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$C_6H_{18}N_4$ BEHAVIOUR ON REINFORCING-STEEL CORROSION IN CONCRETE IMMERSED IN 0.5 M H_2SO_4

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ABSTRACT

This paper studies $C_6H_{18}N_4$ (Triethylenetetramine; TETA) corrosion-resistance behavior on reinforcing-steel in concreteimmersed in 0.5 M H_2SO_4. Analyses showed that the corrosion inhibition efficiency increases as theconcentration of $C_6H_{18}N_4$ admixture increases, whereby the inhibition efficiency also portrayed excellent correlation model (at r = 98.82%, *Nash Sutcliffe Efficiency* (*NSE*) = 97.65%, Analysis of Variance (ANOVA) p-value = 0.0350) with function of the $C_6H_{18}N_4$ concentration admixed in the concrete. The optimal resistance to reinforcing-steel corrosion, in the study, was exhibited in the concrete sample having 0.1824 M $C_6H_{18}N_4$ admixture, from which inhibition efficiency η = 94.78% was attained. The results support the suitability of $C_6H_{18}N_4$ for inhibiting reinforcing-steel corrosion in concrete for the industrial/microbial medium, simulated by the 0.5 M H_2SO_4.

Keywords: Triethylenetetramine, steel-reinforced concrete, reinforcing-steel corrosion, microbial/industrial simulating-environment, corrosion rate, electrochemical monitoring analyses.

INTRODUCTION

Acidic sulphate environment is one of the known aggressive medium causatives of steel-reinforced concrete degradation through an attack of the concrete structure and corrosion of the reinforcingsteel embedded in the concrete.[1-5] This kind of environment could be encountered from the reaction of SO_2 effluent with water vapor in the atmosphere ofthe industrial environment to form acid rain or from activities of sulphur oxidizing bacteria in sewage and in underground environments.[4-9] These produce sulphuric acid that converts hydrated cement products from steel-reinforced concrete to gypsum and ettringite that are expansive materials of much lower structural integrity and of degradable nature that renders the reinforcing steel prone to accelerated corrosion attacks.[5,10-12] Repairs and maintenance for averting insidious failure that could ensue from corrosion degradation of steel-reinforced concrete, induced byacidic sulphate, gulp huge costs in developed countries where such costs are monitored, apart from the usually unmonitored costs from the developing countries.[10,13]

The use of admixtures and as corrosion inhibitors have been recognized as an easy and low-cost technique that has also been found effective severally for mitigating acidic sulphate attacks on steel-reinforced concrete and for protecting the reinforcing steel material from corrosion degradation.[4,13-14] For these, while some authored reports deliberated on improving the concrete resistance,[12] other reported studies investigated anticorrosion performance on the reinforcing steel metal.[4,8,14,15] For the anticorrosion studies, research interests are increasing towards the use of organic chemicals for many of the benefits accruing

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from such usage and for the characteristics corrosion resistance by lone pair/π-electrons in their structure that have an affinity for the iron in reinforcing steel.[16-17] Of specific interests are the N-, S- and O- or multiple bonds containing compounds due to their ability to chemically interact with metallic materials in such a way as to provide effective protection against corrosion attacks.[18-20]

Triethylenetetramine ($C_6H_{18}N_4$; TETA) is an $–NH_2$ containing, lone-pair rich organic chemical, shown in its 3-D optimized molecular structure in Figure 1, from which corrosion inhibiting derivatives had been synthesized, in report work,[21] for protecting zinc material in acidic chloride environment. Aside from this, $C_6H_{18}N_4$ had been utilized in another study[22] for the inhibition ofsteel reinforcement corrosion in sodium chloride medium, among many other organic chemicals but from which TETA was identified to be among the most effective corrosion inhibiting compounds. In a more recent study, and as a motivation for the present work, $C_6H_{18}N_4$ was used as an inhibitor of carbon steel corrosion at elevated temperature in mixtures of acidic sulphide, acidic chloride and water (i.e. H_2S-HCl-H_2O) test-solutions.[23] However, there is a scanty report in the literature studying the effects of $C_6H_{18}N_4$ on reinforcing-steel corrosion in concrete for the acidic sulphate (i.e. H_2SO_4) environment. Thus, this paper investigates the behavior of $C_6H_{18}N_4$ admixture concentrations on the corrosion of reinforcing-steel embedded in concrete, and which was immersed in 0.5 M H_2SO_4 (i.e. an industrial/microbial simulating service-environment).

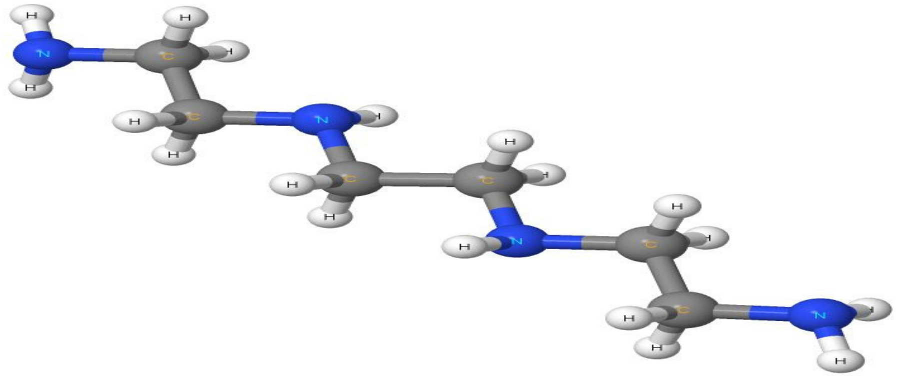

Fig.-1: Triethylenetetramine ($C_6H_{18}N_4$; TETA) in 3-D optimized Molecular Structure

EXPERIMENTAL

Materials and Data Measurement Method

Reinforcing steel employed in this paper was of 12 mm diameter that was cut into steel-rebar specimens of 190 mm length and for which surface preparations for corrosion experiment were according to ASTM G109 – 07,[24] and as detailed in studies.[25-27] From the 190 mm length, 150 mm was embedded in cast 100 × 100 × 200 mm slabs of steel-reinforced concrete specimens, and thus remaining protruding steel of 40 mm length from the concrete. The protrusion was covered in cellophane tape,[28] tapped with a hole having a bolt for electrochemical connection. The concrete preparation, casting and admixture addition were as per ASTM C192/192M-18,[29] and as described in the literature.[26,30-31]

Analytical grade $C_6H_{18}N_4$, used in the study, was sourced from Oxford Laboratory Chemicals®. By this, six different concentrations ranging from 0.0 M (the blank sample) in increments of 0.0365 M to 0.1824 M were utilized as an admixture in the requisite steel-reinforced concrete sample.[32] Each steel-reinforced concrete slab was immersed in bowls, along their longitudinal dimension, containing 0.5 M H_2SO_4, usually used in studies for an industrial/microbial simulating-environment. From each of the setup, electrochemical monitoring of corrosion rate measurements were obtained every seven days for the seven weeks of experimental period, using the Model 1500 LPR data logger (Metal Samples®).[33] This instrument utilizes the 3-electrode configuration whereby: an Ag/AgCl SCE (Direct-ION®) serves as the reference electrode, a brass plate serves as the auxiliary electrode, while the reinforcing-steel in concrete serves as the working electrode.[34-35]

Data Analysis Procedures

Measured electrochemical datasets of corrosion rate readouts, from the linear-polarization resistance instrumentation of the data logger employed, were analyzed by the Normal and the Weibull probability distribution functions (pdf's). This analysis approach follows the standard procedure prescribed in ASTM G16-13(2019).[36] Each of these pdf's has their formula respectively as:[35,37-41]

$$f(x)_{Normal} = \frac{1}{\sigma(2\pi)^{1/2}} \exp\left\{-\frac{(x-\mu)^2}{2\sigma^2}\right\} \tag{1}$$

$$f(x)_{Weibull} = \frac{k}{c}\left(\frac{x}{c}\right)^{k-1} \cdot \exp\left\{-\left(\frac{x}{c}\right)^k\right\} \tag{2}$$

Apart from the estimations of expected values, via the mean ($\mu_{\text{distribution}}$), and variations from such expected values, via the standard deviation ($\sigma_{\text{distribution}}$), from these descriptive Normal and Weibull statistical models, the reliability of the obtained expected values followed from their respective cumulative distribution function:[35,37-38,42]

$$F(x)_{Normal} = \frac{1}{\sigma(2\pi)^{1/2}} \int_{-\infty}^{x} \exp\left[-\frac{(x-\mu)^2}{2\sigma^2}\right] dx \tag{3}$$

$$F(x)_{Weibull} = 1 - \exp\left[-\left(\frac{x}{c}\right)^k\right] \tag{4}$$

As per the ASTM G16-13(2019) designation also,[36] distribution of each dataset,of corrosion tests, like the Normal and the Weibull pdf was ascertained via application of the Kolmogorov-Smirnov goodness-of-fit test-statistics,[42-47] using the formula:[35,40,43-45]

$$D_n = D(x_1,\ldots,x_n) = \sup_{-\infty<x<\infty} \left|F^*(x) - F(x)\right| \tag{5}$$

From the foregoing analyses, the corrosion-resistance by different $C_6H_{18}N_4$ concentrations was assessed through the use of the mean corrosion rate (μ), from the probability distribution of better-fit of the scatter of the measured corrosion data, for evaluating the corrosion inhibition efficiency, η (%), according to:[48-54]

$$\eta = \frac{\mu_{CR,blank} - \mu_{CR,\text{admixed}}}{\mu_{CR,blank}} \times 100 \tag{6}$$

RESULTS AND DISCUSSION

Statistically Analysed Electrochemical Test-data of Corrosion-rate

The results obtained from the analyzing electrochemical measurements of corrosion rate using the Normal and the Weibull probability distributions are shown in Figure 2. This figure details the plots of Normal mean and the Weibull mean of corrosion rate as well as the probability, $F(\mu)$, of obtaining each mean value from the measured datasets of corrosion rate, by each of the pdf models. The plotted mean of corrosion rate showed that the Weibull mean models were higher in values (implying over-prediction) than the Normal mean models for the lower $C_6H_{18}N_4$ admixture concentrations, but that these over-predictions diminished as the $C_6H_{18}N_4$concnetration increases. The probabilistic analyses show that while the probability of obtaining the Normal mean from each dataset of corrosion rate was monotonically 50%,[35] that of obtaining the Weibull mean from the datasets range from 77.21% (by the 0.1459 M $C_6H_{18}N_4$ admixture) to 66.78% (by the 0.1824 M $C_6H_{18}N_4$ admixture). In spite of these, however, both the Normal and the Weibull pdf models show that the corrosion rate of steel-reinforcement in the concrete samples decreases as the $C_6H_{18}N_4$ admixture concentration decreases.[55-57]

The disparity,in Fig.-2, between the modeled corrosion rate by the Normal and the Weibull distributions were observed to be the highest in the 0.0 M $C_6H_{18}N_4$ (blank) sample. This implies that different value of inhibition efficiency would be obtained from the use of either the Normal or the Weibull model for evaluating inhibition efficiency. Avoiding the error that could ensue from the use of the inappropriate probability distribution model requires ascertaining the distribution that fit the datasets of corrosion test-data better among the two, using the Kolmogorov-Smirnov goodness-of-fit test-statistics.

Figure-3 presents the plots from the Kolmogorov-Smirnov goodness-of-fit test-applications for the fittings of the corrosion datasets to the Normal and the Weibull probability distribution functions. Also included in the figure is the line plot of α = 0.05 significant level, for directly identifying dataset scattering like, or otherwise, the Normal and the Weibull distribution. Thus, it is observable from the figure that the corrosion rate datasets from the steel-reinforced concrete sample having 0.0365 M $C_6H_{18}N_4$ admixture and the sample having 0.1459 M $C_6H_{18}N_4$ admixture did not come from the Normal probability distribution.

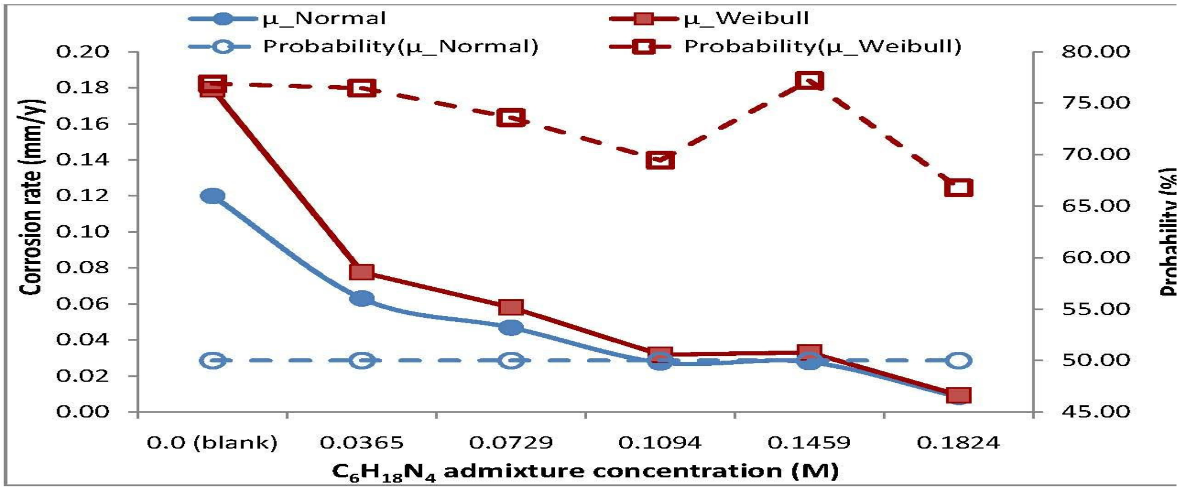

Fig.-2: Corrosion Rate Analyses Using the Normal and Weibull Distributions

For these samples, the Kolmogorov-Smirnov p-values are, respectively, 0.037 and 0.044, both of which are lesser than the α = 0.05 significance level. This indicates that only four out of the six steel-reinforced samples from this work have their datasets following the Normal probability distribution. Unlike this, the Kolmogorov-Smirnov p-values of all the steel-reinforced concrete from this work overshot the α = 0.05 significant level baseline, which indicates that they all have their datasets scattered like Weibull probability distribution, and are thus compatible to the fitting of this distribution model. These findings, therefore, support utilizing the Weibull probability distribution as the descriptive statistics for the presentation of the $C_6H_{18}N_4$ admixture performance on the corrosion steel-reinforcement in the concrete samples being studied.

Corrosion Inhibition Performance Model Computation

The results from the corrosion inhibition efficiency computation, via substitution of the Weibull mean of corrosion rate into Equation (6), are presented in the graphical plot shown in Fig.-4.

It could be observed from the plotted results in Fig.-4, in agreement with the plotted results in Fig.-2, that increase in the $C_6H_{18}N_4$ admixture concentration in the steel-reinforced concrete samples for the H_2SO_4 medium leads to increase in the corrosion-inhibition efficiency.[55-57] Further analyses of this performance model by the $C_6H_{18}N_4$ admixture, via probing of mathematical correlation modeling,[35,46,49,58] as per ASTM G16-13(2019),[36] indicated that the inhibition efficiency, η (%), exhibited a relationship with the $C_6H_{18}N_4$ concentration, ρ (M), which could be represented as:

$$\eta = 386.187\left[\ln\left(\rho+\rho_1\right)^{0.2281} - \rho + 0.7614\right] \qquad (7)$$

In Equation (7), ρ_1 = 0.0365 M, which is the incremental $C_6H_{18}N_4$ admixture concentration utilized for the experimental steel-reinforced concrete samples in this work. Computations from this correlation fitting equation indicate that the correlation coefficient r = 98.82%, while the Nash-Sutcliffe Efficiency NSE = 97.65%.[25] These values interpret to "excellent" model efficiency as per the modeling efficiency classification from the literature.[59] The results from the analysis of variance (ANOVA) computation from the presented mathematical correlation are detailed in Table-1. These results indicate that the p-value = 0.035, and which for the reason that the value is less than 0.05 implies that the relationship between the inhibition efficiency, η, and the $C_6H_{18}N_4$ admixture concentration, ρ, is statistically significant at greater than 95% confidence interval.

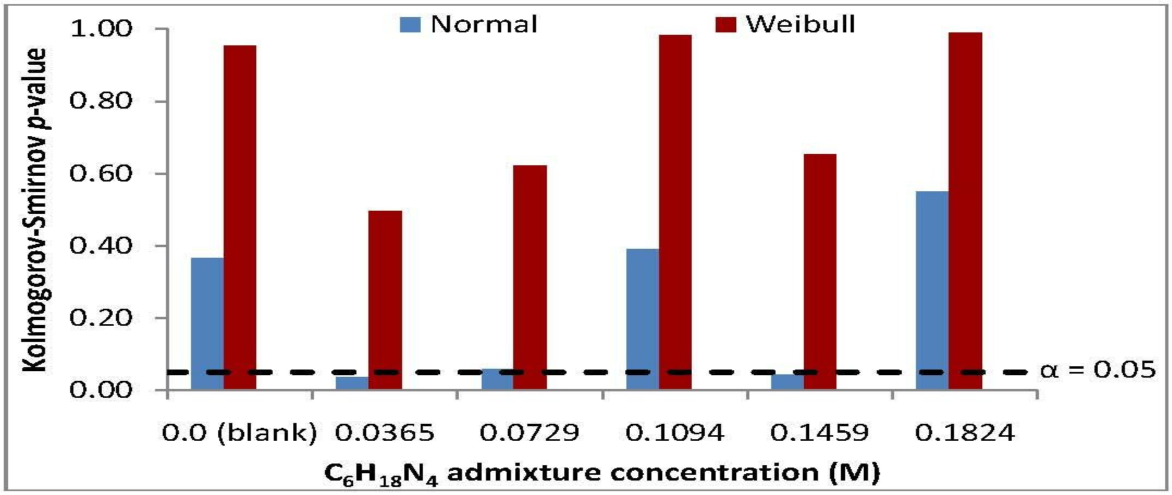

Fig.-3: Plots from the Kolmogorov-Smirnov Goodness-of-fit Test-Statistics Applied to the Corrosion Rate Datasets for Testing Scattering like the Normal and Weibull Distributions

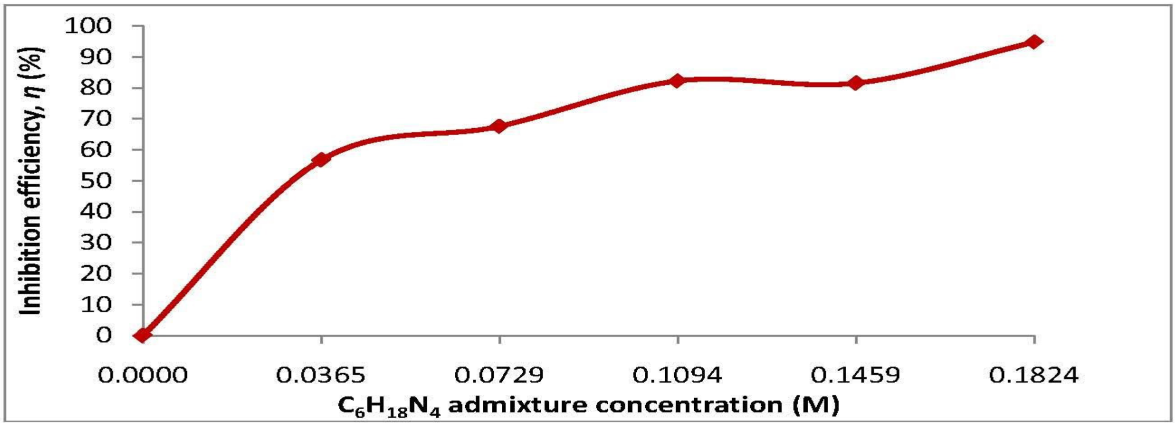

Fig.-4: The Performance Model of the Corrosion Inhibition Efficiency by the $C_6H_{18}N_4$ Admixture Concentrations on Reinforcing-Steel in the H_2SO_4-immersed Concretes Samples

Table-1: Analysis of Variance (ANOVA) for the Correlation Equation.

Source of Variation	DoF	SS	MS	*F*	*p*-Value
Treatment	3	5614.3238	1871.4413	27.7190	0.0350
Residual	2	135.0294	67.5147		
Total	5	5749.3532			

Discussion

By following the Weibull probability distribution more than the Normal probability distribution, in Fig.-3, the corrosion rate data exhibited the behaviour of failure related data that are extreme values by definition[42] and for which the Weibull pdf, as a type of extreme value distribution, finds fitting suitability.[47,60] Thus, estimation of the probability of obtaining the mean using the Weibull pdf model led to values $F(\mu) > 0.5$ (that are otherwise the central limit designation of the Normal pdf model) see Fig.-2. This further corroborates the fact that the modeled Weibull mean values of corrosion rate are in the upper tail of the distribution fitting the datasets from the studied steel-reinforced concretes, which constitute characteristics identified in the previous work[42] for data of corrosion failure-causing physical processes of materials.

The improvement of corrosion resistance in the 0.5 M H_2SO_4-immersed concrete with increasing concentrations of $C_6H_{18}N_4$ admixture exhibit confirmations from studies on the identifications of amine compounds as effective corrosion inhibitor of steel-reinforced concrete, especially in acidic sulphate related environment.[12,18,61] In these cited works,[12,61] amines are shown to exhibit good corrosion inhibition effects on steel-reinforcement in concrete by their concrete pore-blocking or hydrophobic activities that prevent the ingress of the corrosive agent into the concrete. In combination with this feature, amines are known to exhibit preferential adsorption of the $–NH_2$ ligand as well as the unshared lone-pair electrons of their N–bonds, which are well-constituted also in $C_6H_{18}N_4$ (See Fig.-1), with the Fe component in steel.[12,61-62] Additionally, many research findings[12,61-63] indicated that amine-based inhibitors form multi-layered protective-film on the base metal and for which thickness of the protective film layer depends on the inhibitor concentration.[61] This concentration-dependent protective thickness appears to explain the increase of inhibition efficiency, i.e. corrosion resistance, as the $C_6H_{18}N_4$ concentration increases in this study, as was observed from the results plotted in Fig.-4.

Thus, the Normal and the Weibull corrosion rate models for the control sample, having 0.0 M $C_6H_{18}N_4$ admixture, were greater than 0.10 mm/y in Fig.-2, which interprets to "Very high" as per classification of typical corrosion rate of steels in studies,[61,63-64] from which the $C_6H_{18}N_4$ exhibited corrosion rate reductions. The continuous corrosion rate reductions, as the $C_6H_{18}N_4$ concentration increases, culminated in obtaining corrosion rate that ranged into the "low/moderate" typical steel corrosion rate, according to set values in the literature,[18,63-64] by the concrete sample with the 0.1824 M $C_6H_{18}N_4$ admixture. It could also be observed that the best agreements, in the study, between the Normal and Weibull corrosion rate models, were exhibited by this same concrete sample having the 0.1824 M $C_6H_{18}N_4$ admixture. Therefore, the 0.1824 M $C_6H_{18}N_4$ admixture fostered the optimal corrosion resistance by the steel-reinforcement embedded in the studied concrete samples in this work. In the presence of this 0.1824 M $C_6H_{18}N_4$ admixture, η = 94.78% efficiency at inhibiting steel-reinforcement corrosion was attained. This also translates to excellent inhibition efficiency model according to the classification of model efficiency from Coffey et al.[56]

CONCLUSION

In this paper, the behavior of $C_6H_{18}N_4$ varying admixture concentrations on the corrosion-resistance of reinforcing-steel in concrete immersed in 0.5 M H_2SO_4, has been investigated. The results showed that the corrosion test-data portrayed the behavior of failure-causing data by following the Weibull probability distribution more than the Normal distribution, as per the Kolmogorov-Smirnov goodness-of-fit test-criteria of α = 0.05 level of significance. Analysis of electrochemical measurements showed that increasing the $C_6H_{18}N_4$ admixture concentration in the H_2SO_4-immersed steel-reinforced concrete leads to decreasing corrosion-rate and, consequently, increasing corrosion inhibition efficiency. It was also established that the inhibition efficiency portrayed excellent correlation (r = 98.82%, NSE = 97.65%, ANOVA p-value = 0.0350) with the function of $C_6H_{18}N_4$ concentration for concrete admixture. Overall, the 0.1824 M $C_6H_{18}N_4$ admixture, with the inhibition efficiency η = 94.78%, exhibited optimal corrosion-resistance behavior in the study on reinforcing-steel metal in the 0.5 M H_2SO_4 test-environment. These results from the study established the suitability of $C_6H_{18}N_4$ (Triethylenetetramine; TETA) admixture, especially at sufficient concentrations as an effective corrosion inhibiting substance for reinforcing-steel in concrete immersed in the 0.5 M H_2SO_4, the medium used for simulating the industrial/microbial environment in the study.

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REFERENCES

1. J.O. Okeniyi, A.P.I. Popoola and E.T. Okeniyi. in *CORROSION 2018*, NACE International, Paper No 10808, (2018).
2. Y. Xie, X. Lin, W. Pan, T. Ji, Y. Liang and H. Zhang, *Construction of Building Materials*, **188**, 9 (2018), **DOI:**10.1016/j.conbuildmat.2018.08.105
3. J.O. Okeniyi, C.A. Loto and A.P.I. Popoola, *Solid State Phenomena*, **227**, 281 (2014), **DOI:**10.4028/www.scientific.net/ssp.227.281

4. H. Gerengi, Y. Kocak, A. Jazdzewska, M. Kurtay and H. Durgun, *Construction of Building Materials*, **49**, 471 (2013), **DOI:**10.1016/j.conbuildmat.2013.08.033
5. A.K. Parande, P.L. Ramsamy, S. Ethirajan, C.R.K. Rao and N. Palanisamy, in *Proceedings of the Institution of Civil Engineers-Municipal Engineer*, Thomas Telford Ltd, **159**, 11 (2006), **DOI:**10.1680/muen.2006.159.1.11
6. S. Tsouli, A.G. Lekatou, S. Kleftakis, T.E. Matikas and P.T. Dalla, *Procedia Structural Integrity*, **10**, 41 (2018), **DOI:**10.1016/j.prostr.2018.09.007
7. J.O. Okeniyi, *Journal of the Association of Arab Universities for Basic and Applied Sciences*, **20**, 39 (2016), **DOI:**10.1016/j.jaubas.2014.08.004
8. M.A.G. Tommaselli, N.A. Mariano and S.E. Kuri, *Construction of Building Materials*, **23**, 328 (2009), **DOI:**10.1016/j.conbuildmat.2007.12.002
9. W. De Muynck, N. De Belie and W. Verstraete, *Cement and Concrete Composites*, **31**, 163 (2009), **DOI:**10.1016/j.cemconcomp.2008.12.004
10. J.O. Okeniyi, C.A. Loto and A.P.I. Popoola, *HKIE Transactions*, **23**, 138 (2016), **DOI:**10.1080/1023697x.2016.1201437
11. S. Wei, M. Sanchez, D. Trejo and C. Gillis, *International Biodeterioration & Biodegradation*, **64**, 748 (2010), **DOI:**10.1016/j.ibiod.2010.09.001
12. E. Hewayde, M.L. Nehdi, E. Allouche and G. Nakhla, *Proceedings of the Institution of Civil Engineers - Construction Materials*, **160**, 25 (2007), **DOI:**10.1680/coma.2007.160.1.25
13. E. Hewayde, M. Nehdi, E. Allouche and G. Nakhla, *Structure and Infrastructure Engineering*, **3**, 17 (2007), **DOI:**10.1080/15732470500253065
14. Y. Tang G., Zhang and Y. Zuo, *Construction of Building Materials*, **28** 327 (2012), **DOI:**10.1016/j.conbuildmat.2011.08.048
15. J.O. Okeniyi, C.A. Loto and A.P.I. Popoola, *Energy Procedia*, **50**, 429 (2014), **DOI:**10.1016/j.egypro.2014.06.052
16. J.O. Okeniyi, A.P.I. Popoola and C.A. Loto, O.A. Omotosho, S.O. Okpala, I.J. Ambrose, *Advances in Materials Science and Engineering*, **2015**, 540395 (2015), **DOI:**10.1155/2015/540395
17. A.S. Raja, S. Rajendran and P. Satyabama, *Journal of Chemistry*, **2013**, 720965 (2013), **DOI:**10.1155/2013/720965
18. J.O. Okeniyi and A.P.I. Popoola, in *Contributed Papers from Materials Science and Technology 2017 (MS&T17)*, pp. 1070-1077 (2017), **DOI:**10.7449/2017mst/2017/mst_2017_1070_1077
19. A.S. Raja, S. Rajendran, J. Sathiyabama, V. Prathipa, I.N. Karthika and A. Krishnaveni, *International Journal of Nano Corrosion Science and Engineering*, **2**, 26 (2015), **DOI:**10.4152/pea.201106429
20. M. Shahid, *Advances in Natural Sciences: Nanoscience and Nanotechnology*, **2**, 043001 (2011), **DOI:**10.1088/2043-6262/2/4/043001
21. M.D. Shah, A.S. Patel, G.V. Mudaliar and N.K. Shah, *Portugaliae Electrochimica Acta*, **29**, 101 (2011), **DOI:**10.4152/pea.201102101
22. M. Ormellese, F. Bolzoni, L. Lazzari, A. Brenna and M. Pedeferri, *Materials and Corrosion*, **62**, 170 (2011), **DOI:**10.1002/maco.201005763
23. Y. Shao, J. Tang, T. Zhang, G. Meng and F. Wang, *International Journal of Electrochemical Science*, **8**, 5546 (2013).
24. ASTM G109-07(2013), Standard Test Method for Determining Effects of Chemical Admixtures on Corrosion of Embedded Steel Reinforcement in Concrete Exposed to Chloride Environments, West Conshohocken, PA, ASTM International, 2013, **DOI:**10.1520/G0109-07R13
25. J.O. Okeniyi, J.A. Idemudia, I.O. Oladele, C.A. Loto and P.A.I. Popoola, *International Journal of Electrochemical Science*, **8**, 10758 (2013).
26. J.O. Okeniyi, O.M. Omoniyi, S.O. Okpala, C.A. Loto and A.P.I. Popoola, *European Journal of Environmental and Civil Engineering*, **17**, 398 (2013), **DOI:**10.1080/19648189.2013.797927
27. A.K. Singh, S.K. Shukla, M.A. Quraishi and E.E. Ebenso, *Journal of the Taiwan Institute of Chemical Engineers*, **43**, 463 (2012), **DOI:**10.1016/j.jtice.2011.10.012
28. M. Ormellese, L. Lazzari, S. Goidanich, G. Fumagalli and A. Brenna, *Corrosion Science*, **51**, 2959 (2009), **DOI:**10.1016/j.corsci.2009.08.018

29. ASTM C192 / C192M-18,Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory, West Conshohocken, PA, ASTM International (2018), **DOI:**10.1520/C0192_C0192M-18
30. J.O. Okeniyi, C.C. Nwadialo, F.E. Olu-Steven, S.S. Ebinne, T.E. Coker, E.T. Okeniyi, A.S. Ogbiye, T.O. Durotoye and E.O.O. Badmus, in *AIP Conference Proceedings*, AIP Publishing, **1814**, 020035 (2017), **DOI:**10.1063/1.4976254
31. M. Ormellese, M. Berra, F. Bolzoni and T. Pastore, *Cement and Concrete Research*, 36, 536 (2006), **DOI:**10.1016/j.cemconres.2005.11.007
32. J.O. Okeniyi, C.A. Loto and A.P.I. Popoola *Portugaliae Electrochimica Acta*, **32**, 199 (2014), **DOI:**10.4152/pea.201403199
33. V.S. Sastri, Green Corrosion Inhibitors: Theory and Practice, Hoboken, John Wiley & Sons, Inc., New Jersey (2011), **DOI:**10.1002/9781118015438
34. C.A. Trevisol, P.R.P.D. Silva, M.M.D.S. Paula and F. Pelisser, *Matéria (Rio J.)*, **22**, (2017), **DOI:**10.1590/s1517-707620170004.0238
35. J.O. Okeniyi, I.J. Ambrose, S.O. Okpala, O.M. Omoniyi, I.O. Oladele and C.A. Loto, *Sadhana*, **39**, 731 (2014), **DOI:**10.1007/s12046-014-0226-9
36. ASTM G16-13(2019), Standard Guide for Applying Statistics to Analysis of Corrosion Data, West Conshohocken, PA, ASTM International, 2019, **DOI:**10.1520/G0016-13R19
37. K. Krishnamoorthy, Handbook of Statistical Distributions with Applications, 2 ed. Boca Raton, FL, CRC Press, Taylor & Francis Group, (2016).
38. D.C. Montgomery and G.C. Runger, Applied Statistics and Probability for Engineers, 6 ed. Hoboken, New Jersey, John Wiley & Sons, Inc. 2014.
39. J.O. Okeniyi, C.A. Loto and A.P.I. Popoola, *International Journal of Electrochemical Science*, **9**, 4205 (2014).
40. J.O. Okeniyi, C.A. Loto and A.P.I. Popoola, *Transactions of The Indian Institute of Metals*, **67**, 959 (2014), **DOI:**10.1007/s12666-014-0424-5
41. J.O. Okeniyi, U.E. Obiajulu, A.O. Ogunsanwo, N.W. Odiase, and E.T. Okeniyi, *Mitigation and Adaptation Strategies for Global Change*,**18**, 325 (2013), **DOI:**10.1007/s11027-012-9365-7
42. F. Haynie, 2005, Statistical treatment of data, data interpretation, and reliability, in R. Baboian, (ed), Corrosion Tests and Standards: Application and Interpretation, ASTM Manual Series MNL20, 2nd Edition, West Conshohocken, PA, ASTM International, pp. 83-88, **DOI:**10.1520/mnl11011m
43. J.O. Okeniyi and E.T. Okeniyi, A.A. Atayero, *Journal of Software and Systems Development*, **2015**, 238409 (2015), **DOI:**10.5171/2015.238409
44. J.O. Okeniyi, E.T. Okeniyi and A.A. Atayero, in *Vision 2020: Sustainable Growth, Economic Development, and Global Competitiveness - Proceedings of the 23rd International Business Information Management Association Conference*, IBIMA 2014, Valencia; Spain, **1**, 2561 (2014).
45. J.O. Okeniyi and E.T. Okeniyi, *Journal of Statistical Computation and Simulation*, **82**, 1727 (2012), **DOI:**10.1080/00949655.2011.593035
46. D. Izquierdo, C. Alonso, C. Andrade and M. Castellote, *Electrochimica Acta*, **49**, 2731 (2004), **DOI:**10.1016/j.electacta.2004.01.034
47. P.R. Roberge, 2003, Statistical interpretation of corrosion test results, in: S.D. Cramer and B.S. Covino, Jr. (eds), Corrosion:Fundamentals, Testing, and Protection, Vol 13A, ASM Handbook, Materials Park, OH, ASM International, pp. 425-429, **DOI:**10.31399/asm.hb.v13a.a0003641
48. D. Jalajaa, S. Jyothi, V.R. Muruganantham and J. Mallika, *Rasayan Journal of Chemistry*, **12**, 545 (2019), **DOI:**10.31788/RJC.2019.1224096
49. J.O. Okeniyi, E.T. Akinlabi, J.O. Ikotun, S.A. Akinlabi, E.T. Okeniyi, M.E. Ojewumi, *Jurnal Teknologi*, **81**, 11 (2019), **DOI:**10.11113/jt.v81.10468
50. V. Swarnalatha and P. Pazhanisamy, *Rasayan Journal of Chemistry*, **11**, 929 (2018), **DOI:**10.31788/RJC.2018.1132073
51. J.O. Okeniyi, C.A. Loto and A.P.I. Popoola, *Metals*, **6**, 255 (2016), **DOI:**10.3390/met6110255
52. J.O. Okeniyi, C.A. Loto and A.P.I. Popoola, *Solid State Phenomena,* **227**, 185 (2015), **DOI:**10.4028/www.scientific.net/ssp.227.185
53. A.A. Farag and M.A. Hegazy, *Corros. Sci.*, 74, 168 (2013), **DOI:**10.1016/j.corsci.2013.04.039

54. A.S. Fouda, M. Abdallah and A. Attia, *Chemical Engineering Communications*, **197**, 1091 (2010), **DOI:**10.1080/00986440903412944
55. K. Rathidevi, S. Jyothi, D. Jalajaa and S. Karthikeyan, *Rasayan Journal of Chemistry*, **12**, 565 (2019), **DOI:**10.31788/RJC.2019.1224098
56. R. Thilagavathi, A. Prithiba and R. Rajalakshmi, *Rasayan Journal of Chemistry*, **12**, 431 (2019), **DOI:**10.31788/RJC.2019.1225133
57. S. Jyothi, Y.V.S. Rao and P.S.S. Ratnakumar, *Rasayan Journal of Chemistry*, **12**, 537 (2019), **DOI:**10.31788/RJC.2019.1225000
58. J.O. Okeniyi, A.P.I. Popoola and C.A. Loto, in *NACE-International Corrosion Conference Series*, National Association of Corrosion Engineers (NACE) International, 2015, Paper No 5590 (2015).
59. R. Coffey, S. Dorai-Raj, V. O'flaherty, M. Cormican and E. Cummins, *Hum. Ecol. Risk Assess.: An Int. J.*, **19**, 232 (2013), **DOI:**10.1080/10807039.2012.701983
60. K. Bury, Statisitcs. in *Statistical Distributions in Engineering*, Cambridge, UK, Cambridge University Press, pp. 18-26 (1999), **DOI:**10.1017/cbo9781139175081
61. T.A. Söylev and M.G. Richardson, *Construction of Building Materials*, **22**, 609 (2008), **DOI:**10.1016/j.conbuildmat.2006.10.013
62. A.A. Al-Amiery, A.A.H. Kadhum, A. Kadihum, A.B. Mohamad, C.K. How and S. Junaedi, *Materials*, **7**, 787 (2014), **DOI:**10.3390/ma7020787
63. T.A. Söylev, C. Mcnally and M. Richardson, *Cement and Concrete Research*, **37**, 972 (2007), **DOI:**10.1016/j.cemconres.2007.03.010
64. J.H. Bungey, S.G. Millard and M.G. Grantham, Testing of Concrete in Structures, 4 ed., Taylor & Francis, New York (2006).

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