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EFFECT OF GREEN INHIBITOR ON THE CORROSION BEHAVIOUR OF REINFORCED CARBON STEEL IN CONCRETE

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ABSTRACT

The effect of inhibitors on the corrosion behaviour of carbon steel reinforced in concrete exposed to 3.5% NaCl solution has been studied by electrochemical and weight loss tests. The corrosion potential and concrete resistivity were determined during the immersion time of the concrete specimens. The results show that calcium nitrite and vernonia amygdalina extract reduced corrosion rate compared to sodium nitrite. However, vernonia amygdalina inhibition was more effective in the immersed concrete in simulated seawater. The weight loss results demonstrated that 2% sodium nitrite was more effective with efficiency of 96% in comparison with 2% calcium nitrite and 6% vernonia amygdalina with efficiency of 92% and 75%, respectively. From the results, it is evident that the extract from vernonia amygdalina (bitter leaf) acts as an excellent corrosion inhibitor for rebar steel, efficiency increases with increase in dosage, and the inhibition mechanism was by physical adsorption which is responsible for shielding the specimen from corrosive environment.

Keywords: carbon steel, corrosion, inhibitor, vernonia amygdalina, chloride, concrete.

1. INTRODUCTION

The degradation of reinforcing steel due to corrosion is predominant in concrete structures. Generally, most structures are contaminated with chloride as a result of deicing during the winter and also in chloride laden environment such as marine for offshore and coastal structures [1]. Corrosion damage causes the weakening and quickens the aging of high way structures [2]. Environmental modification by means of inhibitors is one of the most effective measures for corrosion control in rebar embedded in concrete. Cost of inorganic inhibitor is low, however some of them are very toxic (harmful to both human and environment) such as chromate, mercride, arsenate etc.

Corrosion inhibitors are chemical substances which can prevent or reduce corrosion rate when present in adequate proportion. The use of inhibitors seems to be more promising due to their simplicity in application, and relatively less expensive. Generally, inhibitors are classified according to which electrode reaction they influence. They can be identified as anodic, cathodic or mixed inhibitors [3]. So far many investigations have been carried out on the effect of inorganic inhibitors on the corrosion of carbon steel in concrete. Calcium nitrite was able to reduce or inhibit initiated localized corrosion of rebar steel chloride contaminated concrete as reported in previous study [4]. Calcium nitrite can inhibit the initiated, localized corrosion of reinforcing steel in solutions simulating chloride contaminated concrete, if it is present in sufficient concentration in early stages of the corrosion process. Nitrites (calcium or sodium salt) are anodic inhibitors, they compete with chloride ions for the ferrous ions at the anode to form a film of ferric oxide (Fe_2O_3) [5]. Sodium nitrite inhibitor was found to be effective in reducing corrosion rate in reinforced steel concrete at 4%wt dosage [6]. However, efficiency decreases as chloride content increases [7]. The inhibitive mechanism of sodium nitrite on carbon steel is similar to that of calcium nitrite.

Moreover, the effect of plant extracts as corrosion inhibitor in mild steel in acidic medium have been studied. It has been reported that vernonia amygdalina (bitter leaf) and Azadirachita (Nee leaf) extracts performed optimally in acidic solution and that inhibition efficiency increases with increase in dosage. The inhibition mechanism of vernonia amygdalina is related to their physical adsorption properties due to the presence of tannin, alkaloid and saponnins [8, 9, 10]. The use of organic compound to reduce the corrosion of carbon steel has great importance due to their applications in prevent corrosion in different aggressive environments [11]. However, no study of vernonia amygdalina inhibition potency in reinforcing steel bar in concrete exposed to chloride laden environment has been reported. Based on the background of knowledge of vernonia amygdalina (bitter leaf), sodium nitrite and the traditional calcium nitrite inhibitors, the present study was designed to identify and evaluate the behaviour of sodium nitrite, vernonia amygdalina and calcium nitrite inhibitors on corrosion performance of carbon steel reinforced in concrete exposed to simulated seawater.

2. EXPERIMENTAL METHOD

2.1. Materials

The following materials were used for the study: substrates metal; carbon steel reinforcing bars, mild steel plates for weight loss test, likewise the inhibitors used were calcium nitrite, sodium nitrites and vernonia amygdalina inhibitors. Similarly, cement, fine and coarse aggregates, tap water and 3.5% NaCl solution were used as test solutions.

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2.2. Method

2.2.1. Preparation of vernonia amygdalina extract

The leaves were obtained from the plant in the neighborhood and thoroughly washed with water to remove unwanted materials and then weighed. The weighed amount (200g) was put in a bottle container, methanol was added and the container was tightly covered to prevent evaporation. The mixture was left for 48 hours to allow proper removal and concentration of the extracts. Afterward, the mixture was filtered to obtain a liquid residue containing methanol. The methanol was removed by heating the resulting solution over a water bath at76°C for 20 minutes in rotary evaporator model Buchi R-200.

2.3. Specimen dimension

In this study, specimen geometry that was used is cylindrical concrete block with dimension 75mm diameter and 150mm height in accordance to ASTM C685M-11. One plain carbon steel bar (grade 40) of diameter 10mm and length 170 mm was embedded at the centre of the each concretes (lollipop).

Both ends of the bars were covered with epoxy and tape with electroplater tape so that the exposure area of each specimen will not corrode; the schematic diagram of the sample dimension is shown in Figure-1(a). In addition, carbon steel plate of dimension 50x25x1.5mm was used for weight loss measurement for bare specimens without concrete as shown in Figure-1(b).

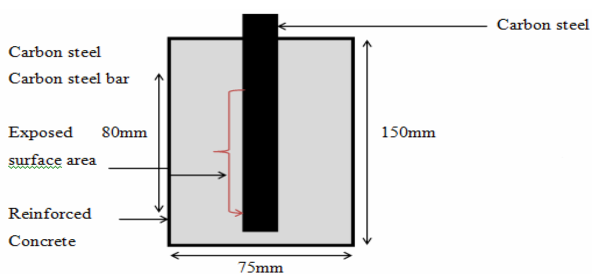

Figure-1(a). Low carbon steel bar embedded in cylindrical concrete block.

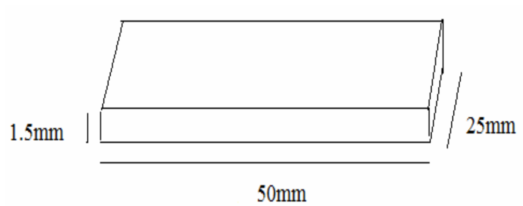

Figure-1(b). Schematic diagram of mild steel coupon.

2.4. Material characterization

The low carbon steel chemical composition was analyzed by Glow Discharge Spectrometer (GDS) model Leco 850A after proper grinding with silicon carbide paper of 220 grit size. The chemical composition wt% is as follows: C 0.032, Mn 0.193, P 0.012, S 0.014, Si 0.018, Fe 99.6. The mild steel plate used for the weight loss measurement was equally analyzed with the same GDS. Similarly, Nikon microphot -FX optical microscope was used to examine the microstructure of the carbon steel before the corrosion test.

2.5. Concrete sample preparation

Figure-2 shows cylindrical reinforced concrete making process. Concrete mix is very crucial in concrete making procedure. Batching, mixing and casting of entire set were done one day, so that all the specimens with same individual inhibitors will have the same age. Prior to the day of casting, all the aggregates were weighed to the nearest kilogram (0.01). Calcium nitrite and the plant extracts inhibitor were measured volumetrically before the mixing in different containers. However, sodium nitrite was weighed in the weighing balance. Hand mixing was used in accordance to ASTM C685 (0.07m³). The cubic volume of the concrete was calculated as follows;

Volume = $\pi r^2 h + 0.3\pi r^2 h$. The concrete mix design is shown in Table-1.

Where r is the radius of the cylinder in millimetres and h, the height of the cylinder.

Volume of the concrete for the cylinder was calculated to be: $8.62 \times 10^{-4} m^3$.

Seven concrete samples were used, each of the samples aggregates were mixed with water containing the inhibitors. Six samples were with inhibitors and one without; the inhibitors used were in proportion of $6L/m^3$ and $12L/m^3$ for each of the two samples containing calcium nitrite and vernonia amygdalina. However, the remaining two samples consist of 2%wt and 4%wt sodium nitrite inhibitors respectively.

Table-1. Concrete batch mix design (Kg/m³), w/c = 0.50.

Constituents	**Weight** (Kg/m³)
Cement	360
Coarse aggregates	1008
Fine aggregates	552
Water	180

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(a) Concrete sample in the mould. (b) Curing process of concretes.

Figure-2. Cylindrical concrete sample making process.

2.6. Corrosion test

Corrosion test was conducted for the sample without concrete (bare samples) and sample with concrete as explained in sections 2.6.1 to 2.6.6.

2.6.1. Visual inspection

Visual inspection is a Non Destructive Evaluation method of corrosion monitoring. It is the simplest and the oldest inspection technique. This method is economical and fast. This simply involves most often the use of the human eyes to observe the surface morphology. Although this techniques has some limitations because it can only provide qualitative results but not quantitative and sometimes pitting corrosion can only be detected with aided human eyes but it is always the first approach in corrosion inspection. In this study, the specimens were inspected prior and after the immersion time to examine surface appearances.

2.6.2. Weight loss measurement

Weight loss measurement was carried out with bare carbon steel prior to reinforced concrete immersion in order to assess the suitability of the inhibitors in 3.5% NaCl solution.

The size of the coupon used for the immersion test was 50x25x1.5mm. Twenty specimens were used, the four set of five coupons were abraided with silicon carbide from 220 to 1200 grits, and were washed with doubly deionized water, degreased with acetone then air dried at room temperature. The specimens were weighed in Ohaus electronic digital weighing balance model PA 214 and each weight was recorded subsequent to immersion. Five coupons suspended by inextensible thread were immersed in a non-conductive container of 3.5% NaCl solution of 2725ml (20ml/cm^2). Four ponding containers were used, each contained vernonia amygdalina extracts (organic inhibitors) 6%v/v, calcium nitrite 2%v/v, sodium nitrite 2%v/v inhibitors and the controlled experiment was without inhibitor. After each immersion period, specimens were removed from the ponding solution; cleaned, thoroughly washed with distilled water, acetone, dried and then weighed in order to evaluate the weight loss, rate of corrosion, inhibition efficiency and degree of surface covering. The weight loss of each coupon was determined at 7 days, 14 days, 28 days, 42 days and 56 days. The study was conducted at temperature range within 28^0C-35.7^0C.

2.6.3. Determination of corrosion rate (mmpy) and inhibition efficiency (%) for weight loss test

The weight loss was calculated by finding the variation between initial and final weight of each coupons after each immersion period from the relationship [3].

$$W = W_i - W_f \quad (1)$$

W_i = initial weight, W_f = final weight

$$CPR = mmpy = 8600W/\rho At \quad (2)$$

W is the weight loss (mg) after exposure time t (h), ρ is density of metal (g/cm³) and A is the area of the specimen (cm²) and t, time of exposure in hours.

The standard expression for measurement of corrosion rate in millimetre per year (mmpy) in equation (2) above was used to determine the corrosion rate as given [3].

The inhibition efficiency (IE %) was evaluated as given in the equation (3) using the relationship [3].

$$IE\ \% = \left(1 - \frac{W}{Wo}\right) \times 100\% \quad (3)$$

Where *W* and *Wo* are the corrosion rates with and without inhibitor, respectively.

2.6.4. Half-cell potential measurement

Electrochemical method for corrosion rate measurement is the simplest monitoring techniques for reinforcing bar embedded in concrete. In this study, copper-copper sulphate half-cell was used to measure the half-cell potentials of the steel rebar according to ASTMC 876-87 G3. Corrosion potential readings were taking by placing the copper-copper sulphate electrode (CSE) firmly on the surface of the concrete at portions of the cylinder block specimens (mapping) at close range interval, the measurement were carried out after the samples have been removed from immersion solutions for 2 hours to allow diffusion of air through concrete pores.

2.6.5. Concrete resistivity measurement

Electrical resistivity of concrete could be an effective parameter to assess the risk of corrosion in reinforcing steel embedded in concrete especially for chloride induced corrosion. AC and DC are the two widely used techniques used for determining concrete resistivity.

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In the study, the Wenner four probes method was used as shown in Figure-3. This was done by placing the four probes in contact with the concrete directly above the reinforcing steel bar. Different readings were taken at different locations at the surface of the concrete. The mean values of the readings were recorded as the final readings of the resistivity in the study.

Figure-3. Concrete electrical resistivity measurement.

2.6.6. Determination of corrosion rate (mmpy) for reinforcing steel bar in concrete

The corrosion rates in each of the specimens were evaluated from corrosion current density $I_{corr.}$ Using the formulae as stated below:

$$Icorr. = B/R_p \quad (4)$$

Stern Geary relationship (Stern 1975).

Where

Icorr = Corrosion current density, μA/cm^2.
B = Stern -Geary constant, 26mV (Andrade, 1978).
R_p = Polarization resistance, Ohm.cm$^{2.}$
Corrosion rates in mm/yr was calculated as; 0.0116 ×Icorr. as given by (Devi and Kannan, 2011).

3. RESULTS AND DISCUSSIONS

3.1. Vernonia amygdalina phytochemical composition

The leaves extract was characterized by IR spectroscopic techniques and quantitative analyses. From the screening, the result shows that the plant extract contains alkaloids, saponin and tannin as shown in Table-2.

Table-2. Phytochemical screening.

Phytochemicals tested	Remark
Alkaloids	Positive
Saponins	Positive
Tannins	Positive

3.2. Microstructural analysis of low carbon steel specimen

Nikon Optical microscopy was used to examine the microstructure of the specimens. Figure-4 shows the results of the microstructure under 200 and 500 magnifications respectively. From these results, it can be visibly observed that the specimen consists of pearlite (dark grey areas) and ferrite (light grey regions). The ferrite matrix consists of larger percentage ferrite and small amount of pearlite by composition. It can therefore be deduced from this result that the microstructure is a typical of low carbon steel.

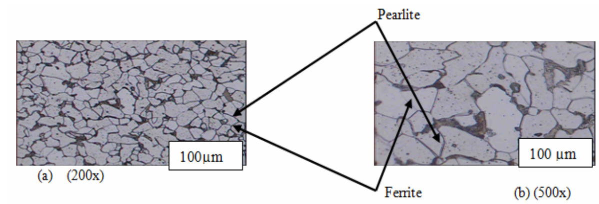

Figure-4. Optical micrograph of low carbon steel.

3.3. Weight loss measurement after immersion duration

From the chart in Figure-5, it can be seen that the weight loss for samples without inhibitor increased linearly from approximately 0.020 to 0.029g with an average loss of 0.111 g/wk. On the other hand, samples treated with 2%v/v sodium nitrite, 2% v/v calcium nitrite and 6% v/v vernonia amygdalina demonstrate a reduction in weight loss in comparison with the sample without inhibitor.

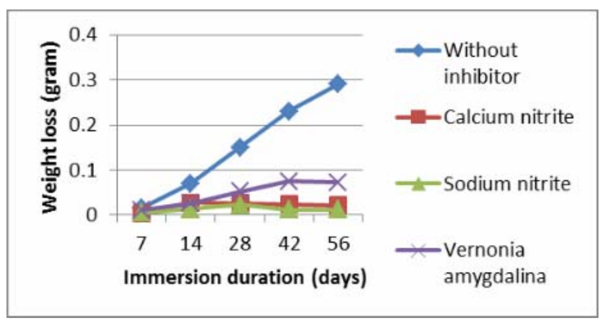

Figure-5. Weight loss as a function of immersion duration for carbon steel immersed in 3.5%NaCl solution.

3.4. Corrosion rate measurement after immersion duration

From Figure-6, it is evident that the corrosion rates of the unprotected carbon steel increased dramatically from 0.04mm/yr to 0.09mm/yr and then reduced slight to 0.088mm/yr at the end of the eight week. On the other hand, sodium nitrite inhibitor proved to be effective at 2%v/v in 3.5% NaCl solution. In similar trend, corrosion rates for samples with calcium nitrite inhibitor increased from 0.001mm/yr in day 7 to 0.033mm/yr in the second week and declined significantly to 0.007mm/yr at the end of the test. The presences of tannins, alkaloids and saponnins in vernonia amygdalina acts as a barrier on the metal surface, thus preventing the diffusion of ions from or to the surface of the corrodent thereby blocking the

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anodic or cathodic site which consequently reduces corrosion rates as reported by [10].

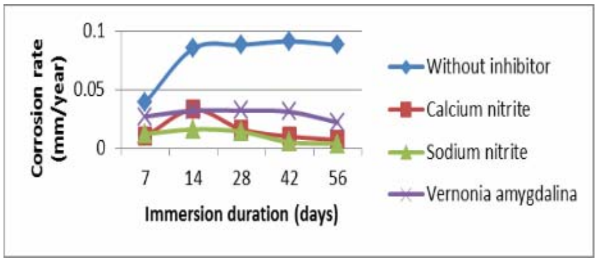


Figure-6. Corrosion rate (mm/yr) versus immersion duration for mild steel immersed in 3.5% NaCl solution for 56 days.

3.5. Inhibition efficiency

Figure-7 shows the inhibition efficiency of the various inhibitors used for the weight loss test within the 56 days of immersion of immersion time. From the chart, it can be inferred that sodium nitrite inhibitor exhibits the highest inhibition efficiency of 96% at the end of the test. The inhibition increases from 68.8% in day 7 to 96% at the end of the study. This shows that the oxide films formed on the carbon surface were thick and stable enough to diminish corrosion rate due to the passivation influence of the nitrite inhibitor. The ferrous ions are converted into stable and insoluble hydrated ferric oxides in the region of stability of the oxide [12].

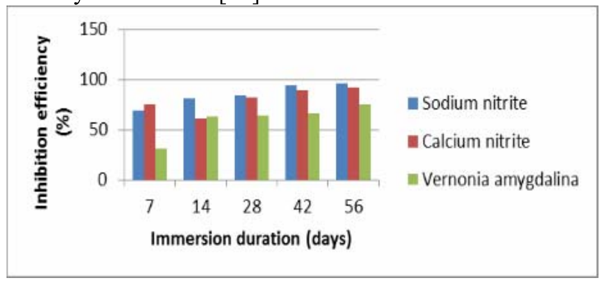


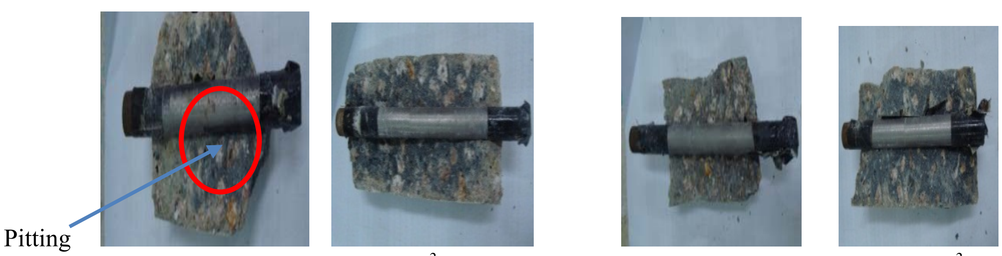
Figure-7. Inhibition efficiency versus immersion duration.

3.6. Corrosion evaluation of reinforced carbon steel in concrete immersed in solution

Corrosion behaviour of reinforced bar was evaluated as explained in sections 3.6.1 to 3.6.4.

3.6.1. Visual inspection

Visual inspection is a Non Destructive Evaluation method for corrosion monitoring. It is the simplest and the oldest inspection technique. Although this technique has some limitations however, it is always the first approach in corrosion inspection. At the end of the end of the experiment, pitting corrosion was observed only on the sample without inhibitor. Figure-8 shows the morphology of the carbon steel after the immersion time.



(a) Sample without inhibitor (b) 12L/m^3 calcium nitrite (c) 2%wt sodium nitrite (d) 12L/m^3 VA.

Figure-8. Visual inspection of reinforcing bar in concrete immersed in 3.5% NaCl solution for 56 days.

3.6.2. Half-cell potential measurement

Figures 9 and 10, it can be ascertained that no pitting corrosion initiation is envisage for all the samples with corrosion inhibitors at the end of the experiment, in an aerated concrete, potential range is normally from +100mV to -200mV SCE [13]. However, for low level of chloride content (0 to 0.3) % by mass of cement, corrosion potential +250mV and below shows that the probability of corrosion initiation is negligible However, there is the likelihood of corrosion initiation and propagation above this corrosion potential in chloride environment [14]. In addition, the visual inspection confirmed minute regions of pitting in the unprotected samples.

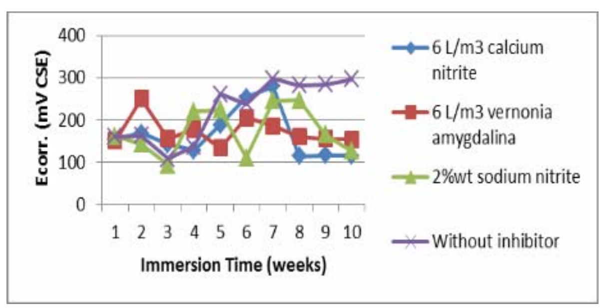


Figure-9. Corrosion potential as a function of time for immersion samples in 3.5% NaCl solution.

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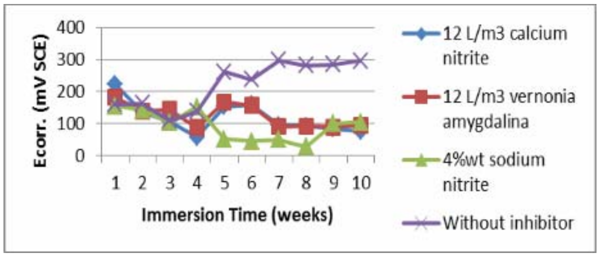

Figure-10. Corrosion potential as a function of time for immersion samples in 3.5% NaCl solution.

3.6.3. Concrete resistivity

Figures 11 and 12 show the plots of concrete electrical resistivity as a function of immersion duration for specimens immersed in 3.5% NaCl solution for 10 weeks. It can be observed that the curves follow the same upward trends. However, sodium nitrite inhibitor assumed a lower resistance position. This is in accordance with the findings of previous study [15]. The lower resistivity is due to higher ions as a result of the presence of Na^+ and NO_2^- in the aqueous phase filling the pores capillary. According to Morris *et al*., [16], reinforcing steels are in active corrosion risk when ρ_c is below 10kΩcm and attain passivity at resistivity greater than 30kΩcm. Previously study [17] reveals that resistivity above 20kΩcm indicates negligible corrosion risk while between 10kΩcm to 20kΩcm suggests low corrosion risk. However, 4%wt sodium nitrite exhibits a lower resistivity of about15kΩcm among the three inhibitors as a result of aforementioned reason. In addition it is also obvious to reiterate that sodium nitrite inhibitor gives optimum inhibition effect in concrete with inhibition composition between 2-3%wt by cement [18].

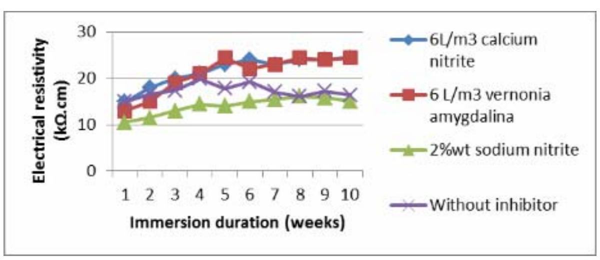

Figure-11. Concrete electrical resistivity versus immersion time in 3.5% NaCl solution.

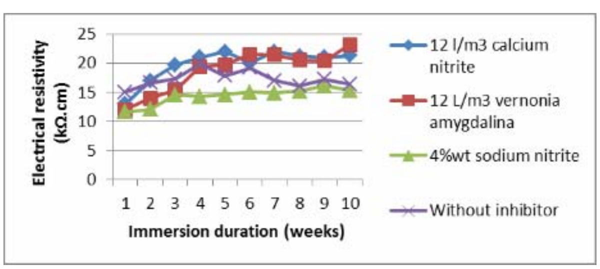

Figure-12. Concrete electrical resistivity versus immersion time in 3.5% NaCl solution.

3.6.4. Corrosion rate

From Figure-13, within the limited period of the study, the corrosion rate was plotted against immersion time for reinforcing bar in concrete samples immersed in 3.5% NaCl solution. Sample without inhibitor shows insignificant protection, this is as a result of diffusion of chloride ions due to differences in concentration gradient between the immersion solution and the concrete pores solution. The corrosion rate decreased significantlly for vernonia amygdalina [19] and calcium nitrite [20] for 12L/m^3 inhibitors dosage. However, increased in sodium nitrite inhibitors dosage to 4%wt reduces the inhibition performance by 8% this attribute agrees with the findings of Devi and Kannan [18]. Overall, the inhibition performance is in order of; vernonia amygdalina □ calcium nitrite □ sodium nitrite □ without inhibitor.

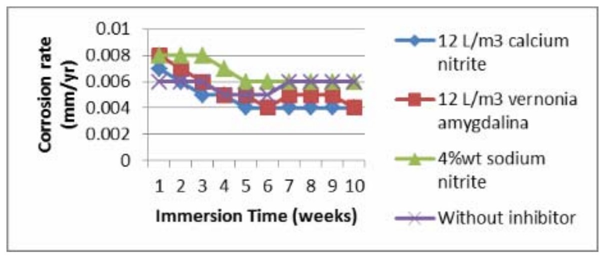

Figure-13. Corrosion rates (mm/yr) versus immersion time for carbon steelin concrete immersed in 3.5% NaCl solution.

4. CONCLUSIONS

a) Vernonia amygdalina (plant extract) inhibitor shows excellent Ecorr of +95mV and calcium nitrite +85mV for 12L/m^3 within the 70 days of immersion. This shows that eco-friendly inhibitor can be harnessed for corrosion inhibition in reinforcing concrete for marine and chloride laden environment. However, 2%wt sodium nitrite falls within +126mV at the end of the study.
b) The degree of reduction in corrosion rates in reinforcing carbon steel was higher in vernonia amygdalina inhibitor compare to calcium nitrite and sodium nitrite within the immersion period.
c) Weight loss test revealed that sodium nitrite inhibitor shows higher inhibition efficiency of 96% followed by calcium nitrite with 91% for 2%v/v inhibitor. However, vernonia amygdalina has inhibition efficiency of 75% with 6%v/v. The nitrites can therefore be used as inhibitors in coolants.

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