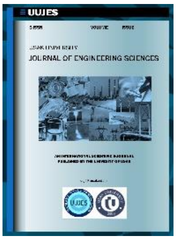

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Research article

CORROSIVE INFLUENCE OF VARIED SALT SOLUTIONS CONCENTRATION ON MILD STEEL

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Abstract

This study is focused on the corrosive effects of varied salt solutions concentration on two mild steel specimens (API 5L X42 and API 5L X60). Gravimetric method was adopted in the evaluation of the weight loss and corrosion rate of the specimens. The hardness of the test specimens after dipping in corrosive solutions was examined. Potentiodynamic polarization technique was also used to compare the corrosive media. API 5L X42 and API 5L X60 specimens exhibited highest corrosion rates of 9.593 mmpy and 9.698 mmpy respectively in solution A that contained 2.0 M NaCl, 3.0 M $CaCl_2$ and 0.5 M Na_2CO_3. They also exhibited the lowest corrosion rates of 7.642 mmpy and 7.836 mmpy respectively, in solution H that contained 0.5 M NaCl, 3.0 M $CaCl_2$ and 2.5 M Na_2CO_3. The API 5L X42 mild steel specimen in solution H exhibited the highest hardness value of 369 HV after immersion in the corrosive medium. The hardness of the corroded specimens decreased compared to their hardness values before immersion in the corrosive media. This is an indication that the chemical/electrochemical reactions of the salt solutions affected the surfaces of the specimens, which enhanced indentation of the specimens.

Keywords: *Mild steel; Corrosion rate; Salt solutions; Molar concentration; Potentiodynamic polarization; Hardness.*

1. Introduction

Corrosion is an irreversible interfacial reaction of a material (metal, ceramic, and polymer) with its environment, which results in its consumption or dissolution into the material of a component of the environment. Often, but not necessarily, corrosion results in effects that are detrimental to the usage of the material considered [1]. When the surfaces of materials deteriorate due to chemical reactions with their surroundings, corrosion has taken place. Rusting can also occur in plastics, concrete, wood, and other materials as

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illustrated in Table 1 but it is more prevalent in metals and alloys. Corrosion is the formation of more solid products as a result of surface reactions in a chemical or electrochemical medium as metals or alloys are attacked in their environments such as air, water, or dust. The most commonly used metal is iron (usually steel), and the corrosion of this metal is the subject of the following question. Corrosion is shown by the rusting of iron ($Fe_2O_3.H_2O$) and the forming of a green carbonate film $CuCO_3.Cu(OH)_2$ on copper[1].

Table 1 A table showing that most of the materials decay overtime [1]

Materials	Types of decay	Mitigation actions
Wood	Fungi, insects, and sun + rain	Chemicals, paint/varnish
Steel	Rust	Galvanizing, painting
Concrete	Cracking/Spalling	Corrosion-resistant rebar
Stone	Wear, damage by pollution	Usually none taken
Glass	Breaks	Tempered glass
Polymer	Become brittle under UV light	Improved polymer grade
Aluminium	Pitting over time, possible galvanic corrosion	Galvanic corrosion can be prevented
Copper	Forms a green patina over time	None required
Stainless steel	No decay	None

In electrochemical corrosion, atoms of metals/alloys are removed because of an electric circuit that is produced. Understanding corrosion processes aids in product design and selection to ensure high levels of safety. The most important factor in corrosion prevention is safety but economy is also important. Corrosion costs money not only because the object must be removed, but also because time and processing materials are wasted [1]. Corrosion of mild steel in particular is of great concern to governments and stakeholders in the industrial sector because of the wide areas of application of mild steel. Corrosion is a problem that usually costs the engineering industry lots of resources to control. Particularly, the oil and gas industries spend huge financial resources to combat corrosion annually [2-4]. When corrosion is not adequately controlled, it may lead to catastrophic failure, environmental hazard and accident with death in extreme cases. Salty and acidic environments promote corrosion. Some salt solutions enhance or promote corrosion because they are acidic by hydrolysis and due to specific catalytic effects or reactions of the anions [5].

Corrosion is more complex in seawater than freshwater due to complicated chemical reactions that often take place during the natural corrosion cycle. A typical seawater majorly consists of six ions of chloride, sodium, sulphate, magnesium, calcium, and potassium. These ions account for about 99 % of all sea salts that often cause corrosion [6]. Materials selection is a promising strategy in corrosion control. Nevertheless, the varying chemical composition of the environment where the material is being used is a major setback. It is possible to predict suitable materials for an environment but unexpected variations in the concentration of the chemical composition of the environment often compromise the reliability of the material. This may result to complications like unplanned maintenance or breakdown because of corrosion. For instance, mild steel is used as pipes to convey water, oil and gas in the petroleum industry [7], the reality of the environment where it is put into application is very severe and varies almost unpredictably.

Many studies have been carried out on the effects of corrosive media on mild steel. Ibrahim [8] investigated the corrosive response of coated mild steel in varied molarity/concentration of sodium chloride (NaCl) solutions and submitted that high concentration (above 3 %) of NaCl decreased corrosion rate because of reduction in the solubility of oxygen in the solution. Gao et al. [9] also investigated the corrosive response of mild steel in sour media at high temperatures and reported that temperature change had a significant effect on corrosion. They submitted that temperature change influenced the type of corrosion product film that was formed. If the film formed had sufficient thickness and good adherence, the corrosion rate would decrease but if the thickness or adherence of the corrosion product film formed was not sufficient, corrosion would increase.

Generally, the corrosion of mild steel structural members often takes the form of electrochemical reactions whereby the metal atoms are dissolved in the corrosive environment at a rather high rate. Sea or ocean water is a corrosive environment that is highly characterised with salt forming ions such as Na^+, Cl^-, Mg^{2+}, SO_4^{2-} and Ca^{2+}. The combination of salts that can be formed affect mild steel in different ways. Hence, it is necessary to investigate the corrosive effects of varied salt concentrations on mild steel and this is the aim of this study.

2. Methodology

2.1. Materials and equipment

Mild steel test specimens, sodium chloride (NaCl) crystal salt, sodium trioxocarbonate IV (Na_2CO_3) and calcium chloride ($CaCl_2$), distilled water, silicon carbide papers of grades 180, alcohol, weighing machine, hacksaw, beakers were used in this study. The mild steel specimens were obtained from Dorman Long Ltd, Lagos, Nigeria and their chemical compositions are presented (Tables 2 and 3). The composition was determined using an optical emission spectrometer. The crystal salts were obtained from a registered vendor in Lagos, Nigeria. The photographs of the prepared specimens and their immersion in the corrosive media are shown in Fig. 1.

Table 2 Composition of mild steel specimen (API 5L X42)

Elements	Fe	C	Si	Mn	P	V	Ni	Nb	Ti	Al	Co
Weight (%)	98.23	0.11	0.25	1.30	0.010	0.05	0.0180	0.02	0.01	0.01	0.001

Table 3 Composition of mild steel specimen (API 5L X60)

Elements	Fe	C	Si	Mn	P	V	Ni	Nb	Ti	Al	Co
Weight (%)	98.04	0.13	0.25	1.45	0.010	0.05	0.0170	0.02	0.01	0.02	0.002

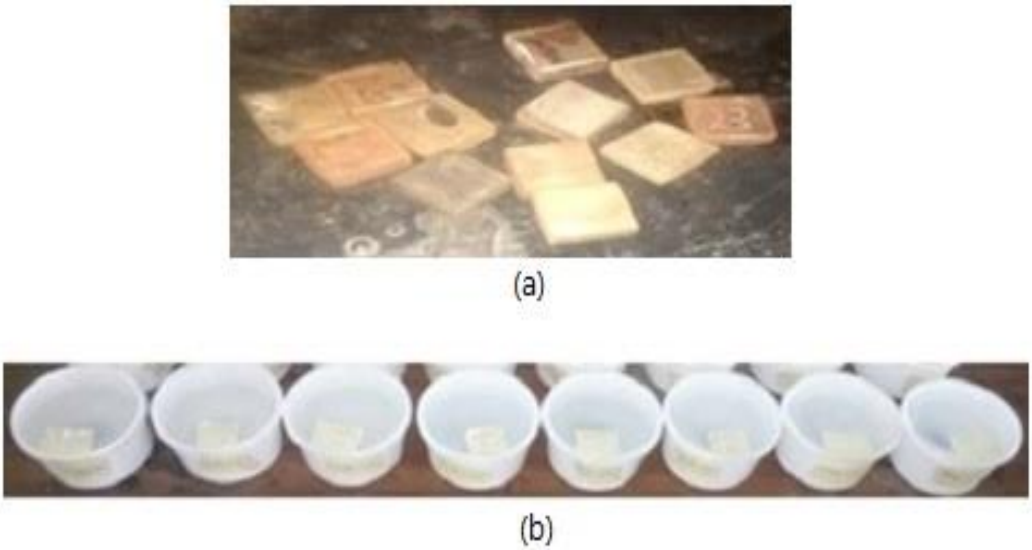


Fig. 1 Photographs of (a) prepared mild steel specimens, and (b) specimens immersed in the corrosive media

2.2. Specimens, solutions and experiment

The as-received mild steel plate was cut to 32 test specimens of dimension 2.5 x 2.5 x 0.80 cm (Fig. 1a) using an hacksaw. The four sides of the specimens were smoothened with a file while their surfaces were sequentially polished using silicon carbide papers of grades 180. The specimens were rinsed in distilled water using a plastic brush. Subsequently, the specimens were soaked in ethanol for 5 mins and allowed to dry for 5 mins [10]. The test specimens were designated for easy identification with an engraving machine. The initial weight (W_1) of the specimens was measured using a digital weighing machine. The concentrations were prepared to low and high levels as shown in Table 4.

Table 4 Range of molar concentrations of crystal salts

Parameter (Salts)	Low level molar concentration (M)	High level molar concentration (M)
NaCl	0.50	2.00
$CaCl_2$	0.50	3.00
Na_2CO_3	0.50	2.50

The amount of salts in grams required to make each concentration was calculated using molarity as follows:

- 0.5 M of NaCl was obtained by weighing 116.95 g of NaCl salt on a digital scale and then dissolved in 4 litres of water (2.925 % NaCl).
- 2.0 M of NaCl was obtained by weighing 467.83 g of NaCl salt on a digital scale and then dissolved in 4 litres of water (11.70 % NaCl).
- 0.5 M of $CaCl_2$ was obtained by weighing 221.97 g of $CaCl_2$ salts on a digital scale and then dissolved in 4 litres of water (5.55 % $CaCl_2$).
- 3.0 M of $CaCl_2$ was obtained by weighing 1331.85 g of $CaCl_2$ salts on a digital scale and then dissolved in 4 litres of water (33.28 % $CaCl_2$).
- 0.5 M of Na_2CO_3 was obtained by weighing 212.09 g of Na_2CO_3 salts on a digital scale and then dissolved in 4 litres of water (5.30 % Na_2CO_3).

- 2.5 M of Na_2CO_3 was obtained by weighing 1060.44 g of Na_2CO_3 salts on a digital scale and then dissolved in 4 litres of water (26.5 % Na_2CO_3).

Thereafter, eight separate solutions were prepared using different combinations of molar concentrations of each salt solution (Table 5). The solutions were poured into eight plastic containers in equal proportions of 40 ml to simulate the corrosive medium. The specimens were dipped in the corrosive media as shown in Fig. 1b. The experiment was executed in accordance with ASTM G1-03(2017) E1 [11] which was earlier adopted by Ikechukwu et al. [12].

Table 5 Molar concentration (M) of the salts

Solutions	**NaCl**	**$CaCl_2$**	**Na_2CO_3**
A	2.0	3.0	0.5
B	2.0	0.5	0.5
C	0.5	0.5	0.5
D	0.5	3.0	0.5
E	0.5	0.5	2.5
F	2.0	3.0	2.5
G	2.0	0.5	2.5
H	0.5	3.0	2.5

2.3. Determination of rate of corrosion of the specimens

Corrosion rate was calculated by gravimetric method. After every 7 days of immersion of the specimens in the various salt solutions, they were removed and their final weights (W_2) was measured and recorded. The weight loss (W_t), which is the subtraction of final from initial weight was recorded and the rate of corrosion rate was calculated using Equation (1) [13].

$$R = \frac{KW_t}{At\rho} \tag{1}$$

Where, K is a constant and its value is 87.6, W_t is weight loss (g), A is surface area (cm^2), t is the duration of immersion of specimens in the solutions (hrs), ρ is the density of specimen (g/cm^3), R is the corrosion rate (mpy). L, B and T are the length, breadth and thickness of the specimens respectively in cm.

2.4. Polarization test

The experiment was carried out at room temperature of 25 ^{0}C using cylindrical test specimens (electrodes) in accordance with ASTM G59-97 [14] standard. The measurement was taken from -1.5 V to +1.5 V at scan rate of 0.0015 V/s in accordance with ASTM G102-89 [15] standard. The corrosion current density (Icorr) and corrosion potential (Ecorr) were obtained from the Tafel plots of potential against log current.

2.5. Hardness test

The hardness of the specimens was obtained by using a hardness tester according to ASTM E92 [16] standard. The tester supported the specimens and made the indenter to have contact with the specimens gradually under an applied load of 1000 g and operating period of 10 secs.

3. Results and discussion

3.1. Corrosion rate

The corrosive effects of the salt solutions on the mild steel test specimens is illustrated in Figs. 2 and 3. The specimens in the various corrosive media experienced sharp increase in corrosion rate during first seven days of immersion. This is an indication that there were reactions between the specimens and the corrosive media. Corrosion rate increased with exposure time in the simulated corrosive media [17]. However, specimens in media F and H exhibited decrease in corrosion rate between 30th and 42nd days of immersion. This is due to the high molar concentration of Na_2CO_3 and the precipitate formed, which hindered corrosion. The API 5L X42 and API 5L X60 specimens exhibited highest corrosion rates of 9.593 mmpy and 9.698 mmpy respectively in solution A that contained 2.0 M NaCl, 3.0 M $CaCl_2$ and 0.5 M Na_2CO_3. They also exhibited the lowest corrosion rates of 7.642 mmpy and 7.836 mmpy respectively, in solution H that contained 0.5 M NaCl, 3.0 M $CaCl_2$ and 2.5 M Na_2CO_3.

Figs. 2-6 further suggest that the solutions that contained 0.5 M Na_2CO_3 were more aggressive than solutions that contained 2.5 M Na_2CO_3. The specimens also manifested the corrosive effect of the media with the curves indicating that the order of corrosion rate in the solutions is NaCl > $CaCl_2$ > Na_2CO_3. This is in agreement with the earlier reported by Ikechukwu et al. [12]. Apparently, corrosion rate increased because of the electrical conductivity of NaCl and $CaCl_2$ salt solutions by enhancing the corrosion current between the anode and cathode [18]. Hence, corrosion rate of the specimens increased more in the chloride media. However, the highly dissolved salt of NaCl solution also reduced greatly the amount of dissolved oxygen, which reduced the high corrosion rate [2]. Hence, the rate of corrosion of the specimens reached its peak when NaCl concentration was 3.5 %. The slow corrosive effect of [solutions E, F, G and H on the test specimens was due to the high molar concentration of Na_2CO_3 (2.5 M). In addition, $CaCl_2$ and Na_2CO_3 reacted during corrosion cycle to form a white precipitate of $CaCO_3$, as shown in Equation (3). The $CaCO_3$ formed a scale in the liquid, which acted as a protective layer. This is similar to the reported by [10]. High molar concentration of Na_2CO_3 in solutions E, F, G and H and the $CaCO_3$ protective film formed is attributed to the low corrosive effect of the solutions on the specimens. This shows that the chemical reaction depends on the molar concentration of the reacting salts. This implies that the higher the molar concentration of the $CaCl_2$ and Na_2CO_3, the more the concentration of white precipitate of $CaCO_3$ formed and hence the more corrosion resistant the solution becomes as noticed in solutions F and H. On the other hand, it is also possible that the reduction in corrosion rate was caused by the formation of Fe_2O_3, Fe_3O_4 and $FeCO_3$ within the corrosive media since Fe^{+2}, Fe_2O_3, Fe_3O_4, and $FeCO_3$ [19] were the corrosion products. The formation of Fe_3O_4, Fe_2O_3 or $FeCO_3$ might have generated adherent passive films on the mild steel specimens, which acted as barriers between the salt solutions and the surfaces of the specimens, which protected them from experiencing corrosion. This is similar to the earlier report by Alves and Brett [20] and Saba et al. [21].

$$CaCl_2 + Na_2CO_3 \longrightarrow 2NaCl + CaCO_3 \quad (3)$$

Furthermore, Na_2CO_3 is an inhibitor, which reduced corrosion rate [19]. The more the concentrations of Na_2CO_3, the lesser the corrosive effect on mild steel specimens.

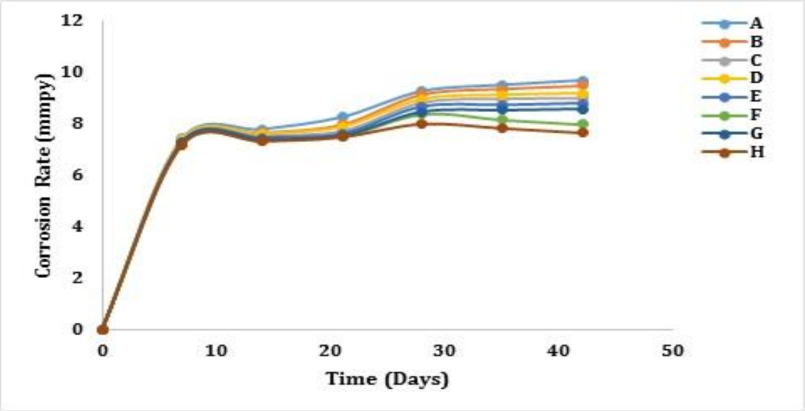


Fig. 2 Rate of corrosion against time for API L5 X42 specimen in the various solutions

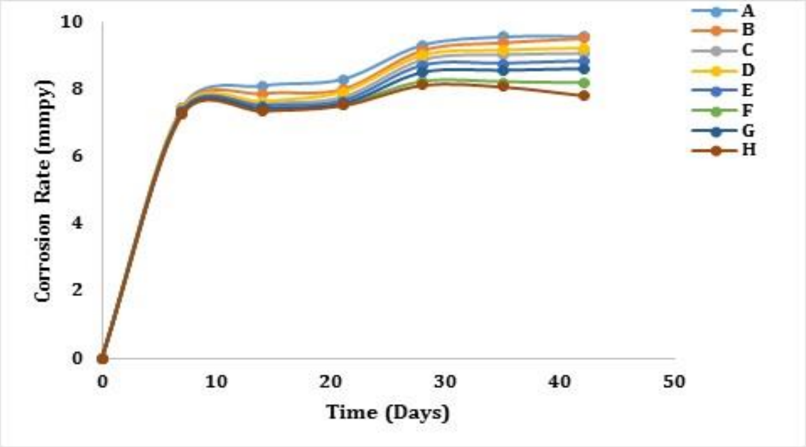
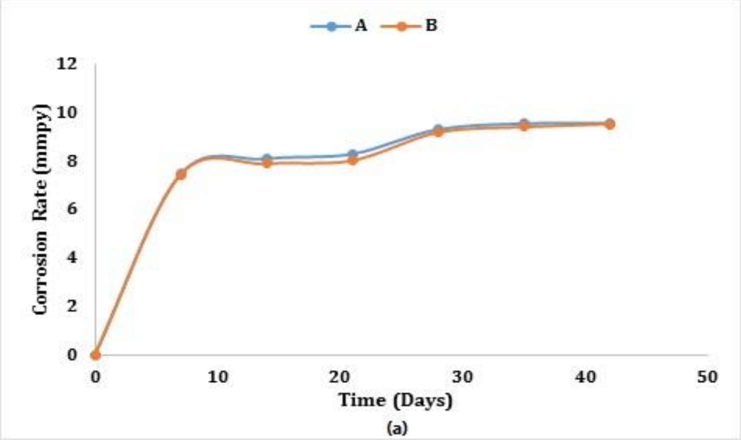


Fig. 3 Rate of corrosion against time for API L5 X60 specimen in the various solutions



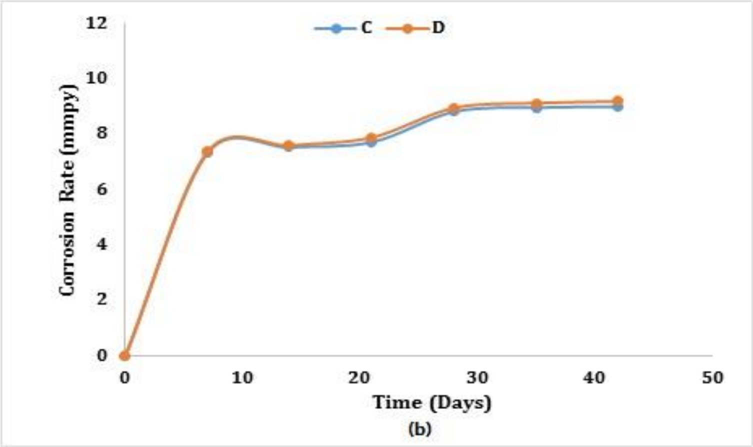


Fig. 4 Effect of variation of $CaCl_2$ concentration on API L5 X42 Mild steel (a) Specimens immersed in solutions A and B (b) Specimens immersed in solutions C and D

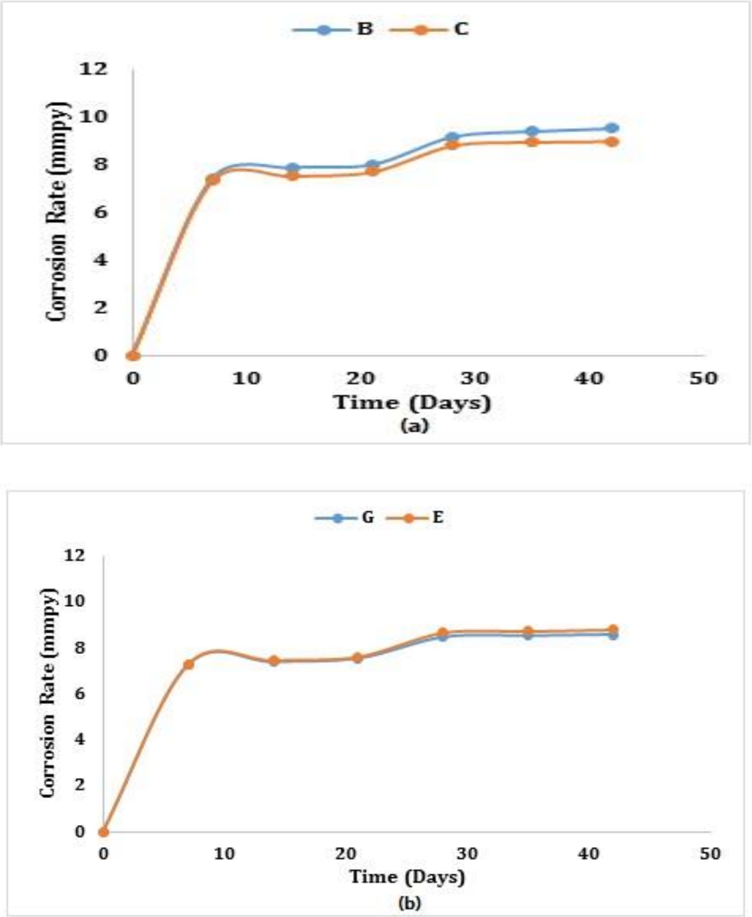


Fig. 5 Effect of variation of NaCl concentration on API L5 X42 Mild steel (a) Specimens immersed in solutions B and C (b) Specimens immersed in solutions G and E

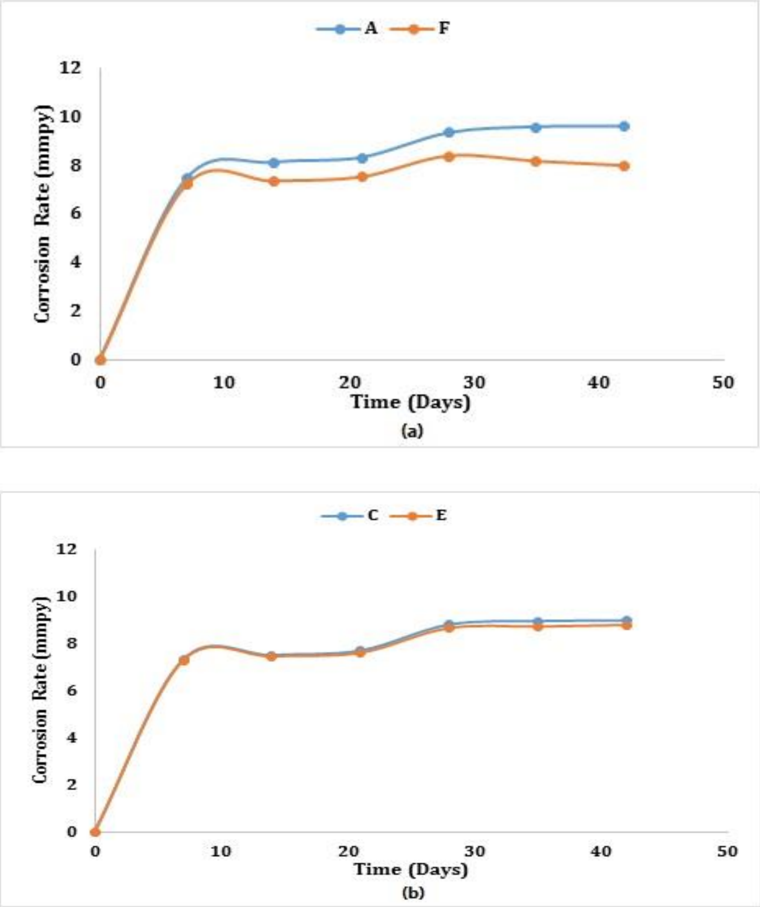


Fig. 6 Effect of variation of Na_2CO_3 concentration on API L5 X42 Mild steel (a) Specimens immersed in solutions A and F (b) Specimens immersed in solutions C and E

Previous study revealed that change in colour of corrosive medium is a characteristic of corrosion rate or control coupled with the presence of iron oxides in the corrosive medium [12]. Among the factors that may activate such features is the molar concentration of the crystal salt constituent used as earlier reported by [12], anions that are present and chemical reactions in the corrosion media [8]. The stimulated corrosive media consisted of mixtures of NaCl, $CaCl_2$ and Na_2CO_3. Fig. 7 shows the resulted corrosive medium after mixing the three crystals salts and before immersion of the steel specimens. The resulted solutions A to H were characterised with a white precipitate phase on formation of calcium trioxocarbonate IV ($CaCO_3$). The resulted solutions after the corrosion cycle is shown in Fig. 8. Solutions A to D are redish brown with sediments of black iron oxide particles while solutions E to H are slightly brown and colourless.

The general disappearance of the white precipitates was due to the decomposition of the $CaCO_3$ and subsequent reaction with water to form $Ca(OH)_2$. Interestingly, both $CaCO_3$ and $Ca(OH)_2$ had low corrosive effect rate that made them to act as inhibitors when they were in contact with NaCl and $CaCl_2$ [3]. Two anions, which may have significant influence on the corrosion characteristics in terms of corrosion rate or colour change of the corrosive media are chloride and carbonate ions. However, $CaCO_3$ decomposed in the presence of weak acid solution formed during corrosion cycle because Na_2CO_3 is a water-soluble sodium salt of carbonic acid. This means that specimens immersed in solutions A to D, which contained 0.5 M Na_2CO_3 at various molar concentration of NaCl and $CaCl_2$ were more susceptible to higher corrosion rate [18]. This is due to the higher corrosive influence of NaCl and $CaCl_2$ and lower inhibition effect of 0.5 M Na_2CO_3. Thus, the colour of the corrosive media was very dark coupled with sediments of iron oxide. The colour change seen in solutions E to H may also be due to the formation of $CaCO_3$ coupled with the inhibition effect of 2.5 M Na_2CO_3. This behaviour is more pronounced in solutions F and H.

Fig. 7 Photograph of the colour of the corrosive medium prior to specimen immersion

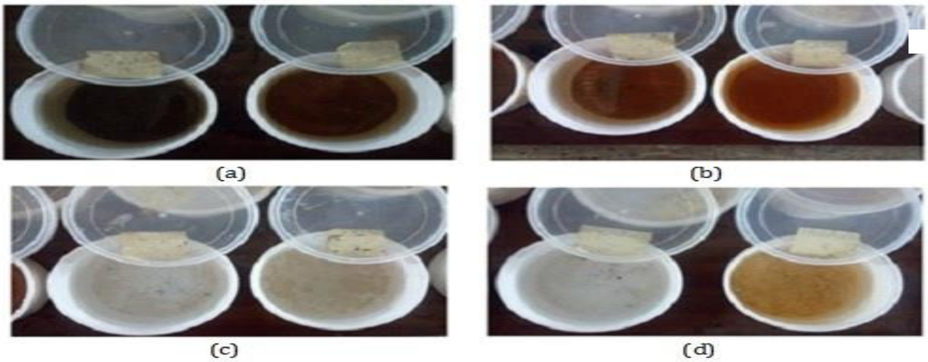
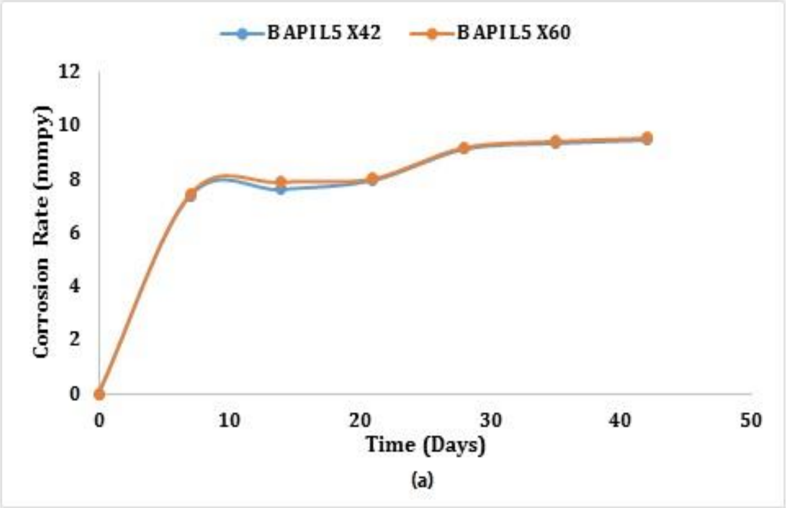


Fig. 8 Photographs of the colour of the corrosive media after 42 days of corrosion cycle for API 5L X60 mild steel specimen (a) solutions A and B, (b) solutions C and D, (c) solutions E and F, (d) solutions G and H

Fig. 9 shows the corrosion rate against exposure time of the specimens using corrosive media B and D. From the experimental results, corrosion rate is faster in API 5L X60 mild steel specimen than API 5L X42 mild steel specimen in the two corrosive media B and D. This could be due to the difference in their chemical compositions. Corrosion rate of these specimens is lower in solution D than B due to the higher molar concentration (3.0 M) of the calcium chloride salt in solution D.



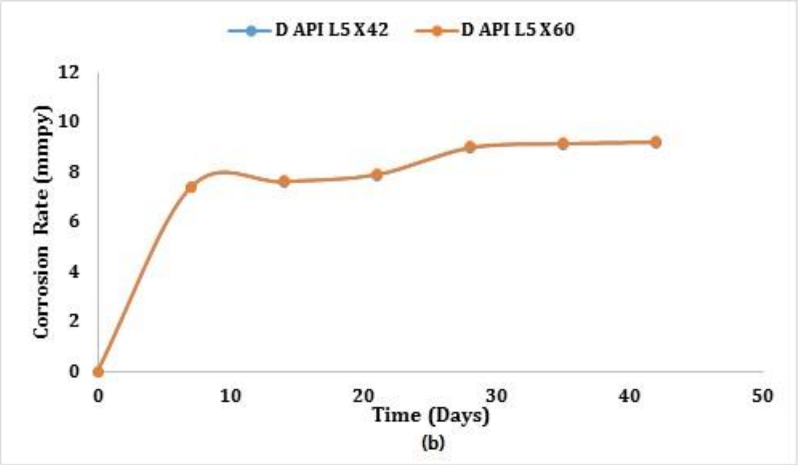


Fig. 9 Rate of corrosion against time of specimen (a) solution B and (b) solution D

3.2. Polarization

The anodic polarization charts of the test specimens are shown in Fig. 10. The corrosion potential of the specimens is more within the negative range for API 5L X60 mild steel specimen than API 5L X42 mild steel specimen, which indicates aggressive corrosion of the specimens. The specimens display active-passive polarization behaviour. The specimens immersed in solutions A and B exhibited more of active polarization while solutions F and H showed passive polarization due to the higher molar concentration of sodium and calcium chlorides salts, which decreased the corrosion rate. This shows that corrosion rate decreased when the specimens were immersed in high NaCl and $CaCl_2$ concentrations. This behaviour could be due to the development or existence of corrosion products, which acted as physical barriers to oxygen diffusion and adsorption of oxygen to the surfaces of the specimens, which decreased corrosion rate. High concentration of sodium trioxocarbonate IV enhanced both low current density and corrosion rate of the steel specimens. This correlates with the findings that anions could serve as inhibitors or plug the pores of passive films at high concentration [18]. Na_2CO_3 of 2.5 molar concentration might have formed thin films on the surfaces the specimens. Apparently, the linear polarization resistance and corrosion current values are functions of the protective films formed. The protective films formed on the surfaces of the specimens caused the linear polarization resistance (LPR) to increase and decreased the corrosion current values.

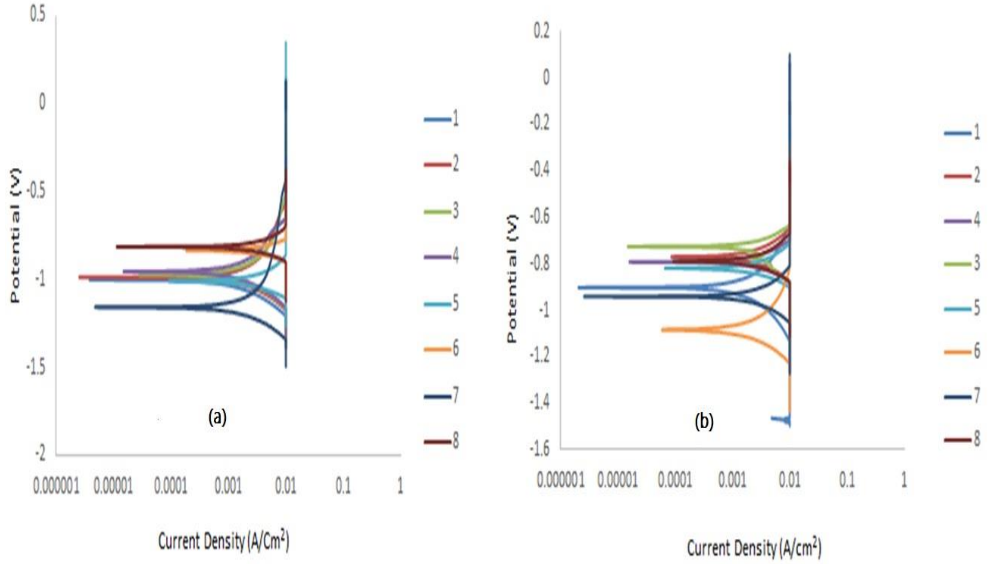


Fig. 10 Potentiodynamic anodic polarization plots of the specimens in media A to H (a) API 5L X42 (b) API 5L X60

3.3. Hardness of the specimens

Figs. 11 and 12 illustrate effects of the various corrosive media on the test specimens. The specimen (API 5L X60) in solution H exhibited the highest hardness value of 366 HV among the specimens in Fig. 11. The API 5L X42 mild steel specimens in solution H exhibited the highest hardness value of 369 HV among the specimens in Fig. 12. Hence, API 5L X42 mild steel specimen exhibited higher hardness than API X60 specimen. As illustrated in the two figures, the specimens demonstrated lower hardness after immersion in the corrosive media when compared to their hardness values before immersion. This is an indication that the chemical/electrochemical reactions of all the salt solutions affected the surfaces of the specimens [1], which enhanced indentation of the specimens. It has been reported that early stages of corrosion largely depends on roughness of the surfaces of metals/alloys because dense oxide films on metals or alloys surfaces that are protecting the metals or alloys from corrosion can be easily destroyed at high surface roughness [22]. It has also been reported that surface roughness of metals or alloys can affect electrochemical or mechano-chemical behaviour of their surfaces, which can enhance interactions thereby influencing corrosion [23]. Hence, the reason for the decrease in hardness after immersion in the corrosive media.

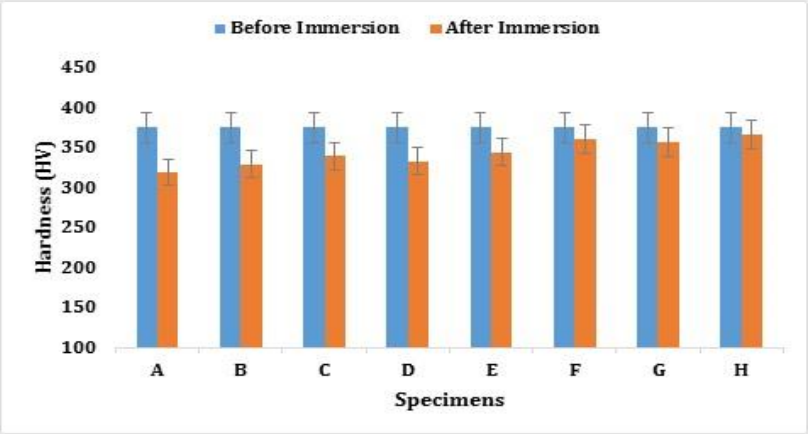


Fig. 11 Hardness of API L5 X60 specimens before and after corrosion

Fig. 12 Hardness of API L5 X42 specimens before and after corrosion

4. Conclusions

In this study, the corrosive effects of varied salt solutions on mild steel specimens was investigated. Test specimens were subjected to different tests to determine the most corrosive medium, corrosion rate, and hardness of the specimens. From the results of investigation and discussion of this research work, the following inferences can be drawn:

- The solutions caused significant increase in the rate of corrosion of the specimens, which was influenced by the molar concentration of the media and exposure time.
- Specimens API 5L X42 and API 5L X60, which were immersed in solution A, exhibited the highest corrosion rates of 9.593 mmpy and 9.698 mmpy respectively. They also exhibited the least corrosion rates of 7.642 mmpy and 7.836 mmpy respectively in solution H.
- Low concentration of NaCl (most aggressive salt) and high concentration of $CaCl_2$ and Na_2CO_3 in solution H reacted and produced $CaCO_3$, which inhibited or hindered the rate of corrosion by forming protective films on the specimens.

- The specimens immersed in solutions A and B exhibited more of active polarization while solutions F and H showed passive polarization due to the higher molar concentration of sodium and calcium chlorides salts, which decreased the corrosion rate.
- The API 5L X42 mild steel specimen in solution H exhibited the highest hardness value of 369 HV after immersion in the corrosive medium
- The hardness of the corroded specimens decreased compared to their hardness values before immersion in the corrosive media. This is an indication that the chemical/electrochemical reactions of all the salt solutions affected the surfaces of the specimens, which enhanced indentation of the specimens.
- For clarity, the IUPAC (International Union of Pure and Applied Chemistry) names of the compounds are illustrated in Table 6.

Table 6 Appendix: chemical formula and their IUPAC names.

Chemical formula	IUPAC Names
$NaCl$	Sodium Chloride
$CaCl_2$	Calcium Chloride
Na_2CO_3	Sodium trioxocarbonate (IV)
$FeCO_3$	Iron (II) trioxocarbonate (IV)
Fe_2O_3	Iron (III) Oxide
Fe_3O_4	Iron (IV) Oxide

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