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Corrosion Rate and Noise Resistance Correlation from $NaNO_2$-Admixed Steel-Reinforced Concrete

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ABSTRACT
The lack of correlation from measurements of corrosion potential with corrosion rate in corrosion monitoring of steel-reinforced concrete has constituted concerns instigating demands for further experiments or additional monitoring techniques in corrosion studies. This paper studies correlation of corrosion rate from linear polarization resistance instrument and noise resistance from the ratio of standard deviations of corrosion potential to that of corrosion current measured from $NaNO_2$-admixed steel-reinforced concrete in 3.5% NaCl. Different concentrations of $NaNO_2$ were admixed in replicates of steel-reinforced concretes immersed in the saline/marine simulating test-solution and corrosion of the embedded steel-rebar were monitored using the three different, non-destructive, electrochemical methods. Measurements of corrosion test-data from these were subjected to the descriptive statistics of the Weibull distribution function, for which test-data compatibility was ascertained using the Kolmogorov-Smirnov goodness-of-fit statistics. The analysed results of the measured corrosion test-variables, exhibited good correlation between the corrosion rate, as the dependent variable and the function of the admixed $NaNO_2$ molar concentration and the noise resistance, as independent variables. Both experimental and prediction, from correlation fitting, models identified 0.1208 M $NaNO_2$, the optimal concentration employed in the study, with optimum effectiveness, of $\eta>90\%$, at inhibiting steel-rebar corrosion in the corrosive solution of concrete test-immersion. These bear suggestions of the suitability of the analysed more easily measured corrosion potential and corrosion current for indicating the corrosion activity of steel-reinforcement in concrete designed for corrosive service-environments.

Key words: Reinforcing steel corrosion, corrosion test-data modelling, weibull distribution, goodness-of-fit test-statistics, correlation fitting analyses

INTRODUCTION
Reinforcing steel corrodes due to aggressive agents in the service-environment of steel reinforced concrete materials, widely used globally for building structures and infrastructures and thus militates against structural integrity of the concrete structures (Yohai *et al*., 2013; Hu *et al*., 2012). Among known aggressive agents causative of steel-reinforcement (steel-rebar) corrosion degradation, ingress of chloride ion from artificial de-icing salt and/or natural marine (sea water) constitutes dominant factor affecting steel-reinforced concrete durability (Romano *et al*., 2013; Yang *et al*., 2013). Among many methods identified in studies for mitigating concrete

steel-reinforcement corrosion, the use of corrosion inhibitors has gained wide acceptability due to ease of applications, relative lower cost than other methods and effectiveness at inhibiting steel-rebar corrosion (Yohai *et al*., 2013; Etteyeb *et al*., 2007; Ormellese *et al*., 2006).

However, ascertaining effectiveness of inhibitors at mitigating steel-rebar corrosion in aggressive environment requires adequate monitoring of the metallic corrosion (Al-Mehthel *et al*., 2009). Among many methods for monitoring concrete steel-reinforcement corrosion, the use of electrochemical techniques has been identified with the greater success in literature due to its sharing the same electrochemical nature as the corrosion process (Romano *et al*., 2013). Another strong point supporting the electrochemical test-techniques of reinforcing steel corrosion in concrete includes the fact that they are also non-destructive methods. A well known, fast and easily applied electrochemical monitoring technique of the corrosion condition of concrete steel-rebar includes the open circuit (also known as the half-cell) potential (OCP or HCP) method (Okeniyi *et al*., 2012, 2014a; Omotosho *et al*., 2012, 2014; Gulikers, 2010; Burubai and Dagogo, 2007). According to Gulikers (2010) the measurement procedure of the OCP is simple and straightforward. Song and Saraswathy (2007) also described the OCP monitoring technique as the main method of detection of corrosion and the most typical routine inspection of reinforced concrete structures. Although, the principle involved in the OCP monitoring technique is the measurement of corrosion potential of the reinforcing steel in concrete relative to specified reference electrode, the procedure and interpretation of its test-results are described in the standard of the American Society for Testing and Materials, ASTM C876-91 R99 (ASTM., 2005a; Gulikers, 2010; Song and Saraswathy, 2007; Broomfield, 2002). However, this method suffers the setback that it can only provide information of corrosion probability but cannot indicate nor correlate with the quantitative rate of steel reinforcement corrosion in concrete (Gulikers, 2010; Song and Saraswathy, 2007).

The foregoing constitutes reasons for which Elsener *et al.* (2003), in the recommendation of RILEM on half-cell potential measurement, indicated that the evaluation of the effectiveness of inhibitors performance by using the half-cell (i.e. the open circuit) potential is difficult. For tackling these, Song and Saraswathy (2007) mandated complementing of open circuit potential measurements by other methods while Elsener *et al.* (2003) recommended more research for unambiguous evaluation of inhibitor effectiveness on steel-rebar corrosion.

These engender motivation of this research study towards investigation of a simple, fast and easily applied additional measurement for complementing open circuit potential and that will also foster the correlation of the OCP monitoring technique with corrosion rate. For this, the measurement of corrosion current by the Zero Resistance Ammeter (ZRA) relative to the same reference electrode employed for the OCP measurement was conceived based on the idealization of Daniel cell described in Broomfield (2002). The premise in this study was that the porous partition by the semi-permeable membrane of the requisite reference electrode would permit charge exchange, when the reference and working electrodes are joined across the ZRA, while being impervious to ion movement (Okeniyi *et al*., 2013a; Broomfield, 2002). This, according to McCarter and Vennesland (2004), is potent at constituting measurement model of the reinforcing steel dissolution activity in the aggressive system of corrosive test-solution.

In reported study submitted elsewhere (Okeniyi *et al*., 2013b), the measurement model of the corrosion potential from OCP and corrosion current from the ZRA has been employed along with inhibitor concentration for correlation with corrosion rate. However, it is of interest in this study to investigate how the ratio of the standard deviation of the corrosion potential by OCP to that of the corrosion current by ZRA would correlate with corrosion rate obtained from the Linear Polarisation Resistance (LPR) instrument. Such correlation, if it could be established would find usefulness, especially, for quick detailing of inhibitor performance at inhibiting steel-rebar corrosion in chloride contaminated medium. In literature, deliberating on corrosion studies (Song and Saraswathy, 2007; Nunez-Jaquez *et al*., 2005; Eden, 2000; Kelly *et al*., 1996), the ratio of standard deviation of corrosion potential to that of the corrosion current has been identified as the noise resistance. However, in none of these was the descriptive statistics followed by the test-data of corrosion potential and/or corrosion current studied for detailing the measurement of dispersion, i.e. the standard deviation, being employed for the noise resistance evaluation. Such form of study, however, finds necessity from the consideration of ASTM G16-95 R04 (ASTM., 2005b) that prescribed that employing descriptive statistics for detailing test-data distributed in other manner could lead to grossly erroneous conclusion. Therefore, the objective of this study was to investigate correlation of corrosion rate and noise resistance test-data, obtained through requisite study of descriptive statistics followed by those data, for detailing $NaNO_2$ effectiveness on steel-reinforcement corrosion. The choice for $NaNO_2$ in the study, followed from its being well-known at inhibiting concrete steel-rebar corrosion (Okeniyi *et al*., 2013c; Mennucci *et al*., 2009; Soylev and Richardson, 2008) such that the procedures from this study could find applicability for detailing performance for other inhibiting substances that are yet not well-known.

MATERIALS AND METHODS

Steel reinforced concrete materials and $NaNO_2$ admixtures: Steel reinforced concretes blocks used for the experimental were cast in duplicated slabs (Haynie, 2005) of size 100×100× 200 mm, using standard procedures from ASTM C192/192M-02 (ASTM., 2005c) as reported in Okeniyi *et al.* (2013a). In each of the slabs were embedded 150 mm out of the 190 mm length of Ø12 mm deformed steel reinforcement, which have the composition in % 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. The duplicated steel-reinforced concretes totaled twelve specimens and the admixed $NaNO_2$ molar concentration (M) with respect to concrete mixing water for these samples were as presented in Table 1.

Experimental procedure of corrosion testing: Each specimen of steel reinforced concrete samples were partially immersed, longitudinally, in plastic bowls containing 3.5% NaCl test-solution for simulating saline/marine environments (Zhou *et al*., 2012; Mennucci *et al*., 2009). These test-medium were made up in each bowl to just below the reinforcing steel but without touching the rebar.

Electrochemical test-monitoring for which correlation were being sought require measurements from each of the steel-reinforced concretes that were taken at interval for 54-days experimental period which include:

Table 1: Molar concentration $NaNO_2$ admixture in specimens of steel reinforced concretes

Admixture in concrete
0 M $NaNO_2$ (Blank) in NaCl
0 M $NaNO_2$ (Blank) in $NaCl_{(Dup)}$
0.0242 M $NaNO_2$ in NaCl
0.0242 M $NaNO_2$ in $NaCl_{(Dup)}$
0.0483 M $NaNO_2$ in NaCl
0.0483 M $NaNO_2$ in $NaCl_{(Dup)}$
0.0725 M $NaNO_2$ in NaCl
0.0725 M $NaNO_2$ in $NaCl_{(Dup)}$
0.0966 M $NaNO_2$ in NaCl
0.0966 M $NaNO_2$ in $NaCl_{(Dup)}$
0.1208 M $NaNO_2$ in NaCl
0.1208 M $NaNO_2$ in $NaCl_{(Dup)}$

- Corrosion potential (V in mV unit) through Open Circuit Potential (OCP) as per ASTM C876-91 R99 (ASTM., 2005a), versus $Cu/CuSO_4$ reference electrode (CSE) Model 8-A (from Tinker and Rasor®), using a high impedance digital multimeter (Okeniyi *et al*., 2014a)
- Corrosion current (I in μA unit) versus $Cu/CuSO_4$ reference electrode CSE, using ZRA instrument (McCarter and Vennesland, 2004; Broomfield, 2002) Model ZM3P (from Corrosion Service®)
- Corrosion Rate (CR) measurements through direct instrument conversion to mpy using the three-electrode LPR Data Logger (Sastri, 2011), Model MS1500L (from Metal Samples®)

Data analysis

Fitting corrosion test-data to the Weibull distribution statistics: Measured corrosion test-data were subjected to the statistical analysis of the Weibull distribution with probability and cumulative distribution functions (Roberge, 2000, 2003, 2005; Haynie, 2005) given by:

$$f(x) = \left(\frac{k}{c}\right)\left(\frac{x}{c}\right)^{k-1} \exp\left[-\left(\frac{x}{c}\right)^{k}\right] \tag{1}$$

$$F(x) = 1 - \exp\left\{-\left(\frac{x}{c}\right)^{k}\right\} \tag{2}$$

where, x denotes test-data of corrosion test-variable, k and c are the shape parameter and the scale parameter, respectively, of the Weibull distribution model. Estimations of k and c were obtained, for sample size n, from the maximum likelihood equations (Okeniyi *et al*., 2014b; Kvam and Lu, 2006):

$$\frac{n}{\hat{k}} - n\ln(\hat{c}) + \sum_{i=1}^{n} \ln x_i - \sum_{i=1}^{n}\left(\frac{x_i}{\hat{c}}\right)^{\hat{k}} \ln\left(\frac{x_i}{\hat{c}}\right) = 0 \tag{3}$$

$$\hat{c} - \left\{\frac{1}{n}\sum_{i=1}^{n} x_i^{\hat{k}}\right\}^{\frac{1}{\hat{k}}} = 0 \tag{4}$$

The unbiased estimated values of k and c, from these, were used to compute the respective Weibull mean, μ and standard deviation, σ, as (Okeniyi *et al*., 2014a; Ajayi *et al*., 2013):

$$\mu = c\Gamma\left(1 + \frac{1}{k}\right) \tag{5}$$

$$\sigma = \sqrt{c^2\left\{\Gamma\left(1 + \frac{2}{k}\right) - \left[\Gamma\left(1 + \frac{1}{k}\right)\right]^2\right\}} \tag{6}$$

where, Γ(•) is the gamma function of (•).

Goodness-of-fit test statistics: As per recommendation of ASTM G16-95 R04 (ASTM., 2005b) and Roberge (2003), the compatibility of the test-data from the corrosion test-monitoring methods employed to the Weibull distribution were studied using the Kolmogorov-Smirnov (K-S) Goodness-of-Fit (GoF) test-statistics (Okeniyi and Okeniyi, 2012; Izquierdo *et al.*, 2004). This employs measurement of the absolute difference between the empirical distribution function F*(x) and the theoretical distribution function F(x), through the statistics:

$$D_n = D(x_1, \ldots, x_n) = \sup_{-\infty<x<\infty} \left| F^*(x) - F(x) \right| \tag{7}$$

The evaluated D-value from this equation finds usefulness for direct computation of the K-S p-value using the procedure that had been reported in Okeniyi and Okeniyi (2012).

Evaluating test-data of noise resistance (R_n): Distribution of the corrosion potential and the corrosion current like the Weibull pdf would satisfy the condition of ASTM G16-95 R04 (ASTM., 2005b) for using the Weibull pdf for describing these corrosion test-data. By this, the noise resistance test-data prevailing in each concrete samples would be obtained as the ratio of Weibull standard deviation of the corrosion potential (mV) to the Weibull standard deviation of corrosion current (mA), i.e. (Okeniyi *et al.*, 2014b; Song and Saraswathy, 2007; Nunez-Jaquez *et al.*, 2005; Eden, 2000; Kelly *et al.*, 1996):

$$R_n = \frac{\sigma_{V(\text{Weibull})}}{\sigma_{I(\text{Weibull})}} \tag{8}$$

Performance evaluators for correlation fitting model: The performance evaluators employed for the correlation fitting model between the corrosion rate (CR) and noise resistance (R_n) include the correlation coefficient (r) and the p-value from the one-way analysis of variance (ANOVA) modelling (Okeniyi *et al.*, 2013a; Mandal and Jothiprakash, 2012; Izquierdo *et al.*, 2004).

Inhibition efficiency: Inhibition efficiency model, η, was evaluated for the replicates of $NaNO_2$ admixed samples relative to the replicates of control samples, through the formula (Okeniyi *et al.*, 2014c; Zhou *et al.*, 2012):

$$\eta = \frac{\mu_{\text{ctrl}} - \mu_{\text{inh}}}{\mu_{\text{ctrl}}} \times 100 \tag{9}$$

RESULTS AND DISCUSSIONS

Statistical analysis of corrosion test-data: The Weibull mean and standard deviation models for the corrosion potential and the corrosion current are shown in Fig. 1a-b, respectively. In the figure, the standard deviations are shown as error bars in the form '$\mu\pm\sigma$'. In addition, Fig. 1a includes the linear plots of corrosion risk interpretation according to ASTM C876-91 R99 (ASTM., 2005a).

It could be observed from Fig. 1a-b, that the plots of mean model of corrosion potential and corrosion current exhibited similar pattern of corrosion test-responses. However, the severe

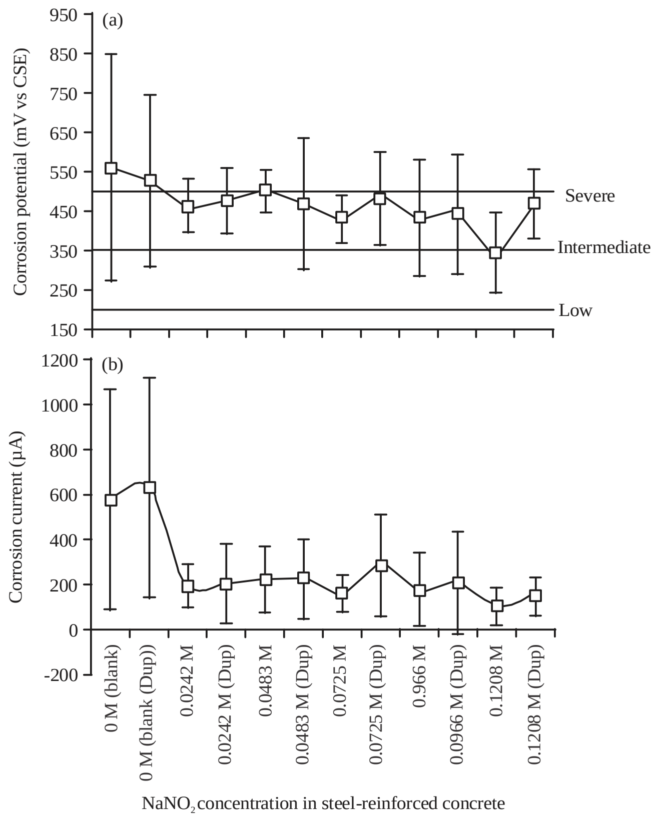

Fig. 1(a-b): Weibull mean and standard deviation models of (a) Corrosion potential and (b) Corrosion current

probability of corrosion risk condition in the 0M $NaNO_2$ (blank) samples, as per the ASTM C876-91 R99 (ASTM., 2005a) interpretation in Fig. 1a, exhibited more pronounced prevalence in Fig. 1b compared to the other $NaNO_2$-Zadmixed steel-reinforced concretes. This showed that the blank samples in the study represent severe condition of corrosive test-system which finds agreement with the preferred practice prescribed by Roberge (2003) for reducing the time for effect to be observed.

The other statistical models that are based on the Weibull pdf are shown in Fig. 2. These include the plot of the Weibull mean models of corrosion rate on which the plot of the noise resistance was superimposed in Fig. 2a and the plot of the K-S goodness-of-fit testing of the compatibility of the measured corrosion test-data to the Weibull pdf in Fig. 2b. In agreement to the inference from Fig. 1, Fig. 2a shows that the corrosion rate model by the Weibull pdf for the 0M $NaNO_2$-admixed (blank) samples also portrayed prevalence of severe corrosion condition above that which obtains from the other $NaNO_2$-admixed samples. Also, notable from Fig. 2a was that the plots of the noise resistance that was also shown in the figure exhibited general trend of lower noise resistance attending higher corrosion rate. This finds agreement with the findings from Kelly *et al.* (1996), where noise resistance was observed as generally tracking the polarisation resistance, which is usually employed for computing corrosion rate. This bears suggestions of the existence of some sort of relation that could be correlated between the corrosion rate and the noise resistance response from the $NaNO_2$-admixed steel-reinforced concretes studied in this work.

Avoiding erroneous conclusion from the Weibull detailing of the corrosion test-results in the study however require that the measured test-data from the corrosion monitoring techniques were

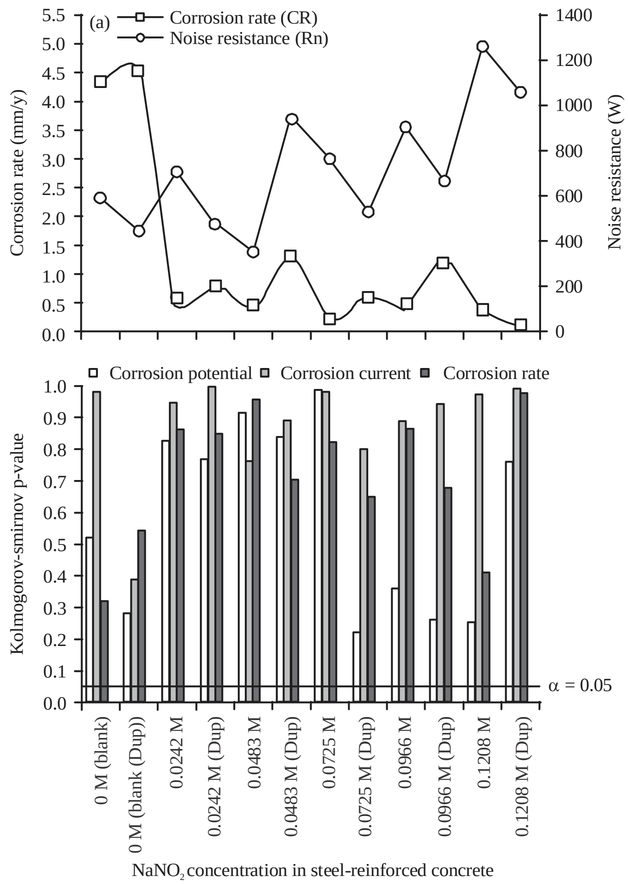

Fig. 2(a-b): Other statistical models based on the Weibull pdf (a) Corrosion rate and noise resistance plots and (b) Kolmogorov-Smirnov goodness-of-fit testing of data compatibility with the Weibull pdf

distributed like the Weibull pdf (ASTM., 2005b; Roberge, 2003). This was ascertained by the K-S GoF test-results in Fig. 2b, where linear plots of significant level, $\alpha = 0.05$, has been included for directly interpreting test-data scattering like the Weibull pdf. This figure shows that the K-S p-value is >0.05 for the measured test-data from the corrosion monitoring techniques that were employed for the $NaNO_2$-admixed steel-reinforced concrete specimens. This implies that each measured datasets of the corrosion monitoring techniques comes from the Weibull distribution, thus bearing supports for the use of the Weibull pdf as the descriptive statistics for detailing the corrosion condition in the concrete samples.

Correlation fitting and analysis of corrosion test-results: Several correlation fittings were employed and studied with fitting performances of correlation coefficient, r and analysis of variance (ANOVA) of the resultant relationship between the dependent and independent variables being studied. Thus, by using Corrosion Rate (CR) as independent variable this finds correlation fitting with the noise resistance (R_n) and the admixed $NaNO_2$ concentration in the steel-reinforced concretes (ζ) as dependent variable that assume the form:

$$CR = 10^3\left\{1.62e^{-\zeta}\left[1-0.016\left(1/\ln R_n\right)\right]+22.31\zeta\left[\left(1/R_n\right)+0.89\left(1/\ln R_n\right)-2.96\left(1/\ln R_n\right)^2\right]-1.61\right\} \quad (10)$$

Table 2: One-way analysis of variance of the correlation fitting model

Source of variations	df	SS	MS	F	p-value
Regression	5	20.64	4.13	4.89	0.04
Residuals	6	5.07	0.84		
Totals	11	25.71			

df: Degrees of freedom, SS: Sum of squares, MS: Mean square, F: Ratio of two mean squares

For this correlation fitting model, the correlation coefficient, $r = 89.61\%$. The results of the one-way analysis of variance modelling based on this correlation fitting are presented in Table 2. From the table, the ANOVA p-value = 0.04 which implies that it could not be rejected that there is significant relationship between the dependent variable (CR) and the independent variables (R_n) and (ζ).

Inhibition efficiency from the experimental and predicted models: The inhibition efficiency modelling from the applications of Eq. 9 to the experimental and the predicted models of CR are presented in ranking order in of admixed $NaNO_2$ effectiveness in Fig. 3. Both the experimental model, Fig. 3a and the predicted model from the correlation fitting, Fig. 3b, of $NaNO_2$ effectiveness ranking showed agreements in the inhibition efficiency estimated versus (vs) the blank sample and the inhibition efficiency estimated vs the blank duplicate sample.

Importantly worth noting from Fig. 3a includes the fact that the experimental model identified the 0.1208 M $NaNO_{2(Dup)}$ with optimal effectiveness, η_exp (vs blank) = 97.15% or η_exp (vs blank$_{(Dup)}$) = 97.27%. However, despite the fact that the 0.1208 M $NaNO_{2(Dup)}$ did not rank as the optimally effective admixture in the predicted model in Fig. 3b, the predicted model still find comparisons to the experimental model by identifying the 0.1208 M $NaNO_2$ as the most effective admixture. This other 0.1208 M $NaNO_2$ admixture exhibited η_pred (vs blank) = 96.89% or η_pred (vs blank$_{(Dup)}$) = 96.73% at inhibiting concrete steel-reinforcement corrosion. By these, the experimental and the predicted models identified the 0.1208 M $NaNO_2$ admixed concretes with inhibition efficiency $\eta > 90\%$ even as both models also identified the other $NaNO_2$ admixtures with highly positive effectiveness at inhibiting steel-rebar corrosion.

These results find agreements with literature (Mennucci *et al*., 2009; Vaysburd and Emmons, 2004), where $NaNO_2$ has been identified as effective inhibitor, although none of these investigated the correlation fitting agreements with the experimental models of the noise resistance with corrosion rate as was done in this study. These bear implications that the measurements of the corrosion potential that had been identified in studies (Song and Saraswathy, 2007; Elsener *et al*., 2003) as not correlating with corrosion rate could find corrosion rate correlation if combined with other measurements as had been suggested by those studies. Such other measurement could simply be that of the corrosion cell current, by ZRA, versus the same reference electrode that was employed for the OCP measurement. This form of correlation could have been made possible by the identification of such current as being proportional to the dissolution model of the steel-rebar (iron) in the corrosive medium by McCarter and Vennesland (2004). In that report also, McCarter and Vennesland (2004) identified the measurement of this form of galvanic current as technically simple to undertake compared to that of the corrosion rate by the polarisation resistance technique. By this, the kind of correlation fitting model, from this study, bearing significant relationship between the CR by LPR with the noise resistance could find usefulness and applications for studying inhibition effectiveness of novel admixtures that are not yet well known.

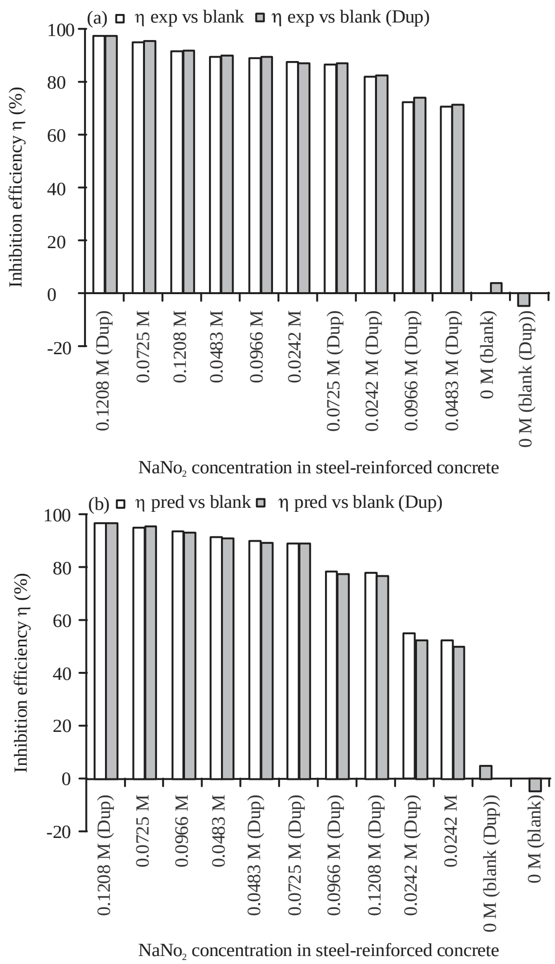

Fig. 3(a-b): Inhibition efficiency models in ranking order performance by the admixed $NaNO_2$ concentrations in steel-reinforced concrete samples (a) By the experimental model and (b) By the prediction from the correlation fitting model

CONCLUSION

Corrosion rate and noise resistance correlation from $NaNO_2$ admixed steel-reinforced concrete has been investigated in this study. From this the following conclusions can be drawn:

- The corrosion potential, corrosion current and corrosion rate test-data measurements from the $NaNO_2$-admixed concretes followed the Weibull probability distribution function at $\alpha = 0.05$ significant level, according to the Kolmogorov-Smirnov goodness-of-fit statistics, this supports the use of the Weibull pdf as the descriptive statistics for detailing the corrosion test-performance of the $NaNO_2$ admixtures
- The corrosion rate from linear polarisation resistance instrument correlated with the noise resistance test-data obtained from the ratio of standard deviation of corrosion potential to that of corrosion cell current as well as the concentration of $NaNO_2$ admixture. This correlation fitting exhibited correlation coefficient, r = 89.61% and ANOVA p-value = 0.04 indicating that the relationship between the dependent variable (corrosion rate) and independent variables (noise resistance and concentration of $NaNO_2$ admixture) is significant

- Both experimental and correlation fitting models identified 0.1208 M $NaNO_2$ admixed steel-reinforced concretes with optimal efficiencies at inhibiting reinforcing steel corrosion. By the experimental model the 0.1208 M $NaNO_{2(Dup)}$ exhibited η_exp (vs blank) = 97.15% or η_exp (vs $blank_{(Dup)}$) = 97.27%, while by the predicted model, from the correlation fitting, the 0.1208 M $NaNO_2$ exhibited η_pred (vs blank) = 96.89% or η_pred (vs $blank_{(Dup)}$) = 96.73% at inhibiting reinforcing steel corrosion in the NaCl-immersed concretes
- The agreements between the experimental and the predicted models, of positive effectiveness of the admixed $NaNO_2$ concentrations at inhibiting steel-reinforcement corrosion in concrete samples immersed in the chloride contaminated solution, bear indication of the use of corrosion potential and corrosion cell current measurements for evaluating noise resistance as useful and more simpler measurements that is potent at exhibiting correlation with corrosion rate. These results suggest that the technically simple measurements of corrosion potential and corrosion cell current could find usefulness and applications for ascertaining effectiveness of novel and not yet well-known admixtures at inhibiting concrete steel-reinforcement corrosion in aggressive medium

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