

## Mild Steel Corrosion Inhibition in H<sub>2</sub>SO<sub>4</sub> Using Ethanol Extract of Vernonia Amygdalina

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### ABSTRACT

The study of inhibition of corrosion of mild steel in 1M H<sub>2</sub>SO<sub>4</sub> medium using ethanol extract of vernonia amygdalina was carried out using weight loss, and scanning electron microscopy. The results reveal that ethanol extract of vernonia amygdalina inhibited corrosion of mild steel. The inhibition efficiency ranged from 83.91% to 89.11%, 81.09 % to 85.00%, 81.08% to 84.90% and 77.45% to 79.66% at 303, 313, 323 and 333K respectively. The inhibition efficiency of the extract increased as the concentration of the extract increases but decreased as temperature and time of immersion increases. Scanning electron microscopy also revealed that ethanol extract of vernonia amygdalina inhibited the corrosion of mild steel by forming a protective film.

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### I. INTRODUCTION

The corrosion of metals remains a worldwide scientific problem as it affects the metallurgical, chemical and oil industries. The increasing interest in the manufacture of sulphuric acid has created the need for obtaining information on the corrosion resistance of mild steel to sulphuric acid attack.<sup>[1]</sup> Corrosion is the deterioration of metal by chemical attack or reaction with its environment. It is a constant and continuous problem, often difficult to eliminate completely. Prevention would be more practical and achievable than complete elimination. Corrosion processes develop fast after disruption of the protective barrier and are accompanied by a number of reactions that change the composition and properties of both the metal surface and the local environment, for example, formation of oxides, diffusion of metal cations into the coating matrix, local pH changes, and electrochemical potential.<sup>[2,3,4,5]</sup> The study of corrosion of mild steel and iron is a matter of tremendous theoretical and practical concern and as such has received a considerable amount of interest. Acid solutions, widely used in industrial acid cleaning, acid descaling, acid pickling, and oil well acidizing, require the use of corrosion inhibitors in order to restrain their corrosion attack on metallic materials. Mild steel is commonly used for industrial structures due to its strength, ductility, weldability and because of its amenability to heat treatment for varying mechanical properties.

However, mild steel corrode easily because most common structural metals form surface oxide films when exposed to atmospheric environment, but the oxide formed on mild steel is readily broken down and in the presence of moisture it is not repaired. Therefore, a reaction between Iron (Fe), Moisture (H<sub>2</sub>O), and Oxygen (O<sub>2</sub>), takes place to form rust. This reaction is complex but can be represented by a chemical equation as follows:



2Fe<sub>2</sub>O<sub>3</sub>·H<sub>2</sub>O is the rust, and as it is not usually protective, the corrosion process is not impeded.

Mild steel corrosion phenomenon has become important particularly in acidic media because of the increased industrial application of acid solution.<sup>[6]</sup> In order to reduce the corrosion of metals, several techniques have been applied. The use of inhibitors during acid pickling procedure is one of the most practical methods for protection against corrosion in acidic environment.<sup>[7]</sup> Most of the efficient and effective organic inhibitors are those compounds containing hetero-atoms in their aromatic or long carbon chain.<sup>[8]</sup> To be effective, an inhibitor must displace water from the metal surface, interact with anodic or cathodic reaction sites to retard the oxidation and reduction corrosion reaction, and prevent transportation of water and corrosion active species on the surface.<sup>[7,9]</sup> Inhibitors which reduce corrosion on metallic material can be divided into three kinds; Inorganic inhibitors, Organic inhibitors, and Mixed materials inhibitors.

The application of these inhibitors for corrosion control, factors such as cost, toxicity, availability and environmental friendliness are very important.<sup>[9]</sup> According to Ebesson et al, the last class of inhibitors (green

inhibitors) are significant because they are non-toxic and do not contain heavy metals. Hence they are environmental friendly<sup>[7]</sup>.

Naturally occurring substances as inhibitors of acid cleaning process have continued to receive attentions as replacement for synthetic organic inhibitors. The greatly expanded interest on naturally occurring substances is attributed to the fact that, not only that they are readily available, ecologically friendly, and pose no threat to the environment but they are cheap and biodegradable and are renewable source of material<sup>[10]</sup>.

Several researches have been done on the use of extract of plant as inhibitor for metals against corrosion in different aggressive media. However, not much has been reported on the use of the extract of Vernonia Amygdalina plant as inhibitor against corrosion of mild steel in H<sub>2</sub>SO<sub>4</sub>. This study is aimed at investigating the efficiency of Vernonia Amygdalina in inhibition of mild steel in acidic environment.

To achieve this aim, the following objectives have to be met: Determining the potential of ethanol extract of Vernonia Amygdalina leaves as corrosion inhibitor of mild steel in tetraoxosulphate (VI) acid, Determining the effect of different concentrations of ethanol extract of Vernonia Amygdalina on the corrosion of mild steel in 1M tetraoxosulphate (VI) acid at different temperatures (303 – 333K), To correlate theoretical/calculated inhibition efficiencies with experimental results, and Propose the mechanism of the corrosion of mild steel in H<sub>2</sub>SO<sub>4</sub>.

## II. MATERIALS AND METHODS

Mild steel sheets of composition (wt %) Mn (0.6), P(0.36), C(0.15) and Si(0.03) were used for the study. The sheet was mechanically cut into coupons with dimension 5cm x 4cm. The coupons were degreased by washing in absolute ethanol, dried in acetone and stored in moisture free desiccators before use. All reagents used for the study were analar grade and double distilled water was used for their preparation. The concentrations of H<sub>2</sub>SO<sub>4</sub> used for weight loss was 1M. The inhibitor, (*Vernonia Amygdalina*) was gotten from Nkwe Town, Awgu L.G. A., Enugu State, Nigeria.

### 2.1 Preparation of Inhibitor

**Extraction of Plants-** Samples of Vernonia Amygdalina was dried, ground and soaked in a solution of ethanol. After 48 hours, the sample was cooled and filtered. The filtrate was subjected to evaporation (to leave the sample free of the ethanol) using a rotary evaporator. The stock solution of the extract so obtained was used to prepare 0.1g/L, 0.2g/L, 0.3g/L, 0.4g/L and 0.5g/L in 1M of H<sub>2</sub>SO<sub>4</sub>.

### 2.2 Preparation of Reagents

The concentrations of the various reagents used for this study were prepared using recommended methods. For weight loss methods, the concentrations of tetraoxosulphate (VI) acid used was 1M. The concentrations of the inhibitor were 0.1g/L, 0.2g/L, 0.3g/L, 0.4g/L and 0.5g/L ethanol extract of Vernonia Amygdalina. Each of these solutions was dissolved in 1M H<sub>2</sub>SO<sub>4</sub> for use in weight loss method.

### 2.3. Corrosion Monitoring

**2.3.1 Weight Loss Method-** A previously weighed metal (mild steel) was completely immersed in 250ml of the test solution (different concentrations of inhibitors) in an open beaker. The beaker was inserted into a water bath maintained at a temperature of 303°K. Similar experiments were repeated at 313, 323 and 333°K. In each case, the weight of the sample before immersion was measured using Scaltec high precision balance (Model SPB31) After every 24hours, each sample was removed from the test solution, washed in a solution of NaOH containing zinc dust and dried in acetone before re-weighing. The difference in weight for a period of 120h was taken as total weight loss. The inhibition efficiency (% $\eta$ ) for each inhibitor was calculated using the formula;

$$\% \eta = 1 - \frac{W_1}{W_2} \times 100 \quad (1)$$

Where, W<sub>1</sub> and W<sub>2</sub> are the weight losses (g/dm<sup>3</sup>) for mild steel in the presence and absence of inhibitor in H<sub>2</sub>SO<sub>4</sub> solution respectively.

The degree of surface coverage  $\theta$  is given by the Eq. (2):

$$\theta = 1 - \frac{W_1}{W_2} \quad (2)$$

The corrosion rates for mild steel corrosion in different concentrations of the acid and other media have been determined for 120h immersion period from weight loss using Eq. 3<sup>[11]</sup>

$$\text{Corrosion rate (mpy)} = \frac{534W}{DAT} \quad (3)$$

where W = weight loss (mg); D = density of specimen (g/cm<sup>3</sup>), A = area of specimen (square inches) and T = period of immersion (hour).

### 2.3.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy of the corroded mild steel in 1M  $H_2SO_4$  and in the presence of *Vernonia Amygdalina* were carried out at National Research Institute of Chemical Technology (NARICT), Zaria, Kaduna State, Nigeria using Scanning Electron Microscope.

## III. RESULTS AND DISCUSSION

### 3.1 Gravimetric Study

Figs. 1 to 12 show plots for the variation of weight loss with time during the corrosion of mild steel in 1M  $H_2SO_4$  containing various concentrations of ethanol extract of *Vernonia Amygdalina* and in the absence of inhibitor at 303, 313, 323 and 333 K respectively. The plots generally reveal that weight loss of mild steel increases with time but decreases with increase in the concentration of ethanol extract of *Vernonia Amygdalina* indicating that the corrosion rate of mild steel increases with increase in the period of contact of the metal with the acid. The result also reveal that ethanol extract of *Vernonia Amygdalina* retarded the corrosion of mild steel in solution of  $H_2SO_4$ , hence it is a good adsorption inhibitor because the corrosion rate decreases with increase in concentration. Comparing Figs. 3, 6, 9 and 12, it is evident that the weight loss of mild steel increases with increase in temperature indicating that the corrosion rate of mild steel also increases with increase in temperature.

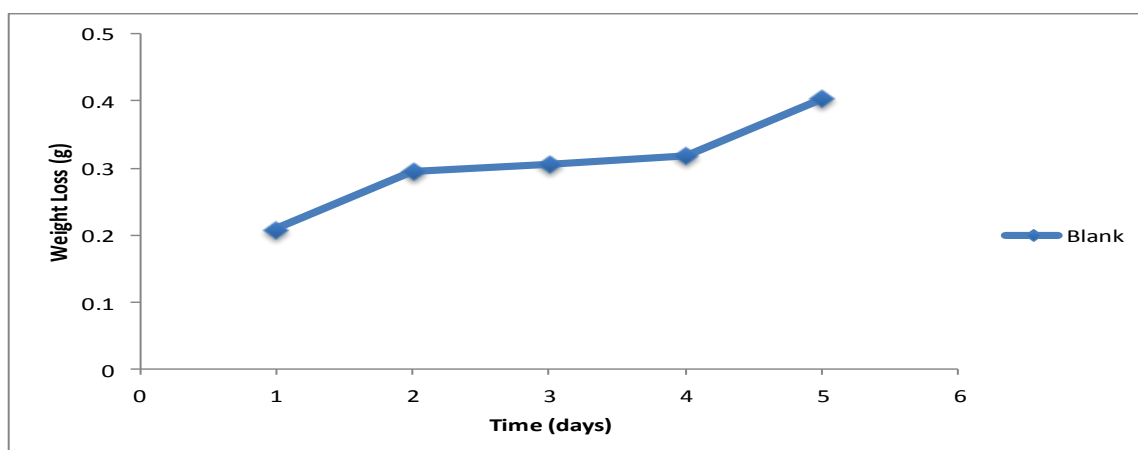


Fig. 1: Plot of Weight Loss with Time for the Corrosion of Mild Steel in 1 M  $H_2SO_4$  in the absence of Inhibitor at 303 K

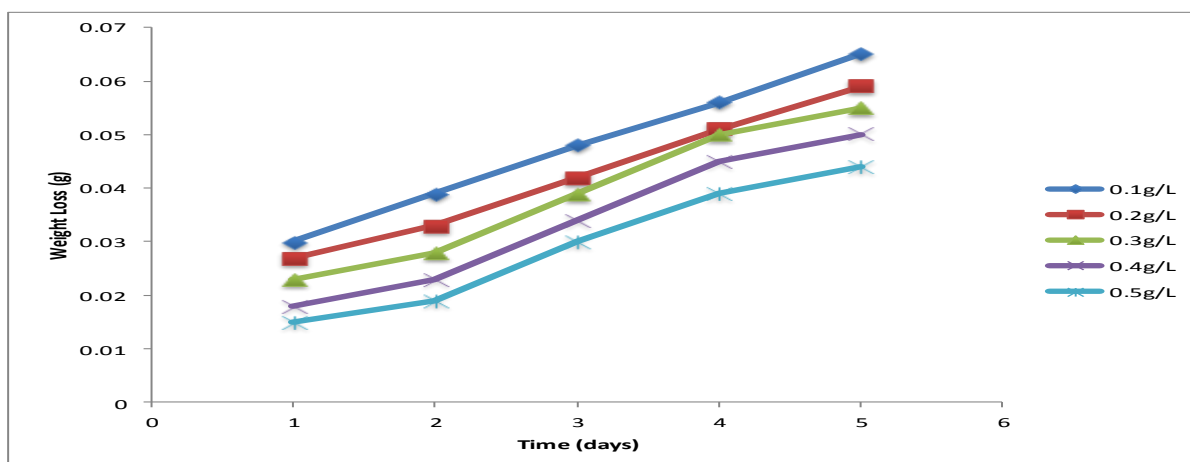


Fig. 2: Weight Loss with Time for the Corrosion of Mild Steel in 1 M  $H_2SO_4$  Containing Various Concentrations of *Vernonia Amygdalina* as Inhibitor at 303 K

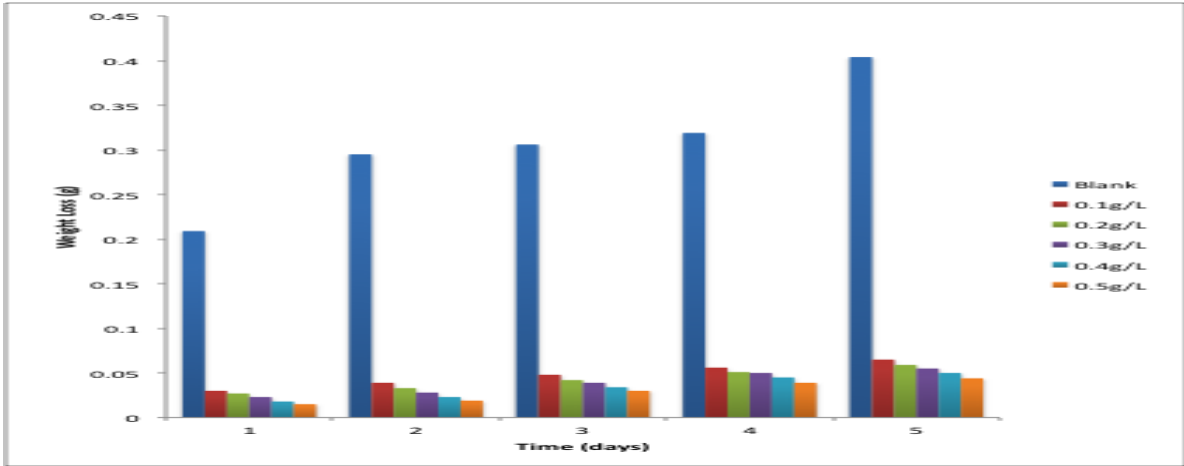


Fig. 3: Variation of Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> Containing Various Concentrations of *Vernonia Amygdalina* as Inhibitor and in the absence of the inhibitor at 303 K

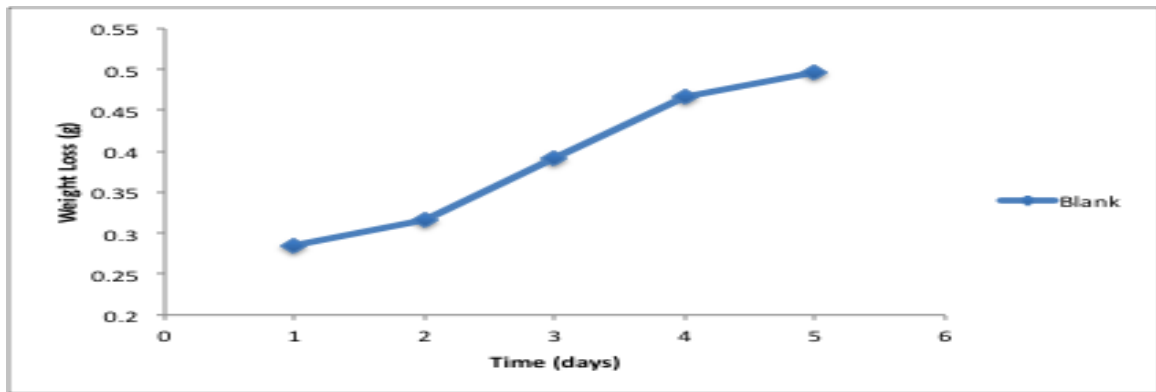


Fig. 4: Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence of Inhibitor at 313 K

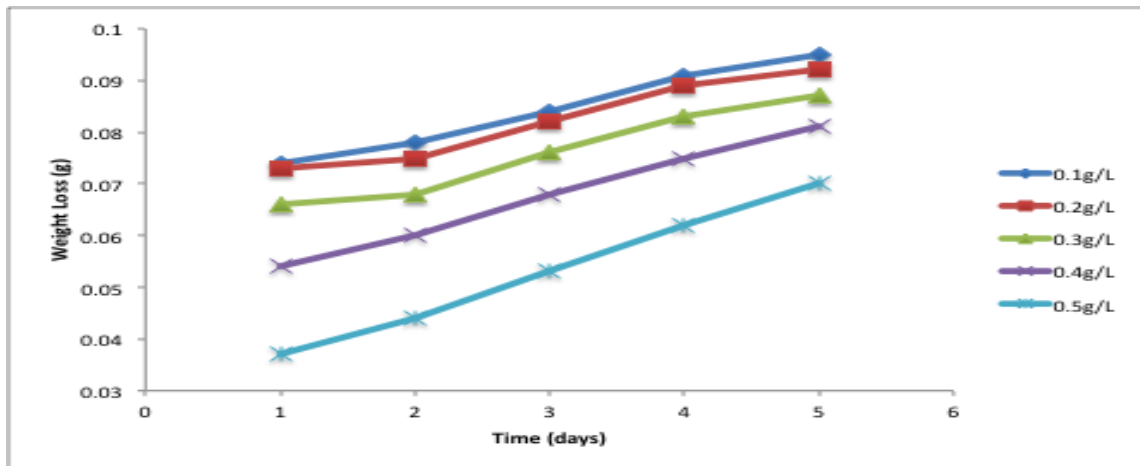


Fig. 5: Variation of Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> Containing Various Concentrations of *Vernonia Amygdalina* as Inhibitor at 313 K

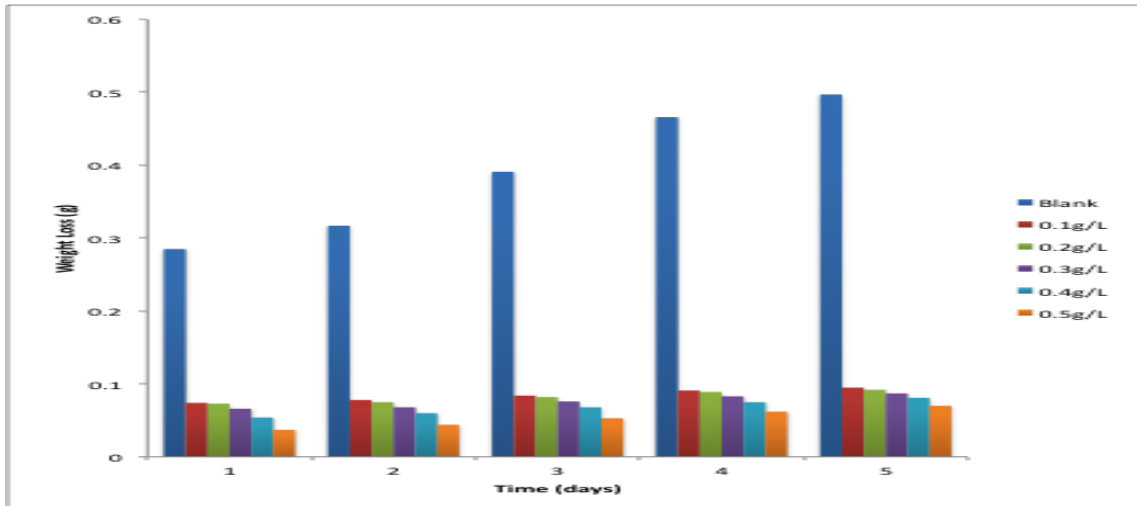


Fig. 6: Variation of Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> Containing Various Concentrations of *Vernonia Amygdalina* as Inhibitor and in the absence of the inhibitor at 313 K

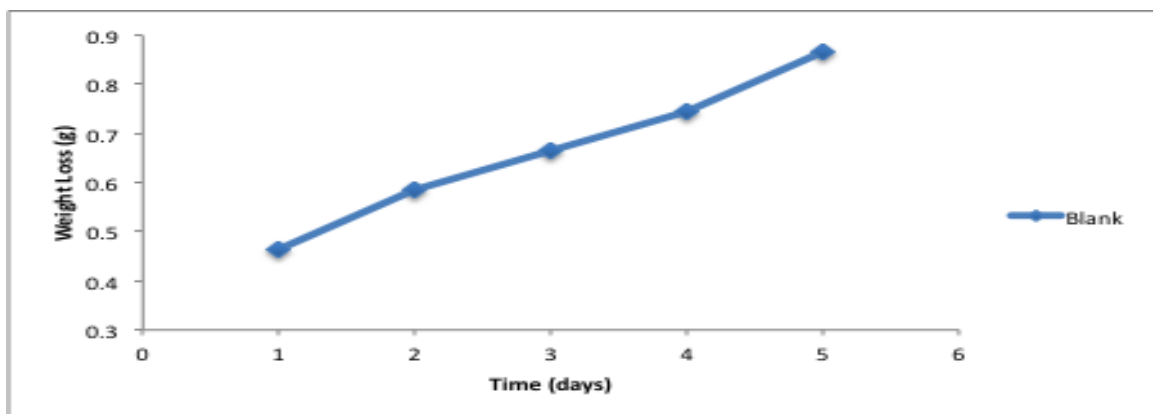


Fig. 7: Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence of Inhibitor at 323 K

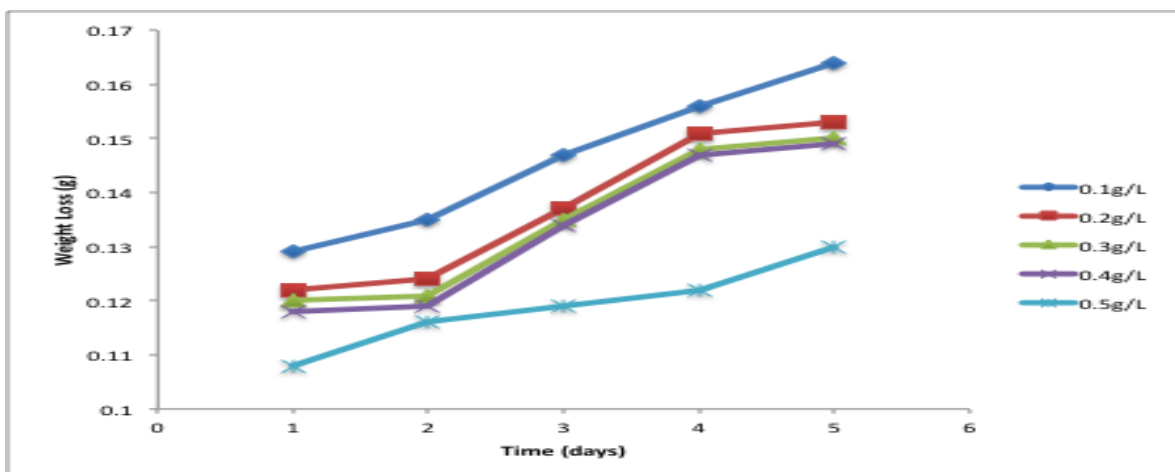


Fig. 8: Variation of Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> Containing Various Concentrations of *Vernonia Amygdalina* as Inhibitor at 323 K

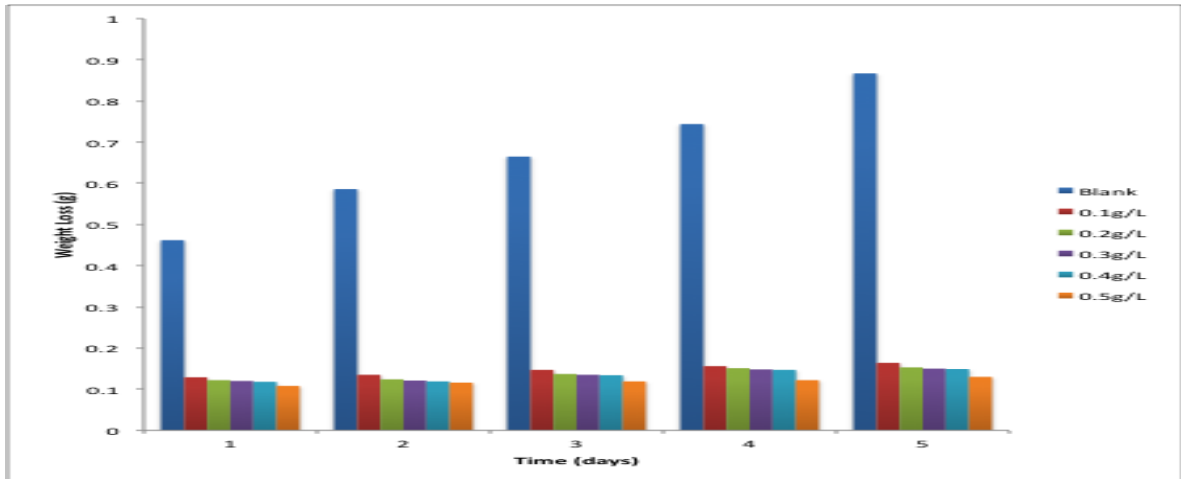


Fig. 9: Variation of Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> Containing Various Concentrations of *Vernonia Amygdalina* as Inhibitor and in the absence of the inhibitor at 323 K

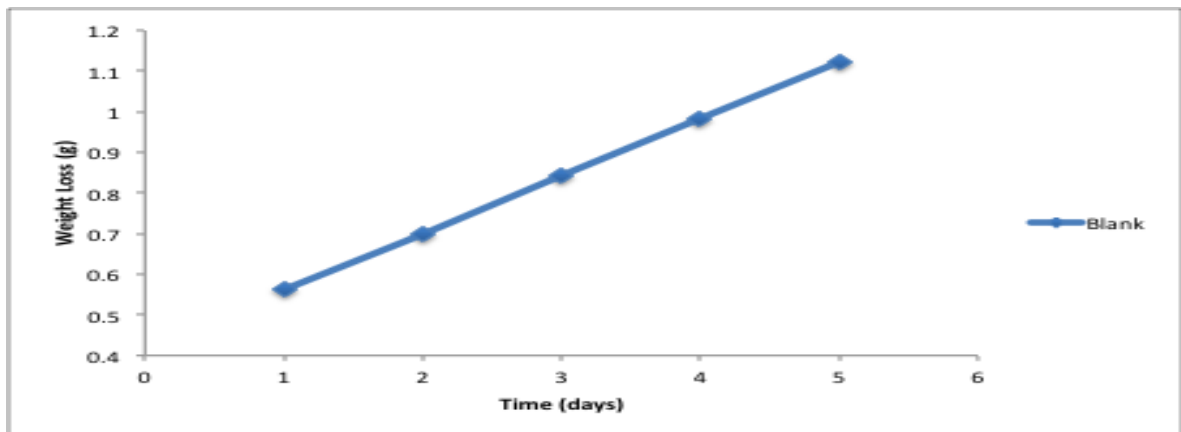


Fig. 10: Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence of Inhibitor at 333 K

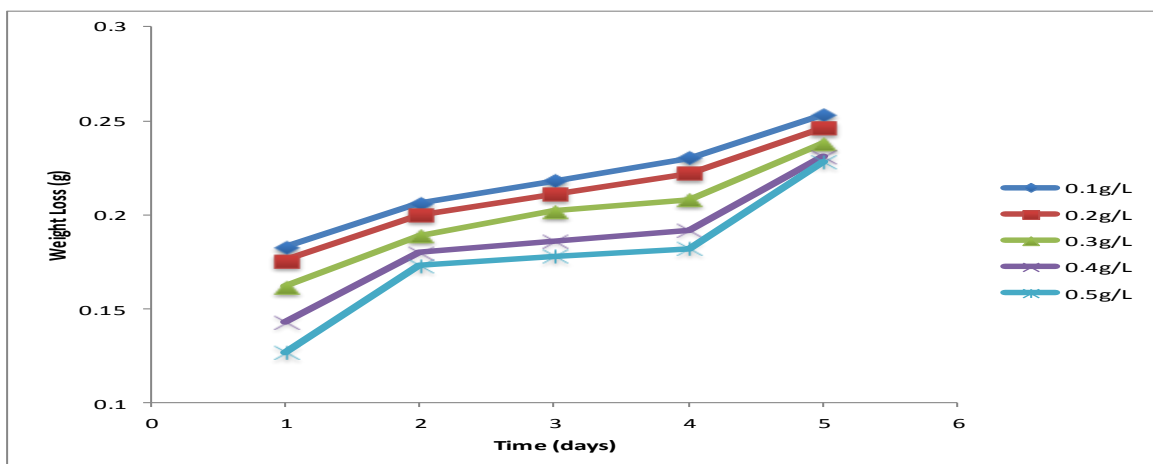


Fig. 11: Variation of Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> Containing Various Concentrations of *Vernonia Amygdalina* as Inhibitor at 333 K

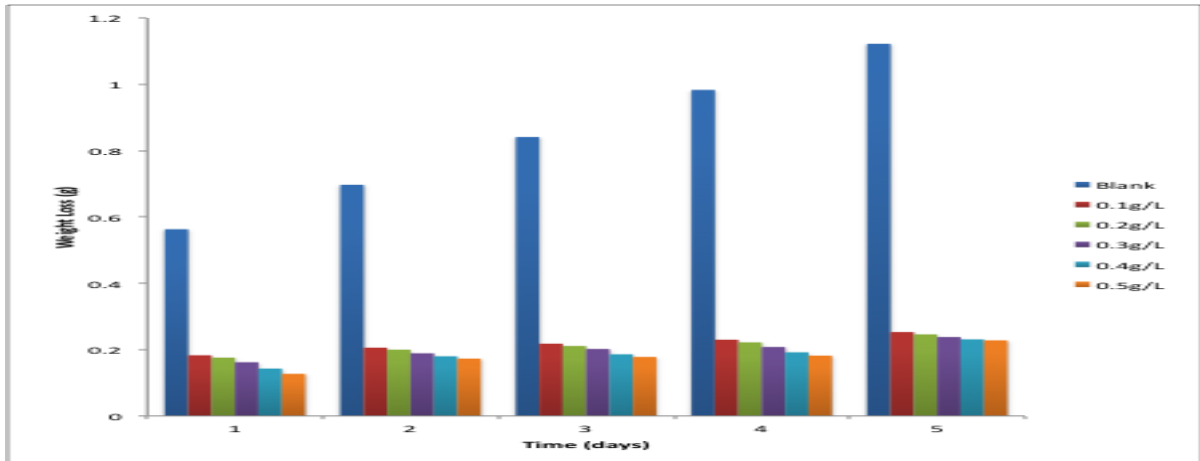


Fig. 12: Variation of Weight Loss with Time for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> Containing Various Concentrations of *Vernonia Amygdalina* as Inhibitor and in the absence of the inhibitor at 333 K

The results in Figs. 13 and 14 indicate that in the presence of ethanol extract of *Vernonia Amygdalina*, the corrosion rate is decreased, even as the concentration of the extract increases.

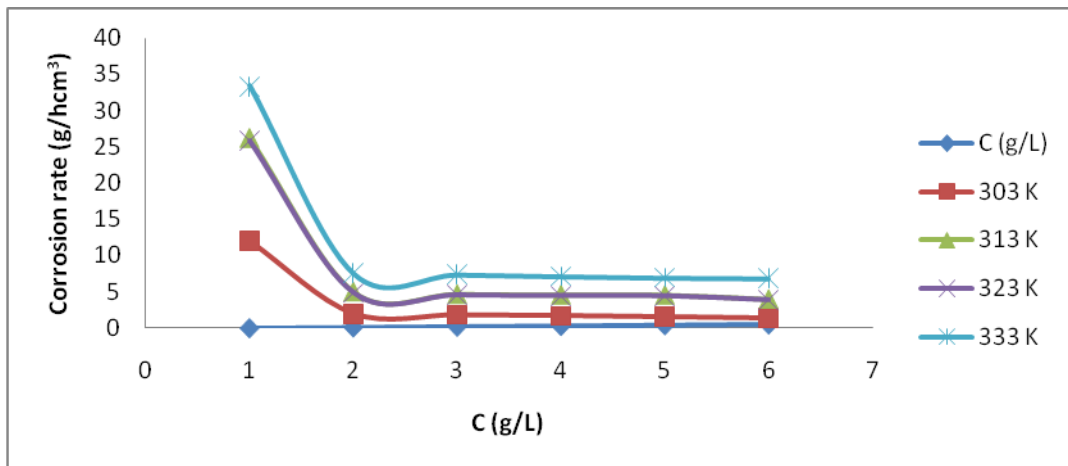


Fig. 13: Variation of Corrosion Rate of Mild Steel with Concentration of *Vernonia Amygdalina* at Various Temperatures

The plot generally reveals that the decrease in corrosion rate varies with the concentration of ethanol extract of *Vernonia Amygdalina*. From the results obtained shows that the inhibition efficiency seems to increase with concentration (Fig. 14) but decreases with temperature indicating that the mechanism of inhibition of the corrosion of mild steel in solution of H<sub>2</sub>SO<sub>4</sub> is physical adsorption.

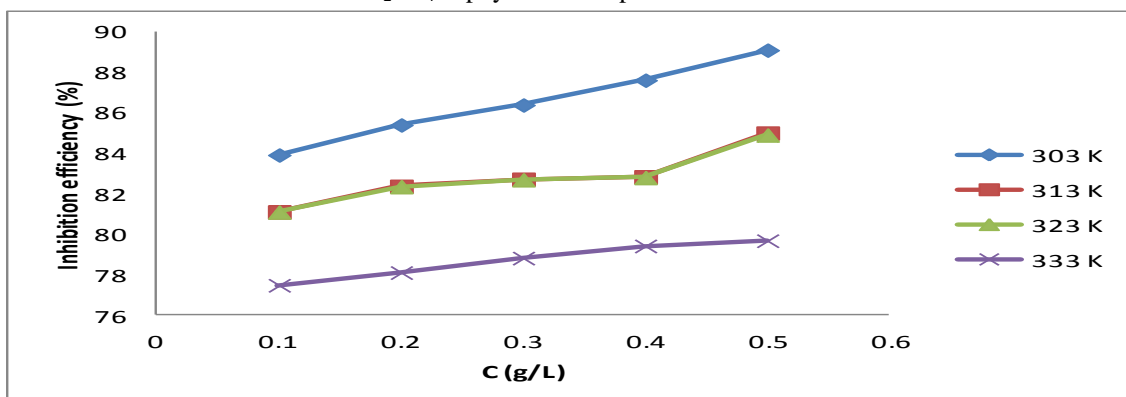


Fig. 14: Variation of the Inhibition Efficiency of *Vernonia Amygdalina* for the Corrosion of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub> at Various Temperatures



A physical adsorption mechanism is characterized with a decrease in inhibition efficiency with temperature as opposed to chemical adsorption mechanism, where inhibition efficiency is expected to increase with increase in temperature [12, 13]

### 3.2 Scanning Electron Microscopy (SEM)

A scanning electron microscope is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. It can achieve results better than 1 nm. In corrosion study, scanning electron microscope has been widely used to study the morphology of the metal before and after inhibition. Fig. 10 shows scanning electron micrograph of mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> while Fig. 11 shows the scanning electron micrograph of mild steel metal in 1 M H<sub>2</sub>SO<sub>4</sub> after inhibition by *Vernonia Amygdalina*.

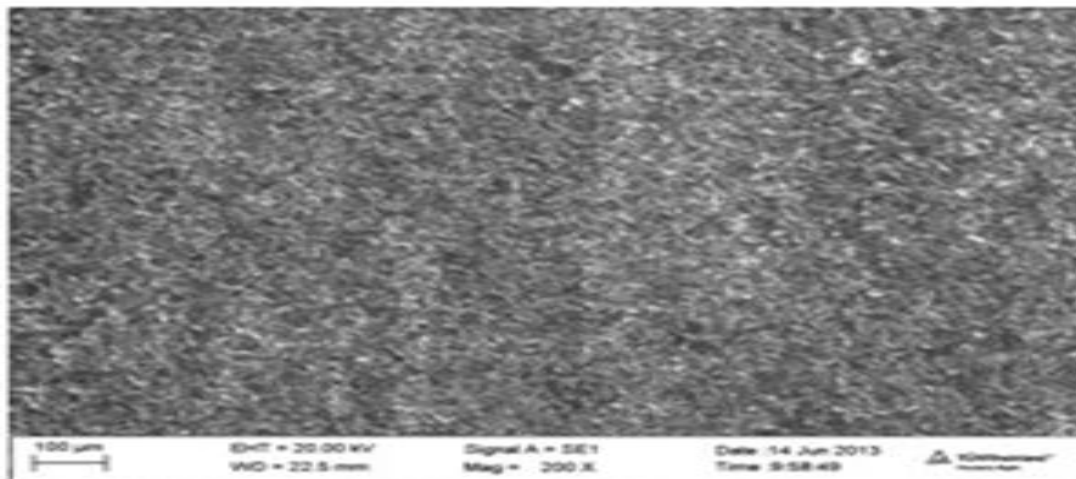


Plate 1: Scanning Electron Micrograph of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub>  
(Without inhibitor)

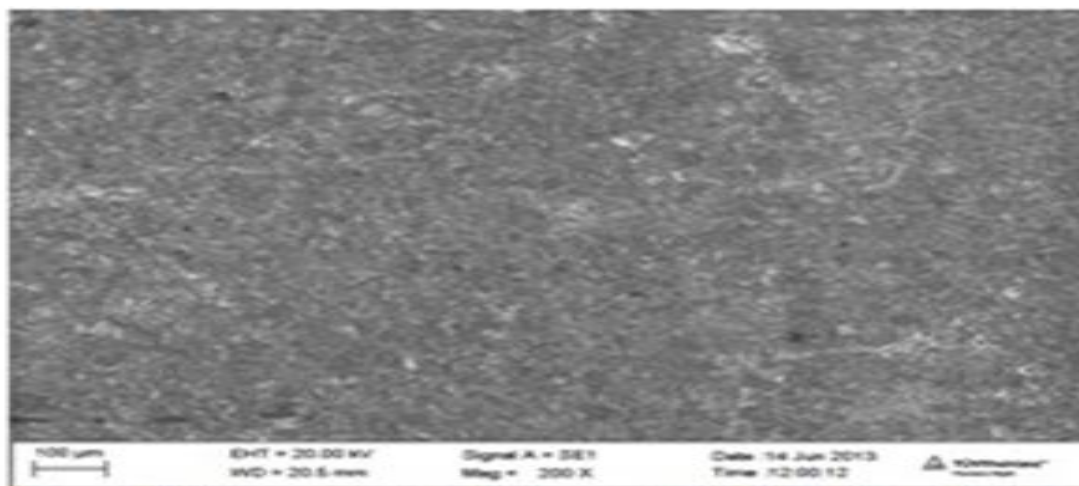


Plate 2: Scanning Electron Micrograph of Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub>, in the Presence of 70 mg/L of *Vernonia Amygdalina* as an Inhibitor

Plates 1 and 2 show the scanning electron micrograph of mild steel in the absence and presence of inhibitor respectively. A close comparison of the two images reveals that *Vernonia Amygdalina* inhibited the corrosion of mild steel by forming a protective film. From Plate 1, flakes showing corrosion product are observed in the micrographs indicating that the surface is strongly damaged by corrosion but in the presence *Vernonia Amygdalina* (Plate 2) as an inhibitor, it can be seen that the damaging effect is greatly reduced and the surface is relatively smooth probably due to the formation of protective coverage by the inhibitor. The results obtained from the scanning electron micrographs of *Vernonia Amygdalina* is consistent with the fact that this compound inhibited the corrosion of mild steel through the mechanism of adsorption. The inhibitor tends to form adsorbed layer on the surface of the metal and protect the metal against corrosion.



#### IV. CONCLUSION

The aim of the present study was to investigate the corrosion inhibition efficiency of ethanol extract of *Vernonia Amygdalina* for the corrosion of mild steel at various temperatures. In order to achieve this, weight loss (gravimetric) and scanning electron microscopy (SEM) of studying corrosion were used. From the results and findings of the study, it can be concluded that ethanol extract of *Vernonia Amygdalina* is an adsorption inhibitor for the corrosion of mild steel in acidic medium. The inhibitor modified the surface of the metal by been adsorbed on it through the mechanism of physical adsorption. The adsorption of the inhibitor on the surface of the metal forms protective layer, which inhibited corrosion reaction. In view of their good inhibition efficiency, the use of ethanol extract of *Vernonia Amygdalina* as corrosion inhibitor for mild steel is advocated in this work. However, future research could be directed towards the potential of ethanol extract of *Vernonia Amygdalina* as an inhibitor for the corrosion of mild steel and other metals in other environment or corrodent (including other acids, bases and salts systems) should be adequately studied. Such studies will provide data that can be useful in industrial application.

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