

Characterization and Corrosion of Work-Hardened Aluminium Nano-Titania Composite in Sulphuric Acid (H_2SO_4): A Case Study in Federal Polytechnic of Oil and Gas Bonny, River State Nigeria

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Abstract

This study focuses on the characterization and corrosion of work-hardened aluminum nanotitania composite in H_2SO_4 media. One of the challenges faced by scientists and engineers is preventing the collapse of metal objects, chemical processing plants, and structures due to corrosion, which not only results in financial losses but also harms the environment and people. The corrosion of work-hardened aluminum nanotitania composite in an aqueous solution of H_2SO_4 at high and low concentrations (0.3M and 0.05M, respectively) was determined by weight loss methods and potentiodynamic polarization. Additionally, surface analysis of the corrosion products of work-hardened aluminum nanotitania composite using scanning electron microscopy (SEM) to determine the morphological changes. Research revealed that the kind and amount of media the samples were submerged in had a substantial impact on their ability to withstand corrosion.

Keywords: Aluminium, Nanotitania, Potentiodynamics, Polarization, Corrosion

1.0 INTRODUCTION

Due to its exceptional qualities, aluminium alloy is one of the lightweight materials that people use most frequently in their daily lives. First off, aluminium alloy weighs only one-third of the amount of iron or steel that is often utilized. This makes it incredibly light. Alloys made of aluminium have a high specific strength despite their low density. Aluminium alloys' high strength-to-weight ratio is very desirable, particularly in the automotive and aerospace industries. Because stable Al_2O_3 frequently forms on its surface when exposed to certain environments, including chromic acid and phosphoric mixes, industrial air gases, sulfur compounds, ammonia, etc., it is also known to be resistant to corrosion in most of these situations. Aluminium and its alloys (AA 1xxx series to AA 8xxx series) have been used in many industries, including packaging,

structural materials, transportation, and the aerospace industry, thanks to these properties (Feyisayo, Samuel, Babatunde, & Peter, 2021; Rambau, Popoola, Loto, Mathebula, & Theron, 2013).

According to Deepa and Padmalatha (2017), researchers have focused a great deal of emphasis on aluminium and its alloys' corrosion studies because of their numerous industrial uses and favourable economic characteristics. Frequently exposed to acids or bases during cleaning, descaling, and electrochemical corrosion, they are extensively utilized in numerous chemical processing sectors due to their broad application. The corrosion of different metals and alloys in HCl and H₂SO₄ environments has been the focus of the majority of reported investigations. (Obi-Egbedi, Obot, & Umoren, 2012; Anozie, Avoaja, Akoma, & Eti, 2011; Deepa & Padmalatha, 2017). Studying the corrosion resistance of aluminium and its alloys in many conditions is crucial due to the wide range of industrial applications for these materials. Various organic acid solutions, including butyric, tartaric, acetic, propionic, gluconic, citric, and malic acids, will be processed using apparatus composed of aluminium and its alloys (Ghali, 2011; Feyisayo, Samuel, Babatunde, & Peter, 2021; Akhil, Khurshid, & Manikanta, 2016).

2.0 LITERATURE REVIEW

Composites are materials composed of two or more separate constituents, each having unique qualities. They combine to form a material whose characteristics are different from those of its constituent parts. One or more discontinuous reinforcements and a continuous matrix make up the composite's constituent parts. The matrix includes reinforcements and gives the finished product its general shape. All or a portion of the load imparted to the composite material is supported by reinforcements (Rajan, Pillai, & Pai, 1998). The precise characteristics, form, orientation, and quantity of reinforcement incorporated into a composite determine its overall characteristics. More dependable composites are frequently utilized in radars, rocket and jet engines, turbine blades, wing fans, helicopter rotor shafts, automobile engines, car bodies, connecting rods, and other components because of their excellent strength-to-weight ratio and resistance to fatigue and corrosion (Prasad & Asthana, 2004). Metal matrix composites are made of fibers or particles that are reinforced within a pure metal matrix or metal alloy. According to Mavhungu, Alinlabi, Onitiri, and Varachia, "Aluminium Matrix composites for industrial use: Advances and Trends, 2017," silicon carbide, titanium oxide, or aluminium oxide particles or fibers are frequently utilized as reinforcements in commercial alloys of magnesium, titanium, and aluminium. Nanocomposite materials are created by adding nanoscale reinforcements to the matrix, which is another advance in composite manufacturing. Comparing nanocomposites to other synthetic materials, their better mechanical strength, toughness, and thermal characteristics are their key advantages. Tribological and mechanical characterisation of aluminium metal matrix composites, Padmavathi & Ramakrishnan, 2016). Composite materials made of aluminium with a volume ratio of up to 70% that are reinforced with other components. By adjusting the types of matrix and reinforcement, reinforcements can be added to satisfy the requirements of specific industrial components. These can be continuous or discontinuous fibers, whiskers, or particulates (Surappa, 2003).

2.2 Aluminium metal matrix composites.

Corrosion Test. For assessing corrosion, various techniques are employed. One way to classify laboratory test methods is electrochemical, which measure the current that results from electrochemical reactions. On the other hand, non-electrochemical techniques rely on the specimen's overall weight loss or surface features like pits, cracks, and microstructural changes that appear after the test due to corrosion.

Weight Loss Test. It is not advisable to use the weight-loss tests to forecast localized attack because they only provide information on a material's resistance to uniform corrosion. Prudent selection of the corrosion test method is necessary; for example, this method could not be appropriate if chosen to evaluate resistance localized corrosion. The latter causes tiny pinholes to grow on the surface, which frequently result in failure (perforation and leaking) and minimal weight reduction. The corrosion rate can be expressed in millimeters per year (mm/y), mils per year (mpy), or other metric units using the data from uniform corrosion. The examination involves preparing the specimens (ASTM, 2020) by polishing, cleaning, and degreasing them. Next, each specimen is weighed, measured, and identified. Large test coupons that can both fit inside the test chamber and provide a noticeable mass loss are ideal. The foundation substrate may be lost in material due to the cleaning process itself. By putting the new cleaning coupon through several cycles, it is possible to overcome such inaccuracies in mass loss. Following the test, the mass loss can be used to calculate the corrosion rate as follows:

$$\text{Corrosion Rate} = (K \times W) / (A \times T \times D)$$

Where;

K = Constant

T = time of exposure, h,

A = area, cm²

W = mass loss, g, and

D = density, g/cm³ (D for steels and stainless steels -7.9 g/cm³, for copper 8.9 g/cm³ for aluminium = 2.7g/cm³).

K = 3.45 x 10⁶ for a corrosion rate in mpy.

Electrochemical Tests Methods: An electrochemical cell with a reference electrode (such as saturated calomel or silver-silver chloride), a counter electrode (such as platinum or graphite), a working electrode (a test specimen), and an experimental solution are required for conducting electrochemical tests. A potentiostat or galvanostat is a device for measuring and applying accurate current and potentials. The specimen-solution interface's potential is measured by the reference electrode, while the counter electrode promotes the flow of electrochemical current

Work Hardening: The majority of machining and shaping processes that are performed on aluminium and its alloys naturally cause work hardening. The strength gained by solid solution and dispersion hardening is increased by the hardening procedure. Work hardening enhances the precipitation strength and heightens the alloy's susceptibility to precipitation hardening in heat-

treatable alloys. In order to give non-heat treatable alloys a strain-hardened temper, work hardening is frequently utilized. Aluminium and its alloys deform due to normal crystal slippage. If the surfaces are polished prior to deformation, evidence of such slippage can be observed in single crystals and coarse-grained materials. A harsher cold working procedure would result in an even greater dislocation density and a smaller piece. The main causes of strain hardening that result from lattice distortions are dislocations and the interaction stresses that arise between them (Total Materia, 2007).

3.0 METHODOLOGY

3.1 Materials and Preparation

The basic metal, aluminum 6063, was acquired from Nigeria Aluminium Extrusion Company (NIGALEX) in Oshodi, Lagos, and titanium dioxide nanoparticles were procured from sciencecommodities.com for this investigation. Stir casting was used to create the 0%, 1%, 2%, 3%, and 5% aluminum nano-titania composite at the Federal Polytechnic of Oil and Gas bonny Rivers State, Foundry Workshop.

3.1.1 Preparation of the Samples

The cast was machined into the following dimensions: length 40mm, breadth 20mm, and thickness 20mm. Then it was work hardened using 5kg and 2kg hammer respectively on each of the each of the composite 0%, 1%, 2% 3% and 5% Aluminium nano-titania. the samples obtained after the work hardening operation were cut to 10 x 10 x 10mm for corrosion test and 20 x 10 x 10mm for SEM. The final operation, employing a manual grinder, was required for material deburring and cleanup. Were given The five separate reinforcement samples are then divided into two samples each, with an average length of 20 mm, which are used for the acid, salt water, and base environments. Then, the specimen is subjected to rough, fine, rough polishing, and finishing polishing using a metallographic grinding and polishing equipment. Emery papers with varied grits, including 240, 320, 400, and 600grit, are used for grinding. The ten samples are polished using 600 SiC materials and emery cloths

3.1.2 Equipment's, Apparatus for Weight Loss Measurement

Equipment, apparatus and reagents used include small plastic containers, weighing balance, hand grinder, funnel, hack saw blade, measuring cylinder, beaker, (H₂SO₄) corrosion media, Metallographic grinder and polisher.

3.1.3 Weight Loss Corrosion Measurement

In this study, two different corrosion media (0.05 M and 0.3 M of H₂SO₄) were used as the corrosion environment to evaluate the corrosion behaviour of Aluminium nano-titania composite with different weight percentages (0%, 1%, 2%, 3% and 5%). Before the samples into the solutions, the weight of each sample was taken before subjecting to the corrosion environment. The weight of each of the samples after immersing where taken through the course of 30days .



Plate 3.1: Stir Cast Aluminium nano-titania Composite



Plate 3.2: Polishing operation



Plate 3.3: Digital weighing balance

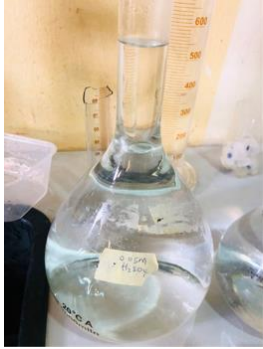


Plate 3.4: Glass cylinder containing 0.05M H₂SO₄



Plate 3.5: Glass cylinder containing 0.3M H₂SO₄

3.1.4 Procedure of Calculating Corrosion rate for Weight Loss Measurements

$$K = 8.76 \times 10^4$$

$$\text{Density} = 2.7 \text{ g/cm}^3$$

$$t = \text{days} \times 24 \text{ hrs}$$

Area = 100 mm² converted to 0.1 cm² for us to have corrosion rate unit to be mm/y.

Therefore, $(K \times W) / (A \times t) = \text{Corrosion Rate (CR)}$ - - - - (i)

Where:

K = a constant determined by the units used.

W = weight loss due to corrosion.

A = surface area metal

t = time exposure to corrosive environment.

3.2 Electrochemical Method of Corrosion Measurement

3.2.1 Sample Preparation for PDP tests

The specimens used were carefully prepared by machining and polishing them with different grits of emery papers, including 60, 120, 400, and 800grits. They were then kept in a desiccator prior to analysis. The specimens were then connected to copper wire for electrochemical measurements. The surfaces of samples from the corrosion test were selected for SEM examination.

3.2.2 Electrochemical Measurements by PDP

Using an Autolab PGSTAT 302N computer-controlled potentiostat/galvanostat, a traditional three-electrode cell was utilized in the Potentiodynamic Polarization (PDP) approach. Counter electrode (CE) made of platinum was utilized.

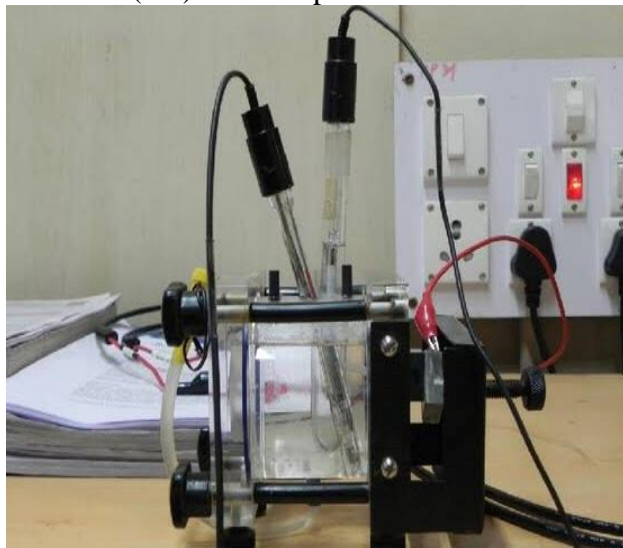


Plate 3.6 : Electropotentiodynamic polarization machine

The prepared sample serves as the working electrode (WE) and Ag/AgCl as the reference electrode (RE). About 1 cm² was the area of the WE exposed to the media. The electrode was left to corrode freely for up to two minutes prior to each electrochemical research, during which time its open circuit potential (OCP) was measured as a function of time. This was enough time to achieve a stable state. Following this, a steady-state OCP was established that matched the working electrode's corrosion potential (corr).

3.3 SEM Examination of Corroded Samples

Using an FEI Quanta 200F scanning electron microscope, backscattered electron (BSE, atomic Z-contrast) investigation was carried out on the Al matrix composite. Selecting micrographs at magnifications of 10,000, 15,000, and 20,000 times magnification, as well as an aperture of 3-5 and a spot size of 4, the SE mode was used for the SEM study.

4.0 RESULTS AND DISCUSSIONS

4.1 Results

4.1.1 Corrosion Test Results by Weight Loss Method

Table 4.1 Result of weight loss of Work hardening (2kg load) in 0.05M H₂SO₄ for 30 days

Days	0%	1%	2%	3%	5%
0	3.9701	4.0738	3.1462	1.9654	2.4275
1	3.9649	4.0668	3.1360	1.9634	2.4229
2	3.9608	4.0606	3.1239	1.9599	2.4196
3	3.9566	4.0546	3.1174	1.9573	2.4135
4	3.9537	4.0530	3.1053	1.9569	2.4100
5	3.9483	4.0401	3.0887	1.9514	2.4025
6	3.9438	4.0346	3.0770	1.9535	2.3993
7	3.9412	4.0231	3.0582	1.9454	2.3967
8	3.9372	4.0171	3.0493	1.9430	2.3961
9	3.9351	4.0109	3.0401	1.9408	2.3950
10	3.9326	4.0010	3.0399	1.9430	2.3950
11	3.9371	3.9986	3.0356	1.9429	2.3996
12	3.9358	3.9940	3.0297	1.9429	2.3992
13	3.9323	3.9893	3.0146	1.9410	2.3992
14	3.9308	3.9839	3.0129	1.9384	2.3965
15	3.9308	3.9809	3.0155	1.9396	2.4025
16	3.9296	3.9778	3.0008	1.9376	2.4008

17	3.9302	3.9760	3.0103	1.9376	2.3997
18	3.9298	3.9744	3.0021	1.9384	2.3979
19	3.9274	3.9740	3.0000	1.9382	2.3977
20	3.9300	3.9703	2.9996	1.9380	2.3976
21	3.9302	3.9688	2.9951	1.9379	2.3975
22	3.9302	3.9671	2.9897	1.9382	2.3975
23	3.9302	3.9660	2.9840	1.9384	2.3975
24	3.9299	3.9648	2.9819	1.9389	2.3974
25	3.9299	3.9636	2.9803	1.9385	2.3978
26	3.9302	3.9627	2.9887	1.9399	2.3979
27	3.9302	3.9599	2.9862	1.9389	2.3976
28	3.9302	3.9596	2.9782	1.9386	2.3970
29	3.9303	3.9596	2.9744	1.9391	2.3970

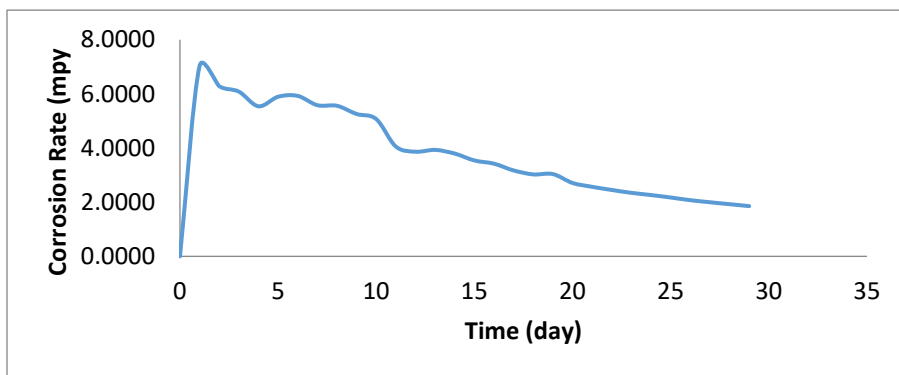


Fig 4.1 A plot of Corrosion rate against Time for Work hardening (2kg load) sample 0% in 0.05M H₂SO₄

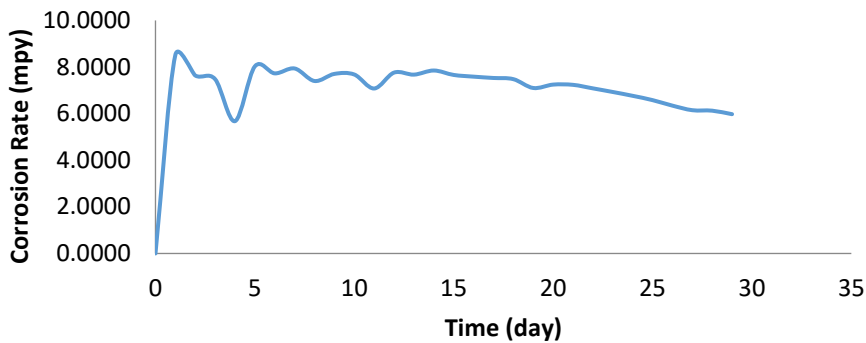


Fig 4.2 A plot of Corrosion rate against Time for Work hardening (5kg load) sample 0% in 0.05M H₂SO₄

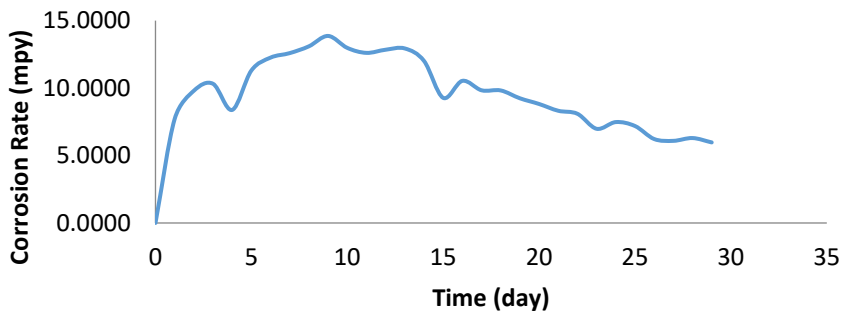


Fig 4.4: A plot of Corrosion rate against Time for Work hardening (5kg load) sample 1% in 0.05M H₂SO₄

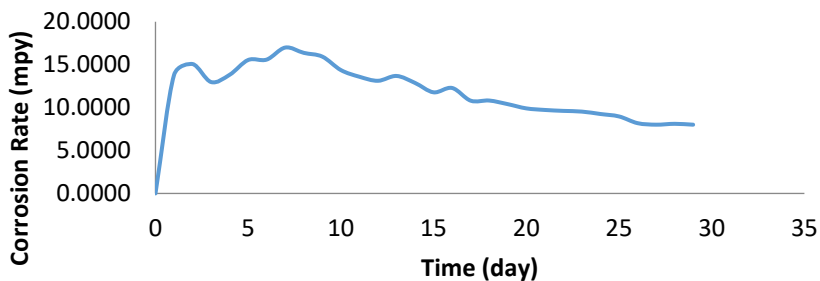


Fig 4.5: A plot of Corrosion rate against Time for Work hardening (2kg load) sample 2% in 0.05M H₂SO₄

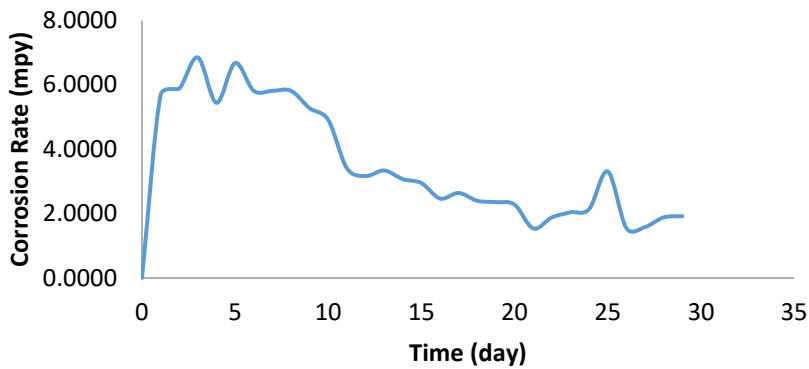


Fig 4.6: A plot of Corrosion rate against Time for Work hardening (5kg load) sample 2% in 0.05M H₂SO₄

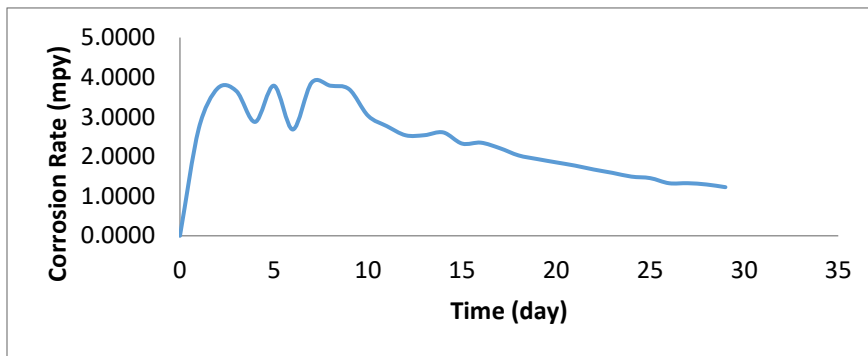


Fig 4.7: A plot of Corrosion rate against Time for Work hardening (2kg load) sample 3% in 0.05M H₂SO₄

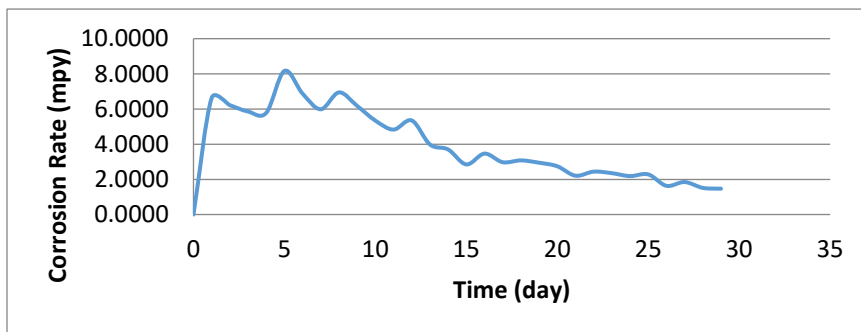


Fig 4.7: A plot of Corrosion rate against Time for Work hardening (5kg load) sample 3% in 0.05M H₂SO₄

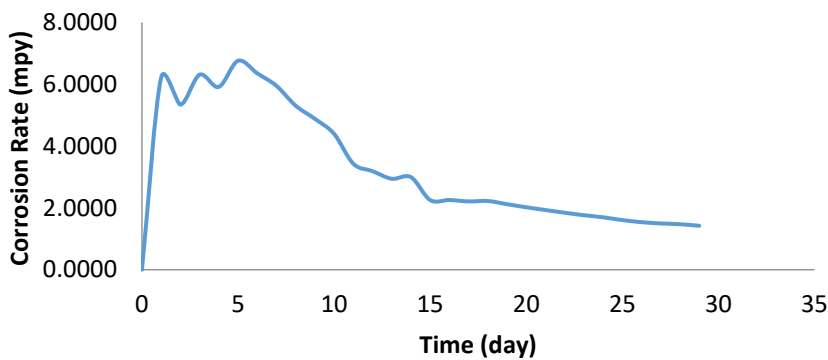


Fig 4.9: A plot of Corrosion rate against Time for Work hardening (2kg load) sample 5% in 0.05M H₂SO₄

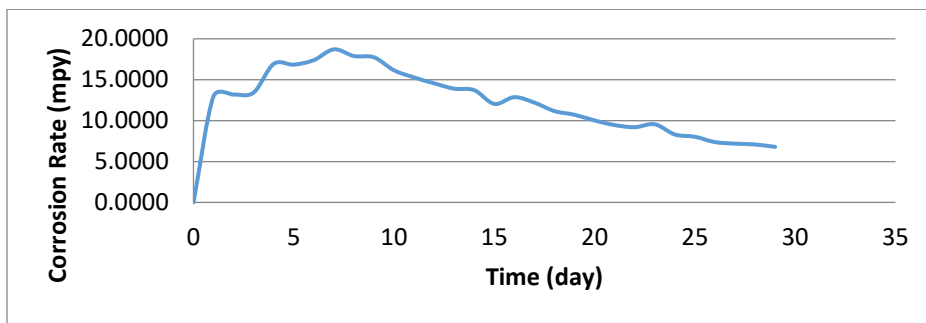


Fig 4.10: A plot of Corrosion rate against Time for Work hardening (5kg load) sample 5% in 0.05M H₂SO₄

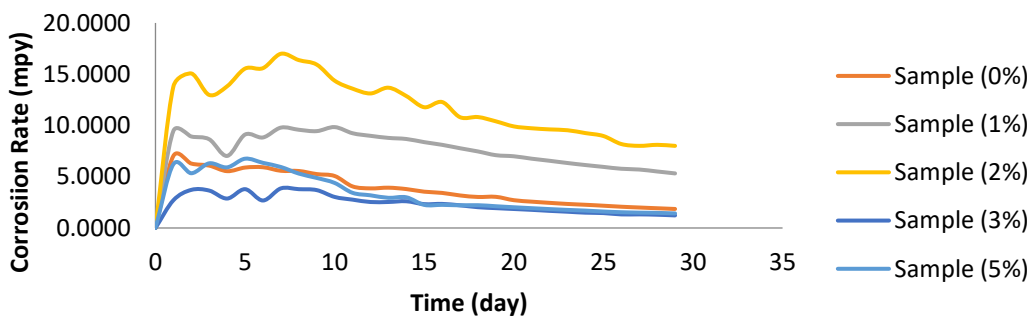


Fig 4.11: Comparative of the Corrosion rate against Time for Work hardening (2kg load) of samples (0%, 1%, 2%, 3% and 5%) in 0.05M H₂SO₄

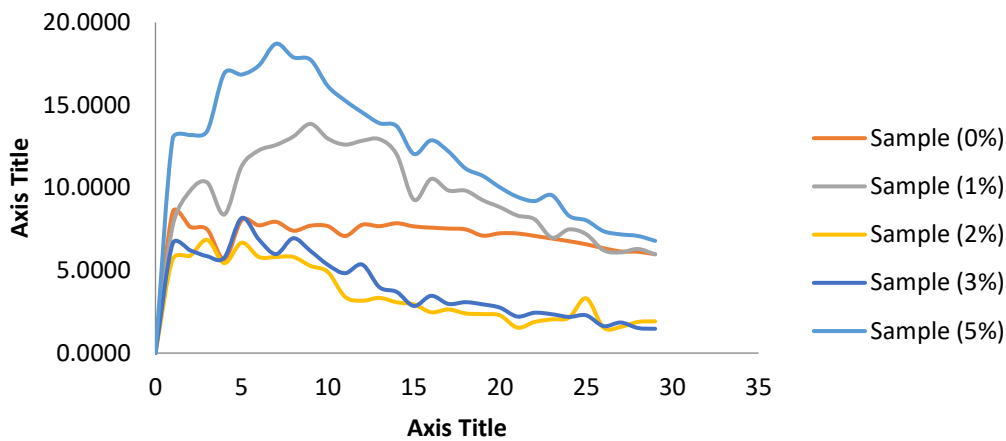


Fig 4.12: Comparative of the Corrosion rate against Time for Work hardening (5kg load) of samples (0%, 1%, 2%, 3% and 5%) in 0.05M H₂SO₄

4.1.2 Potentiodynamic polarization Result

Table 4.15 Corrosion properties of Work hardening (2kg load) of Sample (0%, 1%, 2%, 3% and 5%) in 0.05M H₂SO₄ by Potentiodynamic polarization

Sample	0%	1%	2%	3%	5%
E _{corr} (V)	-0.72719	-0.66182	-0.73589	-0.68902	-0.72591
I _{corr} (A/cm ²)	0.00008	0.00002	0.00003	0.00004	0.00002
Corrosion rate (mm/yr)	0.93664	0.27629	0.36107	0.493	0.27902
Polarization Resistance(Ω)	200.44	479.77	472.44	375.45	571.69
ba (V/dec)	0.08378	0.059793	0.07483	0.079817	0.068721
bc (V/dec)	0.066917	0.046846	0.061652	0.067863	0.058528

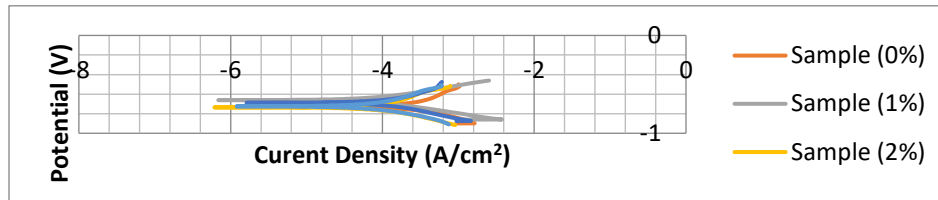


Fig. 4.13 Tafel extrapolation for Work hardening (2kg load) sample (0%, 1%, 2%, 3% and 5%) in 0.05M H₂SO₄ by Potentiodynamic polarization

Table 4.16 Corrosion properties of Work hardening (5kg load) Sample (0%, 1%, 2%, 3% and 5%) in 0.05M H₂SO₄ by Potentiodynamic polarization

Sample	0%	1%	2%	3%	5%
E _{Corr} (V)	-0.72313	-0.77487	-0.69391	-0.67552	-0.70806
j _{corr} (A/cm ²)	0.000046	0.000017	0.000012	0.000019	0.000017
Corrosion rate (mm/yr)	0.53047	0.202212	0.14439	0.22081	0.19842
Polarization Resistance(Ω)	307.48	950.96	1404.2	711.92	927.6
ba (V/dec)	0.067744	0.08302	0.088813	0.0632	0.080554
bc (V/dec)	0.061814	0.070374	0.073369	0.061425	0.066649

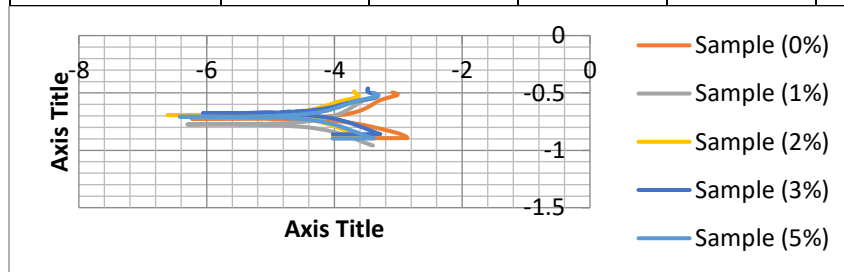
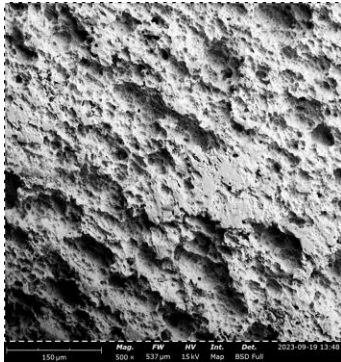


Fig. 4.14 Tafel extrapolation for Work hardening (5kg load) sample (0%, 1%, 2%, 3% and 5%) in 0.05M H₂SO₄ by Potentiodynamic polarization.

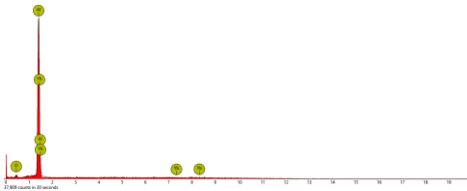
4.1.3 SEM EDX Result

Table 4.17: SEM EDX Result for Work hardening (2kg load) sample 0%, in 0.3M H₂SO₄

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
13	Al	Aluminium	83.58	87.87
8	O	Oxygen	16.11	10.04



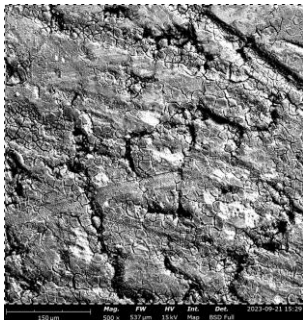
FOV: 537 μm, Mode: 15kV - Map, Detector: BSD Full, Time: SEP 19 2023 13:48



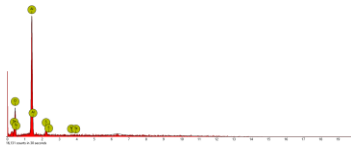
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Table 4.17: SEM EDX Result for Work hardening (5kg load) sample 2%, in 0.05M H₂SO₄

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	55.80	41.89
13	Al	Aluminium	41.01	51.93
16	S	Sulfur	2.88	4.34



FOV: 537 μm, Mode: 15kV - Map, Detector: BSD Full, Time: SEP 21 2023 15:29



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Table 4.17: SEM EDX Result for Work hardening (5kg load) sample 5%, in 0.05M H₂SO₄

Comparing the results

- Fig 4.1 A plot of Corrosion rate against Time for Work hardening (2kg load) sample 0% in 0.05M H₂SO₄, shows moderate resistance.
- Fig 4.2 A plot of Corrosion rate against Time for Work hardening (5kg load) sample 0% in 0.05M H₂SO₄ has the minimum weight loss, indicating it is the least resistant to corrosion in 0.05M H₂SO₄
- Fig 4.4: A plot of Corrosion rate against Time for Work hardening (5kg load) sample 1% in 0.05M H₂SO₄, has the highest weight loss, indicating it is the least resistant to corrosion in 0.05M H₂SO₄.
- Fig 4.5: A plot of Corrosion rate against Time for Work hardening (2kg load) sample 2% in 0.05M H₂SO₄ has the medium weight loss, indicating it is the least resistant to corrosion in 0.05M H₂SO₄
- Fig 4.6: A plot of Corrosion rate against Time for Work hardening (5kg load) sample 2% in 0.05M H₂SO₄ has the average weight loss, indicating it is the least resistant to corrosion in 0.05M H₂SO₄
- Fig 4.7: A plot of Corrosion rate against Time for Work hardening (2kg load) sample 3% in 0.05M H₂SO₄, has the medium weight loss, indicating it is the least resistant to corrosion in 0.05M H₂SO₄
- Fig 4.7: A plot of Corrosion rate against Time for Work hardening (5kg load) sample 3% in 0.05M H₂SO₄. has the lowest weight loss, indicating it is the least resistant to corrosion in 0.05M H₂SO₄.
- Fig 4.9: A plot of Corrosion rate against Time for Work hardening (2kg load) sample 5% in 0.05M H₂SO₄ has the average weight loss, indicating it is the least resistant to corrosion in 0.05M H₂SO₄.

5.0 CONCLUSION

Drawing from the findings of the corrosion test, it can be inferred that the samples' corrosion rate and efficiency differed considerably based on the kind of media. When juxtaposed with 0.3M H₂SO₄ solution, the corrosion rates of the samples submerged in 0.05M H₂SO₄ solution were comparatively low. In summary, the kind and amount of media the samples were submerged in had a substantial impact on their ability to withstand corrosion.

5.1 Recommendation

To ascertain the ideal concentration and kind of reinforcement for particular applications and settings, more research is advised. Further research is advised to completely understand the corrosion behaviors of the reinforced alloy by taking into account the influence of additional variables including temperature, humidity, and the presence of other chemicals in the surrounding environment. It is also advised that the samples be evaluated for a longer duration in order to obtain more accurate and trustworthy results.

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