ISSN:2716-9227 Algerian Journal of Engineering and Technology 03 (2020) 064–068 e-ISSN: 2716-9278

Algerian Journal of Engineering and Technology

Journal homepage: https://jetjournal.org/index.php/ajet

Original Article

Combating chloride ions in reinforced concrete using $K_2Cr_2O_7$ as corrosion inhibitor

Catherine Mayowa Ikumapayi[a]*, Akintayo Adedamola Adeniji[b], Emmanuel Osamudiamen Idehen[b], Abel Adeiza Barnabas[c]

[a]*Civil Engineering Departmnt, Federal University of Technology, PMB 704, Akure, Nigeria*

[b]*Civil and Environmental Engineering, Elizade University, Ilara Mokin, Nigeria*

[C]*Metallurgical and Materials Engineering, PMB 704, Akure, Nigeria*

ARTICLE INFO

Article history:
Received 27 March2020
Revised 27 November 2020
Accepted 28 November 2020

Keywords:
Corrosion;
Chloride ion;
linear polarization;
Weight loss;
Potassium dichromate.

ABSTRACT

The ingress of chloride ions from seawater in contact with concrete reinforced structures is one of the major causes of deterioration in the construction industries worldwide. This gives rise to corrosion of embedded steel in the concrete structures which sooner or later results in dilapidation, deterioration and partial or total failure of the reinforced structure. In this study potassium dichromate ($K_2Cr_2O_7$) solution was used to mitigate the effects of chloride ions in reinforced concrete. Cylindrical concrete samples of size 15mm x 36mm diameter with cement to sand mix ratio of 1:6 and a water cement ratio of 0.5 were prepared. 12 mm high yield reinforcing bars were inserted into the sample. The samples were then immersed in NaCl solution with varying concentration of $K_2Cr_2O_7$. Both weight loss method and linear polarization measurement were performed on the samples. Data acquisition and analysis were carried out using 4 decimal places electronic weighing balance, a potentiostat interfaced and a computer. The results from the weight loss method showed that the weight loss decreased from 0.7629g to 0.1398g for the 7th and 35th day respectively. Results from the potentiodynamic polarization method further revealed the efficiency of $K_2Cr_2O_7$ in mitigating corrosion.

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1. Introduction

Reinforced concrete is a successful steel and concrete combination that has been widely used for over 100 years [1]. Steel provides tensile strength to the structural material while concrete provides compressive strength to the material. Concrete as a high alkalinity material further performs the role of a physical barrier to the reinforcing steel i.e it protects the steel from corrosion and other aggressive agents. Reinforced concrete has been extensively used in the construction industries over the years, although, when subjected to aggressive agents such as carbonic gas and chloride ions it deteriorates. Concrete act as a physical obstruction that shields steel from corrosion. Concrete is alkaline in nature, the alkalinity of concrete leads to the formation of a passive layer around the reinforcement, which increases protection against corrosive processes. However, concrete contains pores that allow the entrance of aggressive agents which destabilizes the passive layer and corrode the steel [2]. Therefore the effectiveness of concrete protecting the steel embedded in it is a function of the quality of that concrete. Corrosion can be defined as the deterioration and loss of a material and its critical properties due to chemical, electrochemical and other reactions of the exposed material surface with the surrounding environment [3]. The corrosion of the steel reinforcement in a reinforced concrete structure is an electrochemical process and occurs when there is difference in the concentration of dissolved ions inside the concrete, creating electrochemical potential cells or

* *Corresponding author.*
E-mail address: mayowaik@yahoo.com

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http://dx.doi.org/ 10.5281/zenodo.4404744

corrosion cells, characterized by a flow of electrons and ions between the cathodic and anodic regions. The corrosion of steel reinforcement is one of the main causes of premature deterioration of reinforced concrete, leading to significant economic losses [4]. Many reinforced structures had failed both partially and totally as a result of corrosion leading to economic losses as well as loss of lives [5]. Rapid deterioration can be caused by chloride ions in a marine zone or due to the use of thaw salts or by carbonation in urban zones. In order to provide additional protection and increase the life span of reinforced concrete structures in such environment, several methods of protection and repair have been developed and some are still in progress to increase the durability of such reinforced concrete structures. The use of additives such as silica fumes, fly ash and other pozzolanic materials are also receiving attention because they can reduce the permeability of concrete to chloride ingression [6]. Another promising method is the use of corrosion inhibitors, they prevent corrosion in the presence of chloride ion and carbonation. Calcium nitrire has been successfully used as corrosion inhibitors in reinforced concrete [7,8]. Corrosion inhibitors are chemical substances that reduce the corrosion rate. They have been widely used, for both prevention and correction of corrosion [9]. Classification of corrosion inhibitors can be based on their chemical composition, mechanism of protection and method of application. Some known corrosion inhibitors used in reinforced concrete structures are sodium nitrite ($NaNO_2$), calcium nitrite [$Ca(NO_2)_2$], sodium mono-fluorophosphate (Na_2PO_3F), amine-based inhibitors, sodium and potassium chromate, sodium phosphate, and bauxite residue (red mud) among others (Lourenço et al, 2014). There is need for attention on how the problem of corrosion in reinforced concrete can be minimized or eliminated, therefore this study investigates the use of potassium chromate in reducing the rate of chloride-induced corrosion in concrete.

2. Materials and Methods

2.1. Material

The materials used for the research include, Potassium dichromate, ordinary Portland cement (OPC), fine aggregates, coarse aggregates, high yield steel of 12mm diameter, Sodium Chloride (NaCl) and water among others. The chemical composition of the high yield steel of 12mm diameter was determined using Optical Emission Spectrometry Test and the outcomes is presented in Table 1. The two main methods used to determine the corrosion rate of the inhibitors on the reinforced concrete are the gravimetric and potentiodynamic polarization methods.

Table 1: Chemical composition of the high yield steel

Element	C	Si	Mn	Cr	Mo	P	S	V	Ti
Content (%)	0.36	0.28	0.66	0.18	0.008	0.046	0.052	0.001	0.0006

Element	Al	Cu	Sb	Ni	B	Sn	Ca	Fe
Content (%)	0.0043	0.27	0.013	0.0992	0.0069	0.037	0.001	98

2.1.Experimental procedures using gravimetric method

The reinforced concrete samples were prepared; the concrete were prepared using mix ratio of 1:2:4 of cement : sand : gravel with water cement ratio of 0.5. The steel samples were cut into 30mm size, weighed using the analytical weighing balance and cast alongside with the concrete with 15mm of its length inside the concrete as shown in Figure 1. The reinforced concrete samples were then demoulded and immersed inside six different solutions. The solutions were prepared with different concentrations of the $K_2Cr_2O_7$ at 0% (control) 2%, 4%, 6%, 8%, 10% and 0.1M NaCl solution as shown in Table 2. Then the reinforced concrete samples were immersed into the solution. A control sample (NaCl without the addition of the inhibitor) was prepared alongside. The sodium chloride introduced into the solutions is to subject the samples to corrosion since the principal cause of corrosion in reinforced concrete is the migration of chloride ions through the pores of the concrete structure [11]. The reinforced concrete samples were removed from the solutions at 7 days interval and the reinforcement were weighed. This was done for six weeks. Weight loss corresponding to each sample was recorded and the corrosion rate was determined using Equation 1. [12,13]

Fig. 1: Reinforced concrete samples cast in place.

Table2: Experimental setup of the solutions for the gravimetric method

Solution	Percentage of $K_2Cr_2O_7$	Percentage of NaCl
Solution 1 (control)	0%	0.1M
Solution 2	2%	0.1M
Solution 3	4%	0.1M
Solution 4	6%	0.1M
Solution 5	8%	0.1M
Solution 6	10%	0.1M

The Corrosion Rate was determined using the relation below:

$$C.R(mm/yr) = \left[\frac{534 \times W}{D \times A \times T}\right] \text{ [14]} \qquad (1)$$

Where C.R represents the corrosion rate
W represents weight loss,
T/365 represents exposure time in days extrapolated to year,
A represents surface area of the specimen (mm^2)
D represent the density of the specimen

2.2. Potentiodynamic polarization method in reinforced concrete

This method is used for determining the electrochemical measurements of the reinforcement. The aim of potentiodynamic polarization study is to evaluate the corrosion current density, $\boldsymbol{i_{corr}}$, corrosion potential and Tafel slope. The polarization resistance of a reinforcement embedded in concrete is defined as the ratio between applied voltage and the step of current when the metal is slightly polarized from its corrosion potential [15]. The experiments was performed using a three-electrode corrosion cell set-up comprising of the steel rod inside the concrete block as the working electrode, saturated silver/silver chloride (Ag/AgCl) as reference electrode, and platinum rod as counter electrode. The working electrodes were prepared by attaching an insulated copper wire to one face of the sample using an aluminum conducting tape, and cold mounting it with epoxy resin. Potentiodynamic polarization measurements were carried out using a scan rate of 1.0 mV/s at a potential initiated at -250mV to + 250mV as shown in figures. After each experiment, the electrolyte and the test sample were replaced. The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential to obtain the corrosion current densities ($\boldsymbol{i_{corr}}$) and corrosion potential ($\mathbf{E_{corr}}$). The test was then repeated for the different samples with the different inhibitor's concentration. The Inhibitor efficiency, P, was calculated using Equation 2

$$P = \left(\frac{W_0 - W}{W_0} \times 100\right) \qquad (2)$$

Where W_0 represents the corrosion rate in the absence of inhibitor, W represents the corrosion rate in the same environment with the inhibitor added.

3. Results and Discussion

3.1. Gravimetric Analysis

The result of the weight loss against exposure time obtained during the gravimetric analysis is shown in Figure 2. The result shows that the NaCl environment without the inhibitor (control) has a greater weight loss curve compared to the curves with the inhibitor ($K_2Cr_2O_7$ solution). The result shows that the higher the inhibitor concentration the more the weight loss experienced with 10% $K_2Cr_2O_7$ solution having the least weight loss. This is in line with the work of Rathi *et al* [16], who deduced that weight loss is a function of the increase in concentration of the inhibitor. Weight loss of the reinforcing bars is an indication of the decrease in the physical, mechanical and chemical properties of the embedded steel.

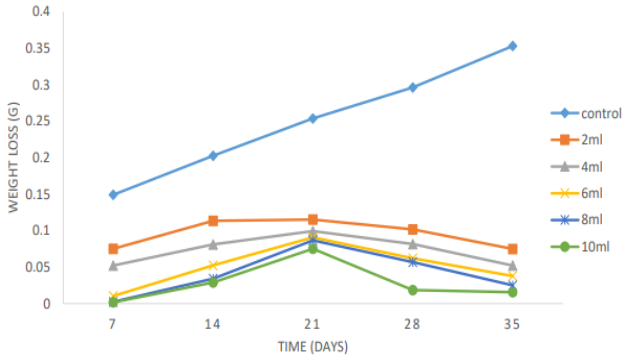

Fig. 2. Weight loss against exposure time with varying amount of $K_2Cr_2O_7$ solution.

The result of the corrosion rate in Figure 3 shows a decrease in the corrosion rate over time for all the conditions of exposure. The more the days of exposure the less the corrosion rate. This pattern of results is in accordance with the outcome of some past researchers who utilised other corrosion inhibitors [17,18]. The corrosion rate increases with increase in the concentration of the $K_2Cr_2O_7$ solution.

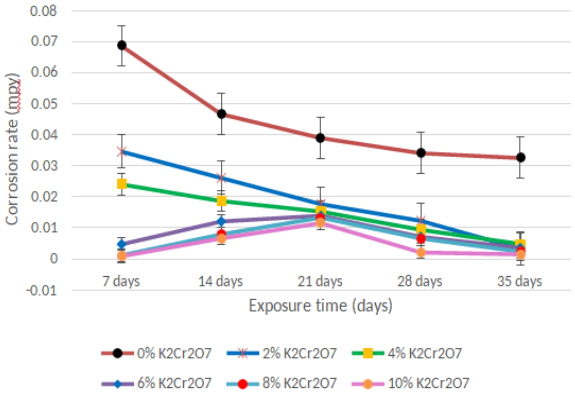

Fig. 3: Corrosion rate of the various concentration of $K_2Cr_2O_7$ over time

3.2. Polarization method

Corrosion parameters obtain from the potentiodynamic polarization values of high yield steel sample in 0.1M of NaCl without $K_2Cr_2O_7$ solution and with varying amount of the $K_2Cr_2O_7$ solution (as the corrosion inhibitor) are shown in Table 3. From Table 3 it can be observed that the presence of the Potassium dichromate lowers the current density (i_{corr}) values obtained for the samples thereby increasing the inhibiting efficiency. The Potentiodynamic curve shown in Figure 4 displays the relationship between the applied voltage and the logarithm of the current density, it shows the corrosion behavior of the steel in the different environment.

Table 3: Result from Tafel polarization

Inhibition concentration (ml)	Ecorr (mV/AgCl)	i (μA/cm^2)	Corrosion rate (mmpy)	Inhibition efficiency (%)
0 (control)	-542	33.347	0.3869	0
2	-533	2.635	0.03058	92.10
4	-511	6.3933	0.07457	80.73
6	-524	5.207	0.06042	84.38
8	-407	3.515	0.04079	89.43
10	-490	5.449	0.06325	83.65

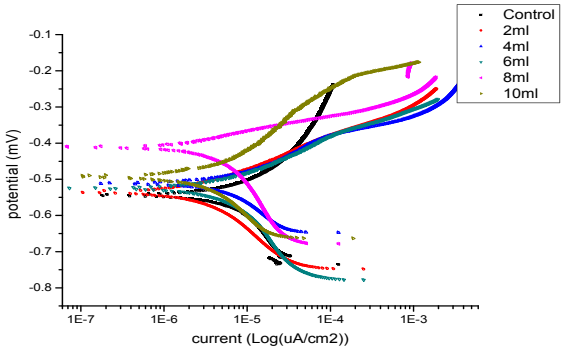

Fig. 4. Potentiodynamic polarization curves with various concentration of $K_2Cr_2O_7$ solution.

Increasing the concentration of K2Cr2O7 solution in the NaCl mixture it can be observed from Figure 4 that there is a corresponding decrease in the current density on the cathodic arm. Also from Figure 4 increasing the concentration of the K2Cr2O7 solution the corrosion potential, (V) decreases which confirms the inhibiting efficiency of K2Cr2O7 solution. This further confirms the results obtained from the gravimetric method.

4. Conclusions

From the experiments carried out on mitigating chloride ions using K2Cr2O7 solution as corrosion inhibitor, the following conclusions can be drawn:

- The potassium dichromate (K2Cr2O7) solution can be used as corrosion inhibitor in reinforced concrete subjected to chloride ion attack in aggressive environment.
- Inhibition efficiency of the Potassium dichromate (K2Cr2O7) solution increases with increase in concentration of the inhibitor. Therefore the higher the concentration of the K2Cr2O7 the more the amount of corrosion it will prevent.
- Two corrosion measurement methods (Linear Polarisation and Gravimetric) were used in this research, and the results obtained from both methods showed similar trend.
- Potassium Dichromate succeeded in lowering the current density of the high yield steel.

Nomenclature

CR corrosion rate in mil per year,
W weight loss,
A surface area of the specimen (mm^2)
T/365 exposure time in days extrapolated to year
W_0 corrosion rate in the absence of inhibitor,
W corrosion rate in the same wwith the inhibitor added.
i_{corr} current densities
E_{corr} corrosion potential

Conflict of Interest

The authors declare that they have no conflict of interest

Author's contribution

All authors have contributed equally to this paper by collecting data, analysis, performing and writing the paper.

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Recommended Citation

Ikumapayi CM, Adeniji AA, Idehen EO, Barnabas AA. Combating chloride ions in reinforced concrete using K2Cr2O7 as corrosion inhibitor. *Alg. J. Eng. Tech.* 2020; 3: 064-068. http://dx.doi.org/ 10.5281/zenodo.4404744

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