corrosion kinetic parameters: anodic and cathodic slope, corrosion rate, and polarization resistance; were

Figure 1: Setup of reinforced mortar samples (Adapted and modified from Alghamdi& Ahmad [21]).
obtained by analyzing the resulted plots using Nova 1.11 software. The obtained corrosion rate (CR) values were used to calculate the IE of CI and GT, according to Equation below.

\[
\%\ IE = \frac{CR\ without\ inhibitor - CR\ with\ inhibitor}{CR\ without\ inhibitor} \times 100\%
\]

Figure 2: Setup of accelerated corrosion using a combination of impressed current and cyclic wetting-drying. The specimens were removed from sodium chloride solution during drying period (Adapted and modified from Guneyisi and Gesoglu [24] and Fayala et al. [27]).

2.2.4 Statistical analyses.
Results on corrosion kinetic parameters were expressed as mean ± standard deviation from three specimens, and were presented as figures with error bars.

3. Results and discussion
The following sections present the results on measurement of corrosion kinetic parameters.

3.1 Effect of CI and GT on corrosion rate
The corrosion rate of CI and GT specimens, and the corresponding IE of CI and GT are shown in figure 5 and 6 respectively. Figure 5 shows that GT specimens had a significantly lower corrosion rate than control and CI specimens. Accordingly, figure 6 shows that GT produced an overall significantly higher IE than CI, which ranged from 75-80% vs. 14-24% at the end of corrosion test. The same figure also shows that there was a sudden decrease in IE of CI at second wetting-drying cycle, due to a sudden increase in corrosion rate of CI specimens (figure 5), before a stabilization (starting from third cycle). This sudden increase in CR indicated a corrosion initiation. Hence, as an admixed corrosion inhibitor, GT was a more effective inhibitor than CI, since the sudden increase in corrosion rate of CI specimens occurred earlier (at second wetting-drying cycle) than did the increase of GT specimens. GT was able to suppress a sudden increase in corrosion rate of GT specimens, and the specimens consistently showed a significantly lower corrosion rate than control and CI specimens, until the end of accelerated corrosion test.
3.2 Effect of CI and GT on anodic slope
Figure 7 shows the effect of CI and GT on anodic slope, which represents the rate of iron oxidation during rebar corrosion [29]. The figure shows that GT specimens had a significantly lower anodic slope than control and CI specimens, even though the reduction diminished after the sixth wetting-drying cycle. On the other hand, figure 3 shows a good correlation between anodic slope and corrosion rate, suggesting that the reduction in anodic slope was a contributing factor to the lower corrosion rate of GT specimens [29].

![Plot of anodic slope vs. corrosion rate of control reinforced mortar specimens and reinforced mortar specimens admixed with calcium nitrite corrosion inhibitor (CI) or green tea extract (GT) at the same inhibitor volume.](image)

3.3 Effect of CI and GT on cathodic slope
The effect of CI and GT on cathodic slope are presented in figure 8. Cathodic slope indicates the rate of oxygen reduction during rebar corrosion [29]. As shown in figure 8, GT specimens showed a lower cathodic slope than control and CI specimens particularly during the first seven wetting-drying cycles. However, in overall there was no significant difference in cathodic slope among control, CI, and GT specimens.

3.4 Effect of CI and GT on polarization resistance
Figure 9 shows the effect of CI and GT on polarization resistance. Polarization resistance indicates the rebar resistance to external potential/current which may change the electrochemical state of the rebar. As shown in figure 9, GT specimens had an overall significantly higher polarization resistance than control and CI specimens. The very strong correlation between corrosion rate and polarization resistance shown in Figure 4, further suggests that the increase in polarization resistance was another factor than reduction in anodic slope, which contributed to the lower corrosion rate of GT specimens. In fact, as shown in figure 9, the increase in polarization resistance was a more prominent factor than the reduction in anodic slope, as GT specimens showed a significantly higher polarization resistance than control and CI specimens until the end of corrosion test. On the other hand, the reduction in anodic slope was reduced after mid-way through the corrosion test (figure 7). The higher polarization resistance is often associated with the formation of a protective film on rebar surface [17, 30, 31].
3.5 Effect of CI and GT on open circuit potential
The effect of CI and GT on open circuit potential are presented in figure 10. Open circuit potential indicates the rebar potential in the absence of interference from external potential [9]. Figure 10 shows the absence of significant difference in open circuit potential among control, CI, and GT specimens.

3.6 Overall effect of CI and GT on corrosion kinetic parameters
GT specimens showed a significantly lower corrosion rate than control and CI specimens, which was contributed by the effect of GT which significantly reduced anodic slope, reduced cathodic slope, and significantly increased polarization resistance without significantly changing the open circuit potential. These changes indicate that GT behaved as a mixed-type corrosion inhibitor [32].

4. Conclusion
Results showed that GT specimens had a significantly lower corrosion rate (CR) than control and CI specimens, which leads to a significantly higher IE of GT than CI (75-80% vs. 14-24% after the last wetting-drying cycle). This higher IE of GT was contributed by a reduction in anodic reaction rate of iron oxidation (βa) and an increase in rebar resistance to external polarization potential/current (Rp). Meanwhile, GT did not significantly change cathodic reaction rate of oxygen reduction (βc) and open circuit potential (OCP) of the rebars. The changes in βa and βc without a significant change in OCP indicated that GT behaved as a mixed-type corrosion inhibitor, which inhibited rebar corrosion by forming a protective layer on rebar surface and increased Rp of the rebars. In fact, the increase in Rp was the most prominent among the aforementioned factors, as GT specimens showed a higher Rp than CI specimens until the end of corrosion test (191-221 vs. 56-64 Ω).
Figure 5: Corrosion rate of control reinforced mortar specimens and reinforced mortar specimens admixed with different doses of calcium nitrite corrosion inhibitor (CI) or green tea extract (GT). Doses of CI are expressed as the ratios of chloride concentration to nitrite concentration ([Cl⁻] / [NO₂⁻]), and doses of GT were kept similar to the CI doses. Therefore, GT doses were expressed similarly to expression of CI doses. Both CI and GT had the same volume and the chloride-to-nitrite ratios are indicated by the values after CI and GT annotations. A lower chloride-to-nitrite ratio indicates a higher inhibitor dose. The presented corrosion rate were the average values from three replicates of samples and the error bars represented the standard deviation in corrosion rate from the three replicates.
Figure 6: Corrosion inhibition efficiency of calcium nitrite corrosion inhibitor (CI) and green tea extract (GT) at different doses. Doses of CI are expressed as the ratios of chloride concentration to nitrite concentration ([Cl⁻] / [NO₂⁻]), and doses of GT were kept similar to the CI doses. Therefore, GT doses were expressed similarly to expression of CI dose. Both CI and GT had the same volume and the chloride-to-nitrite ratios are indicated by the values after CI and GT annotations. A lower chloride-to-nitrite ratio indicates a higher inhibitor dose. The presented corrosion inhibition efficiency were the average values from three replicates of samples and the error bars represented the standard deviation in corrosion inhibition efficiency from the three replicates.
Figure 7: Anodic slope of control reinforced mortar specimens and reinforced mortar specimens admixed with different doses of calcium nitrite corrosion inhibitor (CI) or green tea extract (GT). Doses of CI are expressed as the ratios of chloride concentration to nitrite concentration ([Cl⁻] / [NO₂⁻]), and doses of GT were kept similar to the CI doses. Therefore, GT doses were expressed similarly to expression of CI doses. Both CI and GT had the same volume and the chloride-to-nitrite ratios are indicated by the values after CI and GT annotations. A lower chloride-to-nitrite ratio indicates a higher inhibitor dose. The presented anodic slope were the average values from three replicates of samples and the error bars represent the standard deviation in anodic slope from the three replicates.
Figure 8: Cathodic slope of control reinforced mortar specimens and reinforced mortar specimens admixed with different doses of calcium nitrite corrosion inhibitor (CI) or green tea extract (GT). Doses of CI are expressed as the ratios of chloride concentration to nitrite concentration ([Cl⁻] / [NO₂⁻]), and doses of GT were kept similar to the CI doses. Therefore, GT doses were expressed similarly to expression of CI doses. Both CI and GT had the same volume and the chloride-to-nitrite ratios are indicated by the values after CI and GT annotations. A lower chloride-to-nitrite ratio indicates a higher inhibitor dose. The presented cathodic slope were the average values from three replicates of samples and the error bars represent the standard deviation in cathodic slope from the three replicates.
Figure 9: Polarization resistance of control reinforced mortar specimens and reinforced mortar specimens admixed with different doses of calcium nitrite corrosion inhibitor (CI) or green tea extract (GT). Doses of CI are expressed as the ratios of chloride concentration to nitrite concentration ([Cl\(^-\)] / [NO\(_2\)-]), and doses of GT were kept similar to CI doses. Therefore, GT doses were expressed similarly to expression of CI doses. Both CI and GT had the same volume and the chloride-to-nitrite ratios were indicated by the values after CI and GT annotations. A lower chloride-to-nitrite ratio indicates a higher inhibitor dose. The presented polarization resistance were the average values from three replicates of samples and the error bars represented the standard deviation in polarization resistance from the three replicates.
Figure 10: Open circuit potential of control reinforced mortar specimens and reinforced mortar specimens admixed with different doses of calcium nitrite corrosion inhibitor (CI) or green tea extract (GT). Doses of CI are expressed as the ratios of chloride concentration to nitrite concentration ([Cl⁻] / [NO₂⁻]), and doses of GT were kept similar to the CI doses. Therefore, GT doses were expressed similarly to expression of CI doses. Both CI and GT had the same volume and the chloride-to-nitrite ratios were indicated by the values after CI and GT annotations. A lower chloride-to-nitrite ratio indicates a higher inhibitor dose. The presented open circuit potential were the average values from three replicates of samples and the error bars represented the standard deviation in open circuit potential from the three replicates.
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