
CORROSION PERFORMANCE OF 1014 MILD AND 304 STAINLESS STEELS IN ACIDIC MEDIA

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ABSTRACT

Corrosion is a degradation of metallic materials under the action of the environment which requires oxygen and moisture to occur. This research work determined the corrosion performance of 1014 low carbon and 304 austenitic stainless steels in different concentration of acidic media. Corrosion tests were carried out using gravimetric technique. One hundred and eighty samples of the metals were prepared and immersed in containers of sulphuric acid (H₂SO₄), hydrochloric acid (HCl) and nitric acid (HNO₃) at 1, 2 & 3 M. The samples were then removed every three days for a period of 15 days to measure the weight loss. These were used to calculate the corrosion rates. The chemical analysis was determined using an Energy Dispersive X-ray (EDX). Scanning Electron Microscope (SEM) was used to determine the texture of the samples. The results showed that the corroded samples had pitting corrosion damage and cracks propagated generally on the sample surfaces. The corrosion rates of the samples increased with increase in molarities of the reagents, Stainless steel samples had the least corroded surfaces. The study concluded that the higher the level of concentration of acidic media (1 to 3 M), the higher the corrosion rates of samples in increasing order of HNO₃, HCl and H₂SO₄ especially for mild steel sample (4.35 to 17.90, 0.21 to 2.90 and 10.37 to 0.64 mm/y) after 360 hours of immersion respectively.

Keywords: Corrosion, Ferrous metals, SEM, EDS, Gravimetry, Acidic media

INTRODUCTION

Corrosion is the deterioration of materials by chemical interaction with their environment. The consequences of corrosion are many and varied and their effects on the safe, reliable and efficient operation of equipment or structures are often more serious than simple loss of a mass of a metal. Adetunji et al., 2011 studied the electro-

chemical property of mild steel in cassava fluid. They reported that after 312 hours of immersion, mild steel had corrosion rate of 1.7 mm/y. The consequence of corrosion damage reported was reduction of metal thickness leading to loss of mechanical strength and structural failure or breakdown (Umoren, 2009).

Onen reported in 2000 that corrosion may also be considered as a redox process in which an oxide coating is formed on the surface of the metal/alloy undergoing the process. It requires oxygen and moisture to occur. It involves the transfer of electrons along the surface of the metal under the influence of a potential difference.

Corrosion is a matter of great concern due to the enormous cost involved in the replacement of metallic parts in all kinds of applications (Loto, 2012). The reaction in this corrosive attack requires a medium, usually water, which is capable of conducting a tiny current of electricity. When a metal comes in contact with a corrosive agent and it is connected by a liquid or gaseous path through which electrons may flow, corrosion begins as the metal decays by oxidation. During the attack, the quantity of corrosive agent is reduced and, if not renewed or removed, it may completely react with the metal, becoming neutralized (Katundi et al., 2010).

It was reported that the enormous usage of thin steel sheets for automotive application for reducing car weight necessitates improved material strength in addition to higher corrosion protection. Sanusi and Hussein (2001) investigated on a mild steel alloy with a thickness of 0.1 cm, cut into specimen of sizes of 4 cm by length and 2.5 cm by width and perforated at the centre. The corrosive effect of acidic media on mild steel samples was determined by Osarolube et al. (2008). It was found that mild steel had slightly high corrosion in the acidic media.

Adetunji, et al. (2016) and Loto (2012) studied the electrochemical effect of orange juice on metallic can. It was found that the

relative acidity of the solution is the most important factor to be considered; at low pH, the evolution of hydrogen tends to eliminate the possibility of protective film formation so that steel continues to corrode but in alkaline solutions, the formation of protective film greatly reduces corrosion rate. Badmos and Ajimotokan (2009) also reported their research findings on effect of citrus species on mild steel. Sanusi and Hussein (2001) also worked on the effect of natural orange juice on mild steel. It was found that the weak acid in the juice affected the corrosion rate after exposure. Adetunji et al., 2012 reported research finding on corrosion of austenitic steel in seawater, cassava fluid and maize pulp, low corrosion rate was got in the media. Mild and stainless steel found applications in processing chemical plants like hydrochloric, nitric and sulphuric acid plants. Huge amount of money are spent to repair and replace corroded parts. This research work therefore, determined the corrosion performance of mild and stainless steels in different concentrations of acidic media using gravimetry and SEM. Specifically, corrosion rates and extent of damages were evaluated in HCl, HNO₃ and H₂SO₄ at 1, 2 and 3 molarities.

MATERIALS AND METHODS

Samples Preparation

The two metals used for this research work were stainless steel and mild steel. The samples for weight loss tests were prepared in accordance to the procedure recommended by ASTM G.1, 2003. The samples were cut into rectangular sizes of 20 mm by 20 mm and a thickness of 1.0 mm. These samples were cut from the parent materials with the

help of shears, while a steel rule and scriber were used for the dimensioning. Ninety samples were prepared from each of the metallic samples (stainless and mild steels) making a total of 180 samples.

Reagents

The pure and analytical three reagents used for this study were purchased from a local supplier. These are sulphuric acid (98% volume of H₂SO₄), hydrochloric acid (75% volume of HCl) and Trioxonitrate (V) acid (75% volume of HNO₃).

Materials Characterization

The characterization employed were chemical analysis by EDX, surface morphology by SEM and gravimetry by Adventurer TM balance.

Chemical Analysis

The chemical analyses to determine the composition of the samples were done using Energy Dispersive X-ray (EDX) machine model JEOL 840. The examination was done by cutting samples into square of 15 mm. They were mechanically ground and polished. The mirror-like polished samples were carbon coated before analysis in order to make samples surfaces conclusive.

Scanning Electron Microscopy

A Scanning Electron Microscopy machine was employed to conduct cross sectional analyses of the samples surfaces. The microstructure and morphology of the samples were taken before and after specified period of immersion at a research centre.

Corrosion Studies

Experimental Procedures

All the prepared samples were first rinsed in distilled water to remove all the foreign materials and were dry-cleaned with acetone

solution in order to remove any traces of water on their surfaces. Each of the samples was weighed by electric weighing balance of Adventurer TM (power equipment 50/60Hz, 6VA, model number of AR3130, maximum weight of 310 g and readability of 0.001 g).

Immersion of Samples

Each of the prepared samples was immersed in different containers. They were fully immersed at room temperature after the initial weight has been recorded. Each sample were immersed in acidic media of 1 M, 2 M and 3 M concentrations, and then removed after 3,6,9,12 and 15 days. The samples were properly cleaned and dried. The weight loss and pH of media were determined. Each test was carried out in duplicate and the mean weight losses were reported.

Theory/Calculation

The mathematical formula by Fontana (1987), for calculating the corrosion rates of the samples after the weight loss measurement is stated as follow:

$$CR = 87.6w / \rho AT \text{ (mm/y)}$$

Where, CR = corrosion rate in millimeter per year, W = weight loss in mg, this was done by subtracting the final weight measured from initial weight which gave the weight loss (weight difference), ρ = density of each sample in mg/m³, A = Area in cm² and T = Time in hours, of each of the samples spent inside the different concentrations of the acidic media.

The redox electrochemical reaction for the metals immersed in acidic media is represented below;



RESULTS

**Characterization of Sample Materials
Chemical Analysis**

The percentage chemical compositions of samples used for this research work; stainless and, mild steel were shown from Tables

1 to 2 respectively. In Table 1, the highest percent was Chromium of 20 % after Iron which determined the ability of stainless steel to resist corrosion. In Table 2, the mild steel is a ferrous metal with largest percentage value of iron (98.35%),

Table 1: Percentage Chemical compositions of 304 Stainless steel (SS)

Elements	C	Mn	P	S	Si	Cr	Ni	Al	Fe
Composition	0.08	2.00	0.05	0.03	0.75	20.26	8.00	0.10	68.73

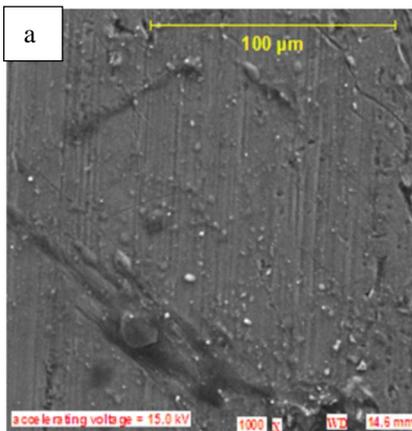
Table 2: Percentage Chemical compositions of 1014 Mild steel (MS)

Elements	C	Mn	P	S	Si	Cr	N	Cu	Fe
Composition	0.14	0.48	0.02	0.01	0.18	0.79	0.01	0.03	98.34

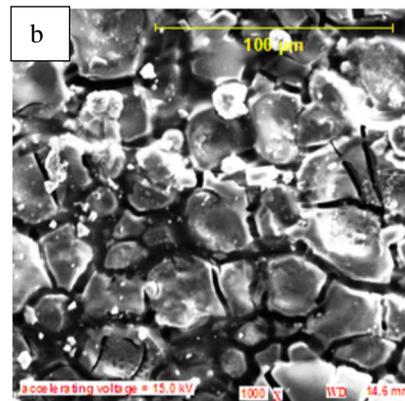
Scanning Electron Microscopy

The scanning electron microscopy (SEM) images of samples surfaces before the time of immersion in different concentration of acidic media are shown in Plates 1 and 2. Plate 1 shows SEM image of stainless be-

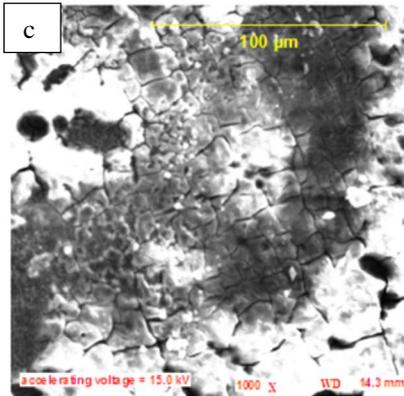
fore (a) and after immersion (b) of stainless steel surface. SEM image of stainless steel after immersion for a period of 10 days shows large grains throughout the surfaces as a result of corrosion attack.



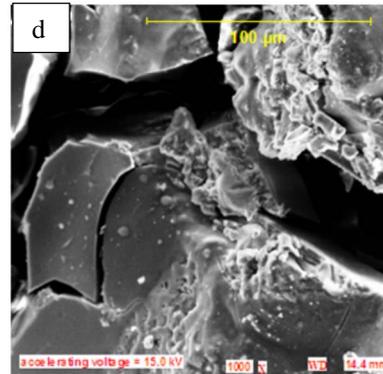
(a) SEM of SS Sample before



(b) SEM of SS Sample after immersion in HNO₃

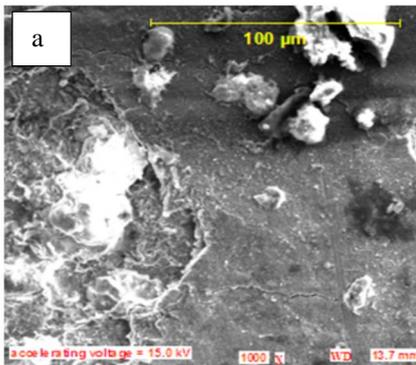


(c) SEM of SS Sample before in HCl

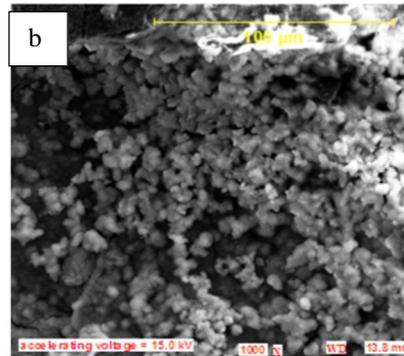


(d) SEM of SS Sample after immersion in HCl

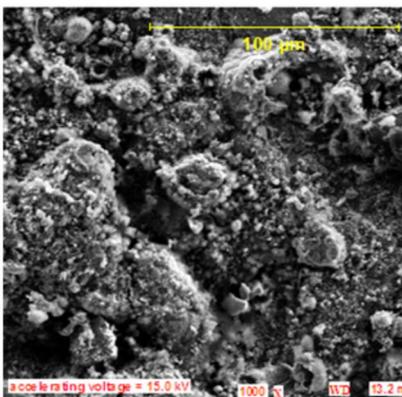
Plate 1: SEM of Stainless Steel (SS) samples before and after immersion in various acidic media in 1M.



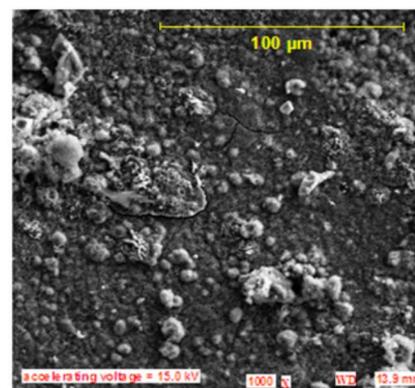
(a) SEM of MS Sample before Immersion



(b) SEM of MS Sample after immersion in HNO₃



(c) SEM of MS Sample after immersion in H₂SO₄



(d) SEM of MS Sample after immersion in H₂SO₄

Plate 2: SEM of Mild Steel (MS) samples before and after immersion in various acidic media in 1M.

Rate of Corrosion of Metal Samples

The results obtained from the investigation of corrosion rates of mild and stainless steel

are contained in Tables 3 to 5 and the trends are illustrated in Figures 1 to 3.

Table 3: Corrosion rate of metal samples (mm/y) in 1.0 Molar of different Solutions

Samples	72HRS	144HRS	216HRS	288HRS	360HRS
SS(HCl)	1.008±0.02	0.185±0.38	0.152±0.07	0.221±0.00	0.209±0.55
MS(HCl)	94.640±5.11	42.012±2.00	28.121±1.33	20.720±1.79	15.451±1.83
SS(H ₂ SO ₄)	0.570±0.01	0.119±0.06	0.149±0.00	0.169±0.00	0.146±0.71
MS(H ₂ SO ₄)	39.813±1.22	23.786±1.33	14.808±1.12	11.963±1.00	10.365±1.90
SS(HNO ₃)	0.314±0.01	0.138±0.07	0.130±0.10	0.121±0.33	0.057±0.01
MS(HNO ₃)	27.220±1.25	13.407±1.11	8.470±0.91	6.681±0.88	4.346±0.54

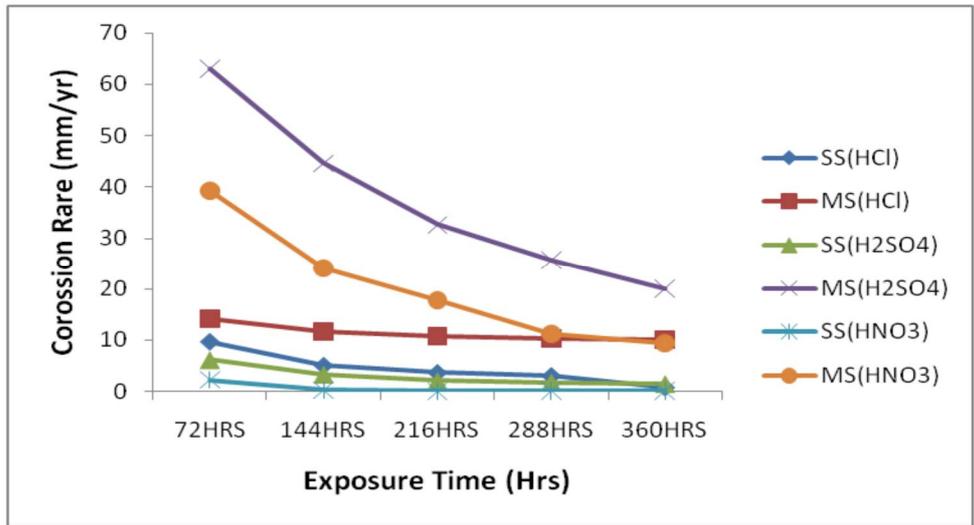


Figure 1: Corrosion rate in 2.0 Molar of different Solutions

Table 4: Corrosion rate of metals in 3.0 Molar Solutions of three media

Samples	72HRS	144HRS	216HRS	288HRS	360HRS
SS(HCl)	10.275±1.44	4.705±0.00	3.454±0.11	3.006±0.44	2.895±0.11
MS(HCl)	44.773±2.06	18.395±1.09	16.523±0.56	25.469±1.00	20.509±1.57
SS(H ₂ SO ₄)	9.325±0.96	6.692±0.00	3.311±0.22	2.690±0.11	2.028±0.33
MS(H ₂ SO ₄)	79.984±2.55	49.321±2.01	33.952±0.81	26.489±0.65	20.939±0.12
SS(HNO ₃)	0.314±0.00	0.152±0.22	0.101±0.99	0.078±0.00	0.061±0.00
MS(HNO ₃)	62.538±3.11	33.274±0.42	26.190±1.00	16.412±0.21	17.895±0.88

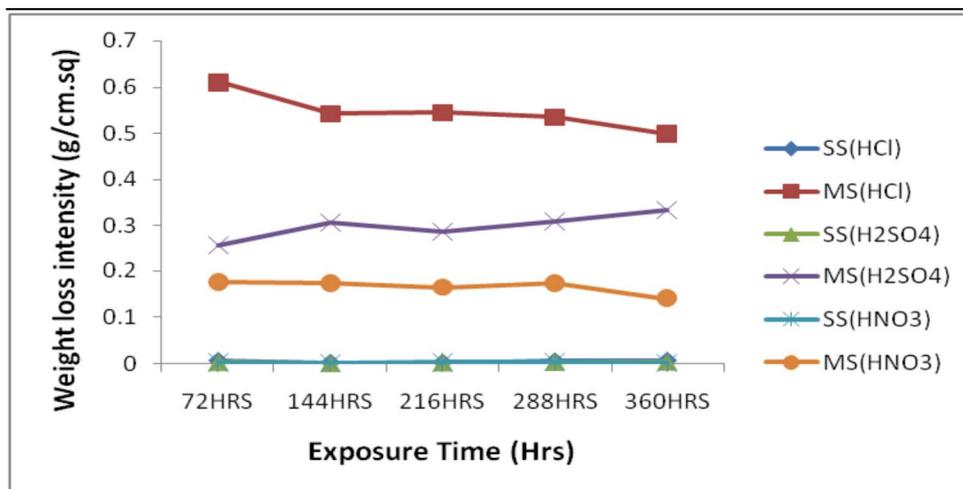


Figure 2: Weight loss Intensity of metals (g/cm.sq) in 1.0 Molar Solutions of three media

Table 5: Weight loss Intensity of metals (g/cm.sq) in 2.0 Molar Solutions of three media

Samples	72HRS	144HRS	216HRS	288HRS	360HRS
SS(HCl)	0.0631±0.01	0.0651±0.00	0.0701±0.01	0.0774±0.00	0.0229±0.11
MS(HCl)	0.0912±0.00	0.1503±0.11	0.2110±0.28	0.2667±0.30	0.3271±0.55
SS(H ₂ SO ₄)	0.0405±1.01	0.0436±0.10	0.0393±0.03	0.0461±0.00	0.0469±0.00
MS(H ₂ SO ₄)	0.4073±0.00	0.5762±0.39	0.6322±1.05	0.6621±1.31	0.6452±1.05
SS(HNO ₃)	0.0144±0.03	0.0019±0.00	0.0005±0.00	0.0018±0.00	0.0020±0.00
MS(HNO ₃)	0.2537±0.15	0.3121±0.20	0.3446±0.10	0.2899±0.00	0.3045±0.51

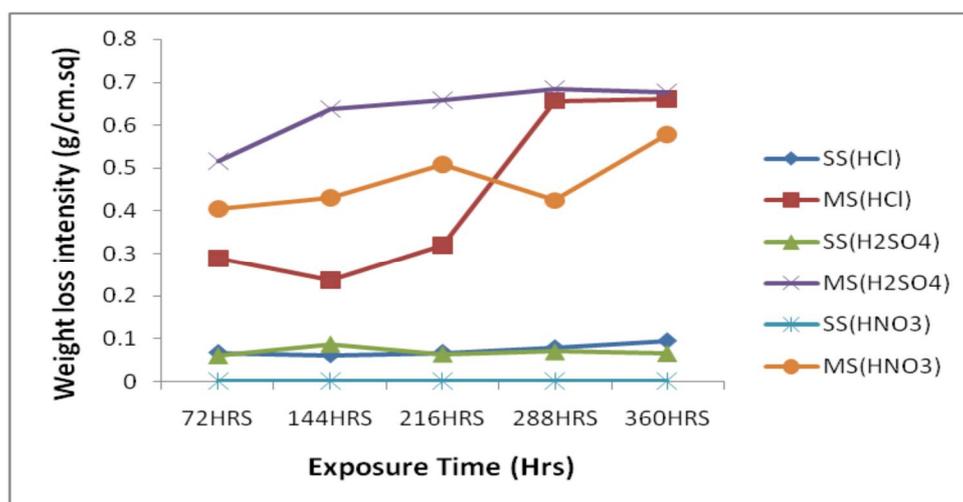


Figure 3: Weight loss Intensity of metals (g/cm.sq) in 3.0 Molar Solutions of three media

Table 3 displayed the corrosion rate of two ferrous metal samples against the time of exposure of samples in 1.0 molar of three acidic media. The corrosion rates of stainless steel and mild steel samples in the three different acidic media generally decreased steadily with an increase in exposure times from 72 to 360 hours. The corrosion rates of samples in acidic media decreased in order of solutions of Hydrochloric acid (HCl), Sulphuric acid (H_2SO_4) and Nitric acid (HNO_3). The corrosion rates of the two samples (stainless steel and mild steel in the same acidic medium of HCl at first exposure time of 72 hours were 1.008 ± 0.02 and 94.640 ± 5.11 mm/y, in acidic media of H_2SO_4 were 0.570 ± 0.01 and 39.813 ± 1.22 mm/y and in solution of HNO_3 were 0.314 ± 0.01 and 27.220 ± 1.25 mm/y respectively.

Table 4 exhibited the corrosion rate of two ferrous metal samples against the time of exposure of samples in 3 molar of three acidic media. The corrosion rates of the samples in three solutions decreased steadily as the exposure time increased from 72 to 360 hours. The corrosion rates of stainless steel in solution of HCl decreased from 10.275 ± 1.44 to 2.895 ± 0.11 mm/y while mild steel samples in the same acidic medium decreased also from 44.773 ± 2.06 to 20.509 ± 1.57 mm/y as the exposure time increased from 72 to 360 hours.

Figure 3 showed the graph of weight loss intensity of two ferrous metal samples in 3.0 molar of three different acidic solutions. The highest weight loss intensity was no-

ticed on mild steel samples in solutions of H_2SO_4 . The weight loss intensity of mild steel sample in the solution of HCl decreased from exposure time of 72 hours to 144 hours and ascended rapidly at this time to compete with highest value of mild steel in solutions of H_2SO_4 at final exposure time of 360 hours. The least weight loss intensity of stainless steel samples in three different acidic media was observed in HNO_3 while the stainless steel samples in solutions of H_2SO_4 and HCl competed with each other as the exposure times increased from 72 to 360 hours.

DISCUSSION

SEM picture of stainless steel in H_2SO_4 after immersion for 10 days displays boundary pitting with 2/3 as a result of corrosion effect while in HCl had a deep-wide cracks as well as little white watery pattern in right hand top corner after 10 days of immersion as shown in Plate 1. This confirmed the susceptibility of stainless steel to pittings in chloride environment. The mild steel sample before immersion exhibits some parts with spot white patches in the natural state. SEM image after immersion in HNO_3 shows conspicuous crystalline grains of $\frac{3}{4}$ of surface erosion. The corrosion effect of H_2SO_4 was so glaring with some big gray patches and blocks crystals of about $\frac{4}{5}$ of sample surfaces while mild steel with HCl had little white patches for about $\frac{1}{2}$ of the sample surfaces as shown in Plate 2.

The corrosion rates of mild steel samples were much higher than that of stainless steel samples. This is in agreement with earlier researchers (Sanusi et al., 2001; Umoren, 2009; Onen, 2000; Loto, 2012). Figure 1 shows the graph of corrosion rates of the

two ferrous metal samples versus the exposure time in three different 2.0 molar solutions. The corrosion rates of all metal samples in three different acidic media declined with an increase in exposure times from 72 to 360 hours. The highest impact of corrosion rate was noticed on mild steel in solution of H_2SO_4 while least impact was observed on stainless steel in solution of HNO_3 . The corrosion rate of mild steel in solution HCl maintained nearly the same values throughout the exposure time. In Figure 2, it shows the weight loss intensity of two ferrous metals samples in 1.0 molar of three different acidic media. The weight loss intensity of all the mild steel samples in different acidic media showed a wide distinct gap with those of stainless steel samples in the same acidic media. The highest weight loss intensity was observed on mild steel samples in solution of HCl and this was followed by the same with solution of H_2SO_4 after 360 hours of immersion.

Table 5 shows the weight loss intensity of two ferrous metals samples in 2.0 molar of different acidic solutions. Generally, there was an increase in weight loss intensity of all the metal samples in different acidic media as period of exposure times increased from 72 to 360 hours. There were significant effects of weight loss intensity of the two metal samples in the solutions of H_2SO_4 ; the weight loss intensity of mild steel samples in this said solution had highest value of 0.4073 ± 0.00 g/cm.sq while that of stainless steel had value of 0.0405 ± 1.01 g/cm.sq at 72 hours of exposure time. It also showed that the weight loss intensity of the metal samples with solutions of HNO_3 had the least effect among three acidic media. At the last exposure time 360 hours, the

highest values of weight loss intensity were noticed on mild steel samples in solutions of H_2SO_4 and HCl as 0.6452 ± 1.05 g/cm.sq and 0.3271 ± 0.55 g/cm.sq respectively while the least effect of samples was noticed in solution of HNO_3 . The highest corrosion rate was observed on mild steel sample in acidic solution of H_2SO_4 (79.984 ± 2.55) at first exposure time and the least rate was noticed on stainless steel sample in solution of HNO_3 (0.061 ± 0.00) at exposure time of 360 hours. It was shown that the highest impact of corrosion rate of stainless steel was in solution of HCl (10.275 ± 1.44) at 72 hours while that of mild steel sample was in solution of H_2SO_4 (79.984 ± 2.55) (Osarolube et al., 2008).

CONCLUSIONS

It can be concluded that the corroded samples had pitting corrosion damage and cracks propagated generally on the sample surfaces. It was observed averagely that the corrosion rates of the samples increased with increase in molarities of the reagents, Stainless steel samples had the least corrosion rate and this was due to its high resistance to corrosion. The percentage composition of chemical analysis of stainless steel showed that it has the highest percentage composition of chromium (20.0%). The research can then be finally concluded that the higher the level of concentration of acidic media, and corrosion rates of samples in acidic media decreased in order of H_2SO_4 , HCl and HNO_3 after fifteen days of immersion for mild steel samples for 2 and 3 M solutions. Austenitic stainless steel is a better choice of materials in acidic media than mild steel.

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