

INVESTIGATIVE USE OF FISH SCALE AS CORROSION INHIBITOR (Mild Steel and Brass as Test Metals)

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Abstract-Investigation of the use of Tilapia fish scale as a corrosion inhibitor has been done in this work. The efficiency of the produced inhibitor was tested on two metal coupons of mild steel and brass, in three concentrations (0.01M, 0.05M, 0.10M) of acid and basic environment for four weeks. Results obtained are as follows: For **Mild steel**: for 0.01M (efficiency of inhibitor ranges from 60%-83% in acidic medium and 71%-86 % in base medium from week 1 to week 4); for 0.05M (efficiency of inhibitor ranges from 67%-86% in acidic medium and 71%-86% in base medium from week 1 to week 4); for 0.10M (efficiency of inhibitor ranges from 67%-86% in acidic medium and 80%-86% in base medium from week 1 to week 4). **Brass**: for 0.01M (efficiency of inhibitor ranges from 40%-88% in acidic medium and 57%-85% in base medium from week 1 to week 4); for 0.05M (efficiency of inhibitor ranges from 66%-89% in acidic medium and 66%-88% in base medium from week 1 to week 4); for 0.10M (efficiency of inhibitor ranges from 71%-89% in acidic medium and 71%-88% in base medium from week 1 to week 4). The fish scale in H_2SO_4 and $BaCl$ medium efficiently inhibits the corrosion and proved to be a zero cost inhibitor, eco-friendly, non-toxic and highly economical.

Key words: Fish Scale, Acid, Base, Corrosion, Inhibitor, Brass, Mild Steel

1.0 INTRODUCTION

The adverse effect of the corrosion process has constituted a major problem of worldwide significance, corrosion which is a form of degradation leads to the loss or contamination of products, plant shutdowns, reduction in efficiency, waste in valuable resources, costly maintenance and can also inhibit technological process. This phenomenon called corrosion can be defined as the process whereby a metallic material depreciates in surface value or size as a result of chemical reactions commonly the oxidation reaction where an exposed metallic surface is attacked by a gas or a liquid corrosive agent. It is also defined as an irreversible interfacial reaction of a material with its environment which results in the dissolution into the material by a component of the environment. It is instigated by certain temperature conditions and the presence of acids and salts which could lead to the loss of production, increased cost of equipment maintenance and reduce material effectiveness. The process of corrosion and the challenges arising from the action of corrosive agents on equipment has propelled the research into a more reliable way of protecting corrosion laden materials from the corrosion process.

Because of the toxic nature and high cost of some chemicals currently in use, it is necessary to develop environmentally acceptable and less expensive inhibitors. Natural products can be considered as a good source for this purpose. The possible replacement of some expensive chemicals as corrosion inhibitors for metal in acid cleaning process by naturally occurring substances of plant origin has been studied by [1]. Natural products of plant origin contain different organic compounds e.g. alkaloids, lignin, tannins, amino acids, pigments and most are known to have inhibitive action. [2], and [3] used the aqueous extracts of some plant parts (fruits, fruit shell, leaves, seeds) as corrosion inhibitors for some metals. The natural inhibitors studied have been found to be highly eco-friendly and possess no threat to the environment. The collagen extract from fish scale has been used as a green organic inhibitor, as an additive to silica sol-gel coatings in order to form a protective film on alloys that are subject to corrosive agents or environment.

1.1 Corrosion

Corrosion can be defined as the process whereby a material breaks into its constituent atoms because of the chemical reactions with its surrounding environment. It is in common terms known as rust, where the metal involved undergoes electrochemical oxidation with oxygen as an oxidant forming a metal oxide also known as rust [4]. Other forms of materials are

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also prone to corrosion attacks such as polymers and ceramics but it is more strongly experienced in iron and steel because the oxide formed loses its adhesive properties to the surface of the metal and begins to break off easily. The phenomenon of corrosion destroys the beauty of metallic materials and lessens its lifespan which also affects technical progress of the company. According to [2] corrosion is the loss of useful properties such as malleability, ductility, electrical conductivity and optical reflectivity of metals due to attack on it by the environment. The process of corrosion involves a weight loss of the material or substance. This loss per unit time is used to express the rate of corrosion. The rate of corrosion depends on the material being corroded.

1.2 Role of Corrosion Inhibitors

Organic and inorganic compounds constitute a large class of corrosion inhibitors which generally affects the entire surface of a corroding metal when present in sufficient concentration. The principle mechanism of inhibition is adsorption where the inhibitor is adsorbed on the entire surface of the corroding metal preventing attack from the corrosive agent. Organic inhibitors are adsorbed according to the ionic charge of the inhibitor on the metal's surface, cationic or anionic inhibitors will be adsorbed preferentially depending on whether the metal is charged positively or negatively. [5,6] The role of inhibitors can be classified into two depending on the nature of protection it provides such as:

- i. Those who form a protective barrier film on the cathodes and anodes by reaction between the metal and the environment, this type of inhibitors function in neutral or in some cases alkaline solution in which the main cathodic reaction is an oxygen reduction reaction in which the corroding metal surface is covered by a film or stabilize an already existing ore.
- ii. The second types are those which are initially adsorbed directly onto the metal surface by interaction between surface charges and ionic and/or molecular dipole charges. This division of inhibitor types results principally from the pH of the solution where they operate.

It has been observed that at certain concentrations, inhibitors lose their efficiency and become corrosion promoters, the efficiency of organic inhibitors can be improved in the

presence of certain halogen ions. Halogen ions are also known to inhibit corrosion to some extent in acid solutions. The synergism of halogen ions can be attributed to the fact that the metal adsorbs halogen ions whose charge shifts the surface in a negative direction thereby causing adsorption of the cationic organic inhibitor. Discovery of corrosion inhibitive compounds requires a lot of hard work, innovation and laboratory analysis/synthesis. [7,8,9]

1.3 Classification of Corrosion Inhibitors

1.3.1 Based on Electrode Process

i. Anodic inhibitors

An anodic inhibitor increases the anodic polarization and hence moves the corrosion potential to the cathodic direction and hence also called as passivating inhibitors. A number of inorganic inhibitors such as orthophosphates, silicates etc. fall under anodic type. Even though anodic inhibitors are widely used, a few of them have some undesirable property. If such inhibitors are used in very low concentrations, they cause stimulation of corrosion such as pitting and for this reason anodic inhibitors are denoted as dangerous.

ii. Cathodic inhibitors

Cathodic inhibitors shift the corrosion potential to the anodic direction. Here the cations migrate towards the cathode surface where they are precipitated chemically or electrochemically and thus block these surfaces. The inhibiting action of the cathodic inhibitors takes place by three mechanisms,

- a. **Cathodic poisons:** The cathodic reduction process is suppressed by impeding the hydrogen recombination and discharge but increase the tendency of the metal to be susceptible to hydrogen induced cracking.
- b. **Cathodic precipitates:** Compounds such as calcium, magnesium will precipitate as oxides to form a protective layer which acts as a barrier on the metal surface.
- c. **Oxygen scavengers:** These compounds react with oxygen present in the system to form a product and reduce corrosion. Eg: As^{3+} and Sb^{3+} on dissolution of Fe in acids.

iii. Mixed inhibitor

These inhibitors retard both the anodic and cathodic processes. The shift in the potential is

smaller and the direction is determined by the relative size of the anodic and cathodic sites. Such inhibitors will have the advantage over other inhibitors in that they control both the cathodic and anodic corrosion reactions and hence are very safe to apply.

1.4 Corrosion Testing Techniques

There are various techniques used in corrosion testing and measurement. [6]

1.4.1 Weight Loss Method

The weight loss coupon method of monitoring is the oldest method for assessing the corrosive potential of an environment on a specific material and involves exposing a specimen (coupon) of the material to the environment for a given duration of time and measuring the resultant weight loss. The coupons can be in the form of discs, rods, plates or of any convenient shape.

Coupons are not an instrumental method and require extensive manual involvement in order to provide information. Also, coupons only provide integrated corrosion loss data. However, the advantages of coupons include:

- i. Visual interpretation of results.
- ii. Deposits can be observed and analyzed.
- iii. Weight can be readily determined
- iv. The degree of localization of corrosion can be observed and measured.

1.4.2 CEION

The CEION is a very ratiometric metal loss measurement device with resolution that is at least 100 times better than existing ER based devices. It is ideal for monitoring oil/gas production and processing systems. A technology that is just as capable of driving the real-time control loop of an inhibitor as it is of operating without maintenance between planned shutdowns. CEION is also ideally suited to measuring sand erosion in producing systems and a specific set of sensor designs has been developed for this application

1.4.3 Electrical Resistance (ER) Monitoring

The ER method of corrosion monitoring is one of the most widely used techniques and consists of determining the change in resistance of a metal element as it corrodes in a process environment. The action of corrosion on the element serves to decrease the cross sectional area thereby

increasing the electrical resistance. The element is usually in the form of a wire, strip or tube and if the corrosion is roughly uniform, a change in resistance is proportional to an increment of corrosion. Estimates of the total corrosion over a period may be obtained from successive readings. A simple formula converts to an average corrosion rate.

Electrical resistance probes are rugged and well adapted to any corrosive environment. The ER technique is well proven in practice and is simple to use and interpret. ER monitoring permits periodic or continuous monitoring to be established for one or multiple number of probes. Corrosion can thus be related to process variables and the method is one of the primary on-line monitoring tools. The major advantage is its ability to measure corrosion in any environment: liquid, gas or particle streams.

1.4.4 Electrochemical Methods

Since corrosion is an electrochemical process, it is not surprising that there are a number of electrochemical methods for corrosion monitoring. The two electrochemical techniques which are most widely used are linear polarization resistance monitoring and galvanic monitoring also known as zero resistance ammetry:

- i. The linear polarization technique attempts to respond on a microscopic scale, the microscopic corrosion cells existing within the plant. It measures the corrosion current flowing between the anodic and cathodic half cells. It provides an instantaneous measure of corrosion rate and may be used as a method for optimizing corrosion inhibitor injection.
- ii. The principle of the galvanic technique relies on the fact that when two different metals are immersed in an aqueous liquid they assume different electrode potentials. If these metals were connected externally, a current will flow between them. This current is the result of the half cell reactions taking place simultaneously causing the corrosion of the more negative metal in the solution and the reduction of any available species such as oxygen at the surface of the more positive electrode. They are particularly used for the detection of the ingress of oxygen into systems protected by de-aeration also they provide a rapid, continuous method for assessing corrosive conditions in aqueous flows.

1.4.5 Hydrogen Monitoring

Hydrogen monitoring is an important facet of corrosion monitoring, since the detection of hydrogen provides an indication that corrosion is taking place.

In particular situation where plant is exposed to wet sour gas or acidic conditions the generation of hydrogen is of primary concern. Hydrogen monitoring probes exist which can be either inserted into the plant in order to measure the presence of hydrogen or conversely attached to the exterior of the plant in a saddle mode capable of detecting the diffusion of hydrogen through the plant.

1.5 FISH SCALES

Fish scales are bio-composites of highly ordered type I collagen fibers and hydroxyapatite $\text{Ca}_{10}(\text{OH})_2(\text{PO}_4)_6$. Collagens extracted from fish scales of black drum, sheep's head sea bream, Red seabream, Red Tilapia, sardine, Japanese sea-bass, skipjack tuna, ayu, yellow sea bream and horse mackerel have been reported. These collagens are mainly type I collagen with a lower denaturation temperatures than the collagen of porcine dermis. These fish are mostly seawater fish and there have been fewer reports about the freshwater fish scales. Carp fish (*Cyprinus carpio*) is one of the primary fish species in freshwater breeding industry in China. During the processing of fish, a great amount of fish scales are dumped, which is a great waste because the scales of carp fish contain a large amount of collagen. [10]

2.0 MATERIALS AND METHOD

This research was undertaken in the Chemical/Petrochemical Engineering Department Laboratory of Rivers State University, Nkpolu-Oroworukwo, Port Harcourt.

Table 1: Materials

S/N	Material	Concentration	Source
1	Ethanol	98%	CHEM. LAB
2	Acetone	98%	CHEM. LAB
3	Tetra-oxosulphate (VI) acid	0.01M, 0.05M, 0.10M	CHEM. LAB

4	Distilled water	100%	
5	Tilapia Fish Scales		Fish Stall
6	Barium Chloride	0.01M, 0.05M, 0.10m	CHEM. LAB
7	Coupons of brass and mild steel	30mm x 50mm x 2mm	MECH. SHOP

2.1 Material Preparation

The following materials were used during the experimental process at required specifications for the production of corrosion inhibitor from fish scale before it was separately mixed with Sulphuric acid and Barium chloride solutions.

- The sample (Tilapia Fish scale) was collected at a fish stall and dried at research quantity and conveyed to the laboratory for the production process.
- The laboratory oven was used to dry the sample before crushing.
- The electric grinder was used for crushing of the dry sample to its finest powder form
- A sieve was used for separating the crushed sample into particles of uniform size, the sieve used is the muslin sieve.
- A foil was used for wrapping both the dried and the crushed sample.
- The grinded sample was weighed in the weighing balance.
- Desiccators were used for the preservation of the sample during the period of the experiment.
- Laboratory beakers were used for the measurement of the volume of the sample and solutions.
- An evaporator was used to evaporate the solvent from the sample after the preparation process.
- A conical flask was used to hold the sample during heating.
- Cotton was used to act as a protective cover for the conical flask.
- Paper cello-tape was also used to mark out the different samples having test coupons.
- Laboratory sterile bottles were used for packaging the samples.

2.2 Experimental Procedure

The following procedures were adopted for the preparation and extraction of the corrosion inhibitor from the fish scales:

1. The sample was collected and washed thoroughly with distilled water for three times to effectively remove impurities.
2. After washing the sample was allowed in the sun for two hours to drain the washing liquid before further heating.
3. The sample was further dehydrated in the laboratory oven at a temperature of about 80°C for 12 hours to completely remove all the moisture in the sample.
4. The dried sample was crushed using the electric grinder to the finest powder form.
5. The grinded sample was then sieved to obtain a uniform sample size.
6. The sample was weighed in a balance to measure 100g worth of sample.
7. 500ml of 98% pure ethanol was measured out using the conical flask to obtain an approximate 1:5 ratio of sample to ethanol respectively
8. The measured out sample was now poured into the ethanol solvent and the mixture was air-tight to avoid sudden evaporation of the ethanol by covering the conical flask with cotton wool, foil and paper cello-tape by wrapping.
9. The mixture was then allowed to stay for four days (96 hours) to allow for proper mixing.
10. The mixture was macerated periodically to enhance effective extraction of the inhibitor from the sample and this extraction method is otherwise known as maceration.
11. The sample after four days was then opened removing all the materials which were used to make it air tight.
12. The mixture was then filtered and squeezed firmly using the muslin sieve and filtrate was collected in a beaker and the residue left in the muslin.
13. The filtrate was then introduced into the evaporated for the ethanol to be completely removed from the inhibitor obtained.
14. The evaporator operating at 80°C was used to remove the ethanol which has a boiling point of 78°C gradually from the inhibitor until it appears completely free of the ethanol mixture.

15. After effective evaporation the sample was put into sterile bottle to preserve and prevent it from contamination by micro-organisms.

2.2.1 Testing of the produced corrosion inhibitor

16. 1700 ml of three concentrations (0.01M, 0.05M and 0.1M) each of Sulphuric acid and Barium Chloride solutions were prepared.
17. 2g of the produced fish scale inhibitor was separately mixed with 200 ml of the different concentrations of Sulphuric acid and Barium Chloride respectively and tagged A1, A2, A3 and B1, B2 B3 respectively. Where A means mixture of inhibitor and acid and B means mixture of inhibitor and Barium Chloride. 1,2 and 3 indicates the different concentrations of the acid and base.
18. Weighed coupons of Brass were then completely immersed in the solutions of (17) above. The same process was also adopted for mild steel coupons.
19. Coupons of brass and mild steel were also immersed separately in the different concentrations of Sulphuric acid and Barium Chloride respectively but without the inhibitor. This is the control experiment.
20. The entire experimental set up of steps 18 and 19 were monitored for a 28-day period with the weights of the coupons being determined and recorded after every 7 days.

2.3 EFFICIENCY OF THE CORROSION INHIBITOR

The efficiency of the inhibitor was determined after weighing using the formula prescribed by [10].

$$IE = \frac{W1 - W2}{W2} \times 100\% \quad (1)$$

Where **IE** is the inhibitor efficiency
W1 is the initial weight of the coupons without inhibitor

W2 is the final weight of the coupons with inhibitor respectively.

3.0 RESULTS AND DISCUSSION

3.1 Mild Steel

From the results obtained as shown in Tables 2 and 3, and Figures 1a and 1b there is greater degree in weight loss of the mild steel coupon in the concentration of sulphuric acid and barium chloride without inhibitor, but slow rate of weight loss of mild steel coupon in concentration of sulphuric acid and barium chloride with fish scale inhibitor. This result implies that inhibition is taking place. It can also be seen from the aforementioned tables and figure that the mild steel coupon in the BaCl environment corroded faster without inhibitor while the metal with inhibitor in the H_2SO_4 environment had a slower rate of corrosion.

Table 2: Weight of Mild Steel Coupons Different Concentrations of Acid And Inhibitor

	H₂SO₄ only (control) + Mild Steel		
	0.01M	0.05M	0.10M
	A ₁	A ₂	A ₃
Initial Weight	9.20g	9.20g	9.20g
WEEK 1	9.15g	9.17g	9.17g
WEEK 2	9.11g	9.12g	9.12g
WEEK 3	9.06g	9.07g	9.06g
WEEK 4	9.00g	9.00g	8.99g

	H₂SO₄ + Inhibitor + Mild Steel		
	0.01M	0.05M	0.10M
	B ₁	B ₂	B ₃
Initial Weight	9.20g	9.20g	9.20g
WEEK 1	9.18g	9.19g	9.19g
WEEK 2	9.17g	9.18g	9.18g
WEEK 3	9.16g	9.17g	9.17g
WEEK 4	9.15g	9.16g	9.16g

Table 3: Weight of Mild Steel Coupons in Different Concentrations of BaCl and Inhibitor

	BaCl only (control) + Mild Steel		
	0.01M	0.05M	0.10M
	C ₁	C ₂	C ₃
Initial Weight	9.20g	9.20g	9.20g
WEEK 1	9.13g	9.13g	9.15g
WEEK 2	9.09g	9.08g	9.07g
WEEK 3	9.02g	9.01g	9.00g
WEEK 4	9.00g	9.00g	8.93g

	BaCl + inhibitor + Mild Steel		
	0.01M	0.05M	0.10M
	D ₁	D ₂	D ₃
Initial Weight	9.20g	9.20g	9.20g
WEEK 1	9.18g	9.19g	9.19g
WEEK 2	9.17g	9.17g	9.18g
WEEK 3	9.16g	9.16g	9.17g
WEEK 4	9.15g	9.15g	9.16g

Tables 4 and 5 and Figures 1a and 1b shows the variation of the inhibitor efficiency of the fish scale with time in acid and the base environment. From the results, it is seen that the inhibitor efficiency increases slightly at different concentrations of H_2SO_4 and BaCl. Also, it can be seen that the inhibitor efficiency increases with time.

Table 4: Determination of Inhibitor Efficiency (%) for Mild Steel at Different Concentration of H₂SO₄

	0.01M	0.05M	0.10M
WEEK 1	60%	67%	67%
WEEK 2	75%	80%	80%
WEEK 3	80%	80%	83%
WEEK 4	83%	80%	86%

Table 5: Determination of Inhibitor Efficiency (%) for Mild Steel at Different Concentration of BaCl

	0.01M	0.05M	0.10M
WEEK 1	71%	71%	80%
WEEK 2	75%	80%	85%
WEEK 3	85%	85%	85%
WEEK 4	86%	86%	86%

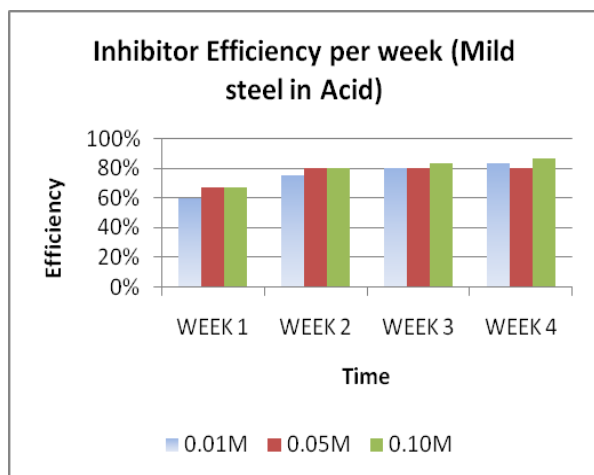


Fig. 1a: Bar chart showing the inhibition efficiency difference for mild steel in acid environment

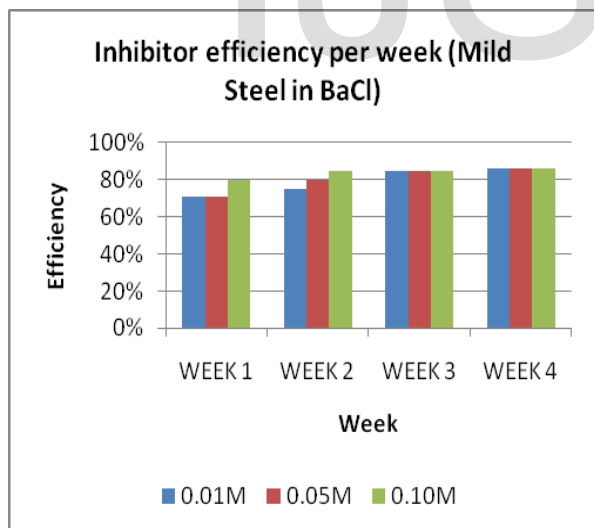


Fig. 1b: Bar chart showing the inhibition efficiency difference for mild steel in basic environment

3.2 Brass

From the results obtained in Tables 6 and 7, and Figures 3a and 3b it is seen that there is greater degree in weight loss of the brass coupon in the

concentration of sulphuric acid and barium chloride without inhibitor, but slow rate of weight loss of brass coupon in concentration of sulphuric acid and barium chloride with fish scale inhibitor. This implies that inhibition is taking place. It can also be seen from the aforementioned tables and figure that the brass coupon in the H_2SO_4 environment corroded faster without inhibitor while the metal with inhibitor in the $BaCl$ environment had a slower rate of corrosion.

Table 6: Weight Loss of Brass Coupon

Weight loss of brass coupons in different concentration of inhibitor and H_2SO_4

	H_2SO_4 only (control) + Brass		
	0.01M	0.05M	0.10M
	A ₁	A ₂	A ₃
Initial Weight	20.31g	20.31g	20.31g
WEEK 1	20.26g	20.25g	20.24
WEEK 2	20.20g	20.19g	20.18g
WEEK 3	20.13g	20.12g	20.11g
WEEK 4	20.05g	20.03g	20.02g

	H_2SO_4 + Inhibitor + Brass		
	0.01M	0.05M	0.10M
	B ₁	B ₂	B ₃
Initial Weight	20.31g	20.31g	20.31g
WEEK 1	20.28g	20.29g	20.29g
WEEK 2	20.27g	20.28g	20.28g
WEEK 3	20.26g	20.27g	20.27g
WEEK 4	20.25g	20.26g	20.26g

Table 7: Weight of Brass Coupons in Different Concentration of Inhibitors and BaCl

	BaCl only (control) + Brass		
	0.01M	0.05M	0.10M

	C ₁	C ₂	C ₃
Initial Weight	20.31g	20.31g	20.31g
WEEK 1	20.24g	20.25g	20.24g
WEEK 2	20.20g	20.21g	20.19g
WEEK 3	20.15g	20.15g	20.13g
WEEK 4	20.08g	20.04g	20.03g

	BaCl + Inhibitor + Brass		
	0.01M	0.05M	0.10M
	D ₁	D ₂	D ₃
Initial Weight	20.31g	20.31g	20.31g
WEEK 1	20.28g	20.29g	20.29g
WEEK 2	20.27g	20.28g	20.28g
WEEK 3	20.26g	20.27g	20.27g
WEEK 4	20.25g	20.26g	20.26g

Tables 7 and 8 and Figures 3a and 3b show the variation of the inhibitor efficiency of the fish scale with time in acid and the base environment. From the results, it is seen that the inhibitor efficiency increases slightly at different concentrations of H_2SO_4 and $BaCl$. Also, it can be seen that the inhibitor efficiency increases with time.

Table 7: Determination of Inhibitor Efficiency (%) for Brass at Different Concentration of H_2SO_4

	0.01M	0.05M	0.10M
WEEK 1	40%	66%	71%
WEEK 2	83%	83%	86%
EEK 3	86%	86%	86%
WEEK 4	88%	89%	89%

Table 8: Determination of Inhibitor Efficiency (%) for Brass at Different Concentration of $BaCl$

	0.01M	0.05M	0.10M
WEEK 1	57%	66%	71%
WEEK 2	75%	75%	80%
WEEK 3	80%	83%	83%
WEEK 4	85%	88%	88%

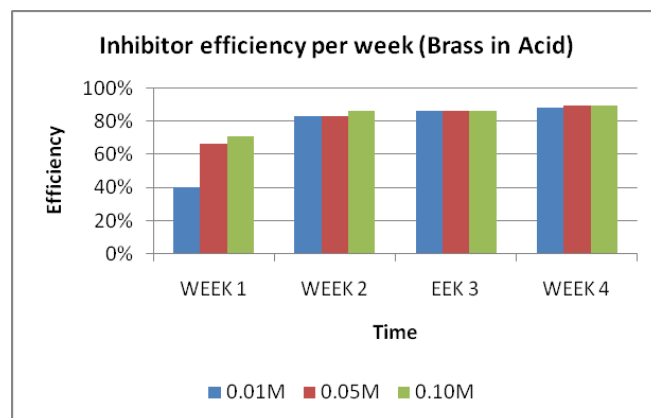


Fig. 3a: Bar chart showing the inhibition efficiency difference for brass in acid environment.

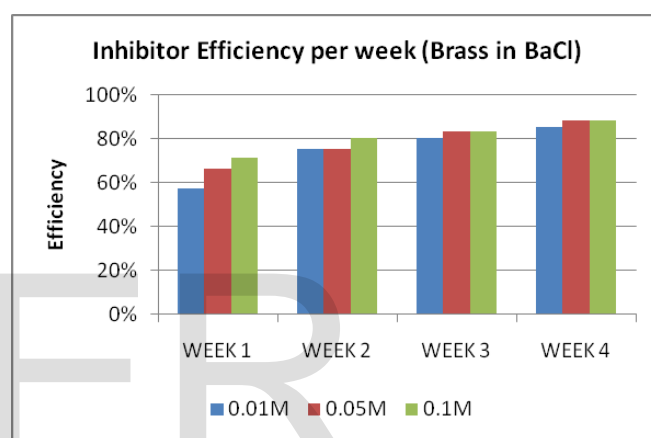


Fig. 3b: Bar chart showing the inhibition efficiency difference for brass in base environment

4.0 CONCLUSION

A study to determine the corrosion inhibitive effectiveness of fish scale was carried out in this work. The analysis of the weight recorded for metal of mild steel, brass, galvanized metal and aluminum coupons, the efficiency of inhibitor for four weeks was determined in both H_2SO_4 and $BaCl$ environment. It was seen that the metallic coupons that was immersed in a H_2SO_4 and $BaCl$ concentration without inhibitor tends to lose more weight which was a clear indication of a high rate of corrosion. But for the coupons immersed in a concentration of H_2SO_4 and $BaCl$ with inhibitor, less weight loss was recorded. This explain that the fish scale tends to slow down the rate of corrosion, therefore inhibiting corrosion for both mild steel, brass, galvanize and aluminum. The inhibitive efficiency of the metals was determined and the result showed that there is increase in efficiency of the inhibitor as the experiment continued.

For mild steel with inhibitor, the inhibitive efficiency was between 60% - 86% in different concentrations of acidic and basic environment.

For brass coupon with inhibitor, the inhibitive efficiency was between 40% - 89% in different concentrations acidic and basic environment. From the above results, it can be seen that fish scales can be effectively used for corrosion

inhibition at all levels. The application of fish scales for corrosion inhibition will also help to reduce or completely eliminate the waste generated by fish scales and to a large extent prevent oil spill and leakages caused by corrosion when applied industrially.

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