

The Evaluation And Investigation Of Green Corrosion Of The Low Carbon Steel And Inhibition Of The Leaf Extract Using Weight Loss Technique

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Abstract – The green corrosion inhibition of buried low carbon steel (LCS) (in different molarities of acid) using orange leaves extract (OLE) as inhibitor has successfully been carried out. The soil sample was characterized to determine its resistivity, chloride and sulphate content, oxidation-reduction potential (ORP) and corrosively towards buried structural material while the absorbed film on LCS surface was characterized using FTIR spectrophotometer to determine the functional groups weight loss measurement, tafel technique were used to determine the corrosion behavior of the metal in the composed medium. The corrosion media were hydrochloric acid (1.0m and 2.0m) an sulphuric acid (1.0m and 2.0m). The weight loss tests were carried out at room temperature and inhibitor concentration of 0.5 to 2.5g/l over a time interval of 25 to 125hours. Result show that OLE is a good corrosion inhibitor of low carbon steel in acidic medium over the time interval of study, the inhibition efficiency of the OLE increased with increase in inhibitor concentration. The highest inhibition efficiency obtained was 89.61% in 1.0m H₂SO₄, it follows that OLE as better as inhibitor in H₂SO₄ than in HCL. Also, the effectiveness of OLE as an inhibitor is higher at lower acid concentration both the open circuit potential and the Tafel plots collaborate the results obtain from weight loss technique. The FTIR spectroscopic analysis reveals the presence of organic compounds, alkyl halides and nitro-compounds these seem to be the functional groups responsible for the inhibition characteristics of the OLE.

Keywords – Investigation, Green Corrosion, Low Carbon Steel, Inhibition Of The Leaf Extract, Weight Loss Technique.

INTRODUCTION

Corrosion is the irreversible deterioration of metals by chemical attack. It occurs when metal interact with its environment causing it or its alloys to return to their unrefined natural forms as minerals and ores (Ogunleye et al., 2019). Metals generally tend to corrode as they always prefer to return to the stable oxide form as a result of corrosion. Low carbon steel is one of the most important metals in existence and has a wild variety of industrial applications. However, it corrodes due to pH, oxidation-reduction potential, chloride and sulfate contents in the acidified moisture in the environment (Popoola et al., 2013, Bhattarai et al., 2016). The Studies on LCS surface reactions in acidified moisture have been the subject of investigation due to areas of applications of LCS for durability in performance and in service (Cheng et al., 2007). Typical situations abound where Synthetic inhibitors have been widely applied to protect metal surfaces against corrosion in the chemical industries, textile wet processing

plants, marine, oil and gas industries (Uchenna et al., 2019); Zhang et al., 2012; Markhali et al., 2013). Most synthetic organic inhibitors contain nitrogen, sulphur or oxygen atoms in their structures (Chigondo and Chigondo 2016). The costs of these synthetic inhibitors are high. This is notwithstanding, they could be toxic to the environment and human lives. Presently, corrosion scientists and engineers are exploring the use of plant extracts inhibitors that are inexpensive, readily available, environmentally friendly and ecologically acceptable, and renewable. The plant extracts are primarily organic compounds composed of carbon, nitrogen, oxygen and sulphur atoms. They are environmentally friendly and constitute a good displacement for the toxic synthetic inhibitors and consequently promote greenness to the environment Frederick et al., (2020). These readily available green inhibitors are nontoxic, inexpensive and can be extracted from various plant parts (Okafor et al., 2011, Oguzie et al., 2013).

Statement of the problems

In spite of the enormous wide range of availability of raw material in Nigeria including orange leaves, most used corrosion inhibitors are imported into the country. The country therefore spends a lot of foreign exchange importing corrosion inhibitors. Nigeria on the other hand has experienced uncountable sudden failure of structure accompanied by huge loss of materials over the years, as a result of corrosion attack especially when low carbon steel are used as structural members. This research is to draw the attention of our country Nigeria on the need to utilize our local raw materials for the production of corrosion inhibitor and consequently reduce high rate of expenditure on importation of corrosion inhibitors. It is known that most conventional corrosion inhibitors constitute environmental hazards. The use of green inhibitors as an alternative to these hazardous though effective inhibitors would help in safeguarding our environment

Summary of current related work

Table 1.0: Summary of current related work

S/N	Authors	Aims	Summary of Finding
1	Uchenna et al, (2019)	Corrosion Inhibition of Mild Steel by Citrus sinensis (Orange) Leaves Extract in Hcl/H ₂ SO ₄ Acid Medium	Citrus sinensis leaves extract was used as inhibitor in 1MHCl and 0.5M H ₂ SO ₄ solution on Mild steel. Potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) tests were carried out. Potentiodynamic polarization and electrochemical Impedence spectroscopy (EIS) showed that the extract concentration from citrus sinensis L. acts as bio-inhibitor in acidic media.
2	Ogunleye et al (2020).	Green corrosion inhibition and adsorption characteristics of luffa cylindrical leaf extract on mild steel in hydrochloric acid environment.	The corrosion inhibition of luffa cylindrical leaf extract (LCLE) was investigated using gravimetric, depth of attack and surface analysis techniques. The constituents of proposed inhibitor were identified by using a GC-MS. The extract was characterized using FTIR spectrophotometer. The extract showed good inhibition characteristic for mild steel in 0.5M HCl solution due to the presence of tannins, phenol, flavonoids and alkanols groups according to the GC-MS and FTIR analyses. The result obtained using weight loss and depth

			of attack methods, the extract proved to be an excellent inhibitor.
3	Divakara, (2017)	Inhibition of mild steel corrosion in acid medium	The inhibiting effect of BPTU and CPTU on mild steel corrosion in 0.1MHCl medium using the Tafel extrapolation technique. It reveals that BPTU and CPTU act as anodic inhibitors for mild steel in the HCl medium and good inhibition efficiency. The investigation shows that there was a good correlation between Tafel extrapolation and linear polarization results.
4	Fatma et al (2019).	Corrosion inhibition of mild steel in acidic solution by leaves and stem extract of Acacia nilotica	The inhibitive action of water extracts of vachellianilotica (VN) leaves and gum Arabic (GA) stem of the Acacia nilotica on the corrosion of mild steel was studied using electrochemical measurements. The plant extract were characterized by FTIR, GC-MS analysis. From the results, it concluded that VN leaves and GA stem show promising results result as an eco-friendly and mild steel corrosion inhibition.
5	Ogunleye et al, (2019)	synthesis of green corrosion inhibitor for mild steel in acidic environment	The inhibitive potential of Mondia whittei roots extract (MWE) on the corrosion of mild steel in 0.5MHCl solution was investigated by weight loss, depth of attack and surface analysis. The identification of constituents of MWE and characteristics of the test solution and adsorbed film were evaluated by GC-MS and FTIR spectrophotometer. Polarization curves reveal that the inhibition efficiency according to the depth of attack given by R_a and R_z increased with increasing inhibitor concentration, temperature, time and volume-area ratio.

6	Gadow and Fakeeh (2022) Green inhibitor of carbon steel corrosion in 1 M hydrochloric acid: <i>Eruca sativa</i> seed extract (experimental and theoretical studies)	The adsorption activity and inhibition effect of <i>Eruca sativa</i> seed extract as a green inhibitor for the dissolution of carbon steel in 1 M hydrochloric acid solution were investigated. In this study, we used a chemical technique (gravimetric method), electrochemical techniques, electrical frequency modulation (EFM), electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization techniques, and theoretical studies. In addition to these techniques, we examined the surface morphology of the carbon steel utilizing different methods. The measurements of the polarization technique indicate that this extract acts as a mixed-type inhibitor. Thermodynamic parameters were calculated and discussed. The adsorption of <i>Eruca sativa</i> seed extracts on the alloy obeys the Langmuir and Henry adsorption isotherms. The extract gives excellent inhibition efficiency 94.8% by a gravimetric method at 0.3 g L ⁻¹ from the extract. The relationship between the calculated % IE from experiments and the theoretical studies was established.
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Research gap

The use of orange leaves as inhibitor has not been adequately explored. Moreso, study has not been carried out on buried steel sheets in acidified environments. This work tries to mitigate this gap.

MATERIALS AND METHOD

EQUIPMENT AND MATERIALS

Equipment

The equipment used were: Electronic weighing balance, stamping machine, measuring cylinder (1000ml), volumetric flask (250ml), plastic containers, nylon thread, blender, emery papers, and digital pH meter.

Analytical equipment includes working electrode low-carbon steel (LCS), Reference electrode (RE) made of silver/silver chloride (Ag/AgCl), counter electrode (graphite rod). CH1604E electrochemical analyzer used. Materials: LCS, Orange leaves, HCl and H₂SO₄ acid.

Sources of Materials

LCS was sourced from new site market in Nsukka, Enugu State, Nigeria and the chemical analyses was carried out at quality assurance and research control express laboratory, Ajaokuta steel Company Limited.

Orange leaves were obtained from a local garden in Amokwe at Nsukka Local Government Area, Enugu State. HCl, H₂SO₄, acetone, distilled water, ethanol were sourced from University of Nigeria Nsukka. Plastic containers, emery paper, thread, paper tape (for labeling) were sourced from Ogige market Nsukka.

Method

Preparation of steel sheet coupons

The low- carbon steel sheet samples 3.0 x 2.0 x 4mm were cut from large sheet as coupons. Holes of about 0.1cm dimension were drilled at one end to enable tying up the coupons with nylon thread. The coupons were mechanically polished with series of emery papers of variable grades starting with coarse and proceeding to the finest (320) grade. These polished coupons were washed with distilled water and dried in acetone and weighed. The weighed coupons were then used for analyses.

Preparation of the inhibitor extract

Fresh orange leaves were washed with distilled water, dried at room temperature (27⁰C) for 14days and ground to powder. The OLE powder was taken to Divine Chemical and Analytical laboratory Nsukka for extraction. 1000g of the sample was placed in the thimble, which is placed inside the soxhlet extractor and 250ml of analytical ethanol was poured into the round bottom flask, which was attached to a soxhlet extractor and condenser see fig 3.1b on an isomantle. The side arm is lagged with glass wool. The solvent was heated at 60⁰C for 3hrs. As the ethanol vapour reached the condenser through the side arm of the soxhlet extractor, it condensed and dropped back into sample in the thimble; the ethanol soluble substance dissolved and was siphoned back into the flask (fig 3.1b). The extraction continued for 4 hours. The flask with the extract was disconnected and heated at 40⁰C for 4 hours until almost all the ethanol evaporated to form a gel, cooled in desiccators and weighed. Total 0f 33g of gel was obtained.



Fig 1.0: (a) Grounded powdered orange leaves and (b) Soxhlet extractor

$$\% \text{ yield} = \frac{\text{wt of flask + extract} - \text{wt of flask}}{\text{wt of sample}}$$

$$W_0 = \text{weight of original sample} = 1000g$$

$$W_1 = \text{weight of empty beaker} = 293.4g$$

$$W_2 = \text{weight of extract} + \text{empty beaker} = 326.40g$$

$$\begin{aligned} \% \text{ yield} &= \frac{W_2 - W_1}{W_0} \\ &= \frac{326.40 - 293.4}{1000} \times 100 \\ \frac{3300}{1000} &= 3.30\% \end{aligned}$$

$$\text{Extract concentration g/L} = \frac{\text{Mass of gel (g)}}{\text{Volume of solution (L)}}$$

Collection of soil

Soil sample was collected from Ngwo in Udi local government area in Enugu State using composite sample method, the soil was taken from the depth of about 1 meter below ground level. The soil collected was crushed to uniform size and sieved. The soil composition analysis was determined in the laboratory like; moisture content, soil type, soil resistivity, soil pH, oxidation-reduction potential (ORP), Chloride and Sulfate contents using appropriate equipment.

Moisture content

Moisture content in the soil was determined using weight loss method in accordance with the ASTM D4959-07 (2007) standards; the weight difference between the soil sample before and after evaporation was regarded as the moisture content. Soil sample was sieved with 2mm mesh and was poured into a pre-weighted beaker as M_2 , meanwhile, the empty beaker was weighed as M_1 . The soil was dried in oven in a certain temperature at 105°C for 2 hours. After the soil sample got dried, the beaker that contained the dried soil was weighed again as M_3 was recorded and used to calculate the percentage moisture content (Buhari et al., 2020). The percentage moisture content MC, is given by

$$\text{Moisture content} = \frac{M_2 - M_3}{M_3 - M_1}$$

$$M_1 = \text{Empty plastic container} = 86.3g, M_2 = \text{Initial weight of soil} = 2014.1$$

$$M_3 = \text{final weight after drying} = 1493.3g$$

$$\text{M.C} = \frac{2014.1 - 1493.3}{1493.3 - 86.3} \times 100\%$$

$$\frac{620.8 \times 100}{1407} = 37.09\%$$

pH measurement

A digital pH meter was used to determine the pH of 1:2 soil-water suspension of the soil sample in accordance with the ASTM G51-95 (2012) standards. 10g of the dry soil was placed in 50 cm³ beaker and 20 cm³ of distilled added. The content of the beaker was thoroughly mixed. The pH of the soil was taken before mixing the soil sample soil. About 20g of acidified acid soil was added into a 50cm³ beakers. 20 cm³ of distilled water was added to the soil sample and the content mixed thoroughly. The pH of the acidified soil was measured. (Buhari et al., 2020).

Preparation of acidified acid soil using (H₂SO₄ and HCL)

The various acid concentrations were added to 707g of soil sample and mixed properly to achieve homogeneous acidified soil.

Preparation of 1MHCl and 2MHCl.

83.6ml of concHCl was added to 916.4ml of distilled water to make it 1M HCl and 167.2ml of conc. HCl was added to 832.8ml of distilled water to make it 2M HCl. See calculations below.

To prepare 1MHCl

$$C_1V_1 = C_2V_2$$

Where

C₁ = initial concentration (molarity) of acid to be prepared = 1MHCl

V₁ = volume of acid to be prepared = 1000ml

C₂ = final concentration of product and

V₂ = volume of raw acid to measure out.

S.G = specific gravity of HCl = 1.18

M.m = molar mass of HCl = 36.5

$$C_2 = \frac{\text{Specific gravity} \times \% \text{ purity} \times 10}{\text{Molar mass}}$$

$$= \frac{1.18 \times 37 \times 10}{36.5} = 11.96M$$

$$C_1V_1 = C_2V_2$$

$$1 \times 1000 = 11.96 \times V_2$$

$$V_2 = \frac{1 \times 1000}{11.96} = 83.6ml$$

Distilled water to be added = 1000 - 83.6 = 916.4ml

To prepare 2MHCl

$$C_1V_1 = C_2V_2$$

$$2 \times 1000 = 11.96 \times V_2$$

$$V_2 = \frac{2 \times 1000}{11.96} = 167.2ml$$

Amount of distilled water needed was 1000 - 167.2ml = 832.8ml

The volume of acid was measured out from concentration of HCl with a measuring cylinder and diluted with 1000ml of distilled water. In table below were different volumes of acid from above equations.

Table 1.1: Preparation of 1M and 2MHCl.

Conc. (molarity) of HCl (mol dm^{-3})	Volume of HCl concentration	Volume of distilled water (H_2O)
1M	83.6ml	916.4ml
2M	167.2ml	832.8ml

Preparation of $1\text{M H}_2\text{SO}_4$ and $2\text{M H}_2\text{SO}_4$.

54.35mls of conc. H_2SO_4 was added into 945.65ml of distilled water to make it 1M H_2SO_4 and 108.7ml of conc. H_2SO_4 was added into 891.3ml of distilled water to make it 2M H_2SO_4 .

To prepare 1M H_2SO_4

$$C_1 V_1 = C_2 V_2$$

C_1 = initial concentration of acid to be prepared,

C_1 = volume of acid to be prepared,

C_2 = final concentration of product and

V_2 = volume of raw acid to be measured out.

$S \cdot G$ = specific gravity of $\text{H}_2\text{SO}_4 = 1.84$

$M.m$ = molar mass = 98.0g/mol

Where $C_1 = 1\text{M H}_2\text{SO}_4$

$$V_2 = 1000\text{ml}$$

$$C_2 = \frac{\text{Specific gravity} \times \% \text{ purity} \times 10}{\text{Molar mass}}$$

$$= \frac{1.84 \times 98 \times 10}{98} = 18.4\text{M}$$

$$C_1 V_1 = C_2 V_2$$

$$1 \times 1000 = 18.4 \times V_2$$

$$V_2 = \frac{1 \times 1000}{18.4} = 54.35\text{ml}$$

Water needed = $1000 - 54.35\text{ml} = 945.65\text{ml}$ of distilled water.

To prepare 2M H_2SO_4 .

$$C_1 V_1 = C_2 V_2$$

$$2 \times 1000 = 18.4 \times V_2$$

$$V_2 = \frac{2 \times 1000}{18.4} = 108.7\text{ml}$$

Water needed $1000 - 108 \cdot 7 = 891 \cdot 3 \text{ ml}$

The volume of acid was measured out from concentration of H_2SO_4 with a measuring cylinder and diluted with 1000ml of distilled water. The tables below were different volume of acid from above equations.

Table 1.2: Preparation of 1M and 2M of H_2SO_4 .

Conc. (molarities) of H_2SO_4 (mol dm ⁻³)	Volume of H_2SO_4 concentration	Volume of distilled water (H_2O)
1M	54.35ml	945.65ml
2M	108.7ml	891.3ml

Preparation of buried coupons in acidified acid with or without inhibitor

707g each of acid soil was filled in 16 plastic containers with 4 containers out of the 16 containers bearing only acid and soil sample used as control (without inhibitor). The total numbers of coupons that were buried was eighty (80) pieces. Five coupons were buried at 60mm depth in each of the plastic containers, the other twelve plastic containers with buried coupons had inhibitors in them weighing 0.5g/L in 1M HCl, 2M HCl, 1M H_2SO_4 , 2M H_2SO_4 , 1.0g/L in 1M HCl, 2M HCl, 1M H_2SO_4 , 2M H_2SO_4 and 2.5g/L in 1M HCl, 2M HCl, 1M H_2SO_4 , 2M H_2SO_4 of the orange leaves extract (OLE).

The buried coupons were monitored every 25hrs. Then, in every 25hrs, the coupons in each of the plastic container were removed, washed with distilled water, dried in acetone and weighed using electronic weighing balance and compared corrosion rate with or without inhibitors. The experiments were repeated 25, 50, 75, 100, 125hours.

RESULTS AND DISCUSSION

Chemical analysis of low carbon steel

The chemical analysis of the low-carbon steel was carried out at quality assurance and research control express laboratory Ajaokuta steel Company Limited. The result is shown in table 1.3.

Table 1.3: Chemical analysis of the low carbon steel

Element	C	Si	S	Mn	P	Cr	Mo	Ni	V	Cu
Composition (%)	0.20	0.27	0.35	0.65	0.35	0.85	-	0.30	-	0.30

Element	W	As	Co	Al	Pb	Ca	Zn	Fe
Composition %	-	-	-	-	-	-	-	96.73

Moisture Content in soil

The moisture content of the soil sample collected was found to be 37.05%. The moisture content of loamy soil ranges from 35 to 40%. The carbon steel was therefore buried in loamy soil.

Soil pH

Initial pH of the soil before mixing with acids was 7.2 (slightly alkaline). Soil pHs after acid treatment are given in table 1.4. Generally, steel is more susceptible to corrosion in acidic than alkaline media.

Table 1.4: pH values of acidified soil.

Acid soil	pH value
1M HCl	5.9
2M HCl	5.4
1M H ₂ SO ₄	5.7
2M H ₂ SO ₄	5.2

Soil Resistivity

The soil resistivity is a soil characteristic that is dependent on the moisture content, concentration of different organic content dissolved in it, degree of compactness and temperature etc.

During the test of soil resistivity, which was performed under standard conditions (i.e. room temperature, 27°C) in order to avoid any form of loss of water or contamination of any form. It was obtained that the resistivity of the soil sample was 15,000 ohm.cm. From the experiment, the resistivity of the soil sample shows that it is mildly corrosive (MiC) due to the measurable quantities of organic content, and moisture content present in the soil (see table 4.2). The resistivity of the soil factor is vital in the soil analysis and has been proven over the years by scientists to be in inverse variation with the corrosive nature of the soil.

Oxidation-Reduction Potential (ORP)

ORP was performed using ORP sensor. When there is low ORP, it indicates that the soil is low in oxygen content, this will increase the chances of survival of sulphate reducing bacteria in the soil which reduces sulfate to sulphide this in turn increases the corrosion of the soil. From the experiment, the ORP value of the soil sample is 250mV which is an indication of mildly corrosive (MiC) (see table 4.2). Therefore ORP of the soil is inversely proportional to corrosive rate of the soil. For effective corrosion in the soil, the ORP should be less than zero or negative values.

Sulphate content in the soil

Sulphate content is a major catalyst that increases electrochemical reactions that occur during corrosion process. It is generally harmful for buried-structural materials as it brings about corrosion in the buried materials. From the result obtained, the sulphate content of the soil sample is 170ppm. This indicates that the soil sample is mildly corrosive (MiC) (see table 4.2). Also it is pertinent to note that soil sulphate increases the soil conductivity. Hence from the result obtained the sulphate content of the soil is less than 200ppm of the sulphate portraying mildly corrosive (MiC) and hence mild conductivity.

Chloride content in soil

The chloride content of the soil is an inevitable factor that indicates corrosion. The chloride content of the soil reduces the passivity of buried structural materials and also partially decreases soil's resistivity. From the experiment it was observed that the chloride content of the soil was 80ppm chloride content, which is an indication that the soil is mildly corrosive (MiC) (see table 1.4).

Table 1.5: Relationship between soil resistivity, chloride and sulphate content, oxidation- reduction potential (ORP) and soil corrosivity towards buried- structural material (JagadeshBhattarai.; 2013).

Soil parameters	Soil corrosivity rate
1. Soil resistivity (Ohm.cm)	
>20,000	Essentially non-corrosive
10,000 -20,000	Mildly Corrosive (MiC)
5,000-10,000	Moderately corrosive (MoC)
3,000-5,000	Corrosive (C)
1,000-3,000	Highly corrosive (HC)
<1,000	Extremely corrosive (EC)
2. Oxidation-Reduction potential (mV)	
>400	Less Corrosive (LC)
201-400	Mildly Corrosive (MiC)
100-200	Moderately Corrosive (MC)
<100	Severe Corrosive (SC)
3. Sulphate Content (ppm)	
< 100	Less Corrosive (LC)
100-200	Mildly Corrosive (MiC)
>200	Corrosive (C)
4. Chloride Content (ppm)	
<50	Less Corrosive (LC)
50-100	Mildly Corrosive (MiC)
>200	Corrosive (C)

Table 1.6: Characteristics of IR Absorption peaks of functional groups

Vibration	Position (cm ⁻¹)	Intensity
Alcohols (O-H)	3550-3200	br, s
Alkanes(C-H stretch)	2990-2850	m to s
Alkenes(C=C stretch)	1680-1620	w to m
=C-H bend	995-685	s
Alkynes(C≡C stretch)	2250-2100	m to w
Aromatic compound(C=H stretch)	3100-3000	m to w

C=C stretch	1625-1440	m to w
C-H bend	900-680	s
Amines (N-H stretch)	3550-3250	br, m
Aldehydes (C-H stretch)	2900-2800	s
Nitriles(C $\equiv$ N stretch)	2280-2200	s
Ketones(C=O stretch)	1750-1705	s
Esters(C=O stretch)	1765-1735	s
Carboxylic Acids (O-H stretch)	3200-2500	br,m to w
C=O stretch	1715-1680	s
Amides (N-H stretch)	3500-3150	m
C=O stretch	1700-1630	s
Anhydrides (C=O stretch)	1850-1800	s
	1790-1740	s
Acid chloride (C=O stretch)	1815-1770	s
Nitro compound (NO ₂ stretch)	1570-1490	s
	1390-1300	s
Alkyl Halides (C-F stretch)	1000-1400	Hidden in
C-Cl stretch	< 600-840	fingerprint
C-Br stretch	< 700	region
C-I stretch	< 600	

The effect of inhibitor concentration on corrosion rate and inhibition efficiency

The effect of inhibitor concentration on corrosion rate and inhibition efficiency are shown in fig 4.4, to 4.8 a,b. It could be obtained that for the two acid concentrations used and the various dosage of inhibitor, the corrosion rate decreases while the inhibition efficiency increases with time. This could be attributed to increase in adsorption of the surface active groups of the inhibition on the LCS.

In 25hrs, of exposure the inhibition efficiency of the OLE was only 23.71%, 40.18%, and 50.00% for inhibition concentration of 0.5g/L, 1.0g/L and 2.5g/L in 1M HCl while at a time of 125hrs the inhibition efficiency had risen to 62.07%, 75.36% and 86.54% for the same inhibitor and acid concentration.

Similarly, for 2M HCl the inhibition efficiency were 18.75%, 33.69% and 39.55% for OLE concentration of 0.5g/L, 1.0g/L and 2.5g/L in 25hrs of exposure and 61.07%, 70.00% and 82.76% in of 125hrs exposure. The same trend was observed when the acid media was 1M and 2M H₂SO₄. Fig 4.5 and 4.6a,b, showed that in absence of inhibitor the corrosion rate increased and decreased with the increase inhibitor concentration. The implication of the foregoing is that OLE is a better inhibitor at low acid concentration.

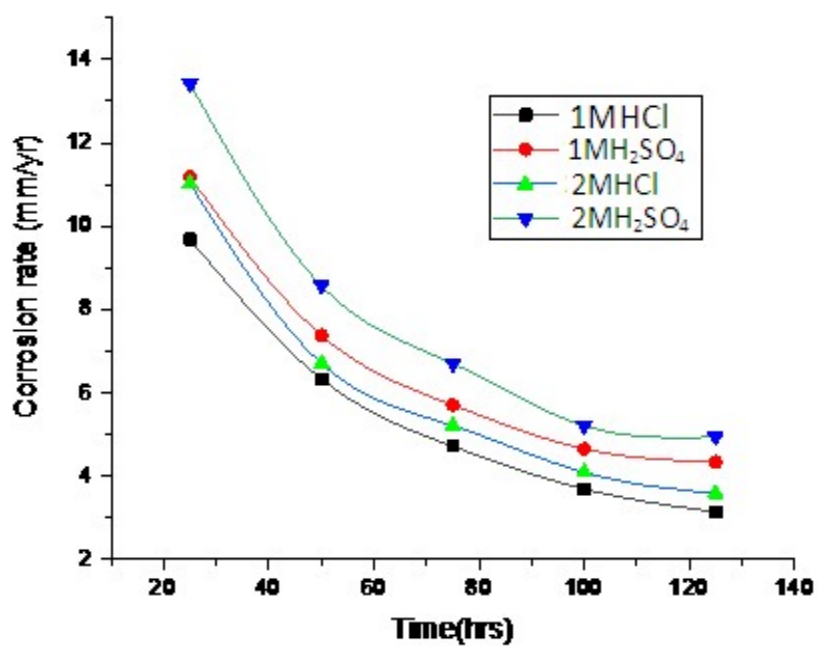


Fig 1.1: Effect of acid concentration on corrosion rate of uninhibited LCS

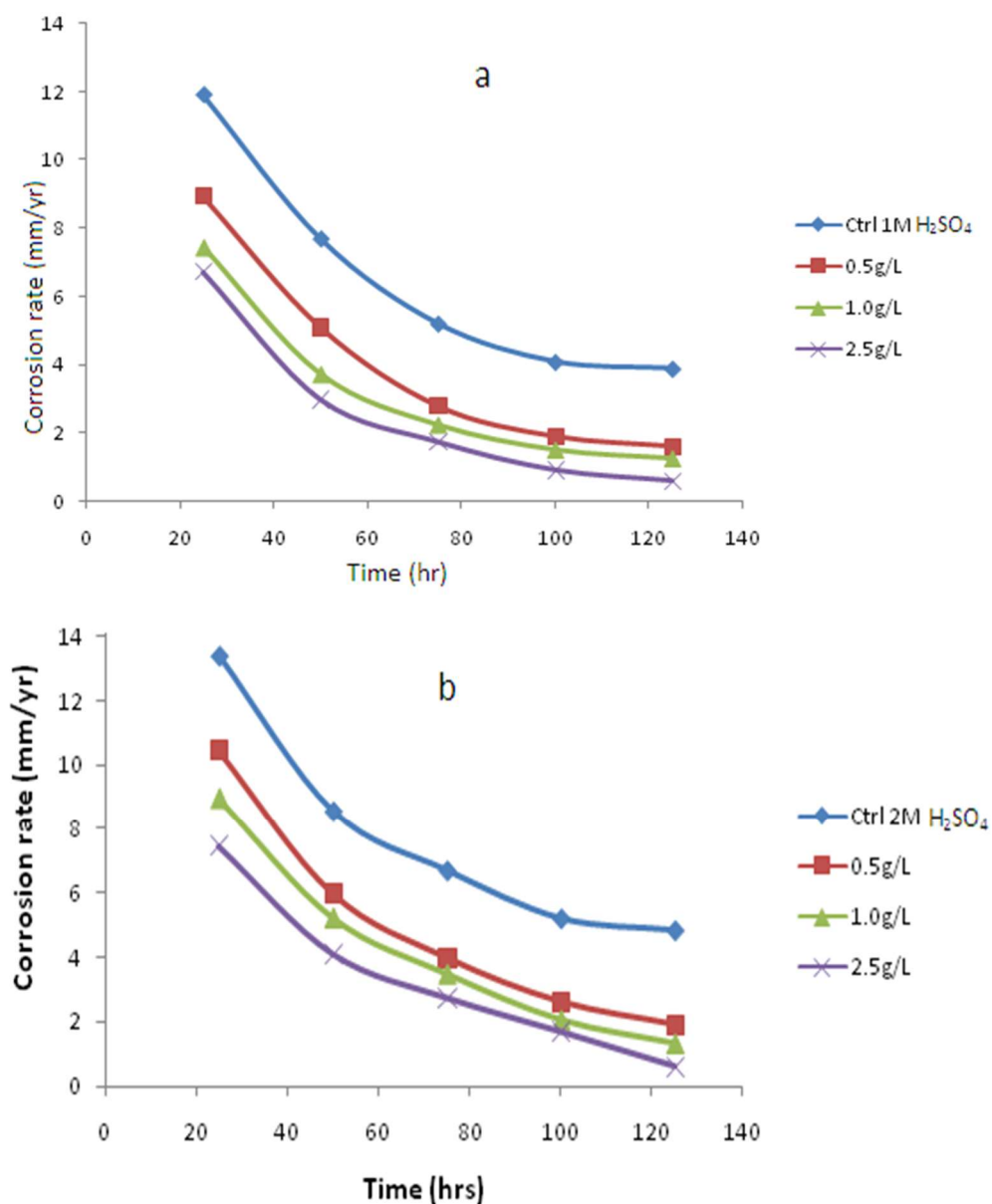


Fig 1.3: Effect of inhibitor concentration on the corrosion rate of LCS in
(a) 1M H₂SO₄ and (b) 2M H₂SO₄.

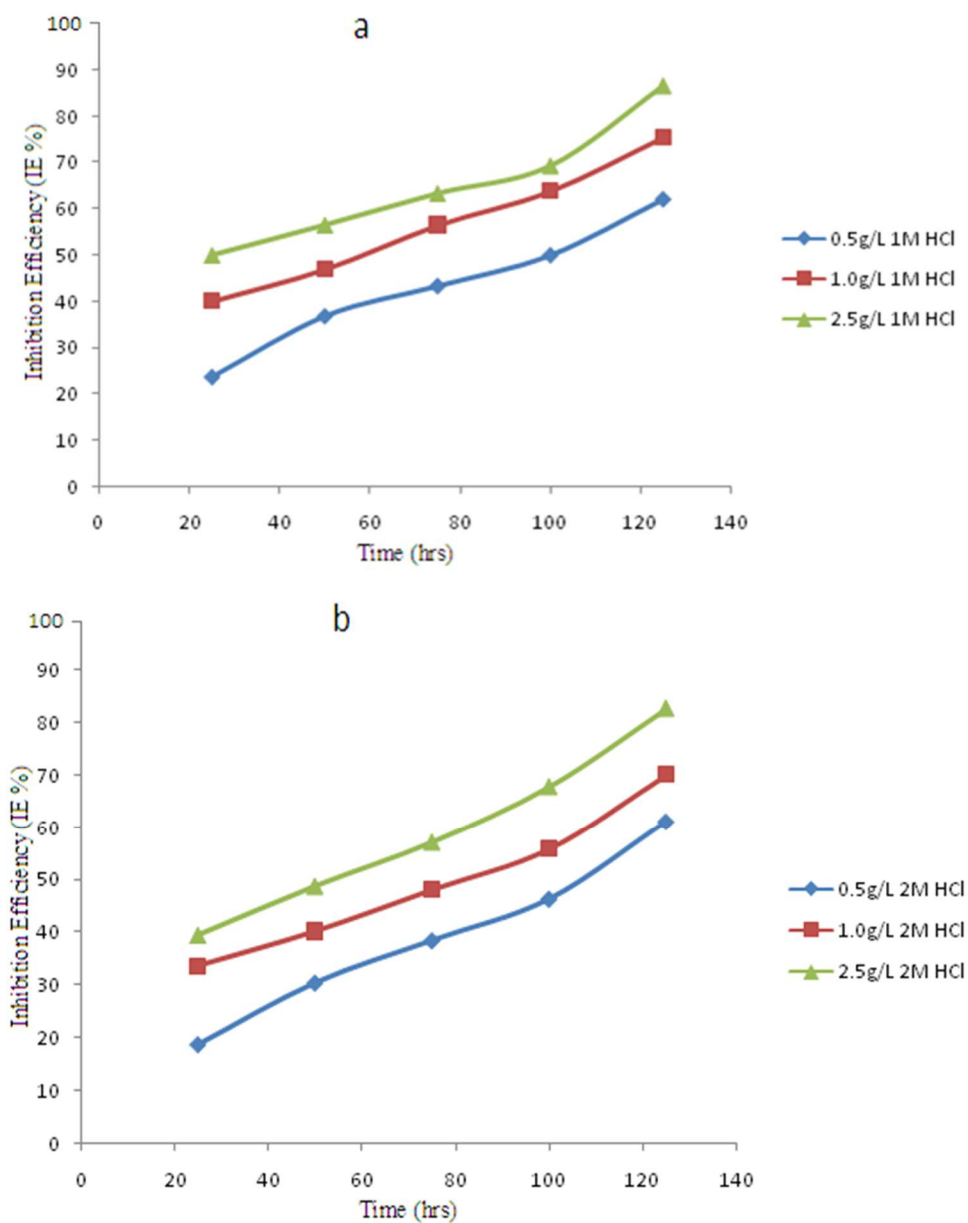


Fig 1.4: Effect of inhibitor concentration on Inhibition Efficiency (I.E %) of OLE in
(a) 1M HCl and (b) 2M HCl.

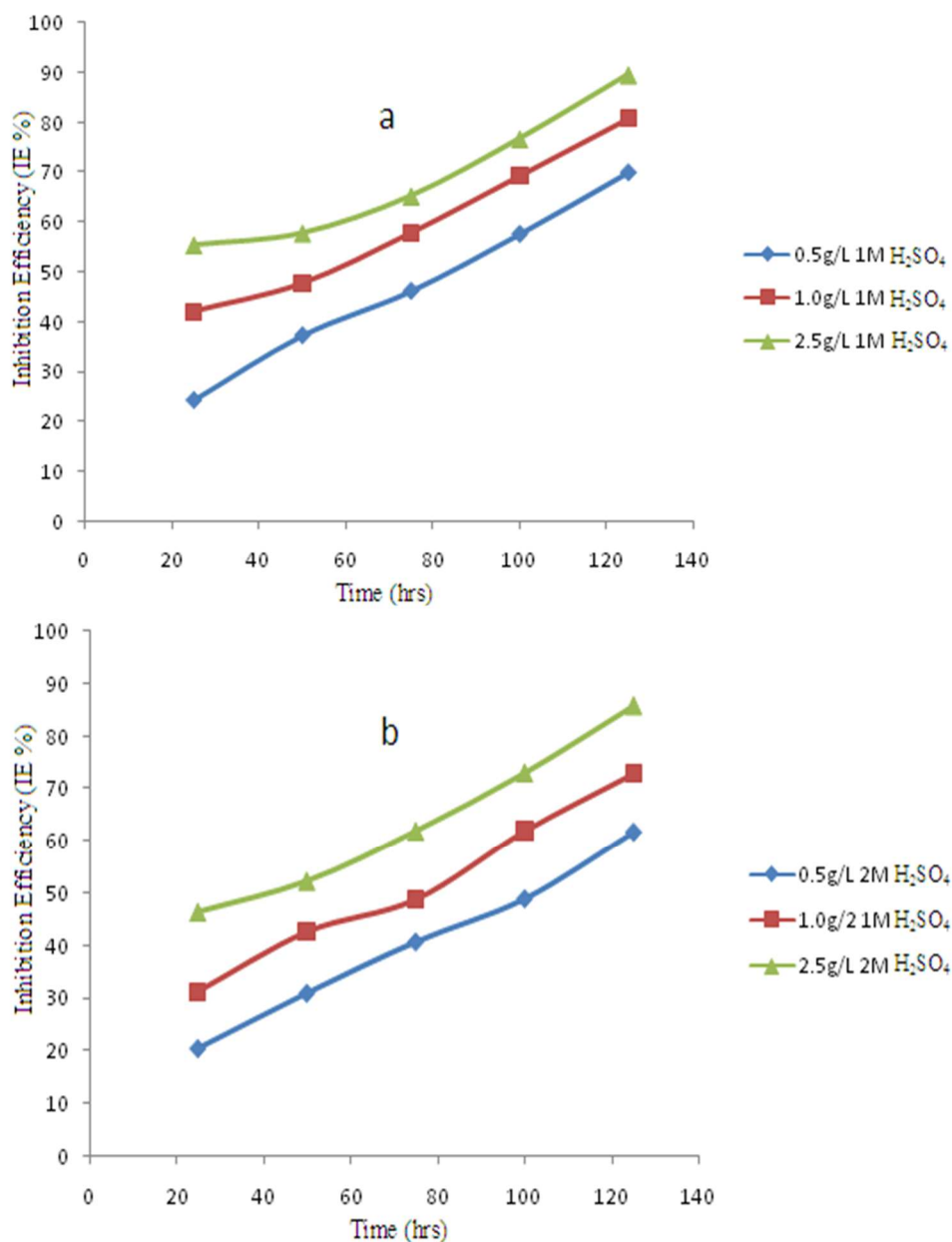


Fig 1.6: Effect of inhibitor concentration on Inhibition Efficiency (I.E %) of OLEin
(a) 1M H₂SO₄ and (b) 2M H₂SO₄.

Comparism between theInhibition Efficiency of 1M, 2M HCl and 1M, 2M H₂SO₄ in inhibitor concentration

Fig 1.7a,b showed the combine plots for comparative analysis in two different concentration of acid 1M (HCl, H₂SO₄) and 2M (HCl, H₂SO₄) in different inhibitor concentrations. It was observed in fig 1.7a, at 125 hours there was an increase in the inhibition efficiency up to 86.57% in 1M HCl and 89.61% in 1M H₂SO₄ with increased 2.5g/L OLE concentrations. It showed that OLE is more effective on the surface of LCS in 1M H₂SO₄ than in 1M HCl.

Similarly, it was observed that the inhibition efficiency of 2M HCl was 82.76% and 2M H₂SO₄ was 85.79%with increased OLE concentration to 2.5g/L at 125 hours. The results showed that OLE is adsorbed more on the surface of LCS in 2M H₂SO₄ than in 2M HCl.

Fig 1.8a,b showed the combined plots for comparative analysis in 1M, 2M HCl and 1M, 2M H₂SO₄. It was observed in fig 4.15a, thatthere was increase in inhibition efficiency up to 86.57% and 82.76% with increased OLE concentration to 2.5g/L in 1M HCl and 2M HCl.

Similarly, in fig 4.15b1M H₂SO₄ and 2M H₂SO₄the inhibition efficiency was 89.61% and 85.79% with increased OLE concentration to 2.5g/L. It was observed that when the concentration of extract increased, it resulted to decrease in corrosion rate and increase in inhibition efficiency with immersion time. This simply means that OLE is adsorbed on the surface of LCS with time thereby protecting the LCS from corroding. This comparative analysis showed that OLE is effective inhibitor in 1M H₂SO₄ than in 1M HCl and in 2M H₂SO₄ than in 2M HCl solutions.

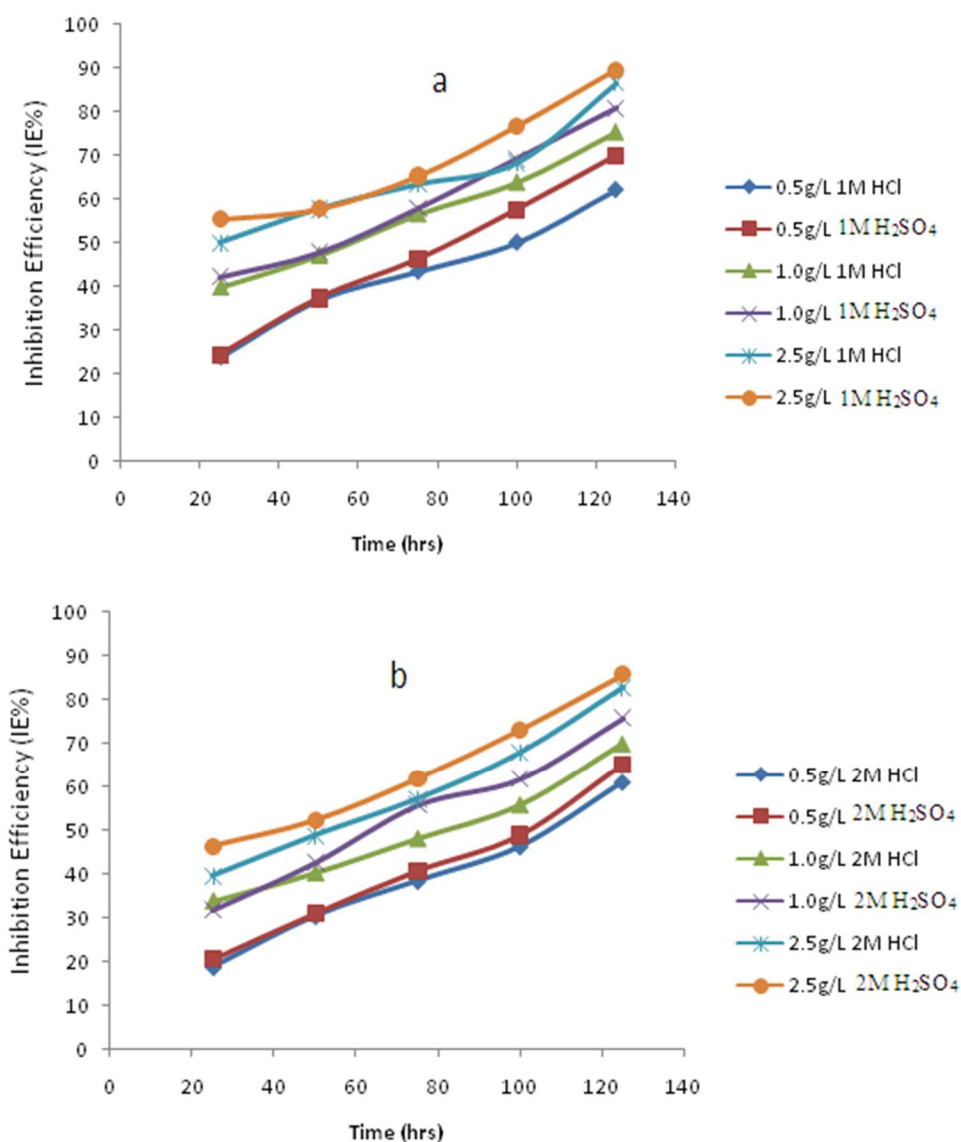


Fig 1.7: Effect of acid type on the inhibition efficiency of OLE on LCS in
(a) 1M HCl, 1M H₂SO₄ and (b) 2M HCl, 2M H₂SO₄ containing different concentrations of OLE.

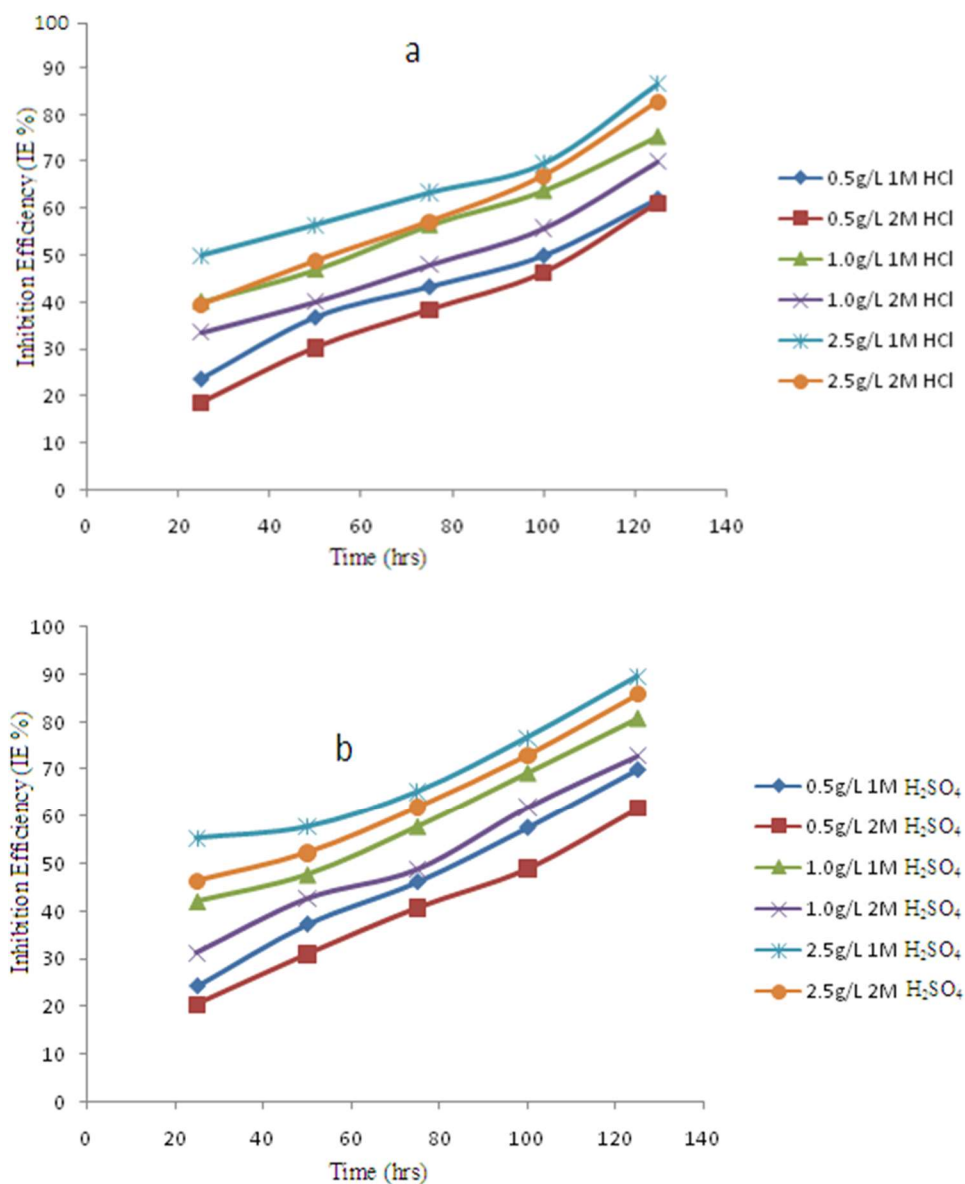


Fig1.8: Variation of inhibition efficiency (IE %) with time for LCS corrosion in (a) 1M, 2M HCl and (b) 1M, 2M H₂SO₄ containing different concentrations of OLE.

Electrochemical Analysis (Tafel extrapolation and OCP) results for inhibitor on LCS corrosion

Fig1.8a,b and fig 1.9a,b shows the Tafel extrapolation plots for corrosion behavior of LCS in various concentrations of HCl, and H₂SO₄ and in the absence and presence of OLE at 27⁰c. An increase in corrosion potential can be attributed to decrease in anodic reaction that resulted to a passive film protection of the mild steel surface and hence a decrease in the corrosion rate. This is synonymous to an increase in cathodic reaction and hence an increase in amount of dissolved oxygen.

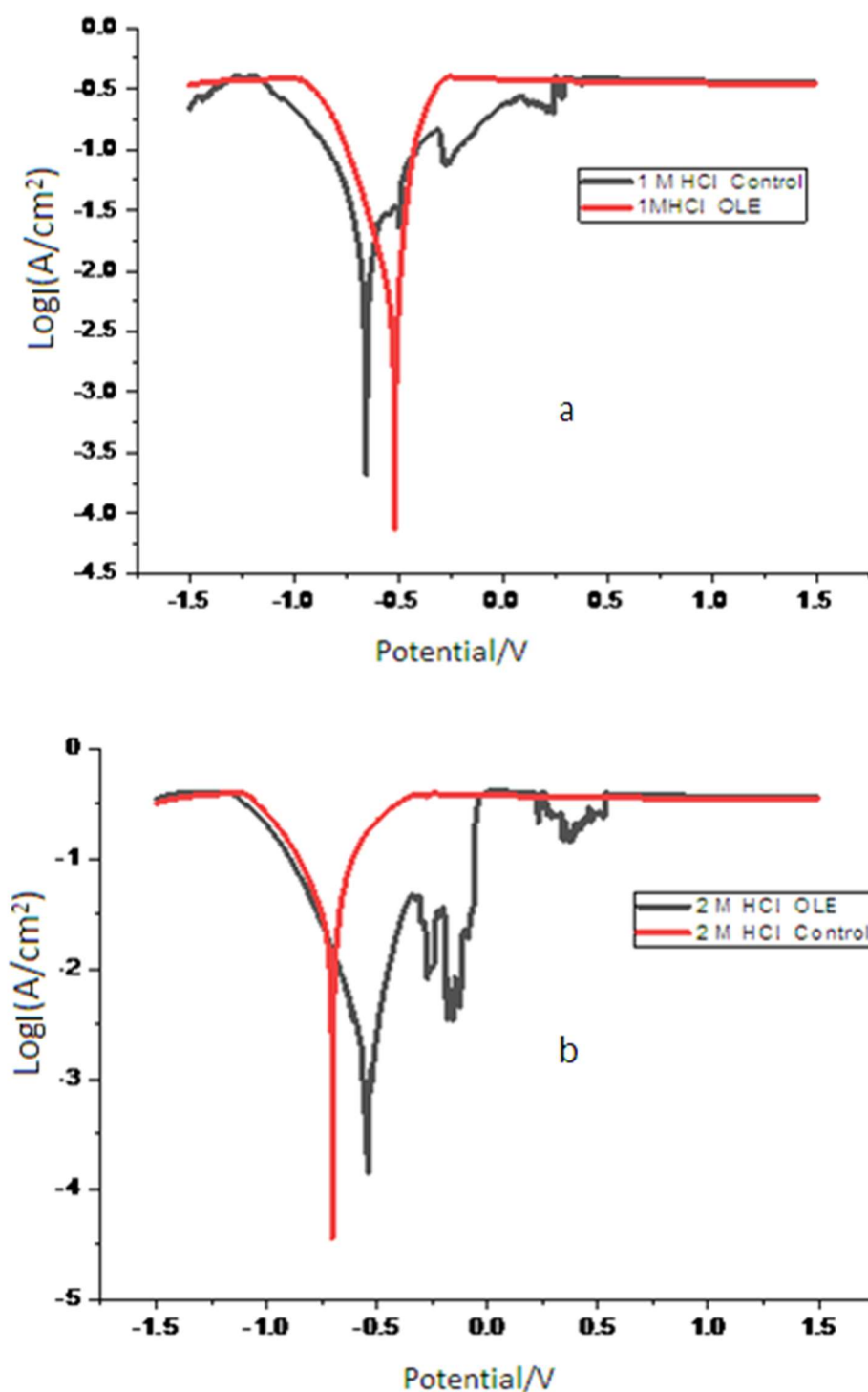


Fig1.9: Tafel extrapolation plots for LCS in (a) 1M HCl and (b) 2M HCl with and without 2.5g/L OLE inhibitor

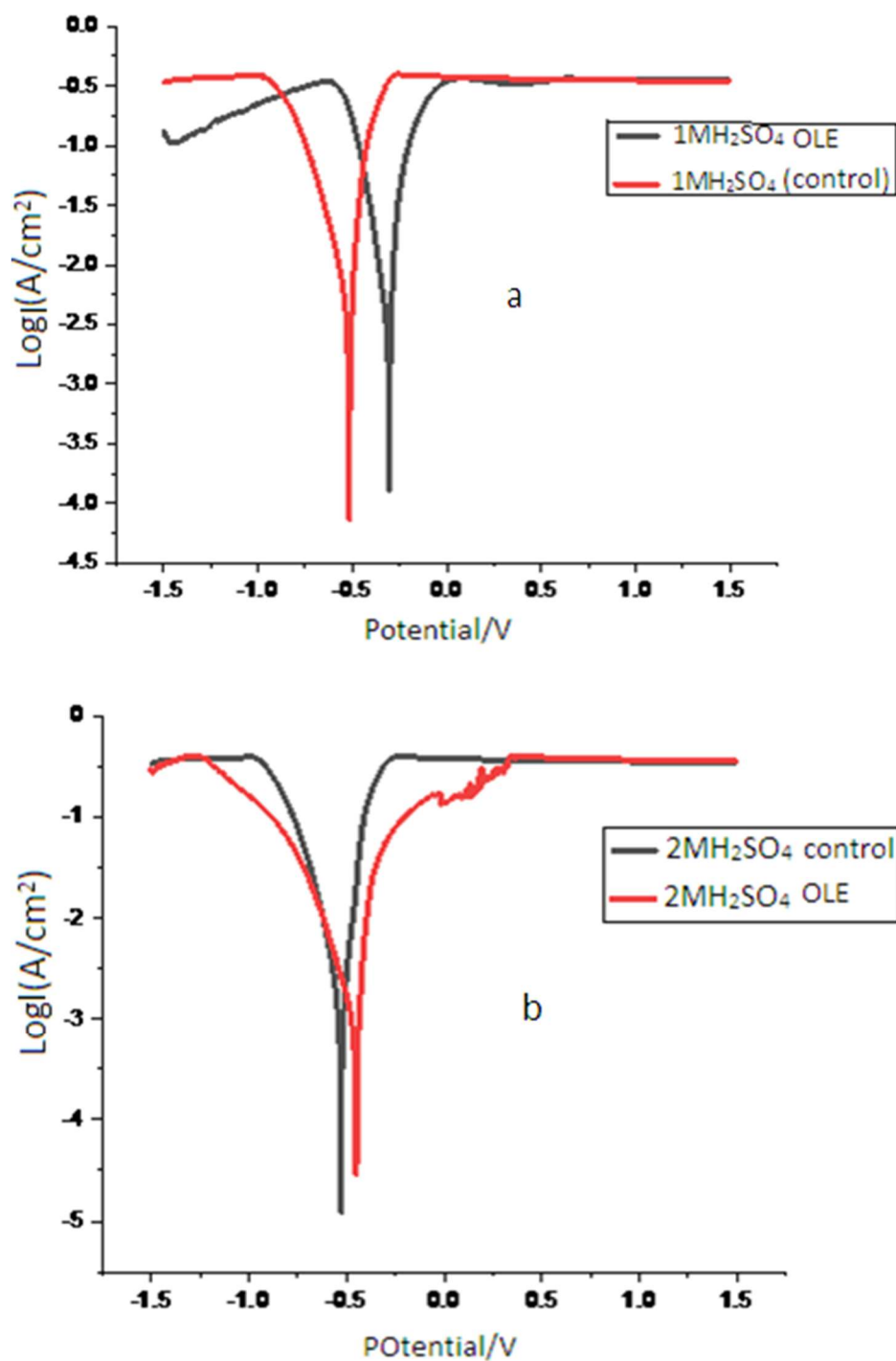


Fig 2.0:Tafel extrapolation plot for LCS in (a) 1M H_2SO_4 and (b) 2M H_2SO_4 with and without 2.5g/L OLE inhibitor

Fig 1.9a,b and fig 2.0a,b show that the Tafel curves for corrosion inhibition of LCS with OLE. It could be observed that the Tafel curve shifted to right along the positive potential (V) direction when compared with the Tafel curve shown for the control/blank (uninhibited system). This demonstrates that there is a noticeable reduction in corrosion current in the presence of OLE inhibitor. It was observed that the corrosion potential (E_{corr}) swings towards the positive direction or in the anodic direction by shifting the Tafel curves of the inhibitor. This swing in E_{corr} towards the positive region indicates that the OLE acts as anodic inhibitor for LCS in H_2SO_4 and HCl medium. It was noticed that the OLE inhibitor reduced corrosion rate to greater extent in $1\text{M H}_2\text{SO}_4$ at -0.25V than in 1M HCl at -0.5V and $2\text{M H}_2\text{SO}_4$ at -0.375V than 2M HCl at -0.5V and solutions. As observed in the fig 4.11a,b and fig4.12a,b, there were increase in potential caused by inhibitive properties of OLE. The Increase in IE maybe attributed to the creation of adsorption film on the surface of the LCS thereby protecting the surface of the LCS which reduces the effective area of attack, hence decrease in corrosion rate. OLE is excellent inhibitor in $1\text{M H}_2\text{SO}_4$ than in 1M HCl , $2\text{M H}_2\text{SO}_4$ and 2M HCl solutions in acid soil this is in agreement with the finding of Shetty et al. (2016).

Open-circuit potential (OCP) result for inhibitor on LCS corrosion

The OCP is a potential between the working electrode and reference electrode while no current is flowing through the cell. It gives information about the initial conditions before you start the experiment. OCP study simply means whether the material is oxidizable or reducible.

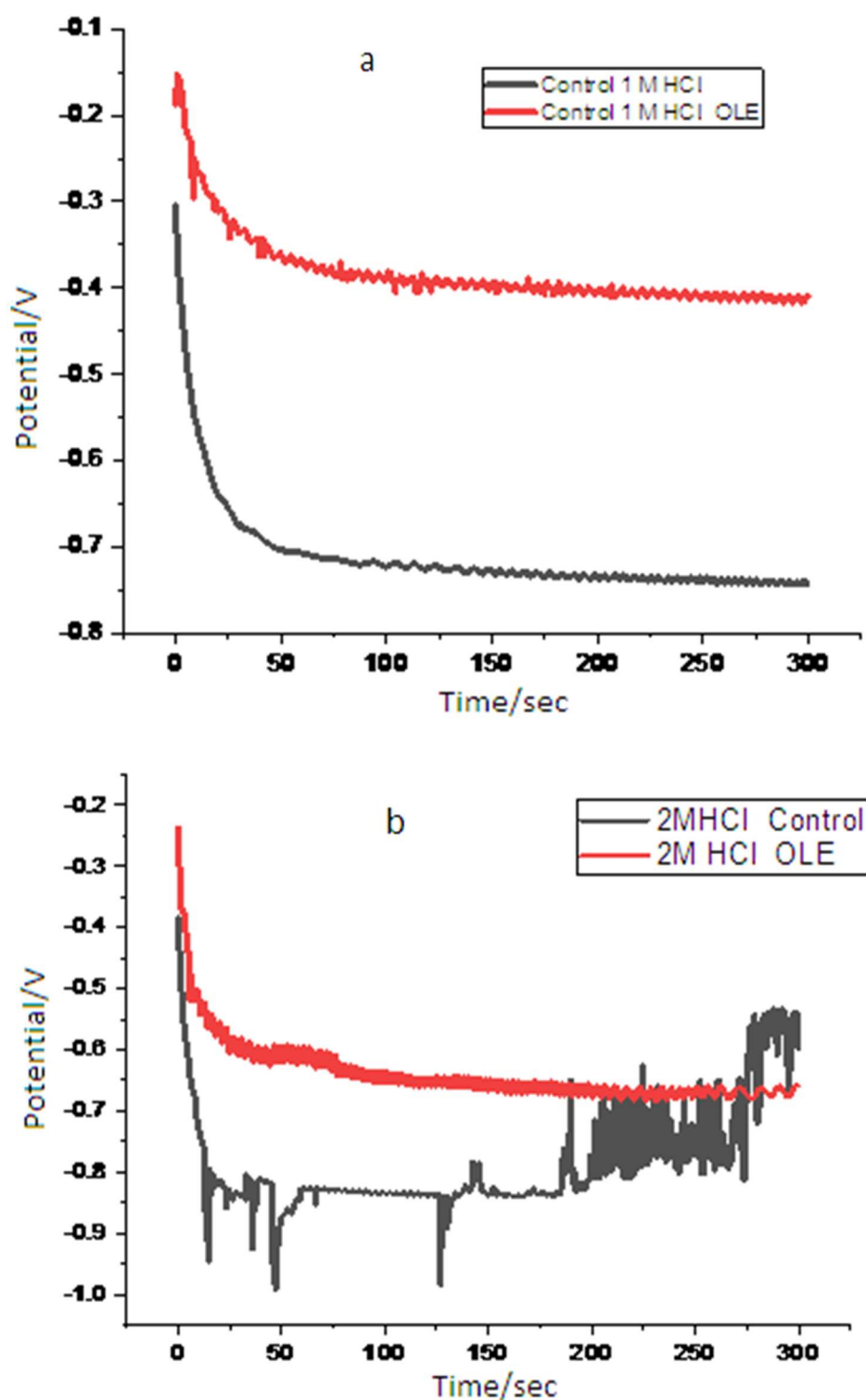
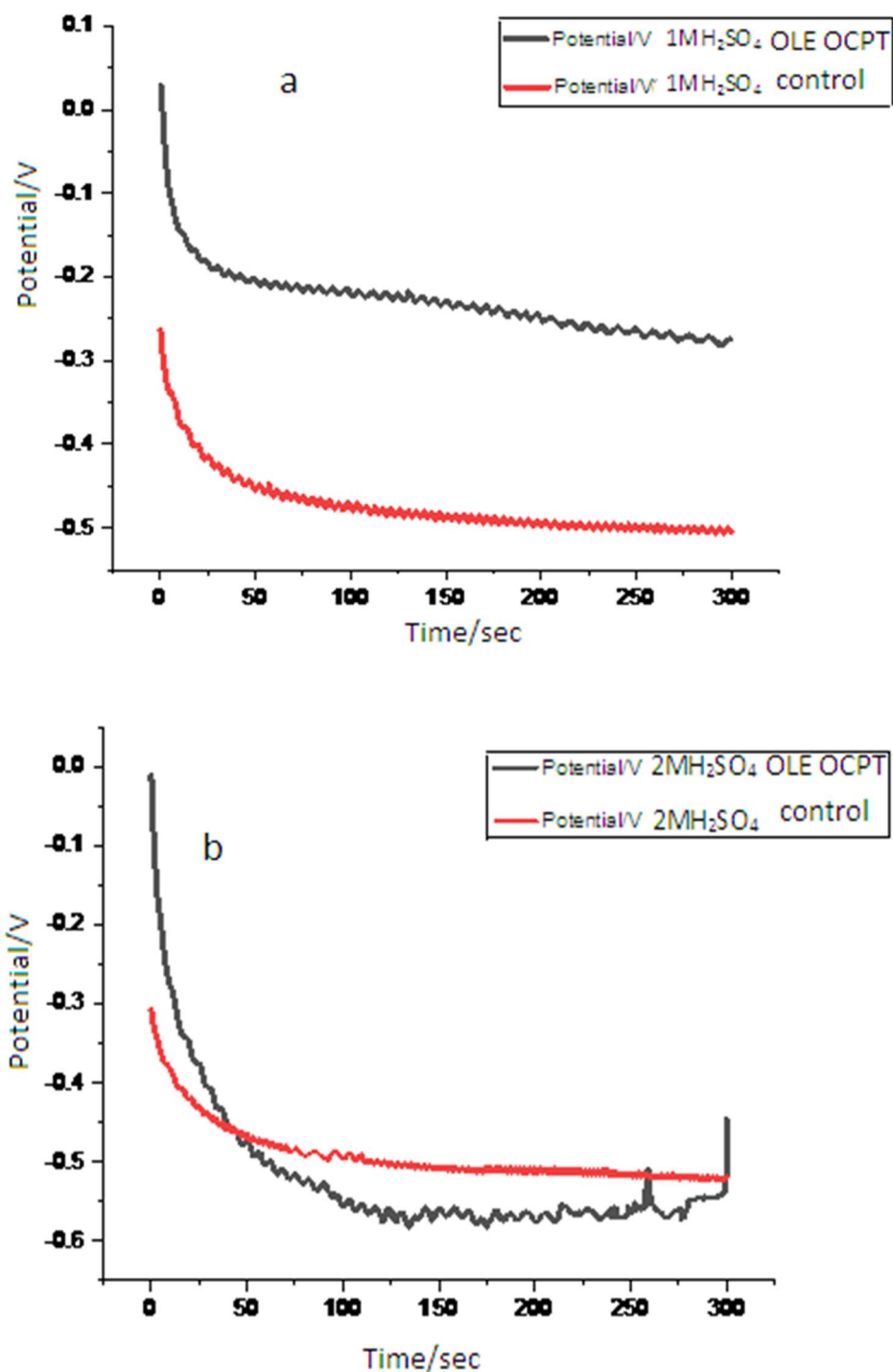


Fig.2.1: OCP analysis for LCS in (a) 1M HCl and (b) 2M HCl with 2.5g/L and Without OLE inhibitor



2.2 OCP analysis for LCS in (a) 1M H_2SO_4 and (b) 2M H_2SO_4 with 2.5g/L and Without OLE inhibitor

Fig 2.1a,b and fig 2.2a,b shows the OCP, OLE corrosion of LCS in HCl and H₂SO₄ without and with OLE inhibitor. It could be observed the E_{ocp} of LCS in solutions without inhibitor, decreases from -0.3 to -0.7v in 1M HCl and from -0.4 to -0.8v in 2MHCl. Similarly it decreased from -0.25 to -0.5v in 1M H₂SO₄ and -0.3 to -0.45v in 2MH₂SO₄ within the time interval of study. After 50s immersion, the open circuit potential (OCP) of LCS stabilized at about -0.7v, -0.8v, and -0.45v and -0.55v for various acid concentrations. This indicates an increase in corrosion rate and hence an increase in dissolve oxygen (Tang j. et al., 2019). The open circuit potential for control and inhibited LCS increased from -0.3 to -0.15v and from -0.4 to -0.25v in 1M HCl and 2M HCl respectively and from -0.25 to 0.5v and from -0.3 to -0.09v in 1M H₂SO₄ and 2M H₂SO₄ respectively. This behavior can be attributed to a decrease in anodic reaction as a result of growth of a passive film on the surface of the LCS.

CONCLUSION

From the results of this study, the following conclusion could be drawn.

1. Soil collected from Ngwo has moisture content of 37.05%, pH 7.2, soil resistivity of 15000 ohm.cm, ORP of 250mV, sulfate content of 170ppm and chloride content of 80ppm which generally would be “mildly corrosive” to the buried LCS used for underground structures like petroleum tanks.
2. The addition of acid to the soil increased the pH values of the soil to -0.10 for 1MHCl, 2.89 for 2MHCl, -0.35 for 1MH₂SO₄ and 1.03 for 2MH₂SO₄, thereby making the soil more corrosive in nature.
3. The OLE contains essential compounds necessary for corrosion inhibition of buried LCS surface in acidic environment according to FTIR.
4. The inhibition Efficiency increased while corrosion rate decreased with increase in extract concentrations for buried LCS used for underground structures like petroleum tanks. OLE is good corrosion inhibitors for buried LCS in acidic soil.
5. The polarization and OCPT study reveals that OLE inhibitor enhanced the corrosion resistance of LCS in H₂SO₄ than in HCl at equal acid concentrations.
6. It was observed that even at a very low concentration of OLE, it could be effectively used as inhibitor for LCS corrosion in both HCl and H₂SO₄.

RECOMMENDATION

Research and observation from this study has shown that OLE has good inhibitive properties. It is therefore recommended that a pilot scale study for industrial use of OLE inhibitor in protecting buried steel pipes should be carried out.

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APPENDIX

Calculated values of corrosion rate and inhibition efficiency of OLE on buried low carbon steel in acid soil.

Time in Hours	Acids	Inhibitor concentration (g/L)	Initial wt M ₁ (g)	Final wt M ₂ (g)	Weight (M ₁ -M ₂)(g)	CR _(mm/yr)	Inhibition Efficiency (IE %)
25	1M HCl	Control/Blank	16.94	16.81	0.13	9.6837	0.00
		0.5	17.00	16.90	0.10	7.4490	23.71
		1.0	16.76	16.68	0.08	5.9592	39.76
		2.5	16.84	16.78	0.06	4.4694	50.00
	2M HCl	Control/Blank	16.91	16.76	0.15	11.1735	0.00
		0.5	16.87	16.75	0.12	8.9388	18.75
		1.0	16.89	16.80	0.10	7.4490	33.69
		2.5	16.89	16.80	0.09	6.7041	39.55
	1M H ₂ SO ₄	Control/Blank	17.01	16.85	0.16	11.9184	0.00
		0.5	16.90	16.77	0.13	9.6837	24.27
		1.0	17.04	16.94	0.10	7.4490	42.18
		2.5	17.00	16.92	0.08	5.9592	55.40
	2M H ₂ SO ₄	Control/Blank	16.99	16.81	0.18	13.4082	0.00
		0.5	17.01	16.86	0.14	10.4286	20.56
		1.0	16.91	16.78	0.12	8.9388	31.69
		2.5	17.02	16.92	0.10	7.4490	46.39
	1M HCl	Control/Blank	16.90	16.73	0.17	6.3316	0.00
		0.5	17.03	16.92	0.11	4.0900	36.84
		1.0	16.80	16.71	0.09	3.3520	47.06
		2.5	16.93	16.85	0.08	2.9796	57.78
		Control/Blank	17.04	16.85	0.19	7.0765	0.00

50	2M HCl	0.5	16.89	16.77	0.13	4.8418	30.43
		1.0	16.92	16.81	0.11	4.0970	40.23
		2.5	16.95	16.85	0.10	3.7245	48.83
	1M H ₂ SO ₄	Control/Blank	16.96	16.78	0.18	7.7041	0.00
		0.5	17.00	16.89	0.11	5.0969	37.27
		1.0	17.04	16.94	0.10	3.7245	47.78
		2.5	16.92	16.84	0.08	2.9796	57.78
	2M H ₂ SO ₄	Control/Blank	16.85	16.62	0.23	8.5663	0.00
		0.5	17.05	16.89	0.16	5.9592	31.03
		1.0	16.85	16.71	0.14	5.2143	42.58
		2.5	16.87	16.76	0.11	4.0969	52.29
75	1M HCl	Control/Blank	16.98	16.79	0.19	4.7177	0.00
		0.5	16.85	16.74	0.11	2.7313	43.36
		1.0	17.00	16.92	0.08	1.9864	56.52
		2.5	17.04	16.97	0.07	1.7381	63.38
	2M HCl	Control/Blank	17.02	16.79	0.23	5.7109	0.00
		0.5	16.90	16.77	0.14	3.4762	38.46
		1.0	17.05	16.93	0.12	2.9796	48.08
		2.5	17.06	16.96	0.10	2.4830	57.22
	1M H ₂ SO ₄	Control/Blank	16.96	16.75	0.21	5.2143	0.00
		0.5	16.90	16.90	0.10	2.7830	46.19
		1.0	16.90	16.81	0.09	2.2347	57.89
		2.5	16.86	16.79	0.07	1.7381	65.23
	2M H ₂ SO ₄	Control/Blank	16.96	16.69	0.27	6.7041	0.00
		0.5	17.03	16.87	0.16	3.9728	40.74
		1.0	17.00	16.86	0.14	3.4762	48.76
		2.5	16.88	16.76	0.11	2.7313	61.90
	1M HCl	Control/Blank	16.94	16.72	0.22	4.0969	0.00
		0.5	16.80	16.70	0.10	1.8622	50.00
		1.0	16.98	16.90	0.08	1.4898	63.83
		2.5	16.97	16.07	0.07	1.3036	68.29

100	2M HCl	Control/Blank	16.98	16.73	0.25	4.6556	0.00
		0.5	16.94	16.81	0.13	2.4209	46.34
		1.0	16.90	16.80	0.10	1.8622	55.84
		2.5	16.93	16.86	0.07	1.3036	67.86
	1M H ₂ SO ₄	Control/Blank	17.04	16.82	0.22	4.0940	0.00
		0.5	16.98	16.90	0.08	1.8898	57.64
		1.0	17.00	16.93	0.07	1.5036	69.28
		2.5	16.95	16.89	0.06	0.9173	76.73
	2M H ₂ SO ₄	Control/Blank	16.87	16.59	0.28	5.2143	0.00
		0.5	16.94	15.80	0.14	2.6071	48.91
		1.0	16.97	16.86	0.11	2.0485	61.83
		2.5	16.87	16.78	0.09	1.6760	73.16
125	1M HCl	Control/Blank	17.01	16.76	0.25	3.7436	0.00
		0.5	17.00	16.91	0.09	1.3177	62.07
		1.0	16.89	16.83	0.06	0.8985	75.35
		2.5	16.87	16.83	0.04	0.4990	86.57
	2MHCl	Control/Blank	16.91	16.62	0.29	4.3426	0.00
		0.5	17.00	16.89	0.11	1.6472	61.15
		1.0	16.90	16.81	0.09	1.3177	70.00
		2.5	17.04	16.99	0.05	0.7487	82.76
	1M H ₂ SO ₄	Control/Blank	16.94	16.68	0.26	3.8933	0.00
		0.5	17.01	16.95	0.06	1.5985	70.00
		1.0	16.90	16.82	0.08	1.2487	80.77
		2.5	16.91	16.87	0.04	0.5990	89.61
	2M H ₂ SO ₄	Control/Blank	16.90	16.57	0.33	4.8415	0.00
		0.5	16.80	16.68	0.12	1.8969	61.15
		1.0	16.93	16.84	0.09	1.3177	72.78
		2.5	17.00	16.96	0.04	0.5990	85.79