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ABSTRACT

This research work uses gravimetric analysis for corrosion tests performed to determine the weight loss and corrosion extent on carbon steel and glass reinforced epoxy (GRE) specimens. Comparative results for both specimens are analysed to justify the use of glass reinforced epoxy pipe as an effective control measure for the corrosion of oil pipelines used for the transportation of hydrocarbon products. The values obtained from the experiment are used to develop regression model which can be helpful to predict the corrosion extents on both materials within the test composition. Coefficient of determination is explored to ascertain the reliability of the regression models. The results obtained indicated that 99 percent of the original uncertainty has been explained by the regression models. From the experimental results, glass reinforced epoxy test specimens had better resistant to corrosion than carbon steel test specimens which makes it attractive alternative to the conventional carbon steel pipe for the transportation of hydrocarbon products in the Niger Delta of Nigeria.

Keywords: Carbon steel, Corrosion extent, Gravimetric analysis, GRE, Regression models

1. INTRODUCTION

Over the years carbon steel pipe has been the major medium for transportation of hydrocarbon products from one location to the others in the Niger Delta region of Nigeria. However, this pipe suffered severe breakdown as a result of corrosion. According to [5], corrosion can be defined as the deterioration and loss of metal and its critical properties due to chemical, electrochemical and other reactions of the exposed material surface with the surrounding environment. [11] also defined corrosion as the destruction of metal by electrochemical action and it cannot take place unless an electrolyte is present. These destructions or leakages are

caused primarily by corrosion as a result of the exposure of the inner surface of the pipe to hydrocarbon products.

However, corrosion of the external surfaces of the pipelines also occurs because of the exposure to their external environment. In this study, corrosion test experiment is conducted to justify the use of a new, economically attractive, light, corrosion-resistance and flexural strength glass reinforced epoxy pipe as an alternative to the conventional carbon steel pipe for the transportation of hydrocarbon products in the Niger Delta so as to lower the expenditure on oil pipeline maintenance activities, reduce the incidence of operational breakdowns as a result of oil pipelines corrosion and ensure trouble-free operation. From the values obtained from the experiment, statistical models are developed to predict the corrosion extents of glass reinforced epoxy and carbon steel materials within the limit of the experimental values.

Carbon steel pipe is a commonly utilised for so many purposes because of its low cost and excellent mechanical properties. However, it suffered severe attack in service in the form of corrosion, particularly in oil and gas exploration and production operations in the Niger Delta region of Nigeria. The effects of corrosion of carbon steel pipe among others include huge economic losses as many companies shut down their oil wells or go out of business, which is detrimental to the growth of the companies. This in turn had serious negative impacts on the growth and productivity of other smaller industries, which most times resulted to mass redundancy, added to the alarming rate of unemployment situation in the country.

2. MATERIALS AND METHODS.

For this research work, corrosion experiment is conducted to determine the weight loss and corrosion extents of glass reinforced epoxy and carbon steel pipes. The test applies the gravimetric method for its analysis. This experiment covers the procedure for preparing glass reinforced epoxy and carbon steel specimens for test, removing corrosion specimens after the test has been completed and evaluating the corrosion damage (weight loss) that has occurred in accordance with ASTM G1. This laboratory corrosion experiment is conducted in a normal atmospheric room temperature, which is similar to the actual field environment and is used to examine, evaluate and predict the corrosion extents of both materials.

2.1 Test Samples

Samples of 0.6cm thickness glass reinforced epoxy and carbon steel pipes were collected for this experiment.

2.2 Test Solutions

The selected test solutions are used to represent acidic, basic and salt environments that influenced the corrosion of oil pipelines in real life situation is shown in Table 1.

Table 1: Test Solutions

Test Solutions	Molecular Weight (g)	Amount (ml)	Molar Concentration (M)
H ₂ SO ₄	98	19.6	1
HCl	36.5	7.3	1
NaCl	58.5	11.7	1
BaOH	154.33	30.9	1
H ₂ O	18	neutral	Neutral

2.3 Preparation of Test Specimens

- Cut 0.6cm thickness glass reinforced epoxy and carbon steel pipes into five (5) test specimens each of 3.4cm long and 3.2cm wide.
- Marked each test specimen with identification label.
- Cleaned the test specimens to remove dirt, rinsed in distilled water and dried with acetone.

2.4 Testing of Specimens

- Measured and recorded the weight of the five (5) test specimens each of glass reinforced epoxy and carbon steel materials.
- Immersed each test specimen of both materials in 50ml solutions of 1M H₂SO₄, HCl, NaCl, BaOH and 50ml of distilled water (for control specimens) contained in beakers.

- Removed the test specimens from the beakers in every seven (7) days, washed them with soapy water, rinsed with distilled water and dried with acetone to remove residual test solution and loose corrosion products.
- Then measured and recorded the weight of each test specimen.
- Repeated the process at an interval of 7days over a period of twenty eight (28) days.

3.0: RESULTS AND DISCUSSIONS

From the corrosion test experiment, the results of weight loss and corrosion extents of both glass reinforced epoxy and carbon steel materials due to the influence of the various test solutions are evaluated and represented in Tables 2 to 6 and Table 7 respectively. The corrosion extent (C_E) is calculated from the weight loss per unit surface area of test specimens as shown in (1).

$$: C_E = W/A \quad (1)$$

Where,

C_E = Corrosion extent of test specimen

W = Weight loss in centigrams

A = Area of test specimen

But Area = $2(3.4 \times 3.2 + 3.4 \times 0.6 + 3.2 \times 0.6) = 29.68\text{cm}^2$

3.1.0: Weight Loss Measurement of Specimens

The weight loss of glass reinforced epoxy and carbon steel test specimens in the various test solutions at an interval of 7 days over a period of 28 days are determined using gravimetric analysis and presented in Table 2 to 6..

Table 2: Weight Loss of Test Specimens in 1M H_2SO_4 Solution

		7 days		14 days		21 days		28 days	
Specimen types	Initial (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)
CS pipe	4735	4670	65	4659	76	4650	85	4643	92
GRE pipe	1237	1231	6	1230	7	1229	8	1227	10

Table 3: Weight Loss of Test Specimens in 1M HCl Solution

		7 days		14 days		21 days		28 days	
Specimen types	Initial (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)
CS pipe	4735	4679	56	4670	65	4662	73	4656	79
GRE pipe	1237	1233	4	1231	6	1230	7	1229	8

Table 4: Weight Loss of Test Specimens in 1M $NaCl$ Solution

		7 days		14 days		21 days		28 days	
Specimen types	Initial (cg)	Final (cg)	Weight loss(cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss(cg)	Final (cg)	Weight loss (cg)
CS pipe	4735	4726	9	4720	15	4715	20	4710	25
GRE pipe	1236	1234	2	1232	4	1231	5	1230	0.06

Table 5: Weight Loss of Test Specimens in 1M $BaOH$ Solution

		7 days		14 days		21 days		28 days	
Specimen types	Initial (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)
CS pipe	4734	4726	8	4720	14	4715	19	4710	24
GRE pipe	1237	1236	1	1235	2	1234	3	1232	5

Table 6: Weight Loss of Test Specimens in 1M H_2O Solution

		7 days		14 days		21 days		28 days	
Specimen types	Initial (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)	Final (cg)	Weight loss (cg)
CS pipe	4734	4726	8	4720	14	4715	19	4712	22
GRE pipe	12.36	12.41	- 5	12.39	- 3	1238	- 2	1237	- 1

3.1.1: Calculation of Corrosion Extents of Specimens

The corrosion extents of glass reinforced epoxy and carbon steel test specimens immersed in the various test solutions at regular interval of 7 days over a period of 28 days are determined using (1) and presented in Table 7.

Table 7: Corrosion Extents (cg/cm²) in Test Specimens after 7, 14, 21 and 28 days

	Carbon steel test specimens				Glass reinforced epoxy specimens			
Days	7	14	21	28	7	14	21	28
H_2SO_4	2.19	2.56	2.86	3.10	0.20	0.24	0.27	0.34
HCl	1.89	2.19	2.46	2.66	0.14	0.20	0.24	0.27
$NaCl$	0.30	0.51	0.67	0.84	0.07	0.14	0.17	0.20
$BaOH$	0.27	0.47	0.64	0.81	0.03	0.07	0.10	0.17
H_2O	0.27	0.47	0.64	0.74	- 0.17	- 0.14	- 0.10	- 0.03

3.1.2 Presentation of Corrosion – Time Graph

Fig. 1 to 5 are the presentation of corrosion extents in both glass reinforced epoxy and carbon steel test specimens immersed in various test solutions at an interval of 7 days of a period of 28 days

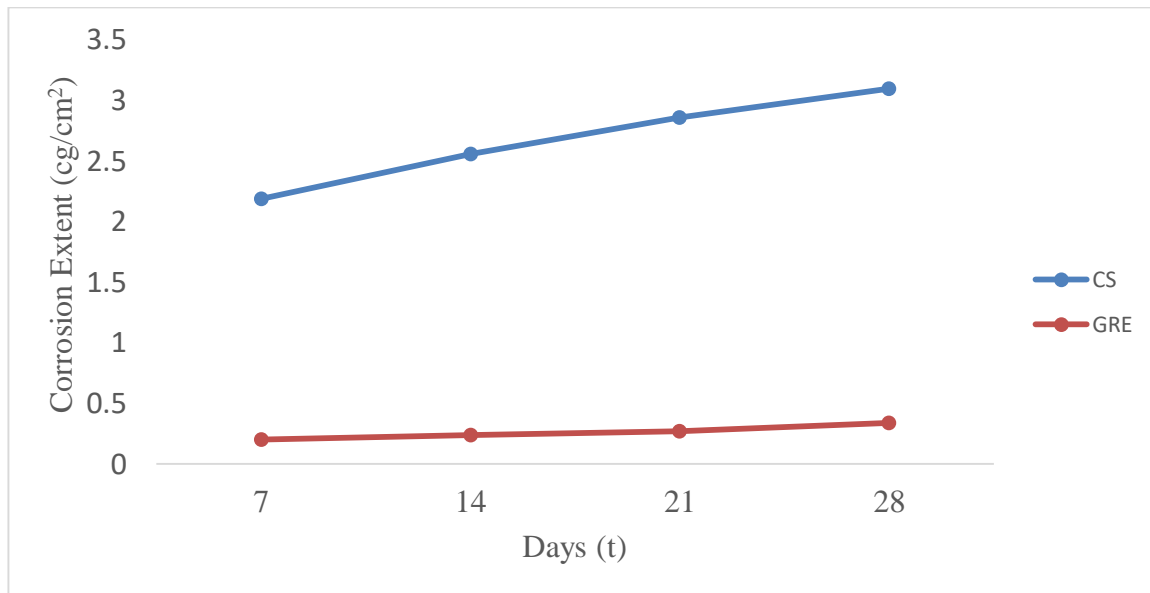


Fig 1: Corrosion extent on glass reinforced epoxy and carbon steel specimen in sulphuric acid

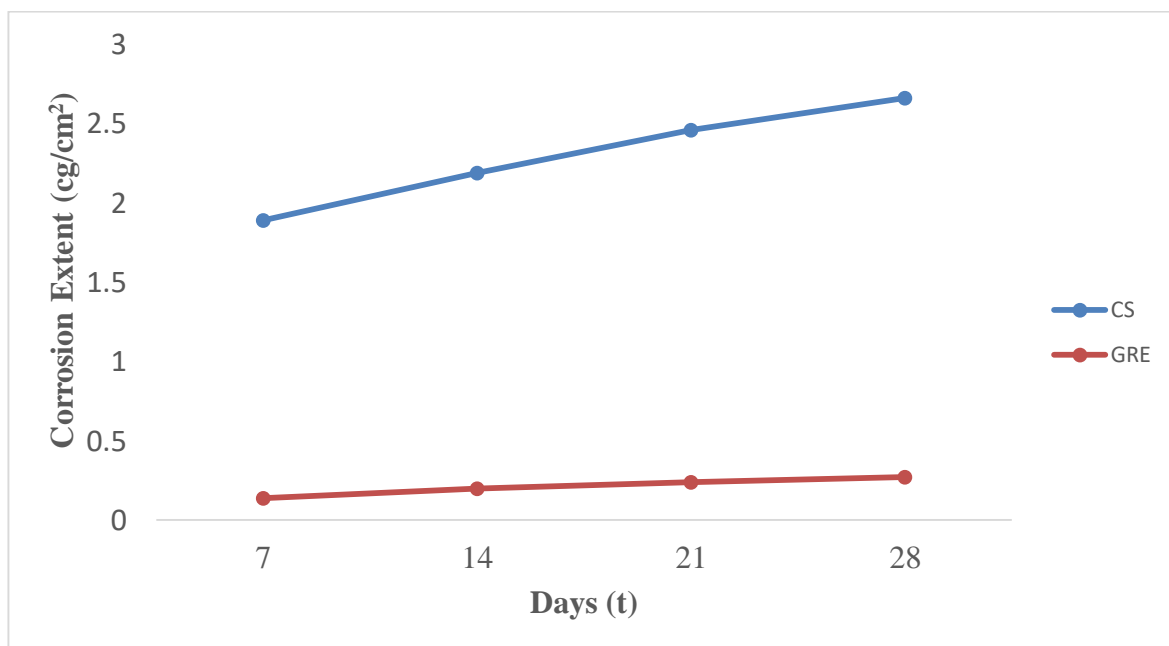


Fig 2: Corrosion extent on glass reinforced epoxy and carbon steel specimens in hydrochloric acid

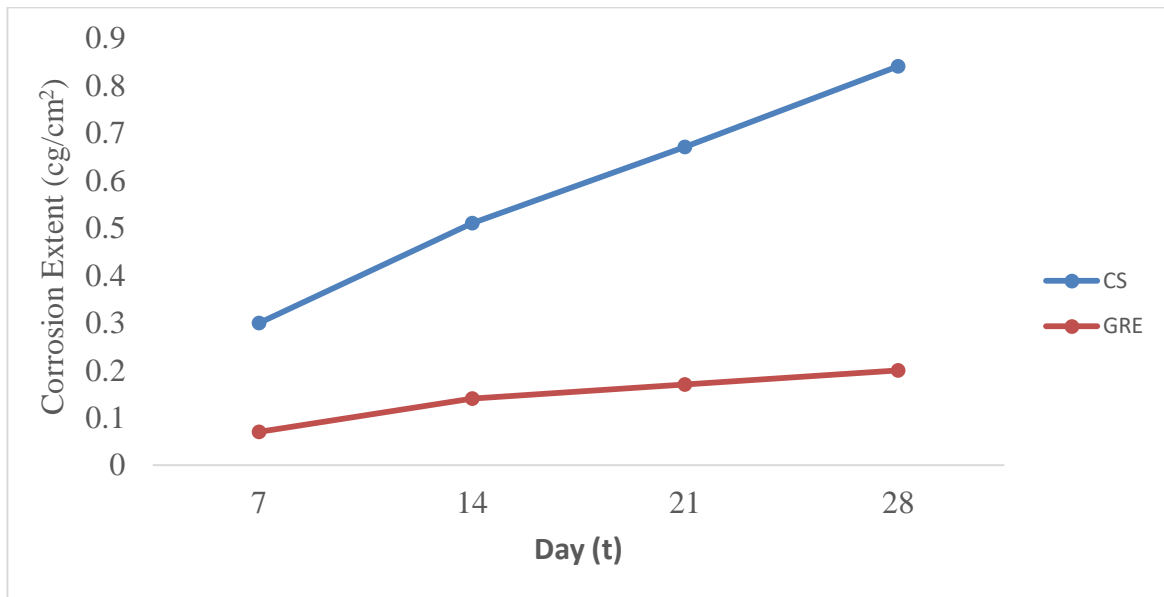


Fig 3: Corrosion extent on glass reinforced epoxy and carbon steel Specimens in sodium chloride

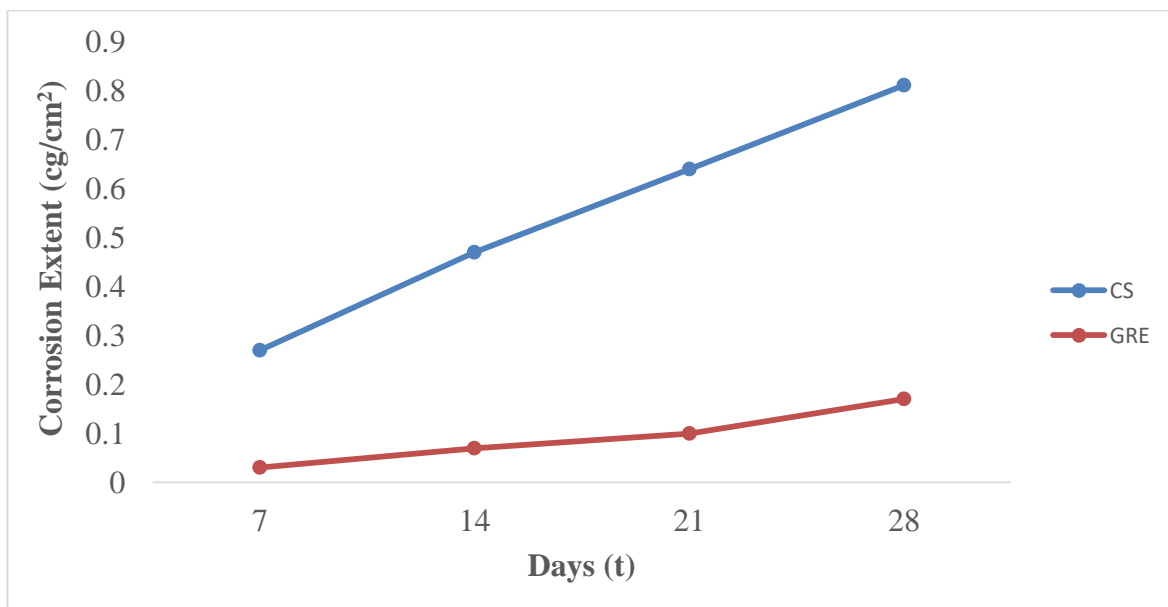


Fig 4: Corrosion extent on glass reinforced epoxy and carbon steel specimens in barium hydroxide

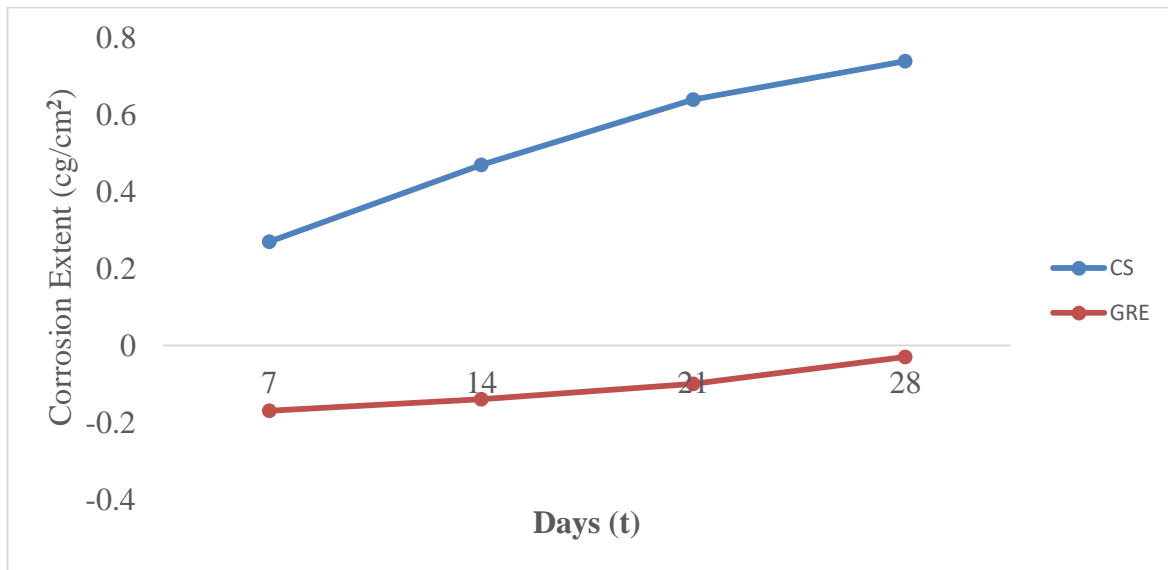


Fig 5: Corrosion extent on glass reinforced epoxy and carbon steel specimens in distilled water

3.1.1.3 Regression Models of Corrosion Trends

The values obtained from the experiment are used to develop statistical models that can be helpful to predict the corrosion extents of glass reinforced epoxy and carbon steel materials over the period of exposure. The corrosion extents in this case is the dependent variable y while the period of exposure is the independent variable represented as t . Applying a second order polynomial regression equation;

$$: y = a_0 + a_1 t + a_2 t^2 \quad (2)$$

The values of a_0 , a_1 and a_2 are obtained by applying the set of simultaneous equations.

$$: \sum y = n a_0 + a_1 \sum t + a_2 \sum t^2 \quad (3)$$

$$: \sum yt = a_0 \sum t + a_1 \sum t^2 + a_2 \sum t^3 \quad (4)$$

$$: \sum yt^2 = a_0 \sum t^2 + a_1 \sum t^3 + a_2 \sum t^4 \quad (5)$$

Where n = number of observational period while a_0 , a_1 and a_2 are unknown variables.

Table 8: Variables for Regression Analysis of Corrosion Extent on Time of Exposure of Carbon Steel in 1M H_2SO_4

Exposure time, t (days)	corrosion extent, y (cg/cm ²)	yt	t^2	yt^2	t^3	t^4
7	2.19	15.33	49	107.31	343	2401
14	2.56	35.84	196	501.76	2744	38416
21	2.86	60.06	441	1261.26	9261	194481
28	3.10	86.80	784	2430.40	21952	614656
$\Sigma = 70$	$\Sigma = 10.71$	$\Sigma = 198.03$	$\Sigma = 1470$	$\Sigma = 4300.73$	$\Sigma = 34300$	$\Sigma = 849954$

Substitute the values of the variables in Tables 8 into (3) to (5) for carbon steel test specimen immersed in Sulphuric acid, we have;

$$: 10.71 = 4a_0 + 70a_1 + 1470 a_2 \quad (6)$$

$$: 198.03 = 70a_0 + 1470a_1 + 34300a_2 \quad (7)$$

$$: 4300.73 = 1470a_0 + 34300a_1 + 849954a_2 \quad (8)$$

Solving for a_0 , a_1 and a_2 using Gaussian Elimination method, we have

$$: y = 1.7575 + 0.0665t - 6.63 \times 10^{-4}t^2 \quad (9)$$

The same method is repeated using the values obtained from the variables for regression analysis of corrosion extent on time of exposure of both carbon steel and glass reinforced epoxy materials in the various test solutions to obtain the statistical models listed in Table 9

Table 9: Statistical Models and R-Squared for Various Experiments.

Corrosion Test	Regression Equations	R-Squared (r^2)
CS in 1M H_2SO_4	$y = 1.7575 + 0.0665t - 6.63 \times 10^{-4}t^2$	$R^2 = 0.9998$
CS in 1M HCl	$y = 1.53 + 0.0547t - 5.1 \times 10^{-4}t^2$	$R^2 = 0.9992$
CS in 1M $NaCl$	$y = 0.085 + 0.03257t - 2.04 \times 10^{-4}t^2$	$R^2 = 0.9932$
CS in 1M $BaOH$	$y = 0.0625 + 0.03093t - 1.53 \times 10^{-4}t^2$	$R^2 = 0.9983$
CS in H_2O	$y = 0.1875 + 0.00107t - 1.53 \times 10^{-4}t^2$	$R^2 = 0.9962$
GRE in 1M H_2SO_4	$y = 0.0575 + 7.143 \times 10^{-5}t + 5.1 \times 10^{-5}t^2$	$R^2 = 0.9645$
GRE in 1M HCl	$y = 0.0675 + 0.0115t - 3.827 \times 10^{-3}t^2$	$R^2 = 0.9924$

GRE 1M <i>NaCl</i>	$y = -0.010 + 0.01314t - 2.04 \times 10^{-4}t^2$	$R^2 = 0.9555$
GRE in 1M <i>BaOH</i>	$y = 0.0175 + 0.00107t - 5.1 \times 10^{-5}t^2$	$R^2 = 0.9629$
GRE in <i>H₂O</i>	$y = 0.0725 - 3.643 \times 10^{-3}t + 1.53 \times 10^{-4}t^2$	$R^2 = 0.9952$

3.1.4 Reliability of the Models

The stability index, also known as the r-squared (coefficient of determination) is used to test the validity of the models using (10) and (11)

$$: r = \sqrt{1 - \left(\frac{S_{yx}}{S_y}\right)^2} \quad (10)$$

$$: r^2 = \left[\sqrt{1 - \left(\frac{S_{yx}}{S_y}\right)^2} \right]^2 \quad (11)$$

$$: S_{yx} = \sqrt{\sum_{i=1}^n \frac{(y_i - y_{ic})^2}{n-3}} \quad (12)$$

$$: S_y = \sqrt{\sum_{i=1}^n \frac{(y_i - \bar{y})^2}{n-1}} \quad (13)$$

where,

y_i = actual experimental value of y of the data set

y_{ic} = value of y computed from the derived models

$n-3$ = degree of freedom, as the number of regression parameters is three: a_0 , a_1 and a_2 .

\bar{y} = sample mean

In order to make it easier to evaluate the correlation coefficient and the coefficient of determination (R-squared) which are useful in testing the reliability of each statistical model, Table 10 is compiled from the actual values of the experiments and the calculated values of the models. This method is repeated to obtain the variables for calculating R-squared of both carbon steel and glass reinforced epoxy specimens immersed in the respective test solutions.

The values obtained are substituted into (12) and (13). For instance, carbon steel test specimen immersed in sulphuric acid will give the following;

Table 10: Variables for Calculating R-Squared of CS Specimen Immersed in H₂SO₄

i	t _i	y _i	(y _i - \bar{y})	(y _i - \bar{y}) ²	y _{ic}	(y _i - y _{ic})	(y _i - y _{ic}) ²
1	7	2.19	0.4875	0.2377	2.1905	-5.0x10 ⁴	2.50x10 ⁷
2	14	2.56	-0.1175	0.0138	2.5585	1.5x10 ³	2.25x10 ⁶
3	21	2.86	0.1825	0.0333	2.8615	-1.5x10 ³	2.25x10 ⁶
4	28	3.10	0.4225	0.1785	3.0995	5.0x10 ⁴	2.50x10 ⁷
		Σ=10.71		Σ=0.4633			Σ=5.0x10 ⁶

$$\bar{y} = \frac{10.71}{4}$$

$$= 2.6775$$

$$S_{yx} = \sqrt{\frac{5.1 \times 10^6}{1}} = 0.002236$$

$$S_y = \sqrt{\frac{0.4633}{3}} = 0.1544$$

$$r = \sqrt{1 - \left(\frac{0.002236}{0.1544}\right)^2}$$

$$r = \sqrt{1 - 0.000209}$$

$$r = 0.9999$$

$$r^2 = 0.9998$$

The coefficients of determination (r² squared) of other test specimens in their respective test solutions are obtained as listed in Table 9 using the same method.

3.2. DISCUSSIONS

From the experimental results, it was found that the corrosion extents of carbon steel test specimens in 50ml solutions of 1M H_2SO_4 , HCl, NaCl, BaOH and distilled water after observational period of 28 days were 3.10 cg/cm^2 , 2.66 cg/cm^2 , 0.84 cg/cm^2 , 0.81 cg/cm^2 and 0.74 cg/cm^2 respectively while the corrosion extents of glass reinforced epoxy test specimens immersed in 50ml of the same solutions over the same period of time were 0.34 cg/cm^2 , 0.27 cg/cm^2 , 0.20 cg/cm^2 , 0.17 cg/cm^2 and 0.03 cg/cm^2 respectively, indicating that carbon steel material is highly vulnerable to corrosion than glass reinforced epoxy material.

The results also showed a general pattern of increase in corrosion extents and a corresponding decrease in corrosion rates in the test specimens of both materials as immersion period increased. The reduction in the corrosion rate of both test specimens is as a result of the formation of protective oxide film from the initial corrosion products on the surface of the test specimens. Table 2 to 6 and Table 7 respectively showed that the weight loss and corrosion extents of carbon steel test specimens immersed in the various test solutions under the observational period of 7, 14, 21 and 28 days are higher than that of glass reinforced epoxy test specimens over the same period of exposure and the same test media, indicating that glass reinforced epoxy pipe is immune to corrosion.

Fig. 1 shows that sulphuric acid as a corrosive medium is more effective corrosive solution on both specimen than hydrochloric acid and other corrosive media used for the experiment due to its extent of corrosion and weight loss of the test specimens. Table 6 and Fig. 5 showed weight increase in glass reinforced epoxy specimen exposed to distilled water unlike carbon steel test specimen, indicating that glass reinforced epoxy pipe material has better corrosion resistant than carbon steel pipe in the same medium.

It was also observed that the initial weight loss of both material was high due to the absent of protective oxide layers on the surfaces of the test specimens. With further exposure to the test media, there was gradual reduction in the weight loss, especially glass reinforced epoxy test specimen immersed in sodium hydrochloride and barium hydroxide as shown in Table 4 and 5. Also, statistical models are developed from the experimental results, which can be helpful to predict the corrosion extents of both materials with time in the different environments. The calculated coefficients of correlation and coefficients of determination (R-squared) indicated perfect agreement between the models predicted values and the experimental values of the corrosion extents.

4: CONCLUSIONS

From the experimental analysis of the corrosion test conducted on both glass reinforced epoxy and carbon steel materials, the weight increase in glass reinforced epoxy test specimen exposed to distilled water is a proof that glass reinforced epoxy pipe does not easily corrode in the presence of water like that of carbon steel pipe in the same medium. Instead of it to corrode in the presence of water, it rather absorbs the water.

Also, from the results of the experiment, the weight loss and corrosion extent of glass reinforced epoxy test specimen in the various test solutions is very insignificant as compared to the weight loss and corrosion rate experienced by the carbon steel test specimen, thereby justifying the use of glass reinforced epoxy pipe as an effective control measure for oil pipelines corrosion.

This research work present glass reinforced epoxy pipe as a superior alternative to carbon steel pipe for the transportation of hydrocarbon products from one location to the others in the Niger Delta region in Nigeria. Furthermore, the corrosion extents of carbon steel and glass reinforce specimens of the test compositions and environments within the values obtained from the experiment can be predicted using the derived statistical models

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