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Mechanism of Corrosion Inhibition of Aluminium in Potassium Hydroxide Using 2.0 M Thiourea.

Okeke P. I., Enemo E. R., Ezenwa T. E.
Department of Pure and Industrial Chemistry, Chukwuemeka Odumegwu Ojukwu University Uli, Anambra State, Nigeria.
Corresponding author: Okeke P. I

Abstract: *The mechanism of corrosion inhibition of aluminium by 2 M thiourea in 0.08 M potassium hydroxide was investigated using gasometric, gravimetric and scanning electron microscope (SEM) methods. Synergistic measurements were also carried out, The result showed that the rate of hydrogen gas evolution and weight loss was greatly reduced while inhibition efficiency increased. These effects were seen to increase as time prolonged. SEM results showed the vivid presence of the absorbed inhibitor molecules on the surface of the metal protecting it from corrosion attack.*
Keywords*: Corrosion inhibition, Aluminium, KOH, Gasometric, Gravimetric, SEM.*

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I. Introduction

Corrosion occurs as a result of the deterioration of metals and its related alloys due to electrochemical reaction with its immediate environment [1-2]. Aluminium is of notable economic, industrial and environmental importance. It is also a useful material for engineering applications due to its low cost, high electrical and thermal conductivity. Its light weight makes it of great choice in aircraft production. Aluminium and its alloys are commonly put to use in many industrial settings such as pharmaceutical industries, food industries, chemical batteries, pipes and reaction vessels. The corrosion of aluminium in alkaline environment can be controlled or reduced by the addition of inhibitors [3-5]. Protection of metals from corrosion using inhibitors has been applied to many systems such as in refinery units, oil and gas production units, boilers, aircraft units, etc. The choice of an appropriate inhibitor for a type of system is actually complicated. This is as a result of their specificity and so many varieties of corrosion-related applications. The highly corrosive nature of an alkaline medium on aluminium requires a good degree of protection to achieve economic maintenance, maximum safety, minimal loss of materials, quality and continuous operation of equipments.

The development of corrosion inhibitors of non-toxic type, with absence of heavy metals and inorganic phosphates is now of a high importance [6-9], inorganic compounds such as nitrates and chromates have been largely used as corrosion inhibitors for different metals in several media, however, the toxicity and non environmentally friendly nature of these inorganic compounds especially chromates have been well reported which limits their applications [10-13]. Effective inhibitors are expected to perform optimally under wide range of conditions, this necessitates special care and attention in the selection of inhibitors for applications of various kinds.

II. Experimental section

2.1 Materials preparation

The aluminium alloy AA1060 used for this experiment is 0.045cm in thickness and composition in weight % as follows: Si (0.35), Fe (0.7), Cu (0.15), Mn (0.05) and Al (balance). The specimen was mechanically pressed cut into 2 x 2 cm coupons. The two faces had a total geometric surface area of 8.0 cm^2. The coupon were degreased in absolute ethanol, dried in warm air, and stored in moisture free desiccators before labouratory analysis.

2.2 Gasometric measurements

Gasometric assembly is an apparatus that measures the volume of gas evolved from a chemical reaction. A reaction system is connected to the burette through a delivery tube. 200 mL of the corrodent was introduced into the flask and the initial volume of the air in the burette was recorded. Then, aluminium coupon was introduced into the corrodent and the flask immediately closed. The volume of the hydrogen gas evolved from the corrosion reaction was monitored by the volume change in the level of paraffin oil in the burette. The change in volume was recorded every 5 minutes for 2 hrs and the results were tabulated.

The corrodent used was 0.08 M KOH solution, 2.0 M thiourea was introduced into the beaker containing the corrodent as the inhibitor and the volume of hydrogen gas evolved was recorded.

2.3 Gravimetric experiments

Previously weighed aluminium metal coupons were completely immersed and suspended in 250 ml open beaker containing 0.08 M KOH, this was used as the uninhibited system. Then another set of aluminium metal coupons were suspended in another open beaker containing 0.08 M KOH + 2.0 M thiourea, this was used as the inhibited system. The aluminium coupons were suspended using polyethylene twines and wooden cross bars. The experiments were carried out at room temperature, coupons were drawn from the test solution at 3 hr intervals progressively for 15 hrs and washed in a solution containing 50% NaOH and 100 g/L zinc dust. The washed coupons were rinsed in ethanol, dried with acetone before reweighing.

The difference in weight for a period of 15 hrs was taken as the total weight loss from the mean value (mean of duplicate determination using 2 coupons for measurements).

Inhibition efficiency was calculated using this formula in the inhibitor/corrodent solution defined as follows.

For gravimetric test

$IE\ (\%) = ((W_1-W_2)/(W_1))\ X100$

Where W_1 and W_2 are the corrosion rates in the uninhibited and inhibited system, respectively.

2.4 Synergistic measurements

The coupons were prepared as in the gravimetric measurements but 0.005 M KI was added to the beaker containing the corrodent and the inhibitor. The coupons were drawn from the test solution at 3 hr intervals progressively for 12 hrs and washed in a solution containing 50% NaOH and 100 g/L zinc dust. The washed coupons were rinsed in ethanol, dried with acetone before reweighing.

The difference in weight for a period of 12 hrs was taken as the total weight loss from the mean value (mean of duplicate determination using 2 coupons for measurements).

2.5 Scanning electron microscope (SEM)

First, on the sample stub a double adhesive was placed on it, thereafter the sample was sprinkled on it, it was then taken to a sputter coater (quorum-Q150R Plus E) with 5nm of gold. It was then placed on a charge reduction sample holder and introduced into the column of the SEM machine (Phenom ProX) where it was viewed from a NavCam before it was sent to SEM mode of different magnification and stored in a USB stick after adjustment of brightness and contrast. Model used is Phenom ProX,by phenomworld Eindhoven Netherlands

III. Results and Discussion

3.1 Gasometric result

Table 1: Change in the volume of hydrogen gas evolved for 0.08 M KOH with and without 2.0 M thiourea as the inhibitor.

TIME (mins)	0.08 M KOH (mL)	2.0 M thiourea
0		
5	0	0
10	1.3	0.2
15	2.5	0.7
20	3.0	1.3
25	5.5	2.0
30	7.2	2.6
35	8.7	3.4
40	10.5	4.1
45	12.3	4.7
50	14.4	5.2
55	16.4	5.9
60	18.2	6.7
65	20.1	7.5
70	22.0	8.4
75	24.0	9.2
80	25.9	10.0
85	27.9	10.8
90	30.3	11.7
95	32.4	12.8
100	34.7	13.7
105	36.7	14.5
110	39.1	15.4
115	41.2	16.5
120	43.4	17.4
125	45.6	18.4

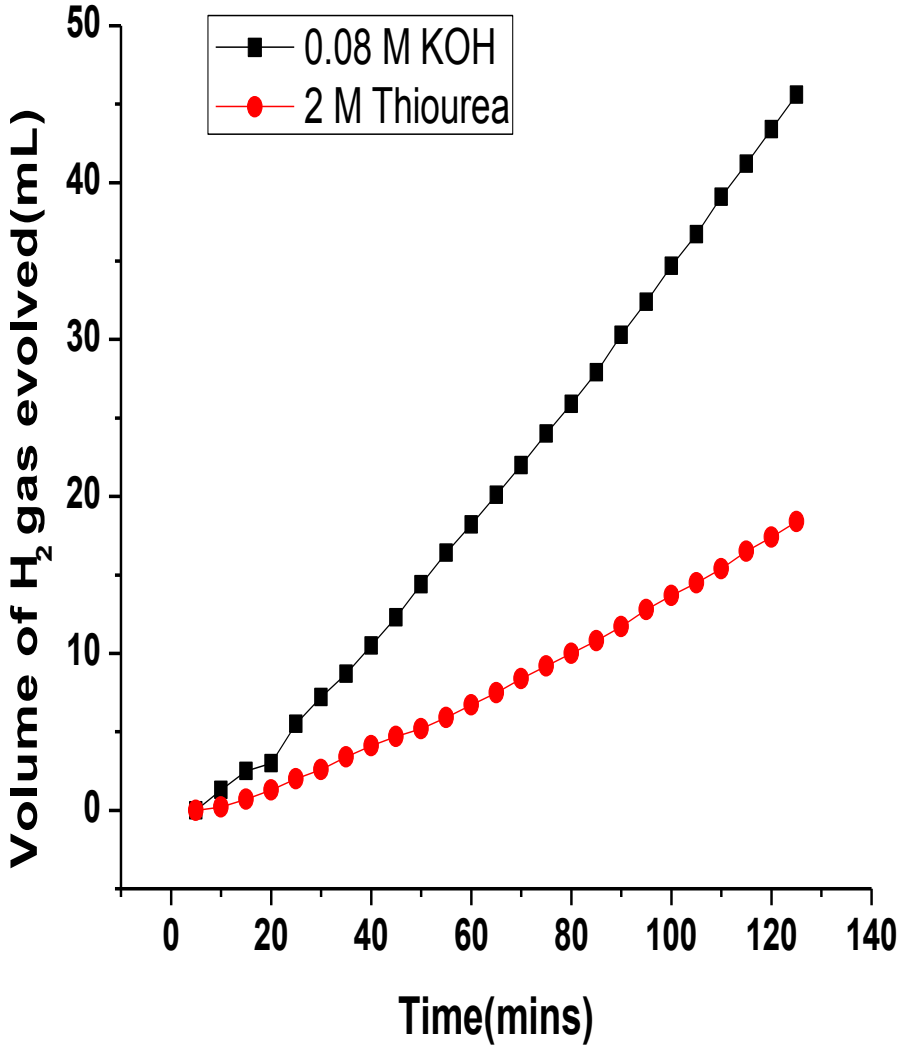


Figure 1: Graph of change in the volume of hydrogen gas evolved for 0.08 M KOH with and without 2.0 M thiourea as the inhibitor.

Figure 1 shows the graph of change in the volume of hydrogen gas evolved for 0.08 M KOH with and without 2.0 M thiourea as the inhibitor after 5 Minutes for 2 hours. This graph further explains table 1, it can also be seen that hydrogen gas evolution increased continuously as time prolonged, addition of thiourea effectively reduced the volume of hydrogen gas evolved.

3.2 Gravimetric result

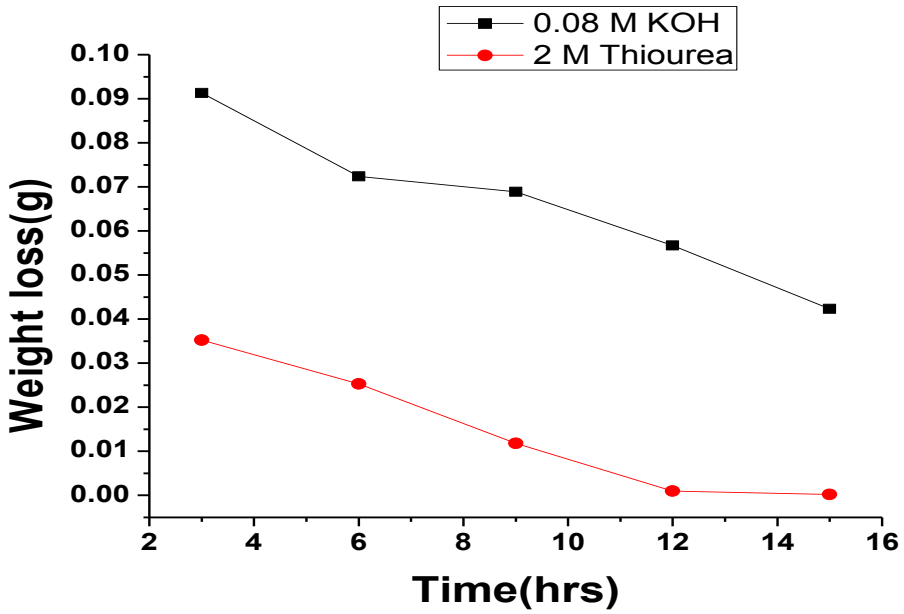


Figure 2: Graph of weight loss against time for aluminium in 0.08 M KOH in absence and presence of 2.0 M thiourea.

The values for the uninhibited system is quite high ranging from 0.0913 g at 3 hrs to 0.0423 g at 15 hrs as can be seen in the graph, figure 2. Weigh loss decreased appreciably in the presence of 2 M thiourea inhibitor from 0.03525 g at 3 hrs to 0.00020 g at 15 hrs. Showing that the inhibitor effectively reduced the weight loss of aluminium in the corrodent.

The inhibiting effect of 2 M thiourea on aluminium in alkaline medium was quantified by calculating the inhibition efficiency IE (%)

Table 2: Inhibition efficiency (%) for aluminium in 0.08 M KOH in the presence of 2.0 M thiourea.

Time (hrs)	3	6	9	12	15
Inhibition efficiency (%)	61.39	65.06	82.36	98.32	99.53

3.3 Synergistic result

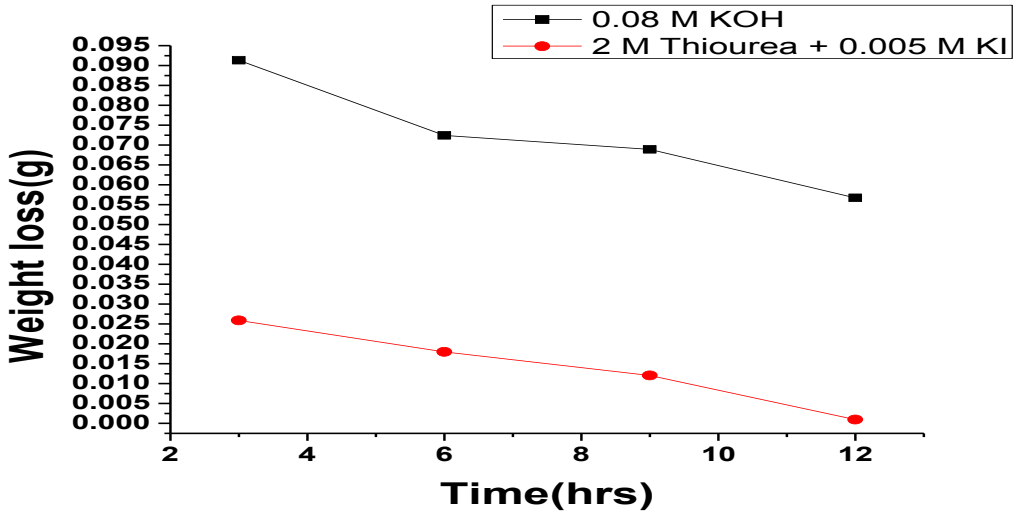


Figure 3: Graph of weight loss of aluminium in 0.08 M KOH + 0.005 M KI in absence and presence of 2.0 M thiourea.

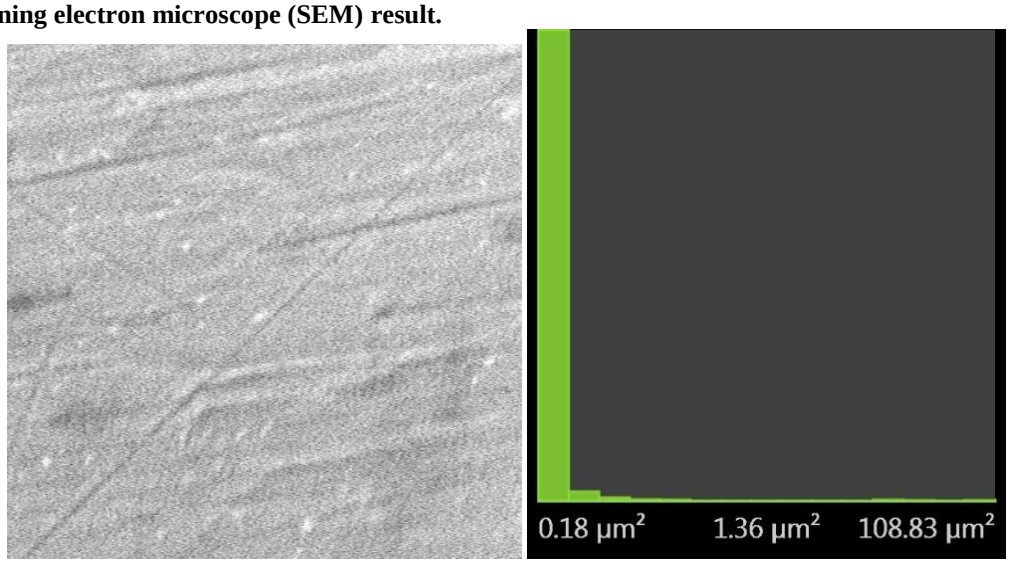
Figure 3: Graph of weight loss of aluminium in 0.08 M KOH + 0.005 M KI in absence and presence of 2.0 M thiourea. Weight loss for the uninhibited system was high ranging from 0.0913 g at 3 hrs and sloped to 0.0567 g at 12 hrs as can be seen in figure 4.18. Weight loss decreased appreciably in the presence of 2 M thiourea inhibitor and 0.005 M KI from 0.02590g instead of 0.03525 g for 2 M thiourea inhibitor without KI at 3 hrs to 0.00095 g 12 hrs. This shows that effect of synergy at 3 to 9 hrs and it gradually stabilized at the 12 hrs.

Table 3: Inhibition efficiency (%) for aluminium in 0.08 M KOH + 0.005 M KI in the presence of 2.0 M thiourea.

Time (hrs)	3	6	9	12
Inhibition efficiency (%)	71.63	75.68	81.99	98.33

The values for the inhibition efficiency in the presence of 2 M concentration of the inhibitor gave 61.39 % after 3 hrs and 71.63 % in the system with KI, the efficiency increased with time such that at 12 hrs, it stabilized to 98.32 %, and 98.33 in the system with KI, Showing very high inhibition efficiency as seen in table 3. From the result, it was observed that efficiency increased due to the synergistic effect and it is pronounced at the early hours

3.4 Scanning electron microscope (SEM) result.



Fibermetric Image Pore Histogram

Figure 4: SEM Result for plain aluminium metal

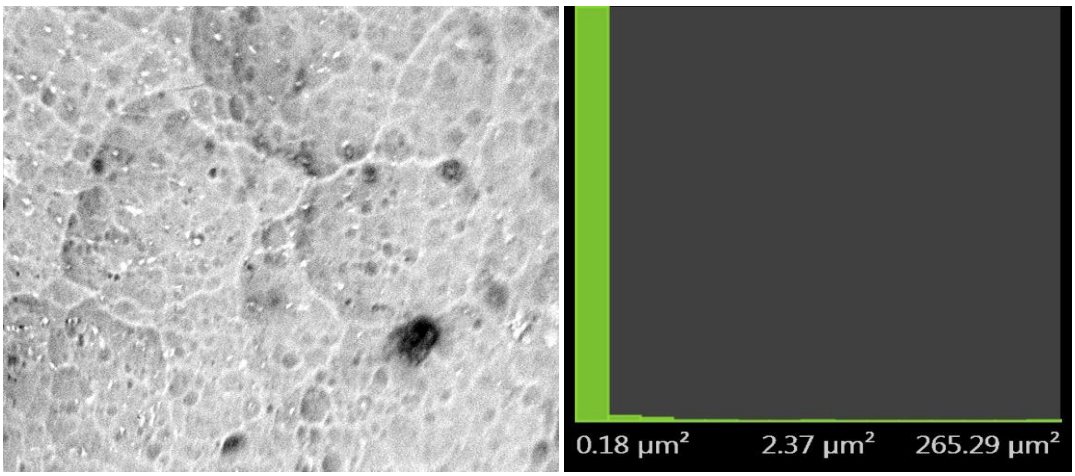


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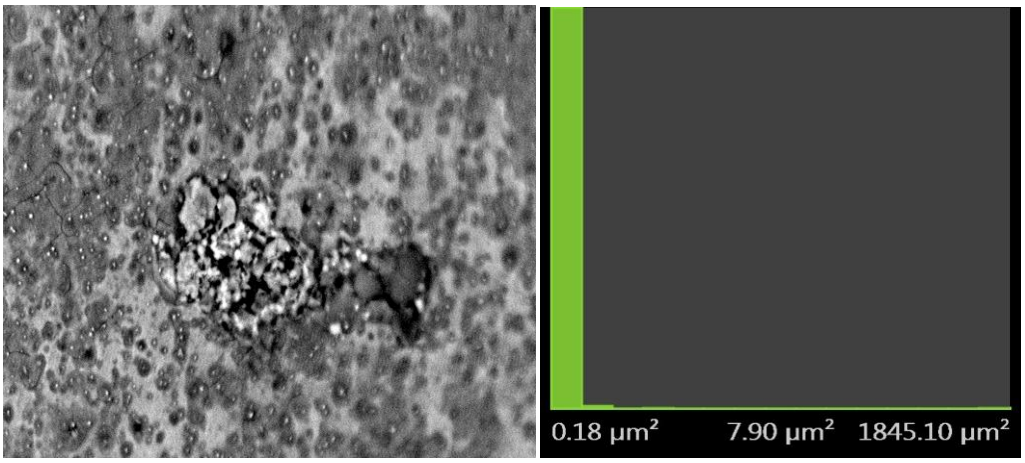
Figure 5: SEM result for aluminium metal in 0.08 M KOH without inhibitor for gravimetric measurements after exposure for 15 hours.



Fibermetric Image Pore Histogram

Figure 6: SEM result for aluminium metal in 0.08 KOH and 2.0 M thiourea as inhibitor.

Figure 4 shows SEM result for plain aluminium metal, it shows the smooth surface of the aluminium metal before exposure (control), figure 5 depicts SEM result for aluminium metal in 0.08 M KOH without inhibitor in the gravimetric experiment for 15 hours, an intensely corroded surface morphology is clearly seen in the uninhibited systems for gravimetric experiments after immersion, this is evident in the roughness of the surface, the corrosion attack is so intense as the grain boundaries on the metal surface became visible due to aggressive attack. But in the presence of 2.0 M thiourea inhibitor as seen in figure 6, the corrosion damage is visibly reduced and there is visible evidence of the adsorbate presence on the metal surface, a distinct and compact corrosion product layer is obvious on the surface of the aluminium metal, this explains the high inhibition efficiency.

A corroding metal surface is usually characterized by multiple adsorption sites, inhibitor molecules may thus be adsorbed at such sites, it is possible that the inhibitor molecules grows inside these pore channels on the aluminum surface, the pore histogram shows the smallest and largest pore areas on the aluminium metal surface from which the smallest and largest pore radius is calculated using (πr^2). The bigger the pore radius, the higher the effect of the inhibitor on the metal surface. The largest pore radius on the plain aluminium metal surface is 5.89 µm, the uninhibited systems for gravimetric is 9.19 µm, with the presence of the inhibitor, the largest pore radius increased to 24.24 µm

The SEM images reveal the agglomeration of the inhibitor molecules on the metal surface.

IV. Conclusion

This research work focuses on the mechanism of corrosion inhibition of aluminium by 2 M thiourea in 0.08 M potassium hydroxide. The result showed that 2.0 M thiourea effectively reduced the corrosion rate and weight loss of aluminium in 0.08 M KOH, highest inhibition efficiency was observed at 15 hrs giving 99.53 %. Effect of synergy was seen to be more pronounced at the early hours. Gasometric experiment revealed that the inhibitor effectively reduced the volume of hydrogen gas evolved from 45.6 mL to 18.4 mL, this was due to adsorption of the molecules unto the surface of the metal, scanning electron microscope clearly showed the presence of grain boundaries on the uninhibited aluminium metal due to corrosion attack and the agglomeration of the inhibitor molecules on the inhibited aluminium metal protecting it from damage. The findings from this research proved that 2 M thiourea is a very good, environmentally friendly, cheap and available inhibitor for aluminium in alkaline environment.

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