Int. J. Electrochem. Sci., 9 **(2014)** 4205 - 4216

International Journal of
ELECTROCHEMICAL
SCIENCE
www.electrochemsci.org

Corrosion Inhibition Performance of *Rhizophora mangle* L Bark-Extract on Concrete Steel-Reinforcement in Industrial/Microbial Simulating-Environment

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Received: 15 March 2014 / *Accepted:* 27 April 2014 / *Published:* 19 May 2014

In this paper, the corrosion inhibition performance of the bark-extract of *Rhizophora mangle* L admixture on steel-reinforcement in concrete, immersed in 0.5 M H_2SO_4 for simulating industrial/microbial environment, were studied. Different concentrations of the plant-extract were admixed in duplicated concrete samples immersed in the acidic test-solution and these were monitored using non-destructive electrochemical techniques, for seventy-five days experimental period. Statistical analyses of the corrosion test-responses, as per ASTM G16-95 R04, showed that the corrosion rate correlated with function of the admixed bark-extract concentration in concrete, the half-cell potential and the corrosion current from the corrosion test-setup. The experimental and predicted model, from the correlation, both identified 0.167% *Rhizophora mangle* L bark-extract (per weight of cement) with optimal effectiveness, η = 78.6% (experimental) or η = 70.7% (predicted) at inhibiting steel-reinforcement corrosion. The experimental model of inhibition efficiency compares well with results from literature, classifies as very good inhibition efficiency model and thus supports *Rhizophora mangle* L bark-extract as inhibitor of steel-reinforcement corrosion in industrial/microbial environment.

Keywords: corrosion of steel-reinforcement in concrete; environmentally-friendly inhibitor; industrial/microbial simulating-environment; ASTM G16 analyses; correlation fitting model; inhibition efficiency

1. INTRODUCTION

Due to its relative low cost and comparative structural integrity, steel-reinforced concrete is the most widely used material for building structures and infrastructures worldwide [1-3]. However, corrosion of the reinforcing steel in concrete affects structural integrity, poses safety risks to life and property and leads to repair and maintenance that constitute costly budgets in many countries [4-6]. Aggressive environments responsible for concrete steel-reinforcement corrosion include sulphuric acid environments from acid rain in industrial [7-8] or microbial activities of sulphate reducing bacteria, e.g. *Thiobacillus* spp., in sewage or underground environments [2,9]. Gypsum and ettringite products from sulphuric acid attack on concrete are expansive and thus crack the concrete exposing the rebar to further corrosion degradation and if unchecked eventual catastrophic failure [2,9-11].

Methods that had been identified in studies for tackling concrete steel-reinforcement corrosion in acidic media include use of protective coatings, use of acid resistant cement and the use of corrosion inhibitors as admixtures in concrete [2,5,7-9]. Among these, the use of corrosion inhibitor admixtures constitutes method that has been found effective with comparably lower cost than the other methods [7-8]. However, many admixtures that had been found effective at inhibiting steel-reinforcement corrosion such as compounds of chromates and nitrites [2,5,8,12] are being restricted in many countries due to their toxicity and hazardousness to the environmental eco-system [13-14]. The search for environmentally-friendly alternatives that will find comparable effectiveness with the well-known but toxic inhibitors constitutes current trend in contemporary studies of corrosion inhibitors [15].

Bark-extract of *Rhizophora mangle* L (Red mangrove) has been identified with both medicinal benefits for human and no toxicity effect to systems of living organs in [16]. That study [16] also identified tannins as a component of this plant extract from which it is worth noting that tannins from bark-extract of *Rhizophora apiculata* have found usefulness for inhibiting corrosion of steel in acidic (HCl) medium in reported works [17-18]. While these constitute motivation for the present study, no reported work have studied the effect of *Rhizophora mangle* L bark-extract on the corrosion of steel-reinforcement in physically cast concrete slab immersed in acidic sulphate environment. This paper, therefore studies corrosion inhibition performance of *Rhizophora mangle* L bark-extract on steel-reinforcement in concrete immersed in sulphuric-acid medium, an industrial/microbial simulating-environment.

2. MATERIALS AND METHODS

2.1. Experimental materials

2.1.1. Rhizophora mangle L bark-extract

Dried barks of *Rhizophora mangle* L (*R. mangle*) *Rhizophoraceae* were finely ground (blended) into powder [18] and from this, solution of methanolic extract [19] were obtained through use of a condenser equipped soxhlet extractor. The extracted solution was then concentrated over water bath and the residue used for concrete admixture in the study.

2.1.2. Steel-reinforcement and specimens of steel-reinforced concrete slabs

Steel-reinforcement employed in the study was of 12mm diameter by 190mm length for each specimen and this have 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. Pre-experimental surface treatments for each specimen of steel-reinforcement were maintained uniform and in accordance with the standard procedures prescribed in [20] and described in [21].

Steel-reinforced concrete slabs for the study were of size 100 mm × 100 mm × 200 mm for each specimen. These were cast in duplicated specimen design, i.e. with similar concentration of *R. mangle* bark-extract admixed in each slab of the duplicated samples, as prescribed by [22]. These duplicates of admixed concrete employs *R. mangle* bark-extract concentration per concrete ranging from 0%, for the duplicates of blank samples, in increments of 0.083% (one part by weight of *R. mangle* bark-extract in 1200 parts by weight of cement) up to 0.417%. Immersed into each concrete slab, during casting of the twelve (in total) specimens, was 150 mm of the length of steel-reinforcement, symmetrically across the width of each of the slab, such that the remaining steel protrusion could be used for electrochemical connections. Each concrete slab were cast as per standard procedure prescribed in [23] using drinkable water, Portland cement, clean natural river sand and granite stones using mix proportion described in [21].

2.2. Experimental Setup for corrosion test-measurements

Each slab of steel reinforced concrete specimens were longitudinally and partially immersed in 0.5 M H_2SO_4 test-solution for simulating industrial/microbial environments [2,5,8,24-25]. The test-medium was made up in each plastic container of concrete slab immersion, to just below the protruding steel-reinforcement from the concrete but without touching the steel-reinforcement. From these specimens, the steel-reinforcement corrosion were monitored for seventy-five days, using electrochemical techniques of half-cell potential (*HCP*), corrosion current (*CC*) and corrosion rate (*CR*) measurements according to standard procedures described in [12,21,26].

2.3. Experimental Data Analyses

2.3.1. Statistical distribution analyses and goodness-of-fit study

The corrosion test-responses obtained from the electrochemical monitoring techniques were statistically analysed using the Normal and the Weibull distribution functions, for which test-data compatibility was studied using the Kolmogorov-Smirnov goodness-of-fit statistics. These followed the prescriptions of [27] for avoiding grossly erroneous conclusions. The descriptive statistics of the Normal probability distribution function (pdf) and that of the extreme value Weibull pdf are respectively given by [28-29]:

$$f_{Norm}(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{(x-\mu)^2}{2\sigma^2}\right] \quad (1)$$

$$f_{Weib}(x) = \frac{k}{c}\left(\frac{x}{c}\right)^{k-1} \exp\left[-\left(\frac{x}{c}\right)^k\right] \quad (2)$$

From which x represents measured corrosion test-data from each electrochemical test-variable, μ and σ are mean and standard deviation of the Normal pdf, while k and c are the shape and scale parameters of the Weibull pdf. These Weibull shape and scale parameters were employed for the estimation of the Weibull mean, μ_{Weib}, using the expression [10,30]:

$$\mu_{Weib} = c\Gamma\left(1+\frac{1}{k}\right) \quad (3)$$

The choice of the statistics for describing the corrosion test-responses from the concrete specimens require testing the probability distribution function followed by the Normal and the Weibull pdf using the Kolmogorov-Smirnov (K-S) goodness-of-fit (GoF) test as per [27,31]. This was done using standard procedure that had been reported in studies [5,21] through the statistics [32]

$$D_n = D(x_1, ..., x_n) = \sup_{-\infty<x<\infty} \left|F^*(x) - F(x)\right| \quad (4)$$

2.3.2. Estimation of Inhibition efficiency performance

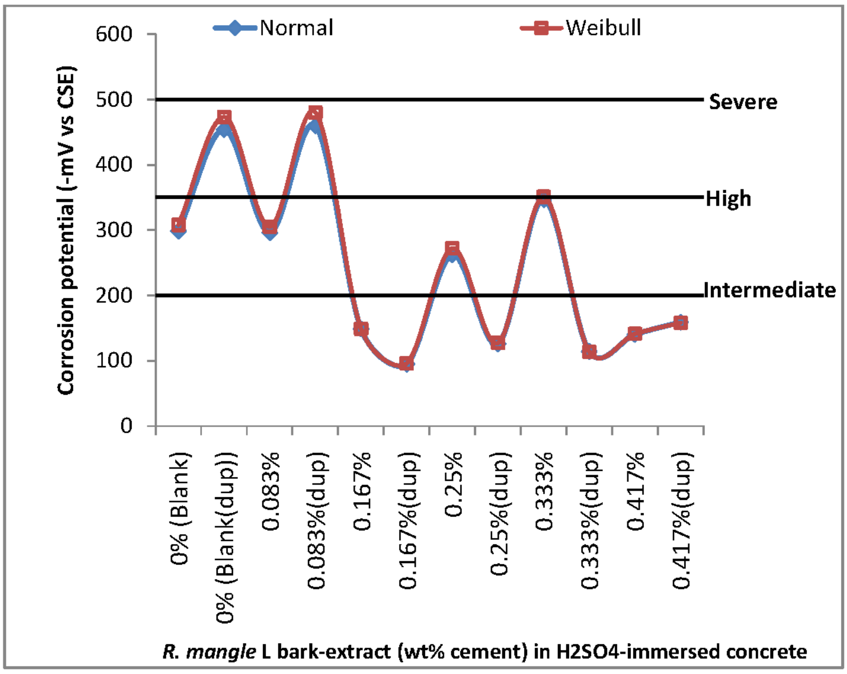
The mean model obtained from the statistical distribution which fits the corrosion rate (*CR*) test-data better was also used for estimating the inhibition efficiency, η, performance by each admixed *R. mangle* bark-extract concentration in concrete relative the blank concrete sample. These estimations employ the expression [21,26,33]:

$$\eta_{(IE)} = \frac{CR_{pdf,blank} - CR_{pdf,admixed}}{CR_{pdf,blank}} \times 100 \quad (5)$$

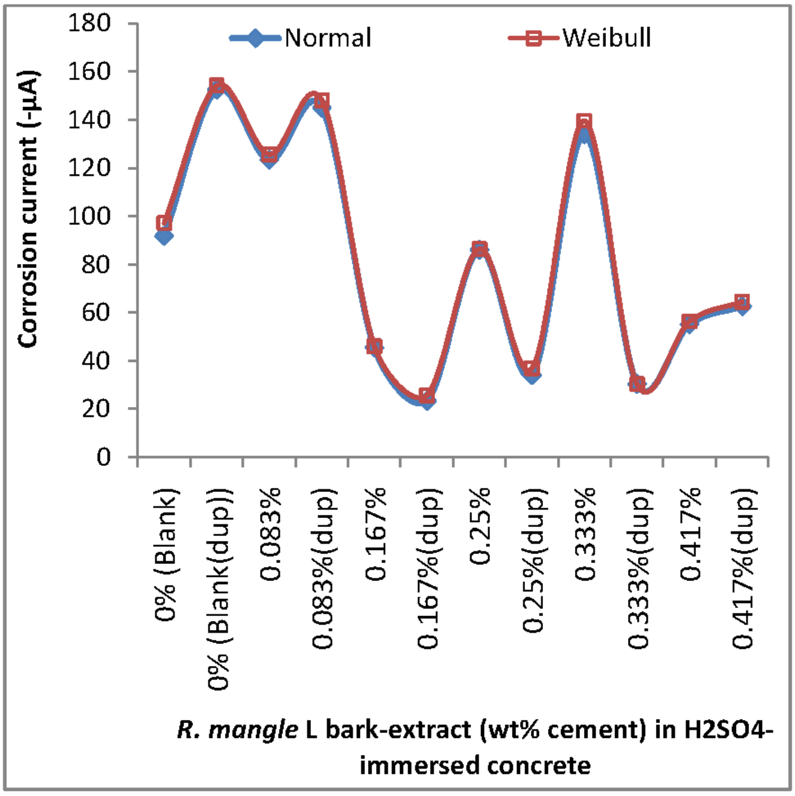
3. RESULTS AND DISCUSSION

3.1. Results of statistical distribution fitting models and analyses

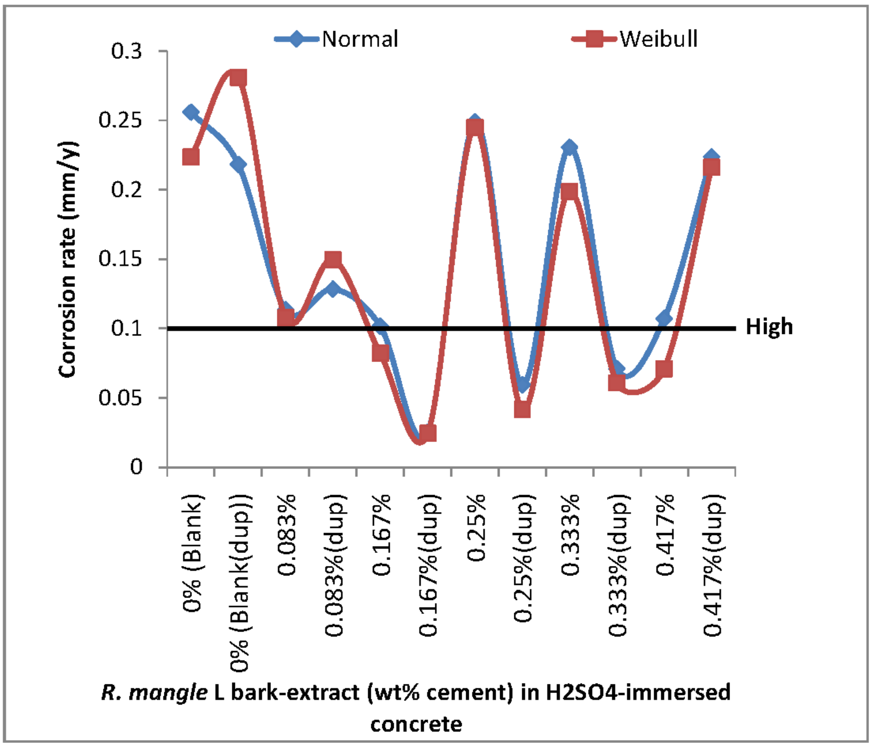
The results of the statistical distribution fitting models of corrosion test-data, obtained from the steel-reinforced concrete specimens, by the Normal pdf and the Weibull pdf are presented in Figure 2 for test-data of *HCP* in Figure 1(a), *CC* in Figure 1(b) and *CR* in Figure 1(c). In the figure, the *HCP* and *CR* plots include the interpretation plots according to standard procedures for interpreting *HCP* in [34] and for classifying *CR* in [35]. From these plotting, it could be noted that the *HCP* plots patterned like the *CC* plots along the *R. mangle* bark-extract concentrations admixed in the steel-reinforced concrete samples. However, in spite of this similar trend of corrosion test-data obtained from different instruments, there were disparities between the Normal and the Weibull models especially in the *CR* models by the two fitting functions. It is based on these forms of disparities that the goodness-of-fit testing of data compatibility to statistical distribution fittings finds usefulness for avoiding erroneous conclusion on the performance of *R. mangle* bark-extract admixture on steel-reinforcement corrosion.


Normal
Weibull
Corrosion potential (-mV vs CSE)
600
500
400
300
200
100
0
Severe
High
Intermediate
0% (Blank)
0% (Blank(dup))
0.083%
0.083%(dup)
0.167%
0.167%(dup)
0.25%
0.25%(dup)
0.333%
0.333%(dup)
0.417%
0.417%(dup)
R. mangle L bark-extract (wt% cement) in H2SO4-immersed concrete

(a)


Normal
Weibull
Corrosion current (-μA)
180
160
140
120
100
80
60
40
20
0
0% (Blank)
0% (Blank(dup))
0.083%
0.083%(dup)
0.167%
0.167%(dup)
0.25%
0.25%(dup)
0.333%
0.333%(dup)
0.417%
0.417%(dup)
R. mangle L bark-extract (wt% cement) in H2SO4-immersed concrete

(b)



(c)

Figure 1. Results of statistical distribution models of corrosion test-data from *R. mangle* bark-extract admixed steel-reinforced concretes (a) *HCP* mean and standard deviation models with corrosion risk plots as per ASTM C876-91 R99 [34] (b) *CC* mean and standard deviation models (c) CR mean models with corrosion rate classification plot as per [35].

The goodness-of-fit tests results of measured corrosion data compatibility as per the Kolmogorov-Smirnov test-statistics [32] are presented in Figure 2, with level of significance interpreting plot of α = 0.05 according to the practice that had been employed in [12,21,26]. The plotting of Kolmogorov-Smirnov *p*-values in the figure showed that the *HCP* test-data distributed like both the Normal and the Weibull probability fitting functions according to the Kolmogorov-Smirnov criteria at α = 0.05 significance level. By this, either the Normal or the Weibull fitting function could be employed as the descriptive statistics for detailing *HCP* performance of the *R. mangle* bark-extract concentrations in the steel-reinforced concrete in their acidic sulphate solution of test-immersion.

However, three datasets of *CC* test-data, as well as 5 datasets of the *CR* test-data were not distributed like the Normal pdf. In contrast, only one dataset of *CC* did not follow the Weibull distribution in the study. Rather, datasets of *HCP* and of *CR* test-data for all the *R. mangle* bark-extract admixed steel-reinforced concretes distributed like the Weibull probability distribution function, according to the Kolmogorov-Smirnov test-criteria at α = 0.05 significance level. By these goodness-of-fit test-results, the Weibull surpassed the Normal distribution statistics at describing corrosion test-data in this study. The Weibull distribution statistics was therefore preferred for detailing *R. mangle* bark-extract performance on concrete steel-reinforcement corrosion in H_2SO_4 test-solution, the industrial/microbial simulating medium employed in the study.

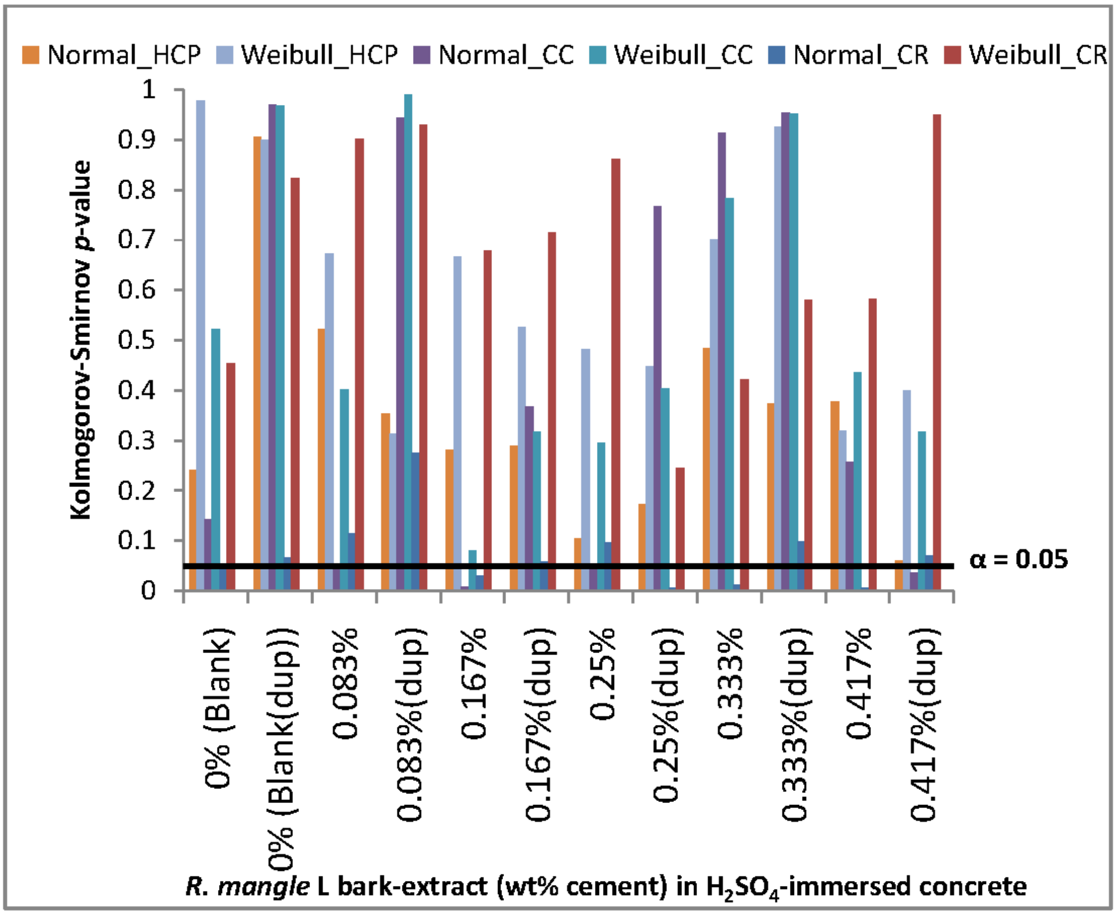


Figure 2. Goodness-of-fit analyses of corrosion test-data compatibility to statistical distribution fittings as per Kolmogorov-Smirnov test-statistics at α = 0.05 level of significance.

3.2. Correlation fitting modelling of corrosion test-variables

In Figure 2, the *HCP* mean models were not only patterned like the *CC* models, it could be also be observed that many *R. mangle* bark-extract admixed concrete with high (identifiable as spikes of) *HCP* also exhibited high (spikes of) *CR*. The only exception to this pattern of high *CR–HCP* model was the 0.417%(dup) specimen of *R. mangle* bark-extract admixed concrete. These engender interests on studying what form of relationship could exist between the models of corrosion rate and the other variables affecting concrete steel-reinforcement corrosion in the H_2SO_4 test-medium employed. For this, several correlation analyses between the variables were investigated [21,26,36], from which it was identified that the corrosion rate (*CR*) correlated with the *R. mangle* bark-extract concentration, ρ (%), corrosion potential, *V* (–mV), and corrosion current, *I* (–μA), according to:

$$CR = \frac{1}{100}\left\{e^{\rho} \cdot \ln I^{1.4242} + 6.246\sqrt{V}\left(1 - 2.4567 \times 10^{-2}\sqrt{V}\right) - 50.1684\right\} \quad (6)$$

For this fitting function in Equation (6), the correlation coefficient, r = 78.9%. Also, from the equation, the correlation of the *CR*, in H_2SO_4-immersed concretes, with the exponential function of admixture concentration and with the logarithmic function of the corrosion current, *I* (–μA), find agreements with findings in [21]. This was in spite of the fact that the admixtures considered in the reported work in [21] were chemical admixtures, the $Na_2Cr_2O_7$ and its partial replacement model, the $C_6H_{15}NO_3$, in contrast to the admixture in this study, which is a natural plant extract. This bears implication of similitude of corrosion inhibition performance by the natural plant-extract being considered in this study with that obtained from the chemical inhibitors of steel-reinforcement corrosion in the H_2SO_4 medium.

In furtherance of testing the significance of the relationship between the *CR* models and other variables attending concrete steel-reinforcement corrosion in the test-solution of study, the analysis of variance (ANOVA) of the correlation fitting expression is presented in Table 1. From this table, the *p*-value of the ANOVA equals 0.0415 a value that < 0.05. This *p*-value implies that it cannot be rejected that the relationship, given in Equation (6), between the *CR* model and the other variables attending concrete steel-reinforcement corrosion in the H_2SO_4 test-medium were not due to chance but were significant at 95% confidence level.

Table 1. ANOVA for the correlation fitting expression in Equation (6)

Source of variations	*df*	SS	MS	*F*	*p*-value
Regression	3	0.053	0.018	4.407	0.042
Residual	8	0.032	0.004		
Total	11	0.086			

3.3. Corrosion inhibition efficiency and performance of admixtures in studied medium

The test-result and the correlation prediction models of corrosion rate (*CR*) were employed for estimating averaged corrosion inhibition efficiencies, which find usefulness for detailing *R. mangle* bark-extract admixture performance on concrete steel-reinforcement corrosion. The results of these experimental and predicted models are presented in ranking order of the experimental model in Figure 3. The plots of inhibition efficiency performance by the extract admixtures, shown in Figure 3, exemplifies the advantages of using duplicated specimen design and correlation prediction models for ascertaining agreements or otherwise from the experimental test-results. For instance, the between-duplicate specimen standard deviations that were shown as error bars in the figure identified the 0.25% *R. mangle* bark-extract admixture as potent at both inhibiting as well as aggravating concrete steel-reinforcement corrosion in the study. The efficiency performance by the experimental model, $\eta_{(exp)}$ = 42.5±48.0%, or by the predicted (correlation) model , $\eta_{(pred)}$ = 31.6±35.5%, for the 0.25% *R. mangle* bark-extract admixture both ranged into corrosion inhibition as well as into corrosion aggravation regions. For this admixture, the experimental test-results find agreements with the prediction from the correlation fitting model. Also, both bear representations of conflicting effectiveness that had been identified in literature [12,37–38] whereby studies report a substance as effective while other studies report the same substance as ineffective at inhibiting corrosion. In furtherance of disagreement identifications, the prediction by the correlation fitting model showed that the 0.083% *R. mangle* bark-extract admixture exhibited negative corrosion inhibition efficiency, $\eta_{(pred)}$ = –3.7±5.9% (i.e. corrosion aggravation). This did not find agreement with the experimental test-results which showed that the 0.083% *R. mangle* bark-extract admixture exhibited the positive effectiveness performance of $\eta_{(exp)}$ = 48.3±11.9%, which implies corrosion inhibition.

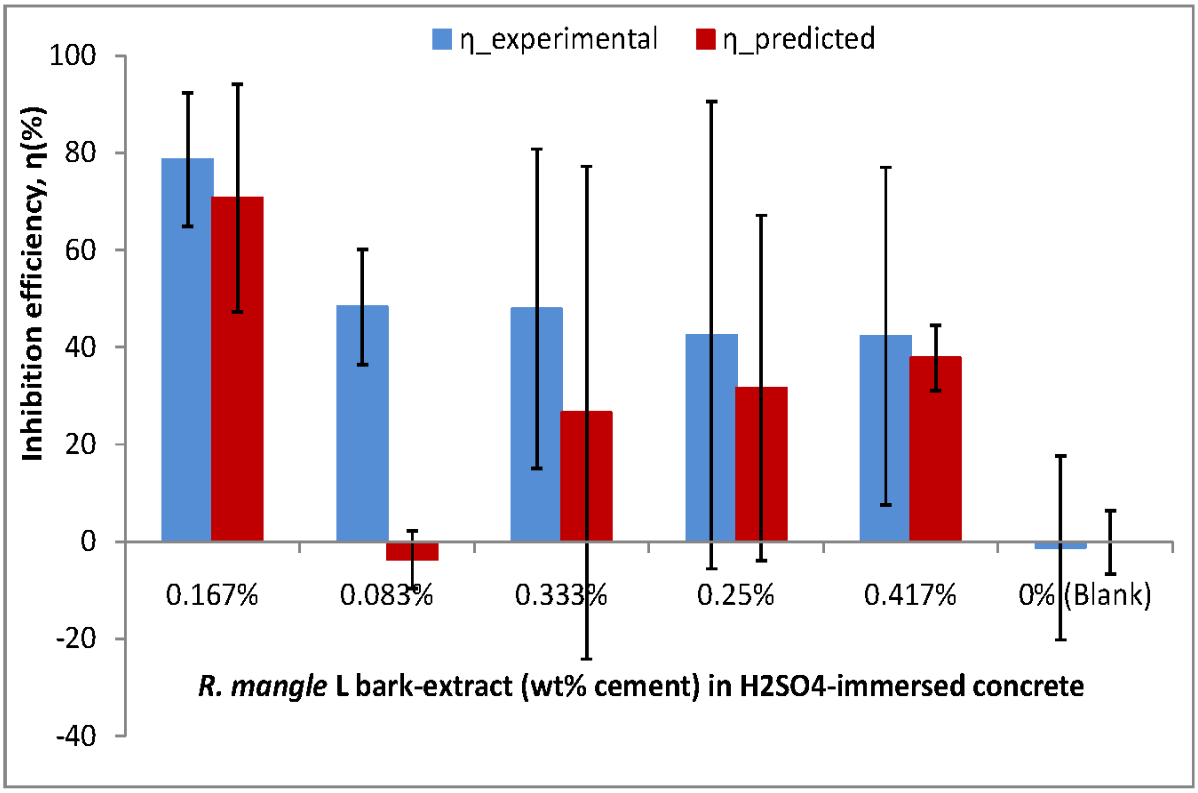


Figure 3. Ranking of inhibition efficiency performance of *R. mangle* bark-extract admixture on concrete steel-reinforcement corrosion

Analytical reasons for this disagreement was that the correlation fitting model of Equation (6) over-predicted the corrosion rate for the 0.083% *R. mangle* bark-extract above both the corrosion rate of the experimental model of the admixture and that of the blank samples. These over-predictions followed from the *HCP* and the *CC* of the duplicated 0.083% *R. mangle* bark-extract admixed specimens which in duplicated agreements either overshot or find comparisons with the *HCP* and the *CC* of the blank samples, Figure 1. These disagreements in test-models highlight the needs for experimental test-results for confirming results from numerical models as well as the use of additional monitoring method, e.g. the *CR* and the *CC*, with the *HCP* for detailing corrosion condition as prescribed in [39].

In spite of the foregoing, both the experimental test-result and the prediction model from the correlation fitting identified the 0.167% *R. mangle* bark-extract admixture with optimal effectiveness at inhibiting concrete steel-reinforcement corrosion in the studied test-medium. These, see Figure 3, showed that the 0.167% *R. mangle* bark-extract admixture exhibited $\eta_{(exp)}$ = 78.6±13.8% or $\eta_{(pred)}$ = 70.7±23.4% effectiveness at inhibiting steel-reinforcement corrosion in the H_2SO_4 test-medium. These inhibition efficiencies find comparisons with the effectiveness results obtained in [8] for sodium molybdate and sodium nitrite, both of which were employed as chemical inhibitors of steel-reinforcement corrosion in acidified concrete pore solution in that study. This comparable effectiveness was still in spite of the consideration that the natural plant extract in this work were admixed directly in physically cast concretes in which there is potency of partial loss of inhibition efficiency [40] due to interactions of the admixture with cement paste [41-42]. This constitutes reasons by which recommendations from the studies in [41-42] prescribed needs for studying effectiveness performance of admixtures at inhibiting steel-reinforcement corrosion in physically cast slabs of steel-reinforced concrete admixed with the admixture.

4. CONCLUSIONS

Corrosion inhibition performance of *R. mangle* bark-extract admixed in steel-reinforced concretes which were immersed in 0.5 M H_2SO_4, for simulating industrial/microbial environment has been studied in this work. The conclusions that could be drawn from this study include:

- The Weibull probability distribution described the distribution of corrosion test-data better than the Normal probability distribution according to the Kolmogorov-Smirnov goodness-of-fit testing of the test-data from the corrosion monitoring techniques employed in the study;
- The corrosion rate models correlated well with the function of *R. mangle* bark-extract admixture concentration, the corrosion potential and the corrosion current with the correlation coefficient r = 78.9% and the ANOVA p-value = 0.0415 which implies statistically significant relationship between the dependent and independent variables at 95% confidence level;
- The experimental model and the prediction from the correlation fitting model both identified the 0.1667% *R. mangle* bark-extract admixture with optimum effectiveness, in the study, at inhibiting concrete steel-reinforcement corrosion in the test-medium of 0.5 M H_2SO_4, simulating industrial/microbial service-environment, the inhibition efficiencies, $\eta_{(exp)}$ = 78.6±13.8% or $\eta_{(pred)}$ = 70.73±23.44% find comparisons with what obtained in literature employing other well-known inhibitors for reinforcing steel corrosion in acidified simulated concrete pore test-environment.

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