Paper No.

5636

NACE INTERNATIONAL CORROSION 2015 CONFERENCE & EXPO

Performance of *Rhizophora mangle* L. leaf-extract and sodium dichromate synergies on steel-reinforcement corrosion in 0.5 M H_2SO_4-immersed concrete

Joshua Olusegun OKENIYI
Mechanical Engineering Department, Covenant University, Ota, Nigeria
Ota 112001, Ogun State
Nigeria

Cleophas Akintoye LOTO
Mechanical Engineering Department, Covenant University, Ota, Nigeria
Ota 112001, Ogun State
Nigeria

Abimbola Patricia Idowu POPOOLA
Chemical, Metallurgical and Materials Engineering Department, Tshwane University of Technology, Pretoria
Pretoria 0001
South Africa

Olugbenga Adeshola OMOTOSHO
Mechanical Engineering Department, Covenant University, Ota, Nigeria
Ota 112001, Ogun State
Nigeria

ABSTRACT

This paper investigates performance of different synergistic combinations of *Rhizophora mangle* L leaf-extract and sodium dichromate ($Na_2Cr_2O_7$) admixtures on the corrosion of steel-reinforcement in 0.5 M H_2SO_4-immersed concrete. Steel-reinforcement corrosion, in concrete slabs partially-immersed in the microbial/industrial simulating test-environment, were assessed using non-destructive electrochemical measurements of corrosion rate (CR) and open circuit potential (OCP) . Probability distribution function (pdf) analyses, as prescribed by ASTM G16-95 R04, of the scatter of corrosion test-data showed that while datasets of OCP distributed like the Normal, the Gumbel and Weibull pdf's, the datasets of CR were best fitted by the Weibull pdf. Results identified 6 g *Rhizophora mangle* L leaf-extract + 2 g $Na_2Cr_2O_7$ synergistic admixture with both optimal inhibition efficiency, η = 90.12%, and synergistic parameter that indicated excellent synergistic interaction of the plant-extract with $Na_2Cr_2O_7$ chemical. That this synergistic admixture out-performed the also effective $Na_2Cr_2O_7$ admixtures in the study suggests suitability of *Rhizophora mangle* L leaf-extract as eco-friendly replacement of toxic $Na_2Cr_2O_7$ inhibitor in H_2SO_4-immersed steel-reinforced concrete. This is potent with the advantage of reducing environmental effect from lower $Na_2Cr_2O_4$ usage for controlling corrosion in steel-reinforced concretes for the microbial/industrial environment.

Key words: *Rhizophora mangle* L leaf-extract, sodium dichromate, concrete steel-rebar, microbial/industrial simulating environment, inhibition efficiency, synergistic parameter

INTRODUCTION

Steel-reinforced concrete is a preferred construction material globally for building structures and infrastructures because of its economical advantage of relatively low cost and technological ease of manufacture.[1-4] However, corrosion of steel-reinforcement (steel-rebar) in concrete, by aggressive agents in the service-environment of the reinforced concrete structure, affects durability, induces costly repair/maintenance and generates concerns of safety risks and loss of properties.[5-7] Aggressive agents inducing concrete steel-rebar corrosion include sulfuric acid environments from microbial activities of sulfate reducing and sulfur oxidizing bacteria in sewage/underground or from acid rain in industrial service-environments.[2,8-10] By-products of the reaction from these with concrete include gypsum and ettringite that are expansive within the concrete and induce loss in concrete material and exposure of the rebar to corrosion attacks and degradation.

Among many methods employed for protecting steel-reinforced concrete from corrosion degradation in acidic environments, the use of corrosion inhibitors has been identified as a simple, less costly and effective technique for mitigating steel-rebar corrosion.[5,8-11] Inhibitors that had been employed in studies[5,8,11] as reference for mitigating acidic corrosion attacks on steel-reinforced concrete include compounds of chromates and nitrites but the use of these is being restricted in many countries due to their toxicity and hazardousness to the environmental ecosystem.[12-14] These have provoked research interests for non-toxic and environmentally-friendly alternative that could be suitable for replacing the toxic chemicals as inhibitors of steel-rebar corrosion in aggressive environments.[14]

Toxicological evaluation of the active composition of extract from *Rhizophora mangle* L in study[15] showed that the extract from this natural plant exhibited no sign of toxicity. In another reported work,[16] it was shown that mangrove extracts contained flavonoids and tannins that exhibited positive potential as inhibitors of steel corrosion in 0.5 M HCl medium in that study. These engendered motivation in this paper for studying effects of different concentration models of the leaf-extract from *Rhizophora mangle* L and sodium dichromate ($Na_2Cr_2O_7$) chemical inhibitor at mitigating corrosion in steel-reinforced concrete in the sulfuric acid medium. This paper, therefore, investigates the performance of *Rhizophora mangle* L leaf-extract and sodium dichromate synergies, compared with individual concentration effects, on steel-reinforcement corrosion in concrete immersed in 0.5 M H_2SO_4, for simulating microbial/industrial environments.

EXPERIMENTAL PROCEDURE

Experimental Materials

Fresh collection of the leaves of *Rhizophora mangle* L. (*R. mangle* L.) Rhizophoraceae were obtained from Ehin-more, Ilaje eseodo, in Ondo State, Nigeria. These leaves were identified at the Forestry Herbarium Ibadan (FHI), Nigeria, where a sample was deposited with FHI. No. 109501. The leaves were dried under cover maintained at 20 °C and then blended to powder. Extracts (by methanol at 40 °C) from this were then employed as individual and synergistic admixture designs with sodium dichromate in 100 mm × 100 mm × 200 mm samples of steel reinforced concrete slabs. These admixture designs entailed the use of 2 g, 4 g and 6 g individual admixtures of the plant extract and of the sodium dichromate chemical as well as the synergistic combinations of these into 2 g *R. mangle* L + 6 g $Na_2Cr_2O_7$, 4 g *R. mangle* L + 4 g $Na_2Cr_2O_7$ and 6 g *R. mangle* L + 2 g $Na_2Cr_2O_7$. Performance from steel-reinforced concrete samples admixed with these admixture models were then compared with steel-reinforced concrete specimen without any of these admixtures (i.e. 0 g admixture) as the blank or control (Ctrl) specimen. The steel-reinforced concrete samples employed in this study totaled ten samples.

Diameter 12 mm reinforcing steel used for corrosion experiment in the study has the composition (%) of: 0.27 C, 0.40 Si, 0.78 Mn, 0.04 P, 0.04 S, 0.14 Cr, 0.11 Ni, 0.02 Mo, 0.24 Cu, 0.01 Co, 0.01 Nb, 0.01

Sn and the balance Fe. This was cut into 190 mm rods of specimens, for each of which surface preparation was maintained uniformly according to standard procedure prescribed in ASTM G109-99a.[17] Each of the rods has 150 mm of its length centrally embedded in the 100 mm × 100 mm × 200 mm concrete slab. Each of the sample of steel-reinforced concretes have the formulation 300.0 kg/m^3 cement, 890.6 kg/m^3 sand, 1106.3 kg/m^3 granite stones and 149.7 kg/m^3 water (i.e. w/c ratio = 0.499), and was cast as per standard procedure prescribed in ASTM C192/192M-02.[18] The remaining 40 mm protrusion of the steel-rebar, which served as connector for electrochemical test-measurements, was painted with glossy paint.

Experimental Setup

Each of the steel-reinforced concrete slabs was partially immersed in plastic bowls containing the 0.5 M H_2SO_4 test-medium that was made up to just below the embedded steel-rebar in the concrete, but without touching the rebar. Electrochemical measurements were then taken from each of the steel-reinforced samples in five days interval for 40 days, then in seven days interval for four weeks. These totaled 12-point measurements in 68 days. The electrochemical test-techniques employed in the study include:

- Open circuit potential, OCP, was measured versus $Cu/CuSO_4$ electrode (CSE) from Tinker & Rasor[†] using a high impedance multimeter according to ASTM C876-91 R99;[19] and

- Corrosion rate, CR, was measured using a 3-electrode LPR Data Logger from Metal Samples,[†] through direct instrument conversion to mpy, and which was connected to the concrete test-system as had been described in a previous study.[20]

Experimental Data Analyses

The scatter of corrosion test-data, from each of the test-variables of electrochemical measurements, were subjected to the probability distribution function (pdf) analyses of the Normal, the Gumbel and the Weibull distributions, as prescribed by ASTM G16-95 R04[21] and detailed in a reported study.[20] Compatibility of test-data fittings of each test-variable to each probability distribution were studied using the Kolmogorov-Smirnov goodness-of-fit test-statistics at α = 0.05 significant level.[22-23]

Inhibition Efficiency Estimation.

Mean values, μ, estimated from the probability distribution of best-fit for the scatter of CR datasets of the steel-reinforced concrete samples were employed for evaluating inhibition efficiency, η, as:[14,20,24-25]

$$\eta(\%) = \frac{\mu_{(\text{control concrete, pdf})} - \mu_{(\text{admixed concrete, pdf})}}{\mu_{(\text{control concrete, pdf})}} \times 100 \qquad (1)$$

Synergistic Parameter Evaluation.

Also, mean value estimates, μ, from the pdf of best-fit for the scatter of CR datasets of the steel-reinforced concrete samples with admixtures were used for evaluating synergistic parameter, S, as:[25-27]

[†] Trade name.

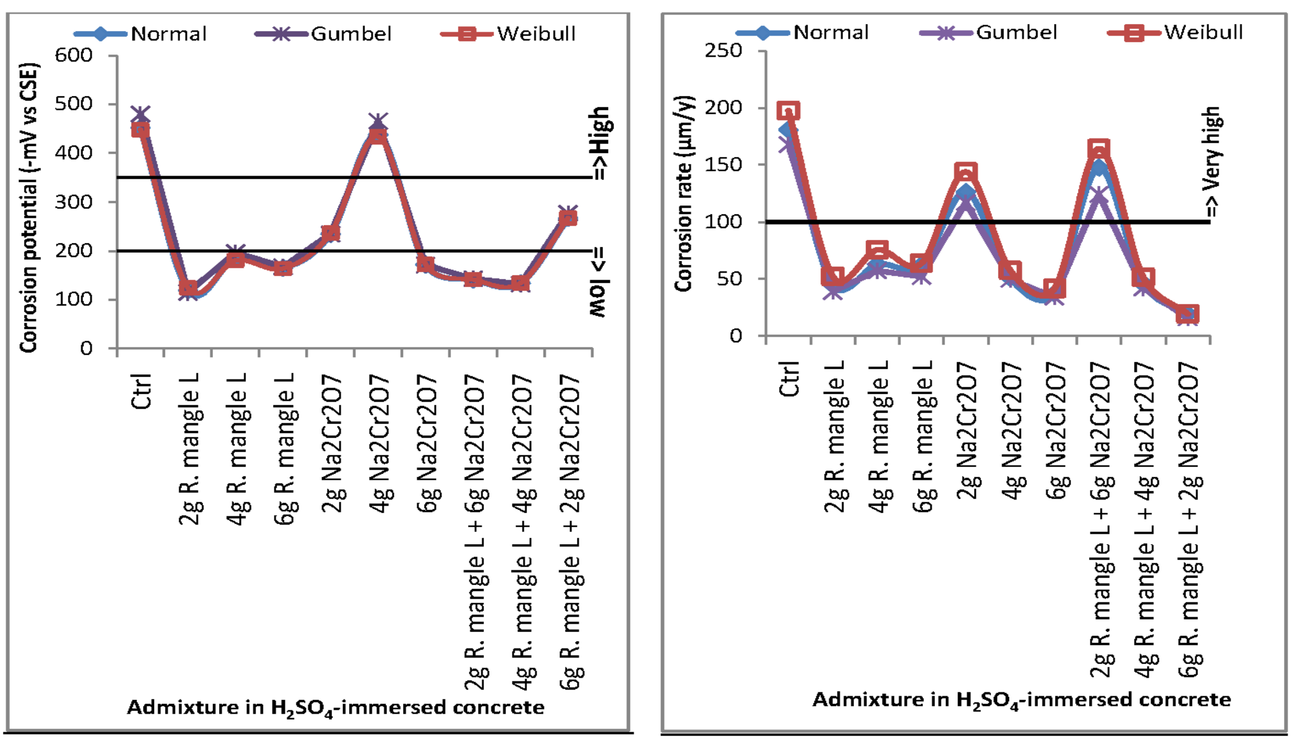
$$S = \frac{\mu_{(R.\ mangle\ \mathrm{L\ admixture})} \times \mu_{(\mathrm{Na_2Cr_2O_7\ admixture})}}{\mu_{(R.\ mangle\ \mathrm{L} + \mathrm{Na_2Cr_2O_7\ admixture})}} \tag{2}$$

By interpretations from studies, [25,28] the synergistic parameter indicates prevalent synergistic interaction between *R. mangle* L and $Na_2Cr_2O_7$ admixtures when $S > 1$, prevalent antagonistic interaction between both admixtures when $S < 1$, or lack of interaction between the two admixtures when S approaches 1.

RESULTS AND DISCUSSION

Probability Distribution Modeled Results

Figure 1 shows plots of the modeled results from the probability distribution function analyses of electrochemical test-measurements obtained from the steel-reinforced concrete samples. Linear plots are included in Figure 1(a), for interpreting modeled results of OCP as per ASTM C876-91 R99[19] and in Figure 1(b) for interpreting modeled results of CR according to studies.[29-30] It could be observed from the figure that the modeled results of probability distribution function analyses portrayed similar patterns among the different distribution functions employed for both corrosion test-variables of OCP, Figure 1(a), and CR, Figure 1(b). In spite of this, however, OCP modeled results were mostly over-predicted by the Gumbel pdf in Figure 1(a) while the CR modeled results were mostly over-predicted by the Weibull pdf in Figure 1(b). These discrepancies necessitate needs for ascertaining, by goodness-of-fit statistics, the pdf fitting best the scatter of test-data of corrosion test-variables for each steel-reinforced concrete samples. This best-fit pdf model could then be employed for detailing the prevailing corrosion condition in each sample.



(a) (b)

Figure 1: Modeled results of probability distribution function analyses of electrochemical test-measurements from steel-reinforced concrete samples (a) OCP (b) CR

Goodness-of-fit Test-results

Results of Kolmogorov-Smirnov goodness-of-fit test-statistics applied to the pdf models of the test-data of corrosion test-variables for each steel-reinforced concrete samples are presented in Figure 2. The figure also includes the linear plot of α = 0.05 significant level for direct interpretation of test-datasets that followed each of the probability distribution models of the corrosion test-variables. By this, it could be deduced from the figure that all test-datasets of OCP measured from all the steel-reinforced concrete samples studied distributed like the Normal, the Gumbel and the Weibull distribution functions. However, five test-datasets of CR, measured from the three steel-reinforced concrete admixed with *R. mangle* L leaf-extract and from two out of the three with synergistic admixtures, were not scattered like the Normal distribution. Also, the CR test-dataset measured from the steel-reinforced concrete admixed with 6 g *R. mangle* L did not scatter like the Gumbel distribution. In contrast, all the CR test-datasets from the steel-reinforced concrete samples in this study distributed like the Weibull distribution according to the Kolmogorov-Smirnov goodness-of-fit test-criteria at α = 0.05 significant level. This supports the use of the Weibull distribution model of corrosion rate for describing the corrosion conditions, which include inhibition effectiveness and synergistic interactions by the inhibitor admixtures, in the studied steel-reinforced concrete samples.

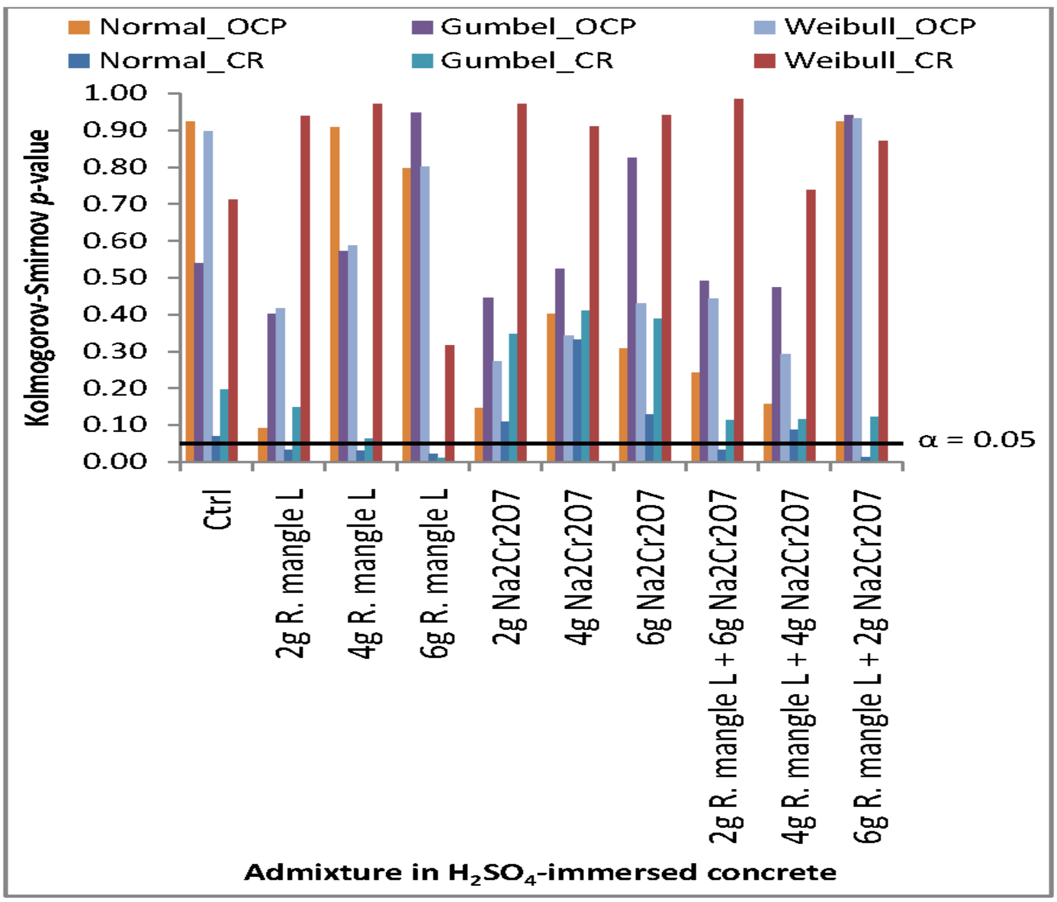


Figure 2: Kolmogorov-Smirnov goodness-of-fit test-results of probability distribution fittings of corrosion test-data

Modeled Results of Inhibition Effectiveness

Weibull mean models of the CR test-variable from the steel-reinforced concrete samples are applied using Equation 1 for inhibition efficiencies on steel-rebar corrosion by the *R. mangle* L and $Na_2Cr_2O_7$ admixtures. These results are presented in Figure 3 in ranking order of effectiveness performance. This figure shows that all the admixtures employed in the steel-reinforced concrete samples studied exhibited at least some inhibitions relative to the Ctrl sample that has no inhibitor admixture. This figure also shows that inhibition effectiveness increased with concentration of $Na_2Cr_2O_7$ alone. The 6 g $Na_2Cr_2O_7$ admixture, that exhibited inhibition efficiency η = 78.87%, out-performed the individual admixtures of $Na_2Cr_2O_7$ at lower concentrations as well as all three levels whereby *R. mangle* L had been used alone. This confirms that the chromate compound is an effective inhibitor of steel-rebar corrosion in the H_2SO_4-immersed steel-reinforced concrete samples.

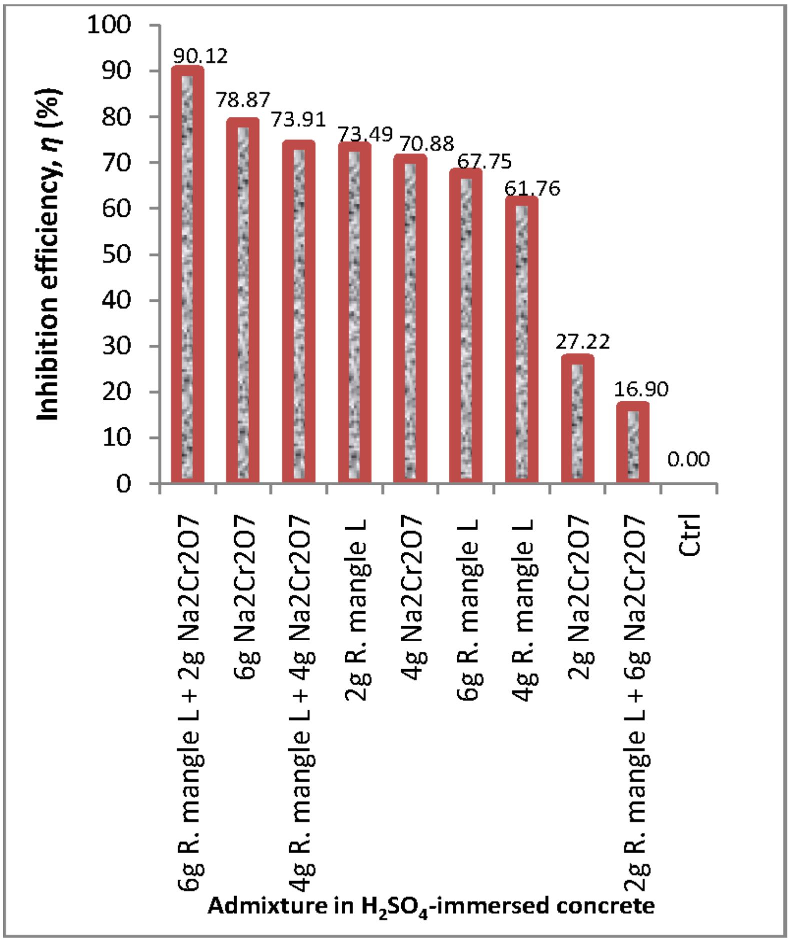


Figure 3: Ranking order of corrosion inhibition effectiveness of *R. mangle* L and $Na_2Cr_2O_7$ admixture models in steel-reinforced concrete

Only the admixture of 6 g *R. mangle* L + 2 g $Na_2Cr_2O_7$ was synergistic, exhibiting the optimal inhibition efficiency η = 90.12%. Figure 3 shows that this admixture exceeded the effectiveness of all three concentrations of $Na_2Cr_2O_7$ at inhibiting steel-rebar corrosion in the H_2SO_4-immersed concrete samples. This was in spite of the relatively low inhibition efficiency, η = 67.75%, exhibited by the 6 g *R. mangle* L admixture alone, and the much lower inhibition efficiency, η = 27.22%, exhibited by the use of

2 g $Na_2Cr_2O_7$ admixture alone. This inhibition effectiveness performance for the synergistic admixture of 6 g *R. mangle* L + 2 g $Na_2Cr_2O_7$ makes it possibly suitable for replacing the higher concentration of 6 g toxic $Na_2Cr_2O_7$ alone for inhibiting steel-rebar corrosion in the H_2SO_4-immersed concrete. This reduction in the required amount of sodium dichromate inhibitor is potent with the advantage of reduction in the portion of the environmentally-hazardous chromate compound, offset by the synergistic addition of the non-toxic and environmentally-friendly *R. mangle* L leaf-extract.

Synergistic Parameter Modeling

Figure 4 shows results of the synergistic parameter evaluations. A horizontal line is also plotted for S = 1 to aid in interpreting the prevalent synergistic interaction between the *R. mangle* L and the $Na_2Cr_2O_7$ admixture. From this, the admixture of 2 g *R. mangle* L + 6 g $Na_2Cr_2O_7$ exhibited an antagonistic interaction, $S = 0.07 < 1$. The increased amount of *R. mangle* L in an admixture of equal masses of 4 g each of $Na_2Cr_2O_7$ and *R. mangle* L resulted in the synergistic parameter of S = 0.43 that indicate increase towards S = 1, the threshold of lack of synergistic interaction. However, the further increase of the of the amount of admixed *R. mangle* L to 6 g and related reduction of the admixed $Na_2Cr_2O_7$ to 2 g resulted in the synergistic parameter $S = 2.38 > 1$, indicating a prevalent synergistic interaction in this case.

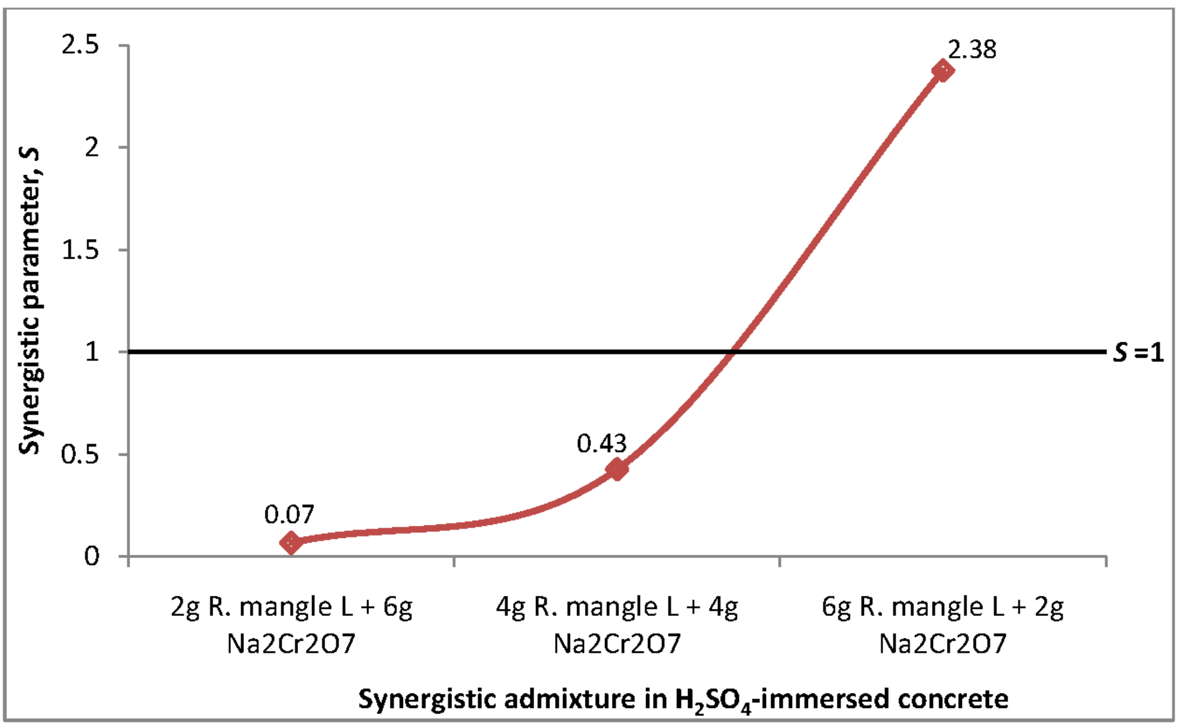


Figure 4: Synergistic parameter from *R. mangle* L and $Na_2Cr_2O_7$ admixtures in steel-reinforced concrete samples

The modeled results of inhibition efficiency, from Figure 3, corroborate the synergistic parameter models, in Figure 4. For instance, the antagonistic interaction (S = 0.07, which is less than unity) between the 2 g *R. mangle* L and the 6 g $Na_2Cr_2O_7$ admixtures resulted in the reduction of inhibition efficiency by the synergistic combination in comparison with the inhibition efficiencies by the individual admixtures. Also, the lack of interaction between the equal mass model of 4 g *R. mangle* L + 4 g $Na_2Cr_2O_7$ synergy, (S = 0.43, which indicates increase of S towards unity), resulted in a just slightly increased inhibition efficiency from the efficiencies of the individual admixtures. In contrast to these, the prevalent synergistic interaction between the 6 g *R. mangle* L (η = 67.75%) and the 2 g $Na_2Cr_2O_7$ (η = 27.22%) admixtures resulted in the highly increased inhibition efficiency to η = 90.12% by the synergistic 6 g *R. mangle* L + 2 g $Na_2Cr_2O_7$ synergistic admixture.

CONCLUSIONS

Although the well-known but toxic sodium dichromate chemical was confirmed as effective inhibitor of steel-rebar corrosion in H_2SO_4-immersed concrete, suitable amount of *R. mangle* L leaf-extract could be employed for reducing use of the toxic dichromate chemical and its consequent environmental effects. This study established that such suitable combination of *R. mangle* L leaf-extract with sodium dichromate, used as an admixture in H_2SO_4-immersed concrete, has potent synergy in out-performing the also highly effective but toxic sodium dichromate inhibitor alone. In this work, this synergistic combination takes the form of 6 g *R. mangle* L + 2 g $Na_2Cr_2O_7$ admixture in concrete. Careful choice of concentrations in such an admixture, informed by experiment, is required because unsuitable combinations of the plant-extract and the dichromate result not only in lack of synergistic interaction but also antagonistic interaction in some cases.

Future Work

That the combination of *R. mangle* L with $Na_2Cr_2O_7$ resulted in synergistic effect with highly effective performance at inhibiting concrete steel-reinforcement corrosion bare suggestion of prospects from further research for total replacement as inhibitor of the toxic chromate by this eco-friendly plant extract.

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