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ORIGINAL RESEARCH ARTICLE

CORROSION INHIBITION OF MILD STEEL DETERMINED USING BLENDED BITTER LEAF (*Vernonnia amygdalina*) EXTRACT AND HONEY IN DILUTE H_2SO_4 AND HCL ACID SOLUTIONS

M. A. Abba-Aji, A. B. Muhammad and D. D. Onwe
Department of Mechanical Engineering, University of Maiduguri, Maiduguri, Nigeria
**Corresponding author's email address: birma4real2004@yahoo.com*

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ABSTRACT

The inhibitive effect of bitter leaf extract (Veronia amygdalina) and honey hybrid on the corrosion of mild steel in dilute H_2SO_4 and HCl solutions was investigated using weight loss method. Various concentrations of the leaf extract and honey (5 to 25ml), and immersion times (96 to 240hrs) were recorded at room temperature (32°C), which were used to calculate the corrosion rates and inhibition effects. Inhibition was found to increase with increase in the inhibitor concentration. The inhibitive effect of honey as inhibitor on the corrosion of mild steel in dilute hydrochloric acid (HCl) solution with 54% efficiency and dilute H_2SO_4 acid solution with 48.03% inhibition efficiency were not as effective as that of hybrid with 72.48% inhibition efficiency. The results obtained indicated that both bitter leaf extract and honey could serve as an effective inhibitor for the corrosion of mild steel in dilute H_2SO_4 acid and dilute HCl solution.

1.0 Introduction

Corrosion is a menace that has plagued engineering structures and systems. Corrosion has remarkably caused severe damage to metallic components or parts such as automobile parts, pipelines, machine parts and lots of others exposed to moisture (rain water) and air. Electrochemical process is set up, causing carbon dioxide (CO_2), or sulphuric oxide (SO_2 or SO_3) to react with the water to form trioxocarbonate (IV) acid, or sulphuric acid respectively (Raja et al., 2008). Corrosion is a contributing factor to economic losses and pollution of our environments. Many manufacturing industries such as automobile industries have spent hundreds of dollars on aftermarket corrosion protection for new automobiles to delay the dreaded rust appearance (Raja et al., 2008).

Bammou et al. (2018) worked on the development of cheap, nontoxic and environmentally friendly natural products as corrosion inhibitors to replace the inorganic and synthetic inhibitors which are hazardous to the environment. The developed organic inhibitor will be a good replacement for the imported inorganic inhibitors. It could as well serve as a means of providing an alternative income to the population.

The use of inhibitors is one of the most practical methods for protection against corrosion in acidic environments (Bammou et al, 2018). Different organic and inorganic compounds have been studied as inhibitors to prevent metals from corrosive attacks. The known hazardous effects of most synthetic organic inhibitors and restrictive environmental regulations have now made researchers to focus on the need to develop cheap, nontoxic and environmentally friendly natural products as corrosion inhibitors. Usually, organic compounds that possess a significant influence on the extent of adsorption on the metal surface are used as effective corrosion inhibitors.

These natural organic compounds are either synthesized or extracted from aromatic herbs, spices and medicinal plants. Plants extracts are viewed as incredibly rich sources of naturally synthesized chemical compounds that can be extracted by simple procedures with low cost and are biodegradable in nature. The use of these natural products such as extracted compounds from leaves or seeds as corrosion inhibitors have been carried out by many researchers (Karthikaiseivi et al., 2018). Mejeha et al., (2010) studied the inhibitive effect of solanum melongena L. leaf extract on the corrosion of Aluminium in tetraoxosulphate (VI) acid. Similarly, El-Etre (2005) studied the corrosion inhibition of various metals like Copper (Cu), Steel, Nickel Zinc and lawsonia extract. Aditionally, Oguzie (2008) studied the corrosion inhibition of leaf extracts of Hibiscussabdariffa, Occimumviridis, Telferiaoccidentalis, and Azadirachtaindica and as well as extracts from the seeds of Garcinaia kola on mild steel corrosion in acidic solutions. Different classes of organic compounds are used as corrosion inhibitors for metallic alloys in various acid media (Abdel-Aal et al., 2001, Mthar et al., 2002, Selvi et al., 2003; Karthikaiseivi et al., (2018); Noor, 2005; Li et al., 2006, Oguzie; 2008, Nnanna et al, 2010, Lebrini et al; 2011, Loto et al;2012, Yusuf et al; 2013, Oloruntoba et al; 2015). Most of these organic inhibitors are nitrogen, sulphur or oxygen containing compounds.

The aim of this study is to investigate the corrosion inhibition of mild steel by Vernonia amygdalina (bitter leaf) extract and natural honey, in dilute sulphuric acid and hydrochloric acid solution at room temperature using weight loss method.

2.0 Materials and Methods

2.1 Materials and Equipment

The bitter leaf extraction was carried out in the Department of Chemical Engineering, University of Maiduguri, Nigeria. The materials used were: Fresh Bitter Leaf, 500ml Ethanol, H_2SO_4 M&B, 98%AR grade, HCl M&B, 98%AR grade, Mortar and Pestle, Digital Weight Balance, Measuring Cylinder (100mm), Beaker (250ml), Silicon Carbide Paper, Soxhlet Extractor, Honey, Distilled Water, syringe, Mild Steel (AISI 1018), Flat bottom Flask (500ml)

2.2 Methods

2.2.1 Extraction and Preparation of Specimen

2.2.2 Extraction Procedure

Fresh bitter leaf (*Vernonia amygdalina*), having a composition of soponin, tanin and flavonoid, were harvested, washed and pounded to reduce the particle size using mortar and pestle to enable the extraction process to be fast (Farombi and Owoeye, 2011). Two hundred grams (200g) of the crushed bitter leaf was extracted against 500ml of ethanol for 6 hours. The extraction method adopted was ASTM D6405-99 as applied by Farombi and Owoeye, (2011). The 200g weight of the pounded bitter leaf was poured into the Soxhlet extraction chamber containing 500ml of ethanol and heated to 60°C, which was the boiling point of ethanol. The condenser condenses the vaporized ethanol and it falls into the sox let extraction chamber containing the crushed bitter leaf, therefore extracting the extract. The extract is a mixture of ethanol and the extract of the bitter leaf. This mixture followed the reflux tube and dropped into the flat bottom flask containing the ethanol as a mixture of ethanol and bitter leaf extract, after the extraction the mixture of ethanol and the bitter leaf extract in the flat bottom flask was separated using distillation process to get the pure extract. After the distillation, the extract was poured in a beaker and allowed to cool down so as to allow any remaining ethanol in the extract to escape as the extract was exposed to the atmosphere leaving behind the pure bitter leaf extract.

2.2.3 Preparation of Specimen

The set up for this study were twenty four (24) coupons of mild steel plate of dimension 4cm x 3cm for each as shown in Figure 1, is in agreement with the work of Bammou et al., (2018)

with density of 7.85g/cm3, Young's modulus of 210GPa and the weight of each coupon is 6.10g. The chemical composition of the mild steel is shown in Table 1. The samples were polished using silicon carbide paper of grades 60 to 220 respectively. The coupons were rinsed with ethanol and dried at room temperature. Ethanol was selected based on its industrial and economic usage.

Table 1: Chemical Composition of Mild Steel Coupon

Steel	% composition					
Mild Steel	C	Mn	Si	P	S	Fe
	0.16	0.30	0.21	0.04	0.03	99.257

The sheared coupons for the experiment are shown in Figure 1

Figure 1: Sheared coupons for the experiment.

2.2.4 Experimental Procedure

The experiment was conducted following the procedures adopted by Bammou et al., (2028), with specimens sheared from mild steel of chemical composition as shown in Figure 1, having a surface area of 12cm^2. Different solution of Tetraoxosulphate (vi) acid (H_2SO_4) and hydrochloric acid (HCL), seventy five millilitre (75ml) of the HCl was used to prepare an acid solution of 150ml of 1 molarity in 900ml of solution of 150ml from 900ml of distilled water. The mild still plates were all weighed; the beakers to which were inserted, were all labelled to differentiate them (ASTM). 150ml of the diluted H_2SO_4 and dilute HCl were poured separately into six 250ml beakers; this volume was kept constant for all beakers. The mild steel plates were each tied with a string to a small stick that will enable them suspend when placed across the mouth of the beaker. Different concentration of 5ml, 10ml, 15ml, 20ml, and 25ml of the inhibitor (honey and bitter leaf extract in the ratio of 50:50) were poured into the diluted acid in the beakers, honey was also used separately in the concentration of 5ml, 10ml 15ml 20ml and 25ml as inhibitor in both acid(dilute H_2SO_4 and dilute HCl).

Previously weighed coupons were each suspended in each of the beakers of capacity 250ml via a string at room temperature. The first set of coupons were retrieved from their corrosive environments after 48hours (two) days. This was followed by the second, third, fourth and fifth sets being retrieved at intervals of 96, 144, 192 and 240 hours immersion time respectively. At each interval, the coupons were mechanically brushed and rinsed in 34.4% ethanol to remove the corrosion product on the surface of the coupon, the coupons were allowed to dry and their weights were taken and recorded. The weight loss (w_0-w_1) for each of the specimen was obtained by finding the difference between the final (w_1) and initial weight (w_0) and recorded as weight loss. Figure 2 presents the experimental setup for honey inhibitor.

Corresponding author's e-mail address: birma4real2004@yahoo.com

Figure 2: Experimental Set- up for honey (inhibitor)

Figure 3: Experimental set up for honey and bitter leaf (inhibitor)

2.3 Experimental Analysis

The following test and analysis were carried out using bitter leaf extract and honey as an organic inhibitor on mild steel.

1. Weight loss, in gram
2. Corrosion rate, in mm/day
3. Inhibition efficiency (IE), in % (Fontana, 1994)

2.3.1 Weight loss and corrosion rate

Weight loss method was used to determine the corrosion rate of the coupons using two different inhibitors separately. The mixture of bitter leaf extract with honey as inhibitor and that of honey alone was considered. The corrosion rate was calculated from weight loss of the coupons at room temperature (32°C) at various concentrations and immersion time, using the relation due to Fontana (1994) as presented in Equation 1

$$mm/day = \frac{87.6W}{\rho AT} \quad (1)$$

The weight loss can be determined using Equation 2 as:

$$WI = {}^{w_b - w_a}/_{S} \quad (2)$$

Where: w_b and w_a are weights before and after immersion, S = total surface area of the specimen (mild steel)

Corresponding author's e-mail address: birma4real2004@yahoo.com

2.3.2 *Inhibition Efficiency (IE)*

The inhibition efficiency of the organic inhibitor (bitter leaf extract with honey and honey alone) was calculated from weight loss measured at different inhibitor concentration at 32°C (room temperature). The percentage of inhibitor efficiency (IE) was calculated using Equation (2).

$$IE = \frac{CR_{UN} - CR_{IN}}{CR_{UN}} \times 100 \quad (3)$$

3.0 Results and Discussion

3.1 Corrosion Rate

The corrosion rates of the uninhibited and inhibited as a function of inhibition concentration were observed. The inhibition efficiency increases with increase in inhibitor concentration from 5ml to 25ml. Corrosion rate for uninhibited mild steel in H2SO4 experienced a decrease from the beginning of the experiment to the 25ml inhibitor concentration. It was observed that weight loss also decreases as the immersion time increases in the uninhibited acid and the inhibited acid solution. This could be attributed to the presence of dissolved air as represented by the reaction:

$$Fe \rightarrow Fe^{2+} + 2e- \quad (4)$$

$$2H_2O + O_2 + 4e- \leftrightarrow 4OH^- \quad (5)$$

This type of corrosion can, therefore, be controlled by:
1. Eliminating oxygen from the corroding medium, or
2. Retarding the diffusion to the cathode areas (Palanna, 2009).

The corrosion rate of the inhibited coupons showed a sharp decrease in corrosion rate and becomes constant as immersion time increases. As shown in Figure 4, corrosion rate of the uninhibited coupon decreases with time. The sharp decrease in the corrosion rate of the inhibited acid solution was as a result of the alteration (passivation) in the mechanism of corrosion due to the presence of inhibitor as confirmed by Rosliza (2010) and also because the exposure time under investigation falls within the active region of corrosion for these materials as reported by Orthorho et al (2017).

3.2 Inhibition Efficiency

Hybrid Inhibition Efficiency in H_2SO_4 Solution

Inhibition efficiency in control solutions are always zero. The number for beakers for each inhibitor is six including that of the control solution.

$$\text{Inhibition efficiency at 48 hours} = \frac{0 + 43 + 44 + 45 + 48 + 51.6}{6} = 31.5\%$$

$$\text{Inhibition efficiency at 96 hours} = \frac{0 + 40 + 40 + 40 + 42 + 48.3}{6} = 35.05\%$$

$$\text{Inhibition efficiency at 144hours} = \frac{0 + 59 + 60 + 62 + 66 + 69}{6} = 52.67\%$$

$$\text{Inhibition efficiency at 192hours} = \frac{0 + 76 + 79 + 80 + 84.2 + 93.2}{6} = 68.7\%$$

$$\text{Inhibition efficiency at 240hours} = \frac{0 + 83 + 84 + 85 + 87.2 + 95.7}{6} = 72.48\%$$

Corresponding author's e-mail address: birma4real2004@yahoo.com

Figure 1 shows a plot of the inhibitor efficiency against concentration (i.e. using the hybrid inhibitor). From the figure, it was observed that the inhibitor efficiency increased with increase in inhibition concentration. Figures 1, 2 and 3, show the representations of the uninhibited and inhibited solutions of the mixture of honey and bitter leaf extract (hybrid) as the inhibitor at various concentration of 5ml, 10ml, 15ml, 20ml and 25ml in dilute H_2SO_4 solution. Figures 4, 5 show the behaviour of the mild steel plate in dilute hydrochloric (HCl) acid solution in the presence of the inhibitor (hybrid).

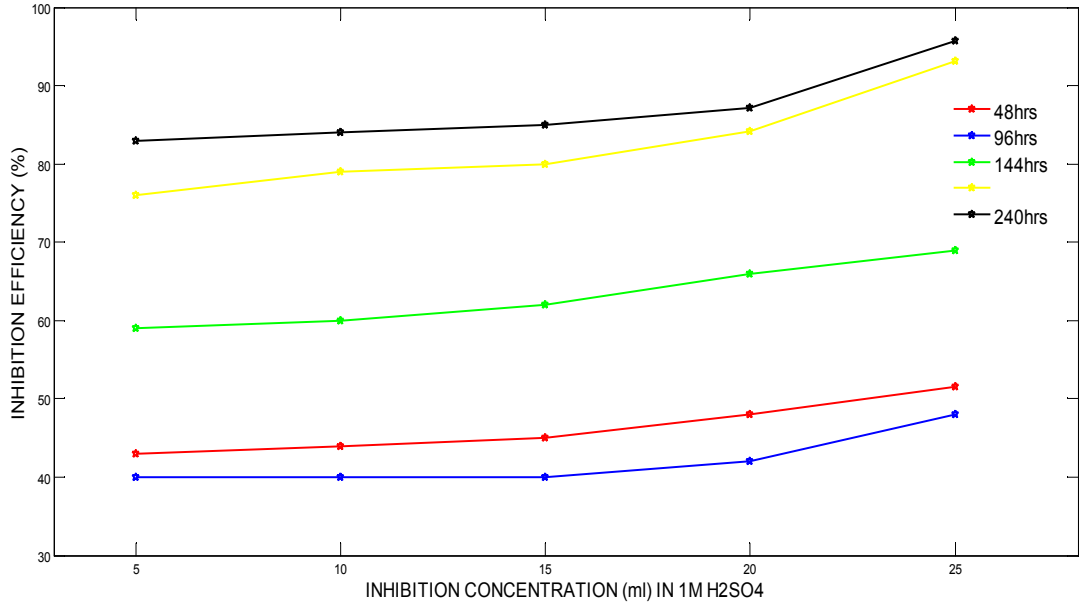

Figure 1: Inhibition efficiency against inhibitor concentration of hybrid

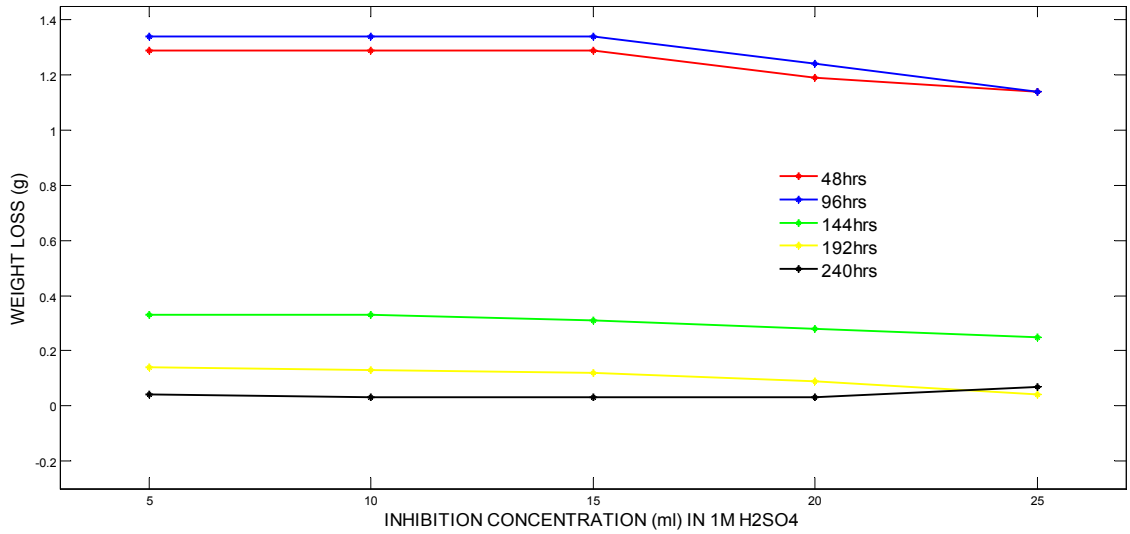

Figure 2: Weight loss against inhibitor concentration of hybrid

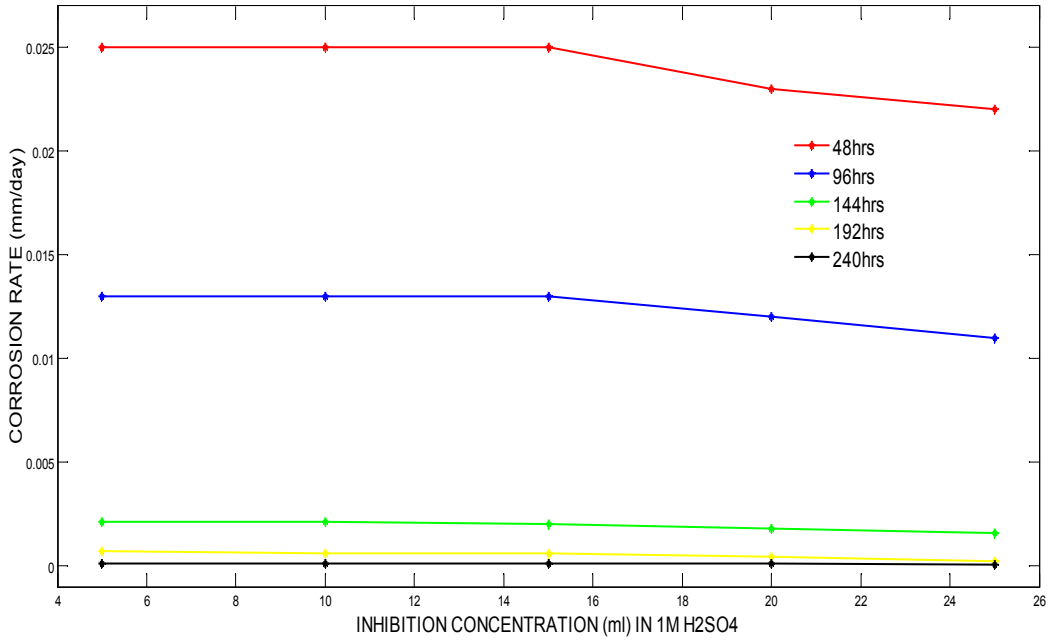

Figure 3 Corrosion rate against inhibitor concentration of hybrid

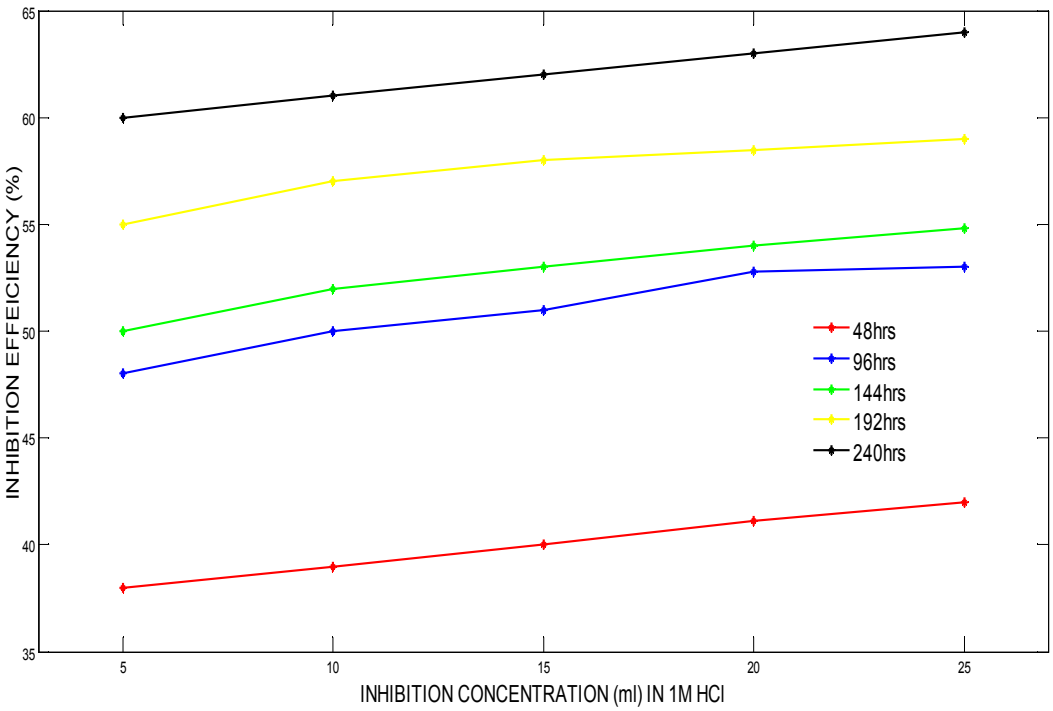

Figure 4: Inhibition efficiency against inhibition concentration of hybrid

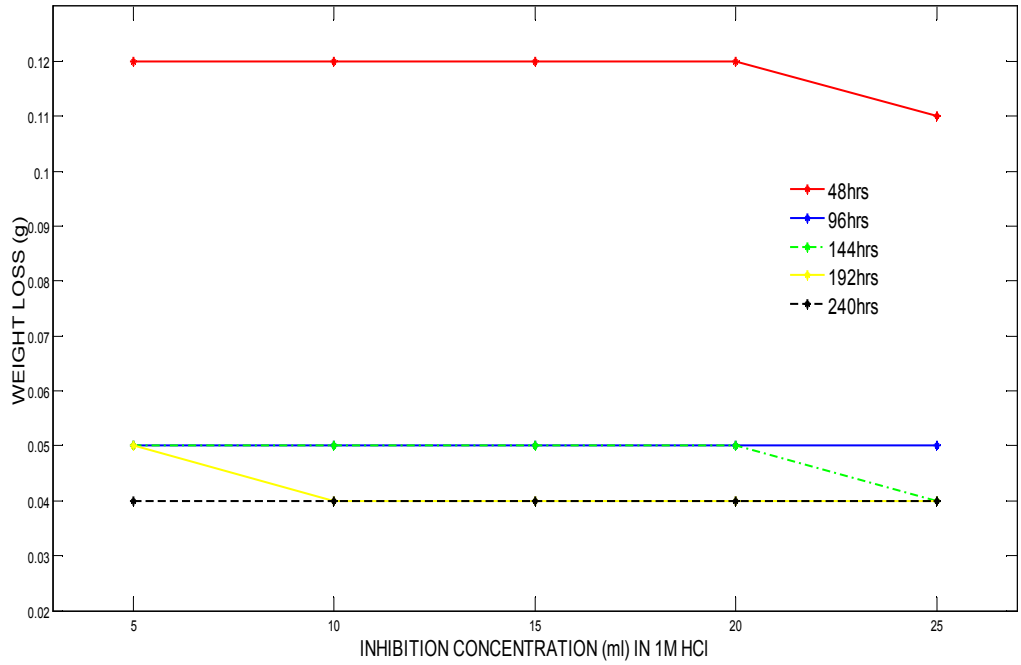

Figure 5: Weight loss against inhibition concentration of hybrid

The result of the corrosion rates and weight loss of mild steel in bitter leaf extract and honey for different mixture of coupon, 5, 10, 15, 20, and 25ml inhibitor concentration for dilute H_2SO_4 and dilute HCl solution were observed.

Figure 3 shows plot of weight loss as a function of inhibition concentration of H_2SO_4. Figure 4 shows the corrosion rate as a function of concentration of H_2SO_4. Consequently, the plot of corrosion rates as a function of time, weight loss as a function of immersion time and also the plot of corrosion rates as a function of time for HCl concentration are shown in Figure 5 and Figure 6. The inhibition efficiency is optimum at 240 hours, and is 72%, similarly inhibition efficiencies for hybrid and honey in HCl and H_2SO_4 were presented in Table 1

Table 1: Inhibition efficiencies of honey and hybrid (bitter leaf/ honey) in H_2SO_4 and HCl

Time (hours)	Hybrid in H_2SO_4	Hybrid inhibition in HCl	Honey inhibition efficiency in H_2SO_4	Honey inhibition efficiency in HCl
48	31.50	33.35	27.16	32.33
96	35.05	42.47	30.33	36.67
144	52.67	43.67	32.23	38.38
192	68.70	47.97	43.33	48.47
244	72.48	51.67	48.03	54.13

As the concentration of inhibitor produced increases, the corrosion rate decreases and the inhibitor has the highest efficiency of 72% while in dilute H2SO4 solution, which is in agreement with Mejeha et al, (2010), it has an inhibition efficiency of 52% in dilute HCl.. Honey has an inhibition efficiency of 48% in dilute H_2SO_4 solution and 54% n dilute HCl solution, which proved that the hybrid inhibitor in tetraoxosulphate (VI) acid solution is effective and its usage in automobile industries will reduced drastically the corrosion rate.

4.0 Conclusion

This study has shown that hybrid of honey and bitter leaf extract can be used as corrosion inhibitor. In this work, honey as an inhibitor alone is more effective in hydrochloric acid solution than in tetraoxosulphate (VI) acid solution. Similarly, bitter leaf extract can be used to upgrade honey in the prevention of corrosion in tetraoxosulphate (VI) acid environment. The

Corresponding author's e-mail address: birma4real2004@yahoo.com

inhibitor has a characteristic which indicated an increase in concentration of inhibitor resulted in a significant decreased corrosion rate with inhibitor efficiency of 72% while in dilute HCl solution, it has an inhibition efficiency of 52%. Honey has an inhibition efficiency of 48% in dilute H_2SO_4 solution and 54% in dilute HCl solution, which proved that the hybrid inhibitor in tetraoxosulphate (VI) acid solution is effective and it's usage in automobile industries will remarkably reduce corrosion rate.

Recommendation

Bitter leaf extract and honey are eco-friendly, acceptable and poses no threat to the environment as the chemical and synthetic inhibitors, as such should be used for corrosion inhibition.

If bitter leaf extract and honey are adopted as corrosion inhibitor, it will increase the gross domestic product (GDP) of our country hence the standard of living will be improved and it will help our environment to be less prone to pollution.

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Corresponding author's e-mail address: birma4real2004@yahoo.com