Metallographic Examination Of Inhibition Potentials By Sulphuric And Perchloric Di-Vinyl Ether Extracts Of C. Tinctorium For The Severe Corrosion Of Steel Alloy

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***Abstract*—This research is focused on the inhibitive effect of bio-based polymer on the morphology of low carbon steel subjected to strong acidic environments. Surface morphology of the mild steel specimens in the presence and absence of the inhibitor was evaluated by SEM analysis. It was revealed that the addition of inhibitor retarded the corrosion processes, where the grain boundary attacks were completely hindered by the adsorbed inhibitor molecules. The micrograph in the presence of CTE showed a near smoother surface with pit morphology lower than in the absence of plant extract. At the inhibitor concentration of 0.5g/dm^{-3},weight loss measurement shows the maximum IE% was 97.3141 % for 0.5M H_2SO_4 and 92.7007 % for 0.5M $HClO_4$ all which shows that CTE act as a good corrosion inhibitor for mild steel in both acid corrodents. However, from the result, inhibition efficiency of CTE is higher in 0.5M H_2SO_4 compares to that of 0.5M $HClO_4$. This could be due to the fact that $HClO_4$ is a stronger acid (super acid) than H_2SO_4. This result suggests that the increase in efficiencies with increase in inhibitors concentration was due to increase of the number of molecules adsorbed onto mild steel surface and reduces the surface area that is available for the direct acid attack on the metal surface, this was confirmed by the surface morphology studies by scanning electron microscope.**

Keywords—Corrosion, Green inhibitor, Microstructure and Cochlospermum tinctorium.

I. INTRODUCTION

A. Background

Metallographic examination is the main method for examining creep cavities. It has been applied almost worldwide for several years, the main reason being that it provides direct visualization of defects, which is still not possible with other methods. Procedures involved in surface preparation include: cleaning and degreasing, pre-polishing, final polishing and chemical attach.

Corrosion is the deterioration of materials by chemical interaction with their environment. It is the destruction or deterioration of a material because of its reaction with its environment [1]. The term corrosion is sometimes also applied to the degradation of plastics, concrete, and wood, but generally refers to metals. The most widely used metal is iron (usually as steel). Corrosion can cause disastrous damage to metal and alloy structures causing economic consequences in terms of repair, replacement, product losses, safety and environmental pollution. Due to these harmful effects, corrosion is an undesirable phenomenon that ought to be prevented [2]. Corrosion control of metals is technically, economically, environmentally and aesthetically important. The best option is to use inhibitors for protecting metals and alloys against corrosion. Since inorganic corrosion inhibitors are toxic in nature, some green inhibitors which are biodegradable without any heavy metals and other toxic compounds are being promoted [3]. Also plant products are inexpensive, renewable, and readily available. Tannins, organic amino acids, alkaloids and organic dyes of plant origin have good corrosion-inhibiting abilities. Plant extracts contain many organic compounds, having polar atoms such as Oxygen (O), Phosphorus (P), Sulphur (S) and Nitrogen (N). These are adsorbed on the metal surface by these polar atoms and protective films are formed and various adsorption isotherms are obeyed [4].

The relative frequency of occurrence of corrosion depends on the type of industry and environments.

There are various types of corrosion damage. Main forms of corrosion grouped by Einar 2003 are; uniform corrosion, pitting corrosion, crevice corrosion, galvanic corrosion, exfoliation, erosion, cavitation, fretting, intergranular, stress corrosion cracking de-alloying and corrosion fatique [5].

B. Justification

Green corrosion inhibitors are biodegradable and do not contain heavy metals or other toxic compounds. The successful use of naturally occurring substances to inhibit the corrosion of metals in acidic environment and alkaline environment have been reported by many works [2][3][7]. In most of these studies nothing has been reported on the use of diethyl ether extract of *Cochlospermum tinctorium* for the inhibition of the corrosion of mild steel in $HClO_4$ and H_2SO_4. This present study intends to investigate the inhibitive effect of diethyl ether extract of *Cochlospermum tinctorium* on the corrosion of mild steel in H_2SO_4 and $HClO_4$ for mild steels.

Several methods have been derived for preventing or reducing corrosion which include coating, electroplating, painting as well as the use of chemical inhibitors. Of all these the use of chemical inhibitors often remain the most practical and cost effective means of preventing corrosion. Most inhibitors in use are either synthesized from cheap material or chosen from compounds having heteroatom in their aromatic or long chain carbon system. However most of these inhibitors are toxic to the environment hence prompting the search for green corrosion inhibitors [8].

Considering environmental and ecological reasons, green inhibitors are found to be effective.

As inorganic corrosion inhibitors are toxic in nature, so green inhibitors which are biodegradable, without any heavy metals and other toxic compounds are promoted. Also plant products are inexpensive, renewable and readily available. The evaluation of corrosion inhibitor effectiveness is significant in many respects for the purpose of evaluating material performance.

Corrosion inhibition is no longer an academic endeavour; rather the focus must be on prolonging the useful life of a metallic structure in the realistic (or relevant) environment at an acceptable cost advantage. The presence of an inhibitor in the inter-phase between the metal and the liquid environment negates and invalidates in all but perhaps a few cases the electrochemical assumptions underlying electrochemical measuring techniques so prevalent in the industry today. Within the framework of carefully defined quality criteria, it has become necessary to re-interprete the too prevalent weight loss measurements and the methodology of constant inventory test systems and that forms the bases of this current research.

II. MATERIALS AND METHOD

A. Materials

- Thermometer, Beakers,Volumetric flasks (500 ml and 1000 ml),Thermostatic water bath,Measuring cylinder,Aluminium foil,Silicon carbide paper,Electronic weighing balance,Two necked volumetric flask,Stop clock,Conical flasks,Round bottom flask,Storage boards,Hand gloves,Bristtle brush,Dessicator,Wash bottle,Evaporating dish,Measuring cylinders,Zinc dust,Extract of *Cochlospermum tinctorium,* Mild steel sheet: (%wt) Mn (0.6), P (0.36), C (0.15), and Si (0.03).
- Reagents include; Diethyl ether (AR),Acetone (AR),$HClO_4$ (60 % purity) AR,AR H_2SO_4 (98 % purity)AR,Distilled water and NaOH (AR).

B. Collection and Extraction of the Plant Sample

Sample of *Cochlospermum tinctorium* leaves were collected from Abocho, Dekina Local Government of Kogi State, identified in University of Calabar Herbarium with voucher number 029 and family name *Cochlospemaceae* by Effa, Effa Anobela and it was sun dried and grounded to powdered form, 300 g of the pulverized sample was weighed and soaked in diethyl ether for 48 hours. After 48 hours, solvent was filtered and evaporated using a water bath.

The divinylethyl ether extract was used in the preparation of the test solutions by dissolving 0.1 g, 0.2 g, 0.3 g, 0.4 g, 0.5 g of the extract in 1L of 0.5 M H_2SO_4 and 0.5M $HClO_4$ and then used to test for corrosion inhibition properties. This is because the phytochemical screening of *cochlospermum tinctorium* leaves by Ahmad (2011) and Etuk *et al., (*2009) was believed to contain phytochemicals that show the presence of hetero-atoms which could serve as a source of bonding to metal surface to reduce corrosion processes in metals [6][7].

III. MATERIALS PREPARATION

Mild steel materials used were collected from Naval dock Yard Apapa, Lagos. The metals were subjected to elemental analysis. The sheet were mechanically press cut into different coupons, each of dimension 4.0 x 2 .0 x 2.0 cm rectangular specimens and polished with emery papers (silicon carbide paper, 100- 1200) grades.

These polished coupons were subjected to water test (ASTM 2000) to make sure the metal surfaces were free of pits.

Each coupon was degreased by washing with and dipped in acetone and allowed to dry and was preserved in a desiccators. All reagents that were used for the study were of analar grades. The prepared coupons were now subjected to corrosion testing using Scanning electron microscope.

IV. SURFACE MORPHOLOGICAL STUDIES

Surface analysis was performed using scanning electron microscope. SEM images were obtained from MS surface after the immersion in 0.5M H_2SO_4 and

0.5M $HClO_4$ in the absence and presence of *Cochlospermum tinctorium* for two hours at 30^oC.

A. Microstructural Examination

The microstructure of the mild steel coupons were examined before and after the corrosion experiment to assess the effect of corrosion on the microstructure and the effect of the addition of the extract as an inhibitor. The process series of preparatory stages carried out before the actual microscopic examination include sectioning, mounting, grinding (smooth and rough grinding), polishing and etching.

B. Microscopic Examination

This was done by using the metallurgical microscope. The etched specimen was placed on the microscope and the microstructures of the various micrographs were obtained accordingly [8]

V. RESULTS AND DISCUSSION

Metallography is useful in the study of corrosion because it helps to identify the type of corrosion on the material. The surface morphology of the metal was evaluated by Scanning Electron Microscopy (SEM) to show the micrographs of the freshly polished mild steel surface, corrosion specimens with and without CTE inhibitor in 0.5M H_2SO_4 and 0.5M $HClO_4$.

The metallographic micrographs of the mild steel coupons before and after immersion in plain acids and in presence of 0.5g/L plant extracts are shown as Figures 3.0 a, b, c, d and e respectively. Fig. 3.0 (a) shows plain metal surface before immersion. The surface shows a more uniform distribution of ferrite and pearlite structure in the grain structures with distinct grain boundary pits at a magnification of x500.

From Fig. 3.0 (b) and (c) the micrographs in 0.5M H_2SO_4 and 0.5M $HClO_4$ without inhibitor are shown, it is evident that there is a form of grain boundary attack by the acids. Corrosion attacked and distorted the grain boundaries of the mild steel in the absence of plant extract. The micrographs in Fig 3.0 (b) and (c) showed that the attack is localized and affects basically the grain boundaries. This is suggestive that the type of corrosion is a galvanic corrosion.

From Fig. 3.0 (d) and (e) the micrographs in 0.5M H_2SO_4 and 0.5M $HClO_4$ in the presence of inhibitor are shown. The addition of plant extract retards the corrosion process as evidenced in Fig. 3.0 (d) and (e), where the grain boundaries attack has been completely hindered by the adsorbed inhibitor molecules. The micrographs Fig. 3.0 (d) and (e) showed a near smoother surface with a pit morphology lower than those in the absence of plant extract, the result of pores histogram before and after immersion in plain acids and in the presence of 0.5g/L CTE inhibitor obtained are confirmatory evidence.

Furthermore, the histogram revealed the pores diameter of the mild steel coupons. Fig. 3.0 (a) indicates the pores diameter of freshly polished coupon which is les than that of pores diameter of mild steel coupon in acidic media as showned in Fig. 3.0 (b) and (c). Fig. 3.0 (d) and (e) which shows the mildsteel coupons in the presence of plant extract recorded a fewer pores. In this case, the grain boundaries are preserved indicating the effectiveness of the inhibitor.

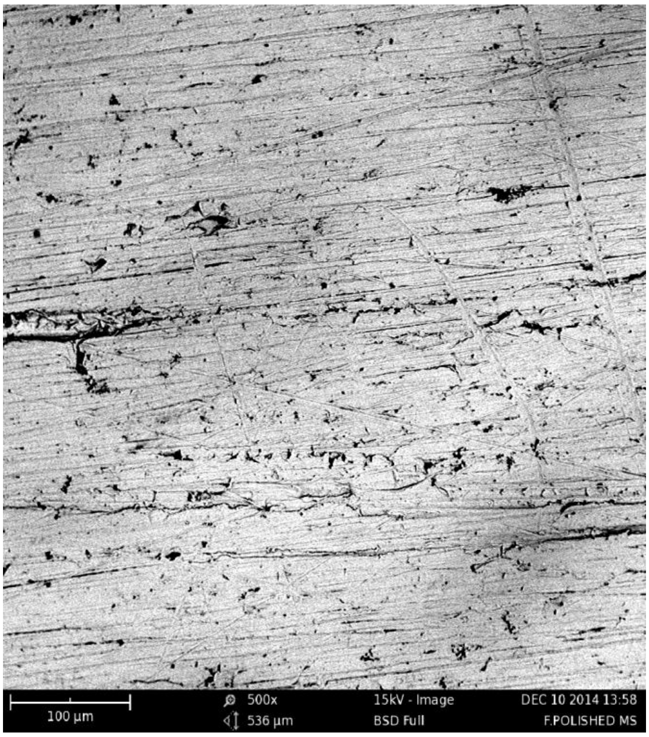

Fig. 3.0.(a) Micrograph for Freshly Polished MS before immersion

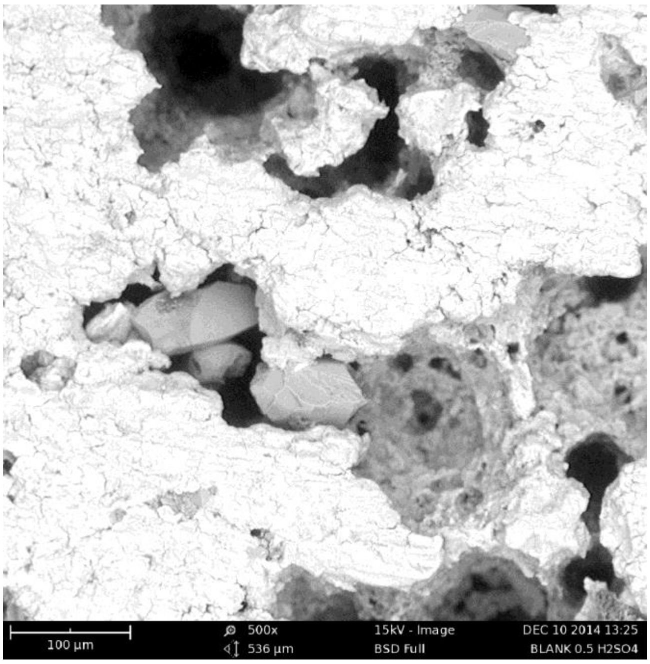

Fig.3.0.(b) Micrograph for MS in 0.5M H_2SO_4 without CTE

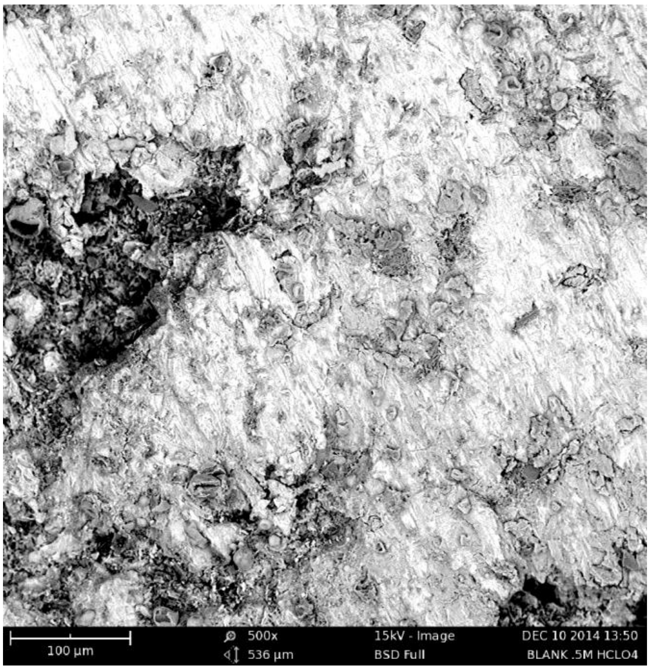

Fig.3.0.(c) Micrograph for MS in 0.5M HClO4 without CTE

Fig.3.0.(d) Micrograph for MS in 0.5M H_2SO_4 with CTE

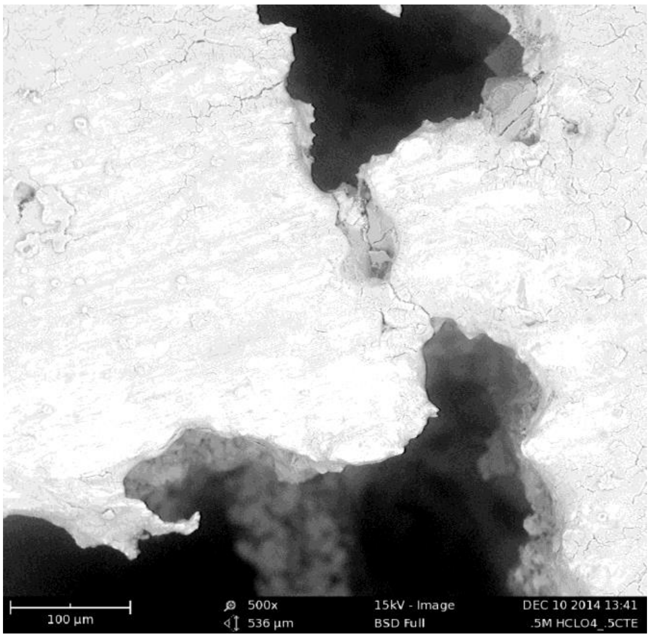

Fig.3.0.(e) Micrograph for MS in 0.5M HClO4 with CTE

CONCLUSION

This research investigated the phenomenon of corrosion of mild steel metal in acidic media. Scanning Electron Microscope was used in this morphological study. From the result and discussions, the following conclusions were derived;

- CTE acts as inhibitor for mild steel in 0.5M H_2SO_4 and 0.5M $HClO_4$ acid medium.
- Inhibition efficiency increases with increase in concentration of inhibitor.
- Compared to several inhibitors in literature, CTE exhibited higher surface coverage for mild steel.
- Corrosion rate decreased with CTE addition in 0.5M H_2SO_4 and 0.5M $HClO_{4.}$
- The metallographic micrographs showed that the morphology pits of mild steel in the absence of CTE demonstrated a higher number than that in the presence of inhibitor.

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