METHODS OF ASTM G16 AND CONFLICTS IN CORROSION TEST DATA: CASE STUDY OF NANO₂ EFFECTIVENESS ON STEEL-REBAR CORROSION

*Okeniyi JO¹, Okpala SO¹, Omoniyi OM¹, Oladele IO¹, Ambrose IJ¹, Menkiti MC², Loto CA¹ and Popoola API³ ¹Mechanical Engineering Department, Covenant University, Ota, Nigeria

² Chemical Engineering Department, Nnamdi Azikiwe University, Awka, Nigeria

³Chemical and Metallurgical Engineering Department, Tshwane University of Technology, Pretoria, South Africa

ABSTRACT

In this paper, applications of the methods of ASTM G16 for addressing inherent conflicts in laboratory measurements of corrosion test data were studied, using the inhibiting effect of NaNO₂ on the corrosion of concrete steel-rebar for the case study. For this, electrochemical monitoring techniques were employed for studying effectiveness of different concentrations of NaNO₂ admixture in replicated concrete samples immersed in NaCl and in H₂SO₄ media for an experimental period of sixty-eight days. The corrosion test data from this experimental setup were subjected to the probability density fittings of the Normal and the Weibull functions as well as to significance testing methods of ASTM G16-99 R04 specifications. Results identified 10g (0.1208M) NaNO₂ admixture with optimal inhibition efficiency model, $\eta = 88.38\pm4.62\%$, in the saline/marine simulating environment and the 8 g (0.0966M) NaNO₂ admixture with optimum effectiveness, $\eta = 13.51\pm83.48\%$, in the acidic environment. The techniques of ASTM G16 adequately identified and addressed conflicting effectiveness from the test data of NaNO₂ admixtures in the studied test environments.

Keywords: Steel-rebar, corrosion inhibition, normal and weibull pdf's, statistical tests.

INTRODUCTION

Corrosion of concrete steel-rebar affects durability of reinforced concrete structures and infrastructures which is generating safety and socio-economic concerns globally (Dong et al., 2012; Tang et al., 2012; Ormellese et al., 2009). Environmental agents of corrosion such as carbonation, chloride ingress and acidic sulphate attack renders steel-rebar in concrete susceptible to corrosion damage by reducing concrete alkalinity and initiating destruction of protective passive layer from cement hydration (Tang et al., 2012; Tommaselli et al., 2009). Methods proposed in studies for mitigating steel-rebar corrosion had include practices for improving concrete durability and measures of preventing concrete steel rebar corrosion (Dong et al., 2012; Shi et al., 2012). For these, use of corrosion inhibitor had been identified as an easy and economical method for preventing reinforcing steel corrosion and prolonging service life of reinforced concrete structures (Ormellese et al., 2009; Tommaselli et al., 2009).

In spite of the ease and cost effectiveness advantages of corrosion inhibitors for mitigating rebar corrosion, its responsible application requires satisfying the critical demand of its presence in suitable concentration for it to be effective in the corrosive environment (Söylev and Richardson, 2008). This dependency of inhibitor effectiveness its suitable concentration in the corrosive system has lead to prevalence of contradictory effectiveness reports in literature (Feng *et al.*, 2011; Ormellese *et al.*, 2009; Söylev and Richardson, 2008; Jamil *et al.*, 2003). Even, well known inhibitors and commercialised ones were not spared from these (Królikowski and Kuziak, 2011; Ormellese *et al.*, 2006; Jamil *et al.*, 2003). While some studies reported effective corrosion inhibitions, others had requested need for further research. Reasons identified by Ormellese *et al.* (2009) for these include lack of standard procedure for evaluating effectiveness and non-availability of reliable test data.

The standard of the American Society of Testing and Materials, ASTM, designation G16 had recognised that corrosion test data could be characterised by inherent scatter that would make interpretation difficult for investigators. These scatter, according to the standard, usually originate from variety of factors which instigate stochastic deviations of measured values from the expected values of the prevailing condition. Without a standard procedure for addressing these randomised deviations, in corrosion test data, interpretations of such data could be contradictory. However, ASTM G16 had proposed statistical evaluation procedures that could be used, as the rational approach, for tackling difficulties arising from unaccounted factors which induce

^{*}Corresponding author email: joshua.okeniyi@covenantuniversity.edu.ng

randomised scatter in corrosion test data.

In spite of these, there is paucity of study where ASTM G16 methods had been employed for analysing corrosion test data for the study of corrosion inhibition of concrete steel-rebar. While Castrellon-Uribe et al. (2008) and Cuevas-Arteaga (2008) had employed ASTM G16 for analysing electrochemical noise test data from the corrosion of stainless steel and of high temperature HK-40m alloy, respectively, none of these involve corrosion inhibition. No study has used the procedures of this standard for investigating NaNO₂ effect on reinforcing steel corrosion. This study therefore examines the applications of ASTM G16-99 R04 methods for studying corrosion inhibition of sodium nitrite (NaNO₂) admixture in steel reinforced concrete. The interests in the work include employing inhibiting properties of NaNO₂ admixture on rebar corrosion to study how procedures of ASTM G16 could be employed for addressing or identifying conflicts in corrosion test results of steel reinforcement in concrete.

MATERIALS AND METHODS

Materials

Steel reinforced concretes and inhibitor admixtures

Ø12mm deformed steel reinforcement was obtained from Federated Steel Rolling Mills, Ota, Ogun State, Nigeria, for use in this study. This rebar, having 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, was cut into 190mm rods for each specimen.

Twenty-eight reinforced concrete block samples used for the experiment were produced as replicated "_R" blocks (Haynie, 2005), each of size $100 \text{mm} \times 100 \text{mm} \times 200 \text{mm}$. Embedded in each block was 150mm length of the steel rebar which was symmetrically placed across the width of the block leaving 40mm steel protrusion for electrochemical connections. This protrusion was painted with glossy paint for each block. The formulation used for the reinforced concrete specimens was 300.0kg/m^3 of cement, 149.7kg/m^3 of water, 890.6kg/m^3 of sand, and 1106.3kg/m^3 of granite stones. The water/cement (w/c) ratio was 0.499. The concentration of NaNO₂ admixed in each of these reinforced concrete samples are shown in Table 1, in which the aggressive solution of their immersion were also indicated.

Experimental Procedures

Corrosion test setup and electrochemical test measurements

Steel reinforced concrete samples were partially immersed, longitudinally, in plastic bowls containing solution of corrosive test environments. Each of the first replicated set of fourteen samples was partially immersed in 3.5% NaCl solution and the second replicated set in $0.5M H_2SO_4$ solution. In each bowl, the test solution was made up to just below the reinforcing steel rebar but was not touching it.

Non-destructive electrochemical measurements (Song and Saraswathy, 2007; Broomfield, 1997) were taken from the experimental setup, first, in five days interval for forty days and thereafter in seven days interval for the following four weeks. This totals sixty-eight days experimental period. The electrochemical test methods employed include:

- i. Half-cell potential (HCP) according to ASTM C876-91 R99, versus Cu/CuSO₄ electrode (CSE), using a high impedance digital multimeter;
- ii. Corrosion cell current (CCC) in the concrete-test solution system, versus CSE, using zero resistance ammeter (McCarter and Vennesland, 2004; Broomfield, 1997);

Table 1. Concentrations of NaNO2 admixed in sample of steel reinforced concretes.

S.No	Admixture in concrete	S.No	Admixture in concrete	S.No	Admixture in concrete	S.No	Admixture in concrete
-							
1.	0 g NaNO ₂ (Control in	2.	0 g NaNO ₂ (Control in	3.	0 g NaNO ₂ (Control in	4.	0 g NaNO ₂ (Control in
	NaCl)		H_2SO_4)		NaCl R)		H_2SO_4 R)
5.	2 g (0.0242M) NaNO ₂	6.	2 g (0.0242M) NaNO ₂	7.	2 g (0.0242M) NaNO ₂	8.	2 g (0.0242M) NaNO ₂
	in NaCl		in H ₂ SO ₄		in NaCl R		in H_2SO_4 R
9.	4 g (0.0483M) NaNO ₂	10.	4 g (0.0483M) NaNO ₂	11.	4 g (0.0483M) NaNO ₂	12.	4 g (0.0483M) NaNO ₂
	in NaCl		in H ₂ SO ₄		in NaCl R		in H_2SO_4 R
13.	6 g (0.0725M) NaNO ₂	14.	6 g (0.0725M) NaNO ₂	15.	6 g (0.0725M) NaNO ₂	16.	6 g (0.0725M) NaNO ₂
	in NaCl		in H ₂ SO ₄		in NaCl R		in H ₂ SO ₄ R
17.	8 g (0.0966M) NaNO ₂	18.	8 g (0.0966M) NaNO ₂	19.	8 g (0.0966M) NaNO ₂	20.	8 g (0.0966M) NaNO ₂
	in NaCl		in H ₂ SO ₄		in NaCl R		in H_2SO_4 R
21.	10 g (0.1208M)	22.	10 g (0.1208M)	23.	10 g (0.1208M)	24.	10 g (0.1208M)
	NaNO ₂ in NaCl		NaNO ₂ in H ₂ SO ₄		NaNO ₂ in NaCl R		$NaNO_2$ in H_2SO_4 R
25.	16 g (0.1932M)	26.	16 g (0.1932M)	27.	16 g (0.1932M)	28.	16 g (0.1932M)
	NaNO ₂ in NaCl		NaNO ₂ in H ₂ SO ₄		NaNO ₂ in NaCl_R		NaNO ₂ in H ₂ SO ₄ _R
		1		1		1	

iii. Corrosion rate (CR) measurements through direct instrument conversion to mpy using the threeelectrode LPR Data Logger, Model MS1500L (Sastri, 2011).

For ensuring good electrical contact for these measurements, a water retentive conducting sponge was employed for the point of contact of the measuring probes and the reinforced concrete (Song and Saraswathy, 2007). This sponge was wetted before measurements using solution containing drinkable tap water, iso-propyl alcohol and detergent (ASTM C876-91 R99).

Data Analysis

Probability distribution modelling

Each measured variable of electrochemical test data, x, were subjected to the statistical analysis of the Normal and the Weibull probability density functions, pdf's (Okeniyi *et al.*, 2013; Roberge, 2008; Roberge, 2005). These have respective cumulative density function given by:

$$F(x) = \frac{1}{\sigma (2\pi)^{\frac{1}{2}}} \int_{-\infty}^{x} \exp\left[-\left(\frac{x-\mu}{2\sigma^{2}}\right)^{2}\right] dx$$
(1)

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

Where μ and σ are the mean and standard deviation of the Normal model, k and c are the shape parameter and the scale parameter of the Weibull model. Estimations of k and c were obtained, for sample size n, from the maximum likelihood equations (Kvam and Lu, 2006):

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

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

These estimates were used to compute the Weibull mean model, μ_W , as (Omotosho et al, 2010)

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

Where $\Gamma(\cdot)$ is the gamma function of (\cdot) .

Goodness-of-fit and significance of difference tests

As prescribed by ASTM G16-95 R04, compatibility of each variable of electrochemical data to the Normal and the Weibull distribution functions were verified using the Kolmogorov–Smirnov (K-S) goodness-of-fit (GoF) test statistics at $\alpha = 0.05$ significant level (Okeniyi *et al.*, 2013; Okeniyi and Okeniyi, 2012; Omotosho *et al.*, 2010; Roberge, 2005). This test measures the absolute difference between empirical distribution function $F^*(x)$ and theoretical distribution function F(x), for *n* data points, through the statistics (Okeniyi and Okeniyi, 2012)

$$D_{n} = D(x_{1},...,x_{n}) = \sup_{-\infty < x < \infty} \left| F^{*}(x) - F(x) \right|$$
(6)

Also from ASTM G16-95 R04, differences of central tendency evaluations for the replicated samples of each NaNO₂ admixtures, 0g to 16g, were studied for significance using student's *t*-test statistics. These include the *t*-test with equal variance (EV) and that with unequal

Table 2. Mean of corrosion test data by the Normal and the Weibull pdf models for samples in NaCl medium.

		No	rmal distribut	ion	Weibull distribution			
S No	Admixture concentration in concrete	HCP	CCC	CR	HCP	CCC	CR	
5.110.	Admixture concentration in concrete	(-mV CSE)	(-µA)	(mm/y)	(-mV CSE)	(-µA)	(mm/y)	
		$(\mu_{N-\text{HCP}})$	$(\mu_{N-\text{CCC}})$	(μ_{N-CR})	$(\mu_{W-\text{HCP}})$	(μ_{W-CCC})	(μ_{W-CR})	
1.	0g NaNO2 (Ctrl) in NaCl	597.31	647.00	2.354	590.38	658.82	5.278	
2.	0g NaNO2 (Ctrl) in NaCl_R	567.62	710.88	2.360	560.34	718.46	5.415	
3.	2g NaNO ₂ in NaCl	523.23	200.81	1.149	518.66	201.13	1.564	
4.	2g NaNO ₂ in NaCl_R	483.72	187.96	1.345	482.84	192.75	1.821	
5.	4g NaNO ₂ in NaCl	512.54	224.85	0.743	508.98	225.71	0.876	
6.	4g NaNO ₂ in NaCl_R	510.51	230.54	1.348	508.04	232.89	1.925	
7.	6g NaNO ₂ in NaCl	444.77	165.65	0.383	443.94	165.56	0.511	
8.	6g NaNO ₂ in NaCl_R	535.38	290.54	0.796	527.07	290.96	1.013	
9.	8g NaNO ₂ in NaCl	447.64	186.66	0.729	446.99	190.39	1.038	
10.	8g NaNO ₂ in NaCl_R	469.44	199.95	0.846	465.12	205.76	1.360	
11.	10g NaNO ₂ in NaCl	356.85	111.82	0.576	355.26	111.86	0.835	
12.	10g NaNO ₂ in NaCl_R	484.03	146.08	0.311	483.90	146.37	0.407	
13.	16g NaNO ₂ in NaCl	496.26	255.88	0.916	489.50	254.56	1.199	
14.	16g NaNO ₂ in NaCl_R	368.31	175.61	0.606	368.23	175.95	0.827	

variance (UV) assumptions. Significance of differences of variability was investigated using one-way (factorial) analysis of variance, ANOVA. These methods test whether differences encountered in replicated samples (the *t*-test) or in concrete samples with different admixtures (the factorial ANOVA) were significant or due to chance or experimental complexities/error.

Inhibition efficiency

Inhibition efficiency model, η , was evaluated for the replicates of NaNO₂ admixed samples relative to the replicates of control samples, through the formula (Obot *et al.*, 2010):

$$\eta = \frac{\mu_{ctrl} - \mu_{inh}}{\mu_{ctrl}} \times 100 \tag{7}$$

RESULTS AND DISCUSSION

Corrosion test data modelling and analyses

Results of corrosion test data by the Normal and the Weibull distribution models are presented, for samples immersed in NaCl in table 2 and samples in H_2SO_4 in Table 3. From the pdf models, the Weibull mean were underestimated, compared to the Normal mean, for the HCP of all samples and the CCC of two other samples (6g and 16g) in NaCl, for the HCP of a replicate of 2g as well as the CR of a replicate of 4g sample in H_2SO_4 medium. Apart from these, the Weibull mean for the test variables

Table 3. Mean of corrosion test data by the Normal and the Weibull pdf models for samples in H₂SO₄ medium.

		Nor	mal distribu	tion	Weibull distribution			
		HCP			HCP			
S. No.	Admixture concentration in concrete	(-mV	CCC	CR	(-mV	CCC	CR	
		CSE)	(-µA)	(mm/y)	CSE)	(-µA)	(mm/y)	
		$(\mu_{N-\text{HCP}})$	$(\mu_{N-\text{CCC}})$	(μ_{N-CR})	(μ_{W-HCP})	(μ_{W-CCC})	(μ_{W-CR})	
1.	0g NaNO ₂ (Ctrl) in H ₂ SO ₄	257.41	77.65	0.270	265.37	83.19	0.316	
2.	0g NaNO ₂ (Ctrl) in H ₂ SO ₄ _R	451.59	146.49	0.181	472.17	146.53	0.198	
3.	2g NaNO ₂ in H ₂ SO ₄	333.08	56.94	0.145	334.27	57.41	0.163	
4.	2 g NaNO ₂ in H ₂ SO ₄ _R	72.64	6.05	0.641	71.49	6.25	0.838	
5.	4g NaNO ₂ in H ₂ SO ₄	132.87	23.97	0.216	142.33	24.35	0.226	
6.	4 g NaNO ₂ in H ₂ SO ₄ _R	70.49	12.80	0.460	71.31	13.03	0.434	
7.	6g NaNO ₂ in H ₂ SO ₄	192.36	44.39	0.423	192.47	44.96	0.571	
8.	6 g NaNO ₂ in H ₂ SO ₄ _R	109.82	20.13	0.470	110.99	20.44	0.634	
9.	8g NaNO ₂ in H ₂ SO ₄	102.33	15.01	0.044	104.44	15.07	0.046	
10.	8g NaNO ₂ in $H_2SO_4_R$	119.56	16.74	0.272	120.59	16.90	0.375	
11.	10g NaNO ₂ in H ₂ SO ₄	424.62	104.75	0.804	440.99	107.38	1.018	
12.	10g NaNO ₂ in H ₂ SO ₄ _R	261.31	44.95	0.617	277.57	45.81	0.795	
13.	16g NaNO ₂ in H ₂ SO ₄	136.82	37.37	0.111	175.99	37.41	0.115	
14.	16g NaNO ₂ in H ₂ SO ₄ _R	208.44	54.97	0.487	209.21	55.41	0.525	

Table 4. ANOVA of corrosion test data of concrete samples.

T (Deg	Sam	ples in NaC	l medium	1	Samples in H ₂ SO ₄ medium			
Test	Source of	of								
Test Data HCP CCC CR	variations	Free dom	SS	MS	F	<i>p</i> -value	SS	MS	F	<i>p</i> -value
НСР	Treatments	13	757509.07	58269.93	2.6610	0.0021	2650592.16	203891.70	14.8553	0.0000
	Residuals	168	3678883.21	21898.11			2305834.63	13725.21		
	Totals	181	4436392.29				4956426.79			
	Treatments	13	5513494.09	424114.93	9.5242	0.0000	266171.70	20474.75	12.2663	0.0000
CCC	Residuals	168	7481092.64	44530.31			280423.73	1669.19		
	Totals	181	12994586.73				546595.43			
	Treatments	13	69.32	5.33	1.8023	0.0462	8.58	0.66	0.8080	0.6512
CR	Residuals	168	497.02	2.96			137.22	0.82		
	Totals	181	566.33				145.79			

of the remaining samples in both media were overestimated compare to their Normal mean. For the CR (mm/y) of the control samples in NaCl medium the Weibull mean overestimations, ($\mu_W = 5.278$; $\mu_{W(rep)} =$ 5.415), more than doubled the Normal mean modelled for these samples, ($\mu_N = 2.354$; $\mu_{N(rep)} = 2.360$). However, other overestimations by the Weibull were in moderate proportions compare to their Normal values.

Kolmogorov-Smirnov goodness-of-fit test results

The graphical representation of the K-S GoF *p*-values was as shown in figure 1, for samples in NaCl, figure 1(a) and in H₂SO₄, figure 1(b). These also include the significant level $\alpha = 0.05$ for identifying test data which follow/did not follow the pdf models. From the figure, it could be observed that all data of the test variables of the studied samples in both test media, scattered like the Weibull pdf. While all the HCP and the CCC test data also followed the Normal pdf, in both test media, eleven, out of the twenty-eight test samples being investigated, had the test data of CR not following the Normal pdf, according to the K-S GoF test statistics. In spite of the CR overestimations of Weibull mean for the replicated control samples in NaCl medium, compared to the Normal, the CR test data of these samples followed the Weibull pdf even with higher K-S *p*-values (0.169, 0.176_(rep)) than that from the Normal (0.063, $0.074_{(rep)}$).

The Kolmogorov-Smirnov GoF tests results were in agreements for many of the replicated samples studied. However, discrepancies in results still abound that could lead to conflicting interpretations of results even between replicates of concrete samples, with the same NaNO₂ admixtures that agreed in following/not following, the pdf models employed. Addressing these conflicts require needs of testing if these differences, encountered within replicates of samples, were significant or not through the use of the student's *t*-test statistics.

Significance of difference testing between replicates Significance test results, obtained through the student's *t*-



Fig. 1. Results of K-S goodness of fit tests for concrete specimens in (a) NaCl medium (b) H₂SO₄ medium.

test statistics, of test data differences between replicated concrete samples are presented in figure 2, for each replicated NaNO₂ admixed concretes in the NaCl, figure 2(a), and H₂SO₄ media, figure 2(b). These also include the significant level $\alpha = 0.05$ for directly identifying significant difference.

Figure 2(a) showed that the differences encountered in the corrosion test data of the replicated samples of the control, the 2g NaNO₂, the 4g NaNO₂ and the 8g NaNO₂ in NaCl medium were not significant. In the H₂SO₄, figure 2(b), medium, only the replicated samples with 8g NaNO₂ admixture exhibited differences that were not significant, according to the student's *t*-test. The CCC data of the replicated samples with 6g NaNO₂ and the HCP data of the 10g NaNO₂ replicates as well as replicates of 16g NaNO₂ had differences that were significant, in the NaCl medium. All concrete samples

immersed in H_2SO_4 had HCP and CCC test data of their replicates exhibiting differences that were significant except all the corrosion test data of samples with 8g NaNO₂ and the CCC data of the 6g NaNO₂ samples. All these were invariant for the *t*-test with equal variance and the *t*-test with unequal variance assumptions.

The corrosion rate (CR) test data for all replicated samples in this study had differences between the replicates of their samples that were due to chance but that were not significant, in their respective test environments, figure 2(a) and figure 2(b). All these constitute statistical bases for differences between replicate samples with same admixture concentration and from this the differences among samples with different admixture concentrations could be investigated using ANOVA, according to specifications of ASTM G16.



Fig. 2. Results of student's *t*-test statistics for replicated concrete samples in (a) NaCl medium (b) H₂SO₄ medium.



Fig. 3. Ranking of inhibition efficiency for modelling inhibitor admixture effectiveness vs. each replicate of control samples: (a) replicates in NaCl medium; (b) replicates in H_2SO_4 medium.



Fig. 4. Ranking of averaged inhibition efficiency for NaNO₂ admixtures (a) NaCl medium; (b) H₂SO₄ medium.

Analysis of variance among different admixture concentrations

The one-way factorial ANOVA results for the variables of corrosion test data of concrete samples, in their test media, are presented in table 4. From this table, it could be deduced that differences were significant in HCP test data (NaCl: p = 0.0021, H₂SO₄: p < 0.0001) and in CCC test data (NaCl medium: p < 0.0001, H₂SO₄ medium: p < 0.0001, H₂SO₄ medium: p < 0.0001) of samples with different NaNO₂ admixtures. However, the CR test data exhibited differences that were significant, p = 0.0462, for samples in NaCl medium only, while modelled CR test data were not significantly different, p = 0.6512, among concrete samples in H₂SO₄ medium.

Inhibition effectiveness modelling for NaNO₂ admixtures

The CR test data followed the Weibull pdf for all samples, figure 1. Therefore the Weibull mean of CR data (μ_{W-CR}) was employed for modelling inhibition efficiency, η . These are presented in figure 3, in ranking order of effectiveness for samples in NaCl, figure 3(a), and in H₂SO₄, figure 3(b). Figure 3(a) showed that the 10g NaNO₂R (replicate concrete) sample had optimum inhibition efficiency, $\eta = 92.29\%$ relative to the control and $\eta_{(rep)} = 92.49\%$ relative to the control replicate, in NaCl medium. Also, inhibition by the other NaNO₂ admixtures were strong such that the least effective admixture in the medium, the 4g NaNO₂ sample, still had $\eta = 63.53\%$, $\eta_{(rep)} = 64.56\%$.

In the H₂SO₄ medium, the 8g NaNO₂ sample lead the effectiveness ranking, figure 3(b) with $\eta = 85.56\%$, $\eta_{(rep)} = 76.92\%$, however, its replicate sample, 8g NaNO₂ (Rep), exhibited negative inhibition efficiency of $\eta = -18.69\%$, $\eta_{(rep)} = -89.73\%$. Also, the other two admixtures with positive inhibition effectiveness, the 16g NaNO₂ and the 2g NaNO₂ samples, had their replicate samples with much

higher negative inhibition efficiencies in the acidic test environment. These results bear implications of the identification of conflicting effectiveness. These seemed to have been presaged by the lack of significant difference, from the ANOVA modelling, among the CR test data of samples with different admixture concentrations in the acidic medium.

The student's *t*-test application on CR test data upheld that the differences between the replicate samples studied were not significant. Thus, it is of interest to moderate their differences through averaging model of inhibition efficiency for each replicated samples (Obot *et al.*, 2010; Haynie, 2005). Results of these are shown for samples in NaCl, figure 4(a) and in H₂SO₄, figure 4(b). In figure 4(a), the averaged model of inhibition efficiency showed that the 10g NaNO₂ admixture retailed optimal inhibition effectiveness, $\eta = 88.38\pm4.62\%$, in the NaCl medium, while the 2g NaNO₂ now ranked as the least effective admixture behind the 4g NaNO₂ admixture.

The averaged inhibition effectiveness model re-affirmed that most NaNO₂ admixtures studied aggravated steelrebar corrosion in H₂SO₄ medium, figure 4(b). In this medium, only 8g NaNO₂ admixture had resultant positive effectiveness of $\eta = 13.51\pm83.48\%$ that was even characterised with variability which ranged highly into the negative inhibition region. All the other NaNO₂ admixtures in the medium exhibited negative efficiency whereby that of the 10g NaNO₂ admixture was as low as $\eta = -272.63\pm112.93\%$. By this, sodium nitrite, an oxidising agent, tends to accelerate corrosion in the H₂SO₄ medium as expressed by Davis (2000). Further studies are needed on corrosion inhibiting admixture for concrete steel-rebar in acidic medium.

CONCLUSION

Techniques of ASTM G16-95 R04 identified prevalent corrosion inhibition of concrete steel-rebar by $NaNO_2$ admixtures in NaCl medium while the methods also identified and addressed conflicting inhibition effect by the admixture on concrete steel-rebar in H_2SO_4 medium.

REFERENCES

ASTM C876-91 R99. Standard test method for half-cell potentials of uncoated reinforcing steel in concrete. ASTM International, West Conshohocken, PA.

ASTM G16-95 R04. Standard guide for applying statistics to analysis of corrosion data. ASTM International, West Conshohocken, PA.

Broomfield, JP. 1997. Corrosion of steel in concrete: Understanding, investigation and repair. E & FN Spon, London.

Castrellon-Uribe, J., Cuevas-Arteaga, C. and Trujillo-Estrada, A. 2008. Corrosion monitoring of stainless steel 304L in lithium bromide aqueous solution using transmittance optical detection technique. Opt. Lasers Eng. 46(6):469-476.

Cuevas-Arteaga, C. 2008. Corrosion study of HK-40m alloy exposed to molten sulfate/vanadate mixtures using the electrochemical noise technique. Corros. Sci. 50(3):650-663.

Davis, JR. 2000. Types of corrosive environments. In: Corrosion: understanding the basics. Ed. Davis, JR. ASM International, Materials Park, OH. 193-236.

Dong, S., Zhao, B., Lin, C., Du, R., Hu, R. and Zhang, GX. 2012. Corrosion behavior of epoxy/zinc duplex coated rebar embedded in concrete in ocean environment. Constr. Build. Mater. 28:72-78.

Feng, L., Yang, H. and Wang, F. 2011. Experimental and theoretical studies for corrosion inhibition of carbon steel by imidazoline derivative in 5% NaCl saturated Ca(OH)₂ solution. Electrochim. Acta 58:427-436.

Haynie, FH. 2005. Statistical treatment of data, data interpretation, and reliability. In: Corrosion Tests and Standards: Application and Interpretation. Ed. Baboian, R. ASTM International, West Conshohocken, PA. 83-88.

Jamil, HE., Montemor, MF., Boulif, R., Shriri, A. and Ferreira, MGS. 2003. An electrochemical and analytical approach to the inhibition mechanism of an aminoalcohol-based corrosion inhibitor for reinforced concrete. Electrochim. Acta 48:3509-3518. Królikowski, A. and Kuziak, J. 2011. Impedance study on calcium nitrite as a penetrating corrosion inhibitor for steel in concrete. Electrochim. Acta, doi:10.1016/j.electacta.2011.01.069.

Kvam, P. and Lu, JC. 2006. Statistical reliability with applications. In: Springer handbook of engineering statistics. Ed. Pham, H. Springer-Verlag, London. 49-61.

McCarter, WJ. and Vennesland, Ø. 2004. Sensor systems for use in reinforced concrete structures. Constr. Build. Mater. 18:351-358.

Obot, IB., Obi-Egbedi, NO. and Odozi, NW. 2010. Acenaphtho [1,2-b] quinoxaline as a novel corrosion inhibitor for mild steel in 0.5 M H_2SO_4 . Corros. Sci. 52:923-926.

Okeniyi, JO. and Okeniyi, ET. 2012. Implementation of Kolmogorov-Smirnov p-value computation in Visual Basic®: implication for Microsoft Excel® library function. J. Stat. Comput. Simul. 82(12):1727-1741. doi:10.1080/00949655.2011.593035.

Okeniyi, JO., Obiajulu, UE., Ogunsanwo, AO., Odiase, NW. and Okeniyi, ET. 2013. CH_4 emission model from the waste of Sus Domesticus and Gallus Domesticus in Nigerian local farms: environmental implications and prospects. Mitig. Adapt. Strateg. Glob. Chang. 18(3):325-335. doi: 10.1007/s11027-012-9365-7.

Omotosho, OA., Okeniyi, JO. and Ajayi, OO. 2010. Performance evaluation of potassium dichromate and potassium chromate inhibitors on concrete steel rebar corrosion. J. Fail. Anal. Prev. 10:408-415.

Ormellese, M., Berra, M., Bolzoni, F. and Pastore, T. 2006. Corrosion inhibitors for chlorides induced corrosion in reinforced concrete structures. Cem. Concr. Res. 36:536-547.

Ormellese, M., Lazzari, L., Goidanich, S., Fumagalli, G. and Brenna, A. 2009. A study of organic substances as inhibitors for chloride-induced corrosion in concrete. Corros. Sci. 51:2959-2968.

Roberge, PR. 2005. Computer based data organization and computer applications. In: Corrosion Tests and Standards: Application and Interpretation. Ed. Baboian, R. ASTM International, West Conshohocken, PA. 89-104.

Roberge, PR. 2008. Corrosion engineering: principles and practice. The McGraw-Hill Companies, Inc., New York, USA.

Sastri, VS. 2011. Green corrosion inhibitors: theory and practice. John Wiley & Sons, Inc., Hoboken, New Jersey, USA.

Shi, X., Xie, N., Fortune, K. and Gong, J. 2012. Durability of steel reinforced concrete in chloride environments: An overview. Constr. Build. Mater. 30:125-138.

Song, HW. and Saraswathy, V. 2007. Corrosion monitoring of reinforced concrete structures: A review. Int. J. Electrochem. Sci. 2:1-28.

Söylev, TA. and Richardson, MG. 2008. Corrosion inhibitors for steel in concrete: State-of-the-art report. Constr. Build. Mater. 22:609-622.

Tang, YM., Miao, YF., Zuo, Y., Zhang, GD. and Wang, CL. 2012. Corrosion behavior of steel in simulated concrete pore solutions treated with calcium silicate hydrates. Constr. Build. Mater. 30:252-256.

Tommaselli, MAG., Mariano, NA. and Kuri, SE. 2009. Effectiveness of corrosion inhibitors in saturated calcium hydroxide solutions acidified by acid rain components. Constr. Build. Mater. 23:328-333.

Received: Feb 12, 2013; Accepted: May 27, 2013