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Comparative Study and Analysis of Carbon Steel Corrosion in co_2 Saturated Environment.

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ABSTRACT

Carbon steel is arguably one of the most efficient, reliable and safer kind of steel used in petroleum and gas industry for production, distribution and transmission of products. Acetic acid (HAc), is also one of the impurities in oil and gas during transportation from the well sites to the refineries. It is formed in the formation water, which also present in oil and gas production and transportation processes. Acetic acid aids corrosion in pipelines and as a result causes environmental degradation. It has been observed that high concentration of HAc increases the rate of corrosion of carbon steel in CO_2 environment. Corrosion slows down production of oil and gas and thereby reduces revenue.

In this work, a comparative study and analysis of carbon steel corrosion in the presence of HAc was carried out at 25°C and 80°C in CO_2 saturated environment. Weight loss and surface analysis methods (XRD, EDX and SEM) were used to characterize the corrosion layers of the carbon steel samples at different conditions. The weight loss results show that the corrosion rate increased initially with the increase in the concentration of HAc and attained a maximum, and then gradually decreased. At 25°C with 500ppm of HAc, the corrosion rate is 1.35 mm/yr, and 1.80 mm/yr when 1000ppm of HAc was added to the solution. At 80°C and 500ppm HAc, the corrosion rate was 1.80 mm/yr and 2.70 mm/yr with 1000ppm of HAc. A further increase was observed at 3.45 mm/yr when 2500ppm of HAc was added to the system. This increase in corrosion rate is attributed to increase in temperature as increased temperature increases the rate of all reactions. The XRD analysis confirmed that the iron is formed in the absence of HAc while siderite ($FeCO_3$), which is an ore of iron is observed on the materials with HAc. The SEM and EDX results confirmed that a fairly dense material of $FeCO_3$ was formed in the absence of HAc and the layers became porous on addition of HAc to the solution.

Key Words: Corrosion, Acetic acid, Carbon steel, CO_2, Environment

1.0 INTRODUCTION

The petroleum industry is faced with a lot of challenges. In fact, it is hard to imagine an industry with greater challenges and complexities than the oil and gas industry. From diminishing reserves, operation in remote and hostile environment, difficulty and high cost of extracting energy to the problems of transportation of products, the list is endless.

One of the recurring and perhaps most challenging problems facing the industry is corrosion. Oil and gas exploration activities will continue to attain deeper depths and tougher hostile terrains as the demand for energy increases. Hence there is an increase in the need to transport petroleum products from the production sites over long distances to the final destination. Pipeline transport is the most feasible, reliable and safer means of transporting enormous quantities of petroleum product from the well sites to either the refineries or to the final consumers. However, like every other iron and steel derivative, oil and gas pipelines are susceptible to damage caused by corrosion. Corrosion does occur in polymers and ceramics, but the effects or impact are quite important in metals especially in carbon steel pipeline. Metallic systems are the predominant materials of construction and are generally susceptible to corrosion.

1.1 Corrosion in the Petroleum Industry

The petroleum industry is faced with varieties of corrosive environments and to a certain degree, all environments are corrosive e.g. air, moisture and others. Corrosion in the oil industry is due more to CO_2, H_2S, and H_2O than to oil. Higher temperatures and pressures usually involve more severe corrosion conditions. Temperature has a great effect on corrosion, and it has been established that in almost all chemical reactions, increase in temperature increases the rate of reaction (Fontana, 1986).

Similarly, the acidic concentration in the formation water gives rise to corrosion. Initially as the concentration of the acid is increased, the corrosion rate also increased. This is due to the amount of hydrogen ions which are active species has increased concentration of acid in the formation water (Fontana, 1986). However, with further increase in the acid concentration, rate of corrosion reaches a maximum and afterward decreases. This is due to the fact that high concentrations of acids e.g. acetic acid ionization are reduced.

1.2 Corrosion in Different Environments

Virtually all environments are corrosive to an extent, and the rate of corrosion of a metal will varies on the kind of operating environment and the material the metal is made up of (Kermani & Harrop, 1996). For a given metal or alloy, it has been observed that the rate of corrosion in one medium or environment will be different when same metal or alloy is exposed to a different kind of environment. As a result, several studies (Fajardo, Canto, Brown, & Nesic, 2007) had been carried out to ascertain the effect of different environmental factors on important engineering materials such as iron and steel. In the oil and gas industry, the focus has been on carbon steels and alloys used for pipelines, tubings, casings, separators as well as fractionating columns used in refineries and process plants, with typical operating conditions being mineral acids, organic acid and high temperature.

1.3 High Temperature Environment

As in almost all chemical reactions, the rate of corrosion of a metal increases with increasing temperature. Studies carried out by (Dugstad, 2006) and (Fontana, 1986) had shown that the relationship between temperature and corrosion rate is directly proportional and exponential in nature. In the case of stainless steel in nitric acid for example, an increase in temperature increases the oxidizing power of nitric acid, which in turn cause a rapid increase in the corrosion rate of the metal. The same situation is applicable in the oil and gas industry as exploration and production activities often involves operating in relatively high temperature environment. It is sometimes desirable to operate in a sensibly high temperature environment so as to reduce the problem of wax formation in production tubing and pipelines.

1.4 Corrosion in Hydrogen Sulphide (H_2s) Environment

Internal pipeline corrosion of steel in the presence of hydrogen sulphide (H_2S) has always been a notable problem in the oil and gas industry. Although significant in pipeline corrosion, H_2S corrosion has not received the same level of attention as given to CO_2 corrosion, and there are currently no generally acceptable corrosion prediction models for H_2S corrosion. Like organic acids such as acetic acid, hydrogen sulphide is a corrosion accelerator, and in certain favourable conditions the deteriorating effects could be very devastating. The corrosion mechanism associated with hydrogen sulphide and hence its overall effect will differ with its operational environment and in the presence of other substances such as corrosion inhibitors or accelerators (Brown, Parakala, & Nesic, 2004). Hence it is very important for one to know what constitutes the operational environment as well as the effect of hydrogen sulphide in such conditions.

1.5 Corrosion in Acetic Acid Environment

Irrespective of the quantity produced, acetic acid is perhaps the most important organic acid used in the oil and gas industry. It is also used to optimise production processes. Acetic acid is considered as a weak organic acid that is ecologically friendly but has corrosive properties especially at elevated temperatures. The most important application of acetic acid is in oil well stimulation (matrix acidizing and hydraulic fracturing). It can be found information water that is present in oil/gas production, separation and transportation processes. However, Acetic acid aids the formation and deposition of scales and causes corrosion in pipelines which may eventually choke off flow.

Several technical papers had been published on organic acid corrosion of mild steel, with a major focus on acetic acid since it is the most prevalent organic acid encountered in the oil and gas industry (Fajardo, Canto, Brown, & Nesic, 2007). Fajardo et al investigated the effect of organic acids in CO_2 corrosion and showed that at constant pH and concentrations of the organic acids, very little difference exists in the behaviour of the different organic acids used when it comes to CO_2 corrosion of mild steel. The organic acids increase the corrosion rate due to an additional cathodic reaction and makes it harder for protective iron carbonate scales to be formed. (Guo, Chen, & Liu, 2005) in a similar work studied the effect of acetic acid and acetate on corrosion behaviour of carbon steel in CO_2 saturated environment at different temperature and pressure. These were carried out using the weight-loss method, electrochemical method, and surface analysis technique. The results obtained showed that the corrosion rate in the acetic acid and acetate were accelerated and independent of pH value of the solutions, rather depends on the temperature, CO_2 partial pressure and the corroded surface area.

1.6 Corrosion in the Presence of Co_2

Corrosion-related failures of production systems constitutes more than 25% of the failures in the oil and gas industry. Besides, sweet corrosion (CO_2 corrosion) and sour corrosion (H_2S corrosion) contributes to more than half of these failures (Kermani & Harrop, 1996).

At some point in the production of oil and gas, formation water is inevitably produced alongside CO_2 and H_2S, which is corrosive depending on the level of these acidic gases produced. The presence of brine, CO_2 and H_2S in crude oil could lead to rapid increase in corrosion rates as well as environmental fracture of the metal involved. Although, it is usually present in formation water, CO_2 is not on its own as corrosive or catastrophic as H_2S, but in the presence of water molecules could lead to very high localized corrosion rates (Popoola, Grema, Latinwo, Gutti, & Gutti, 2013). CO_2 corrosion is by far the most prevalent form of corrosion experienced in the oil and gas industry, and as a result, many studies have been carried out to ascertain the potential impact on production systems. (Kermani & Harrop, 1996). It has been established that the rate of sweet corrosion is dependent on temperature and partial pressure of the CO_2, which in turn determines the concentration and the solution pH for a given temperature. Since metals react differently with respect to changes in the prevailing conditions, the rate of corrosion of such metals should be observed and recorded alongside the operating conditions (Dugstad, 2006). The corrosion of carbon and low alloy steel as a function of temperature based on oilfield experience is given in the table 1 below (Kermani & Harrop, 1996)

Table 1: Corrosion conditions in oilfield experience (Kermani & Harrop, 1996)**.**

Conditions	**Corrosivity**
PCO_2 < psi (0.5 bar)	Corrosion unlikely
7 psi (0.5 bar) < PCO_2 < 30 psi (2 bar)	Possible Corrosion
PCO_2 > 30 psi (2 bar)	Corrosion

The CO_2 corrosion rate of a metal in the presence of acetic acid will differ from that in the presence of H_2S. Therefore, it is necessary to determine the effect of such corrosion factors that closely match the operating conditions. Experiments were conducted by (Brown, Parakala, & Nesic, 2004) to determine the effect of an incremental change in the solution pH on CO_2 corrosion rates of steel in the presence of H_2S in both single phase and multiphase flow in a large-scale multiphase flow loop. It was observed that the presence of small amounts of H_2S

(<500ppm) led to a significant reduction in the CO_2 corrosion rate. At higher concentrations however, this trend is somewhat arrested and then reversed. Studies by (Ikeda, Ueda, & S.Mukai, 1985) suggested that a low concentration of H_2S (<30ppm) significantly accelerates the corrosion rate when compared to similar CO_2 environments, but the effect of the H_2S seems to be insignificant at higher temperatures and higher concentrations of H_2S when a protective film begins to form.

2.0: MATERIALS AND METHODS

2.1: Test Set Up and Procedures

The test specimens used entirely for this study are carbon steel with a surface area of 8.11cm^2 and a circular hole toward the top end. A copper wire of 0.02 mm diameter was spot welded and used on the test coupons to ensure electrical contact between the electrode and electrolyte. The test coupons were suspended in the test solution by passing a plastic wire through the coupon and the wire was hung to a plastic rod held in place of the glass cell top holes. Prior to introduction into the test cell, the test coupons were successively ground with 240, 600 and 800 SiC grit paper and polished with 1200 SiC paper. It was then cleaned with acetone, rinsed with distilled water and dried. The general composition of the carbon steel samples used in this work is given in Table 2.

The experiments were performed in a mini circulation flow loop. A peripheral pump circulates the test solution from the reservoir to the test cell. A small diameter pipe maintained a constant liquid volume flow in the cell. The reservoir tank and the test cell were continuously purged with CO_2 to removed oxygen completely from the test solution. Once de-oxygenation has been achieved, the required amount of HAc were added at the begining of every experiment. The temperatures were controlled by means of a heating plate with a temperature sensor in the cell. The pH of the solution was adjusted to the desired value throughout the experiments by adding sodium bicarbonate ($NaHCO_3$) or hydrochloric acid (HCl) as needed.

Table 2: Chemical composition of carbon steel (wt. %).

Comp.	C	Si	Mn	S	P	Sn	Cr	Ni	Mo	Cu	Al
Val. %	0.08	0.25	1.54	0.001	0.019	0.008	0.04	0.03	0.01	0.12	0.038

Table 3: Compositions of the test matric used for the experiments

Parameters	Values
Test solution	3.5% wt. NaCl
Test material	Carbon steel
P_{CO2}	0.54
Temperature (^oC)	[25, 80]
C_{HAc} (ppm)	[0, 1500, 2500]
Surface area (cm^2)	8.11

Test method	[WL, XRD, SEM and EDX]

2.3: MEASUREMENT TECHNIQUES

The weight loss method was performed on the carbon steel samples in all the test solutions at temperatures of 25 °C and 80 °C respectively. The weight loss is measured, determined and the corrosion rate calculated using equation 1:

$$\mathrm{CR} = \frac{\mathrm{WL(g)}}{\rho \times \mathrm{t} \times \mathrm{A}} \times \mathrm{K}\left(\frac{\mathrm{mm}}{\mathrm{yr}}\right) \quad (1)$$

Where:

CR = corrosion rate, mm/yr,

ρ = density of the material, 7.86 g/cm^3

WL = weight loss, grams,

t = the exposed time, hr

A = the exposed coupon surface area, cm^2,

K = conversion factor, 8.76×10^4

Carbon steel samples with compositions shown in Table 2 of the same dimensions were exposed to acetic acid and NaCl solution in the presence of CO_2 saturated environment. The samples were washed thoroughly with distilled water and further rinsed with acetone to remove all traces of dirt, oil or grease and were then dried thoroughly before weighing. This was done in order to ensure that the obtained weight was accurate and void of impurities.

All the samples were tagged individually and properly labelled using masking tape, for easy identification and recognition and to avoid mixing the samples up. The lid of the beaker was perforated to enable the immersion of the samples in the containers and was sealed throughout the experiment. The salinity of the solution was measured using a refractometer. The samples were weighed individually using a digital weighing scale. It was used for both measurements of initial and final weights. The digital scale has an accuracy of 0.001.

3.0: RESULTS AND DISCUSSION

3. 1 Weight Loss Test

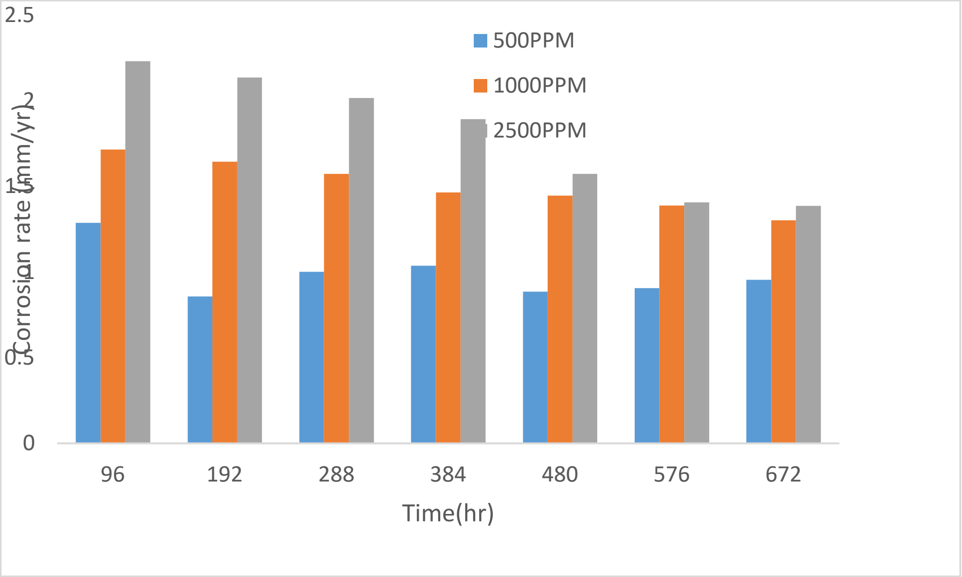

Figure 1: Effects of corrosion rate on carbon steel as a function of time in 3.5% wt. NaCl solution at 25ºC

The results obtained from the experiments are presented in tables and graphs, for ease of presentation and analysis.

Figure 1 show the weight loss result for the corrosion rate of carbon steel as a function of time in 3.5% wt. NaCl solution at 25ºC. As can be seen from the graph (Figure 1), the corrosion rate increased initially with the increase in the concentration of HAc to the solution, attained a maximum and then gradually decreased. At a duration of 12 hours with 500ppm of HAc, the corrosion rate is 1.35 mm/yr, and 1.80 mm/yr when 1000ppm of HAc was added to the solution. As expected, the corrosion rate increased gradually to 2.36 mm/yr on addition of 2500ppm of HAc to the system. It was also observed that the corrosion rate decreases after a long period of the experiment. As observed in Figure 1, after 84 hours duration of the experiment, the corrosion rate reduces to 0.94 mm/yr with 500 ppm of HAc, and to 1.25mm/yr with 1000 ppm of HAc. However, a further reduction of 1.40 mm/yr was recorded on addition of 2500ppm of HAc to the solution. Table 4 show the summaries of average corrosion rate as a function of time for carbon steel in the presence of HAc at 25 ˚C.

Table 4: Summary of average corrosion rate as a function of time for carbon steel in the presence of HAc at 25 ˚C

t (hrs)	500PPM	1000PPM	2500PPM
96	1.286	1.715	2.231
192	0.858	1.644	2.135
288	1.001	1.572	2.016
384	1.036	1.465	1.892
480	0.886	1.446	1.572
576	0.905	1.387	1.407
672	0.954	1.302	1.385

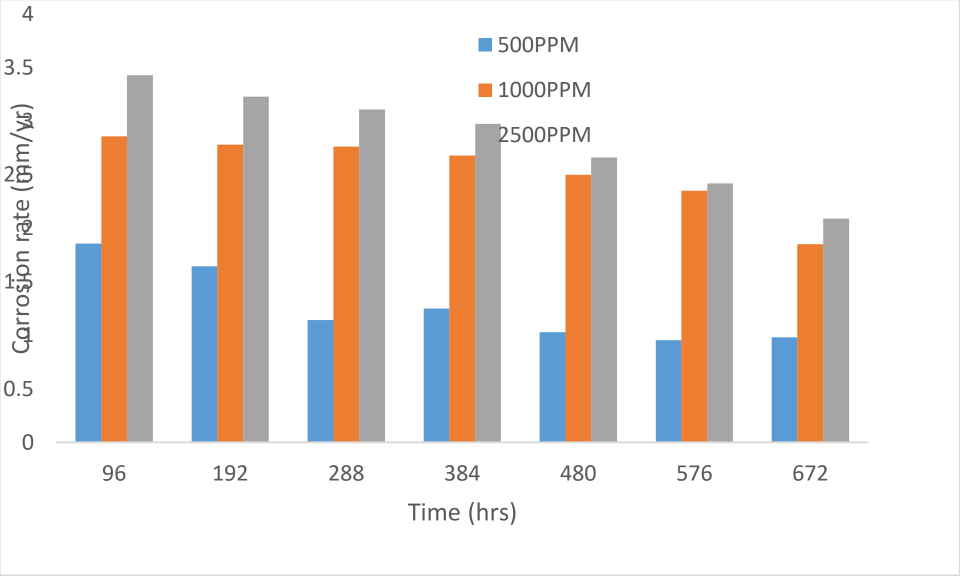

Figure 2: Effects of corrosion rate on carbon steel as a function of time in 3.5% wt. NaCl solution at 80ºC

Similarly, and at 80 °C (Figure 2), the corrosion rate increased exponentially as increase concentration of HAc was added to the solution. After 12 hours period of experiment with 500ppm HAc, the corrosion rate was 1.80 mm/yr and 2.70 mm/yr with 1000ppm of HAc. A further increase was observed at 3.45 mm/yr when 2500ppm of HAc was added to the system. This increase in corrosion rate can be attributed to increase in temperature as increase in temperature increases the rate of almost all reactions. However, a thin film was formed on the body of the carbon steel after a long time (84 hours) which acts as corrosion inhibitor thereby slowing down the rate of corrosion. Table 5 show the summaries of average corrosion rate as a function of time for carbon steel in the presence of HAc at 80 ˚C.

Table 5: Summary of average corrosion rate as a function of time for carbon steel in the presence of HAc at 80°C

t (hrs)	500PPM	1000PPM	2500PPM
96	1.858	2.859	3.43
192	1.644	2.78	3.23
288	1.143	2.763	3.11
384	1.251	2.68	2.975
480	1.029	2.5	2.66
576	0.953	2.35	2.42
672	0.98	1.85	2.09

3. 2 X-Ray Diffraction (Xrd)Analysis Results

The identification of the corrosion films formed on the carbon steel materials was analysed using the X-Ray Diffraction XRD) method. Figures 3, 4 and 5 shows different patterns and phases of materials formed on the carbon steel when exposed to different conditions. For samples without HAc, (in Figure 3), the major component of the products formed is iron, while Figure 4 and Figure 5 show that a new compound was formed. The samples without HAc was seen to be Iron (Fe, JCPDS06-0696), the products occurred in Figures 4 and 5 (conditions with 1500ppm and 2500ppm HAc) are very similar existing as Siderite (FeCO3, JCPDS 29-0696), which is an ore of iron. However, these conditions confirm the fact that the reaction that took place on the metal is a redox reaction since metals tend to go back to their original/natural form through the process of corrosion. Also, an evidence of uneven or localized corrosion was also observed on the samples where some parts will corrode more than other.

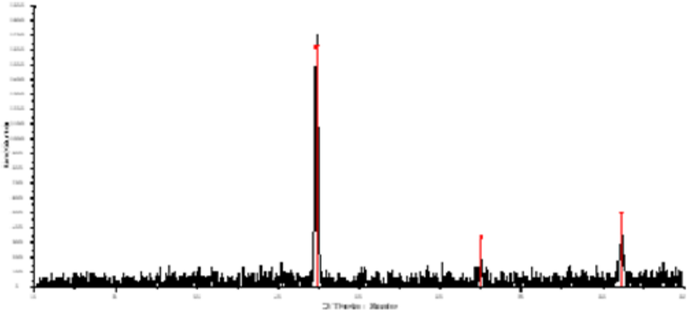

Figure 3: XRD pattern analysis on carbon steel sample in 3.5% NaCl solution with 0ppm oh HAc

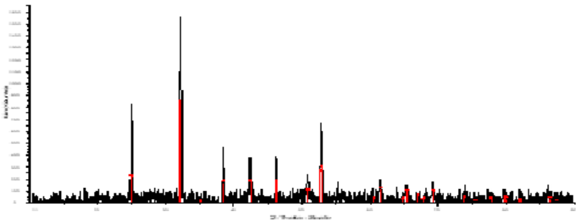

Figure 4: XRD pattern analysis on carbon steel sample in 3.5% NaCl solution with 1500ppm oh HAc

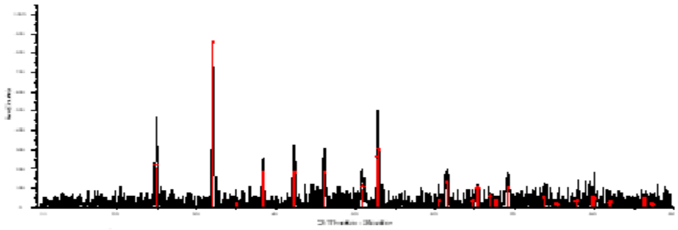

Figure 5: XRD pattern analysis on carbon steel sample in 3.5% NaCl solution with 2500ppm of HAc

3. 3 Sem And Edx Analysis Results

The SEM and EDX analysis results were used to compliment the XRD analysis. Figures 6, 7 and 8 show the morphologies of the samples of carbon steel in NaCl environment after exposure for some period time. In Figure 6B (conditions without HAc), It is observed that a corrosion films were seen on the surface of carbon steel compared to the surfaces where 1500ppm and 2500ppm concentration of HAc were added to the solutions.

In Figures 7B and 8B (conditions in the presence of HAc), it is confirmed that the corrosion products/films are formed as a results of carbon dioxide, water and hydrogen sulphide. Iron carbonate layer and sulphides are formed on carbon steel (Figure6B) without HAc. Addition of different concentration of HAc to the solution result in a less dense layer corrosion film to be formed on the electrode as observed in Figures 7B and 8B respectively. The thin corrosion layers formed is as a result of HAc accelerates the dissolution of the electrode and the solubility of $FeCO_3$ increases in the presence of acetic acid irrespective of the pH of the solution. Similarly, Figures 6A, 7Aand 8A indicate the analysis of EDX on the surface of the material with and without HAc. As can be seen, an irregular discontinuous layers of corrosion film (Figures 7A and 8A) is observed without any orientation. The peak in the Figures 7Aand 8A comprises of iron and oxygen. However, these peaks is an indication that iron oxide and carbonate are formed as a result of corrosion film on the surface of the carbon steel. Also, an element of sulphur was observed based on iron sulphide presence.

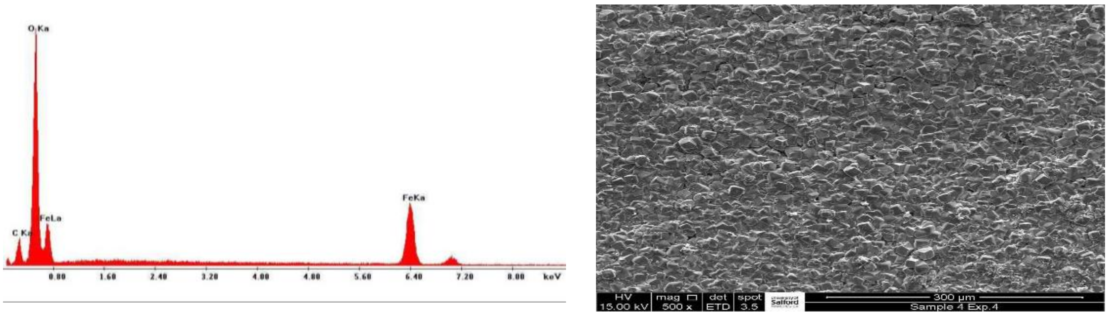

Figure 6: EDX spectrum (A) and SEM image (B) of carbon steel samples exposed to 3.5% wt. NaCl solution and 0ppm HAc

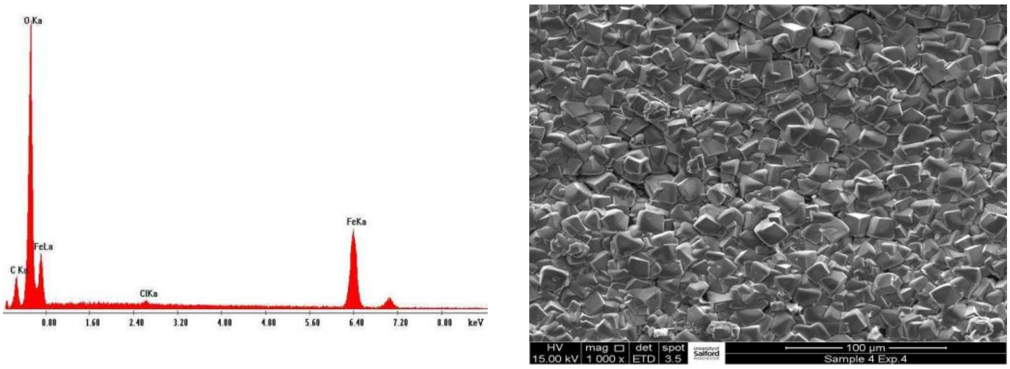

Figure 7: EDX spectrum (A) and SEM image (B) of carbon steel samples exposed to 3.5% wt. NaCl solution and 1500ppm of HAc.

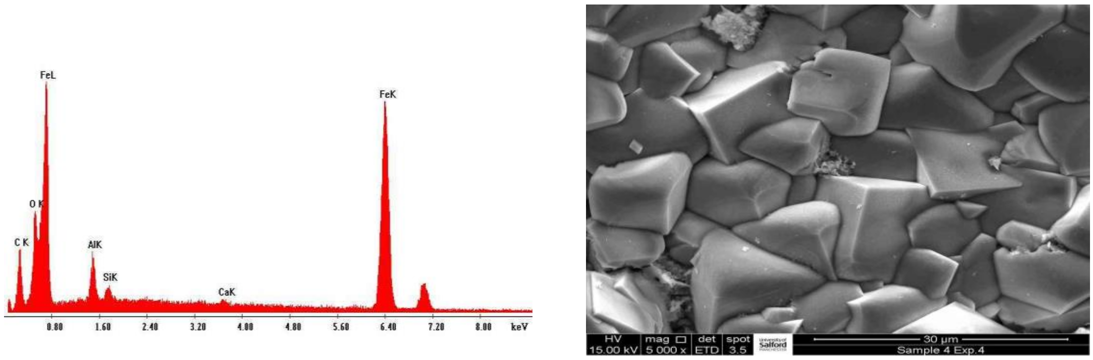

Figure 8: EDX spectrum (A) and SEM image (B) of carbon steel samples exposed to 3.5% wt. NaCl solution and 2500ppm of HAc.

4.0 CONCLUSION

In this study, a comparative study and analysis of carbon steel corrosion in the presence HAc was carried out in CO_2 saturated environment. This was done in order to investigate the corrosion rate of carbon steel, evaluate the effect of temperature and concentration of HAc on the rates of corrosion. Direct method such as the weight loss and surface analysis methods (such as XRD, EDX and SEM) were used to characterize the carbon steel material and type of corrosion that occurred on the materials.

The results of the analysis show that an increase in temperature increases the rate of corrosion of the carbon steel samples and hence the weight loss also increases. This agrees to the general trend that an increase in temperature of a reaction generally speeds up the rate of the reaction, all other conditions being constant.

Secondly, it was also observed that an increase in the concentration of HAc increases the corrosion rate to a peak and then begins to decline with time. This was as a result of a build-up of products of corrosion which acts as a protective layer, hence reducing the rate of corrosion. The SEM analysis confirmed that a fairly dense material of FeCO3 was formed in the absence of HAc and the layers became porous on addition of HAc

Similarly, the EDX analysis on the carbon steel confirmed more lines characteristics which confirmed the presence of carbon and oxygen. The XRD analysis shows that the iron is formed in the absence of HAc while siderite ($FeCO_3$) which is an ore of iron is observed on the materials with HAc. Also, an evidence of localized corrosion was also observed on the carbon steel samples where some area corrodes more than others.

5.0: ACKNOWLEDGMENTS.

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