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Rubber Leaves vs. Corn Cob Extract: A Performance Evaluation of Superior Corrosion Protection for Aluminium in 1.0 M H_2SO_4

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ABSTRACT: In a quest to develop an effective eco-friendly corrosion inhibitor for aluminium rubber leave and corn cob extract were both tested in 1.0 M H_2SO_4 and compared. The following techniques were used weight loss, AAS and gasometric technique. SEM was utilized to determine the surface morphology of the inhibitor on the metal surface before and after immersion in the corrosive medium. Three adsorption isotherm models were used to explain the reaction mechanism, results obtained shows that the highest inhibition efficiency for rubber extract was 97.8% while that of corn cob was 92.22%. This shows that rubber extract has a superior protection for alumibium in 1.0 M H_2SO_4electrolyte. The SEM result shows that the surface morphology of the adsorbent on rubber extract treated surface was better than that of corn cob. The Langmuir and Temkin adsorption isotherm models best explain the reaction mechanism which supports the chemisorption interaction of the inhibitors.

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Aluminium is one of the lightest metals for structural engineers, having strength to weight ratio superior to steel, Bashir *et al.,* (2020); Chen *et al.,* (2021). Aluminium and its alloys are of economic importance because of their low cost, lightness and good corrosion resistance at moderate temperatures. Aluminium is the world's most abundant metal and the third most common element comprising 8% of the earth crust. The versatility of aluminium makes it the most widely used metal after steel. However, aluminium and its alloys are susceptible to intense corrosion processes in acid media, Asan and Asan *(*2020); Fouda *et al.,* (2020). In aeronautical industries, this issue is minimized by applying different types of insulating coatings such as fiberglass to avoid direct contact between the metal and its surrounding, thus providing protection for the metal via barrier mechanism. However, various coating systems contain hazardous Cr (VI), which makes them environmentally unfriendly Wang et al., (2019); Asadi *et al.,* (2019) and Dehghani et al., (2019). This has triggered a lot of research in the area of corrosion and its control. Despite intensive study over many years, the chemistry and mechanisms of corrosion attack is not fully understood. Moreover, the occurrence and severity of highly localized metal degradation cannot currently be predicted and often cannot be rationalized in failure analysis. Thus the corrosion of metallic materials in acidic and neutral solutions continues to cause considerable losses, Cao *et al.,* (2014); Petrović *et al.,* (2017). Corrosion occurs due to the tendency for most metals to return to their natural state e.g. iron in their moist air will revert to iron oxide, known as iron ore. Therefore, in an effort to control this trend a lot of

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investments have been made into corrosion research. Among the findings made by researchers is the use of corrosion inhibitors which has been found easily applicable and cost effective. Corrosion inhibitors are substances which when added in small concentration to an aggressive environment effectively reduces the corrosion rate of a metal exposed to that environment. Most organic compounds having heteroatom(s) (N, S, O, P) in their aromatic or long carbon chain have been successfully used as corrosion inhibitors, Aytac *et al.,* (2005); Babi–Samardizija *et al.,* (2005).Among the inhibition methods, green corrosion inhibitors are currently of immense interest because of rabid increase in environmental awareness and regulations which restrict regular corrosion inhibitors due to toxic contents. Natural products are a good source of green corrosion inhibitors, since most plant extracts contain the needed heteroatoms, which improve their adsorption on metal surface, Beenakumari (2011); Singh *et al.,* (2013); Chakravarthy *et al.,* (2014); Ayodeji *et al.,* (2016). and Akinbulumo *et al.,* (2020); Corn cobs contain approximately 39.1% cellulose, 42.1% hemicelluloses, 9.1% lignin, 1.7% protein, and 1.2% ash; all these compounds contain lots of heteroatoms. Due to their chemical composition, corn cob extract shows a great potential as corrosion inhibitor. On the other hand rubber leaf is a medicinal plant that possesses secondary metabolites such as flavonoids, alkaloids, tannin, cardiac glycosides and steroids similar to that of corn cob, Okafor *et al.,* (2010); Anadebe *et al., (*2021) and Ushie *et al.,* (2022). Following the characteristics of rubber leaf extract shows that it is a potential corrosion inhibitor since it contains already mentioned inhibitive components. Hence, in the present work, we intend to compare the corrosion inhibition capabilities of corn cob and rubber leave extract to establish which is better for aluminium corrosion protection in 1.0 M H_2SO_4corrosive solution.

MATERIALS AND METHODS

Fresh samples of rubber (*Hevea brasiliensis*) leaves and corn cob were collected from a plantation farmland in Opu-oko in Khana L.G.A., Rivers State of Nigeria. Bare mild steel of 0.2 cm thickness used for the study was cut into 2cm by 3cm coupon size for weight loss analysis. The coupons were polished and brushed with an iron brush and a hole was made at the centre of the top edge of each coupon in such a way that the coupon will freely be suspended in the solution. The coupons were soaked in ethanol solution for 5 mins for degreasing and rinsed with distilled water and acetones and finally were kept in desiccators for proper drying. This was necessary to prevent contamination before usage for corrosion studies.

Preparation of Sample Extracts: The fresh leaves from rubber tree (*Hevea brasiliensis*) and corn cob (*Zea mays*) were washed with enough water, sun dried for few days and grinded into powdered form using a grinding machine. About 100 g each of the pulverized sample were measured and soaked in 1200 ml of ethanol in a beaker and was stirred for complete dissolution and allowed to stand for 7days (a week). Within the period, the volume of ethanol was checked constantly and more ethanol was added if the ethanol level dropped. The mixture was then filtered using filter paper and funnel, and the powder product was recovered through evaporation to dryness; same was carried out for rubber and corn cob extracts. Thereafter, 0.2 g of finely grounded sample was weighed and 10 ml of 70% acetone was added and Placed in an ice bath for 10mins.The solution was oscillated with oscillator for 15 mins and allowed to cool for 30 mins and the mixture was filtered into a conical flask and with a Pipette, 0.5 ml of the extract or filtrate was poured into 50 ml volumetric flask. 0.5 ml of distilled H_2O was added to the sample and 1 ml of distilled water was added into another flask as blank.0.5 ml folin–dennis reagent was added to sample and standard. Then 2.5 ml of 20% sodium carbonate solution was added to the sample and standard and mixed properly and made up to mark with distilled H_2O. It was allowed to stand for 40 mins for a bluish–green colour to develop. And the absorbance was read at 725nm. Using FTIR (Fourier Transform Infrared Spectroscopy) analysis, the structural organization of the rubber leaf extract was investigated to identify the functional group present. The structural organization of the rubber leaf extract was determined using (FTIR – Perkins Elmer Spectrum BX II) model.

Weight loss Analysis: The mild steel coupons dried in the desiccators were carefully weighed and their respective weights in grams were recorded as the initial weights. Each of mild steel coupons was placed in different beakers containing the different concentrations of the test solution (10% v/v, 20% v/v, 30% v/v, 40% v/v, 50% v/v, 60% v/v, 70% v/v, 80% v/v, 90% v/v, 100% v/v and control).A rubber thread was used to suspend the coupons in the test solutions. The various test solutions containing the coupons were maintained at room temperature. The coupons were retrieved from the test solutions at every 2 h interval progressively for 16 h. At each interval the retrieved coupons were washed under running tap water and then dipped in ethanol, distilled water, and acetone. After which they were allowed to dry before the final weight was taken. The weight loss was calculated in grams as the difference between the initial weight (w_1) prior to immersion and the final weight after removal of the corrosion product from the surface of the coupons by iron brushing.

Instrumental Analyses: Using atomic absorption spectroscopy (AAS) analysis, the concentration of iron in each test solution was determined using atomic absorption spectroscopy (GBC XPLORAA, model). Each of the labeled coupons were dipped in their respective test solutions for a period of 16 h, after which the amount of iron deposited in each of the test solutions was analyzed using the AAS. Gasometric experiment is used to determine the amount of hydrogen gas evolved when the metal reacts with the acid. 100ml of the 10% test solution was measured into the reacting vessel of the gasometer. Thereafter, the metal coupon was introduced into it and then closed properly. The amount of hydrogen gas evolved was recorded after every interval of one minute for a period of 30mins. The process was repeated for the different test solutions. By utilizing the scanning electron microscopy (SEM), the images of the mild steel coupon samples were taken before and after immersion in the inhibitor using the scanning electron microscope.

RESULTS AND DISCUSSION

Phytochemicals of rubber leaf and corn cob: The different components and their percentages present in rubber and corn cob extracts were investigated via phytochemicals analysis as presented on Table 1. It can be seen that most of the percentage components from alkaloids to oxalate are much higher in rubber leaf than corn cob. This could be attributed to its higher activity compared to the corn cob.

Table 1. Percentage composition of phytochemicals found in rubber leaf and corn cob.

Phytochemicals	Molecular mass of phytochemicals (g/mol)	% Composition in Rubber Leaf	% Composition in Corn cob
Alkaloids	162	9.53	6.01
Flavonoid	293	3.32	2.64
Saponin	1233.3	2.01	1.15
Phenol	94.11	0.749	0.488
Tannin	1701.2	0.401	0.268
HCN	27.03	6.95	1.95
Phytate	660.029	10.12	8.08
Oxalate	128.10	0.71	0.62

FTIR (Fourier transform infrared spectroscopy) of rubber leaf and corn cob: Figure 1 shows the FTIR spectrum of rubber leaf extract performed to analyze the functional groups present in the extracts. A stretch at 3422 falls within the range of N-H (stretching of amines). A stretch at 2926.38 shows the presence of a C-H stretch which suggests the presence of a phytate that has a cyclohexane ring. A stretch at 1655.48 shows the presence of carboxylic acid C=O. Another stretch at at 1053 suggests the presence of an alkylamine. A stretch at 3766.36 indicates the presence of O-H groups of amide. The observed functional groups are common among the phytochemicals screened in rubber leaf.

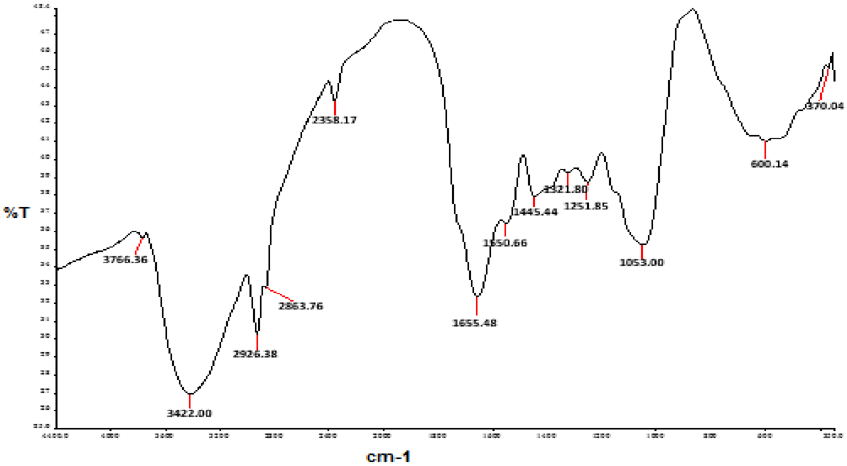

Fig 1. FTIR spectrum of rubber leaf extract carried out at room temperature

The phytochemicals have large molecular mass, contain hetero atoms and pi electrons. All these are responsible for their capacity to inhibit corrosion of metals. On the other hand Figure 2 presents the FTIR spectrum of Corn cob extract. A stretch at 3438 falls within the range of N-H (stretching of amines). A stretch at 2925.63 shows the presence of a C-H stretch this suggests the presence of a phytate that has a cyclohexane ring. A stretch at 1642.43 shows the presence of carboxylic acid C=O. A stretch at 1045 suggests the presence of an alkylamine. A stretch at 2355 shows the presence of C=C conjugated acid. The observed functional groups are common among the phytochemicals screened in corn cob. The phytochemicals have large molecular mass, contain hetero atoms and pi electrons. All these are responsible for the observed corrosion mitigation performance of the extract.

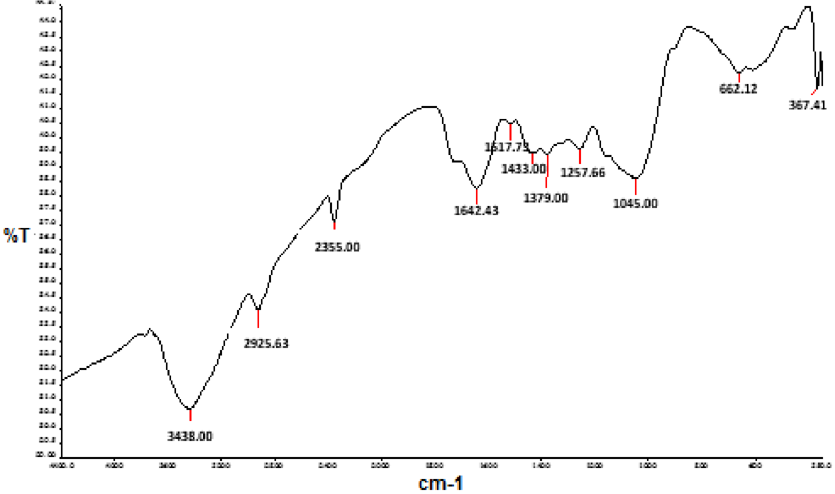

Fig 2. FTIR spectrum of Corn cob extract carried out at room temperature.

Weight loss analysis of corn cob and rubber leaf extracts: Figure 3 shows the weight loss analysis of corn cob and rubber leaf extracts as a comparison of the lost weight versus exposure time for both samples on aluminium in 1.0 M H_2SO_4 at room temperature. Figure 3 indicates that weight loss increases with decrease in inhibitor concentration. This can be attributed to less active inhibitor molecules that are available to bind the metal surface thus exposing the metal to attack by the acid which in-turn leads to dissolution of the metal in the acidic medium. Also, the weight loss increases with increase in exposure time which might be as a result of the inability of the inhibitor to bind to the metal surface for a longer time in the face of aggressive environment with increase in exposure time. At equilibrium the interaction between the adsorbate on the adsorbent and that in the bulk of the solution makes it difficult for more of the adsorbate to adsorb on the adsorbent, hence reducing inhibition efficiency. This is as result of exposing the area of metal not properly covered by the adsorbate for further corrosion attack.

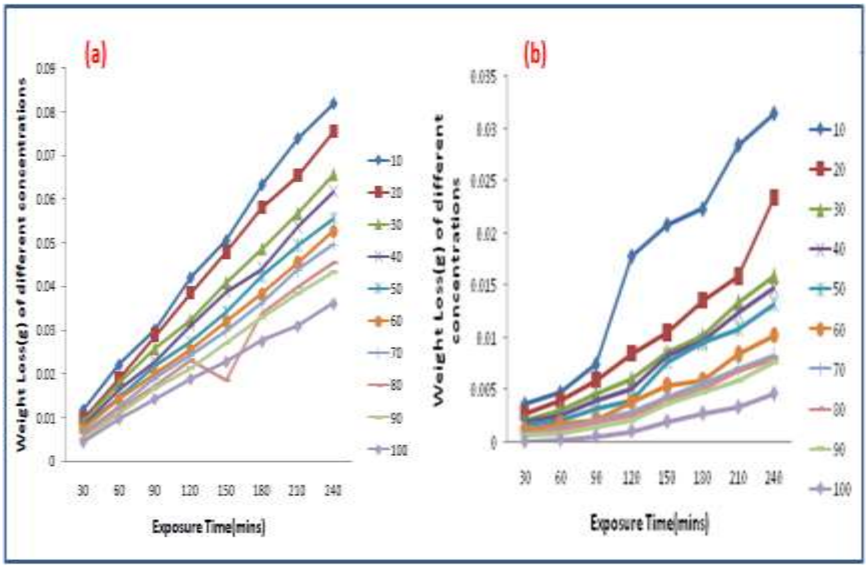

Fig 3. Analysis of weight loss (g) against time of exposure for (a) Corn cob (b) rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature.

The Inhibition efficiency was equally analyzed against exposure time. Figure 4Shows the inhibition efficiency against exposure time during weight loss analysis for corn cob and rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature. Figure 4 proves that inhibition efficiency decreases with increase in exposure time. This might be as a result of the adsorbate being unable to adsorb on the adsorbent for a long period of time which in-turn exposes part of the metal surface not properly covered for attack by the acid.

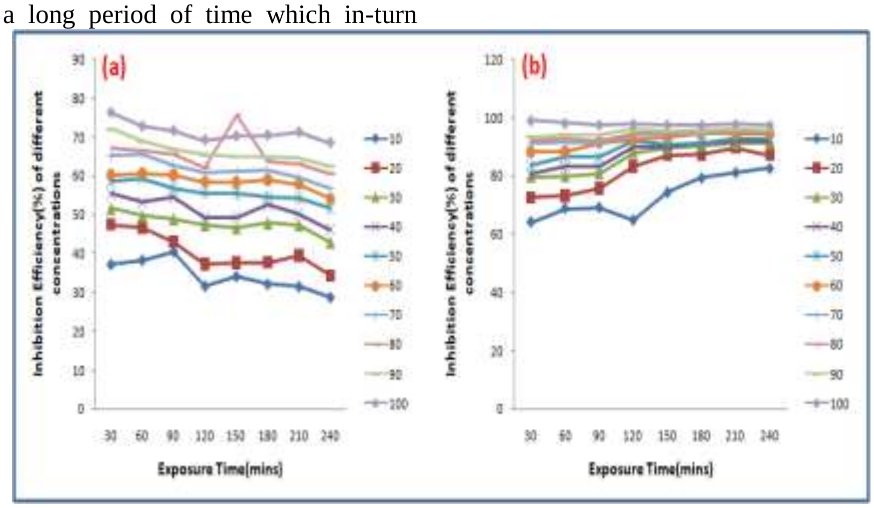

Fig 4. Inhibition efficiency against exposure time during weight loss analysis of (a) Corn cob (b) rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature.

Inhibition efficiency analysis of the different techniques: The inhibition efficiency is very crucial in discussing the performance of a corrosion inhibitor and the technique or method used for the performance evaluation is equally important. Figure 5X-rays the inhibition efficiencies against concentration during AAS, gasometric and weight loss analyses of corn cob and rubber leaf extracts on aluminium in 1.0 M H_2SO_4 at room temperature. The analysis shows that the inhibition efficiencies of the various techniques follow similar trend of increasing with the increase in concentration, confirming the efficacy of the methods. However, the values of the inhibition efficiencies from the methods are different and goes in the following order; AAS>weight loss > gasometric. This pattern can be attributed to the likely experimental errors associated with the methods such as accuracy in weighing, improper brushing of coupons prior to weighing, improper handling of coupons among others for weight loss. While in the gasometric technique the possible leakage of the hydrogen gas from the outlets under the experimental condition might be the major source of error.

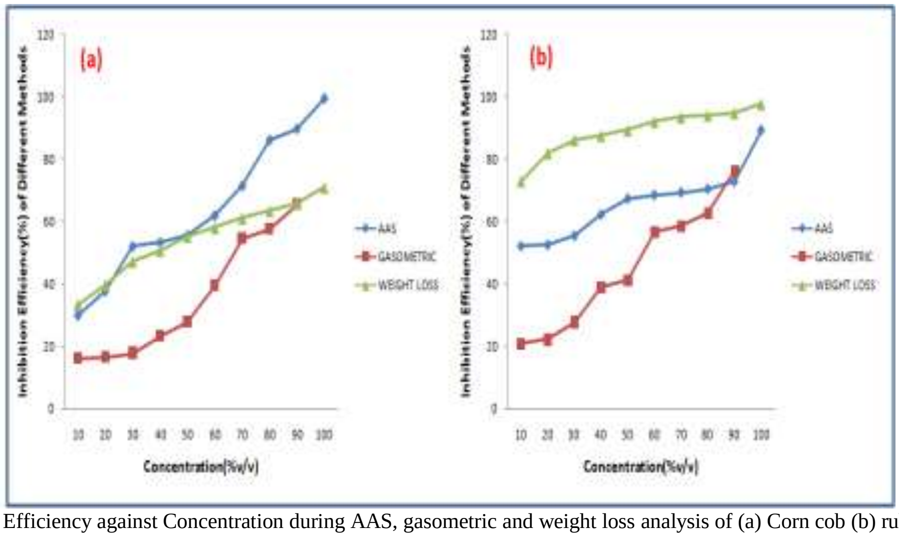

Fig 5. Inhibition Efficiency against Concentration during AAS, gasometric and weight loss analysis of (a) Corn cob (b) rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature.

Scanning electron microscope (SEM) analysis: Figure 6 shows the post immersion SEM images of aluminium in 1.0 M H_2SO_4, without and with corn cob and rubber leaf extract, respectively, at room temperature. The right hand panels (Figures 6a and 6c) are the aluminium metal surface before immersion which as expected shows no evidence of corrosion. However, it is apparent from the SEM morphology (Figures 6b and 6d) that the dissolution of aluminium substrate is remarkably evident. It equally shows that corrosion is higher on corn cob system (Figure 6b) than that of rubber leaf (Figure 6d). This can be confirmed by the number of pits and scratches visible on the surface of the corn cob sample after addition of the inhibitor as evident by the morphology of the corroded parts.

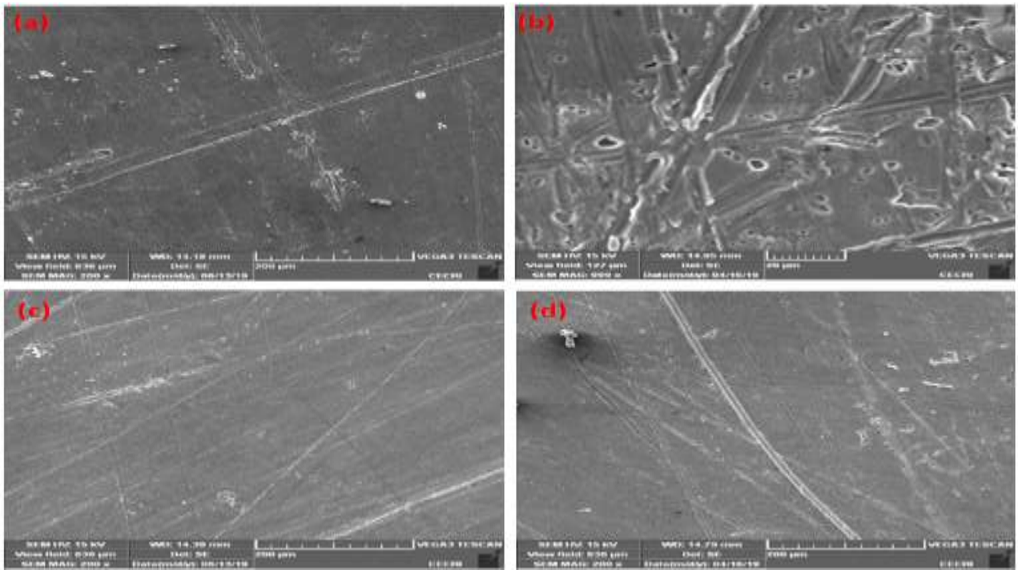

Figure 6. Post immersion SEM images of aluminium in 1.0 M H_2SO_4, (a,b) without and with Corn cob (c,d) without and with rubber leaf extract, respectively, at room temperature.

Surface Coverage: Surface coverage decreases with exposure time and increases with concentration. As concentration increases more of the adsorbate are readily available to cover the metal surface hence reducing the area for acidic attack on the metal. Figure 7 clearly shows **s**urface coverage against exposure time during weight loss analysis for corn cob and rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature. The trend of the curves equally shows that corn cob has less coverage than rubber leaf extract and this confirms the better performance of rubber leaf over corn cob.

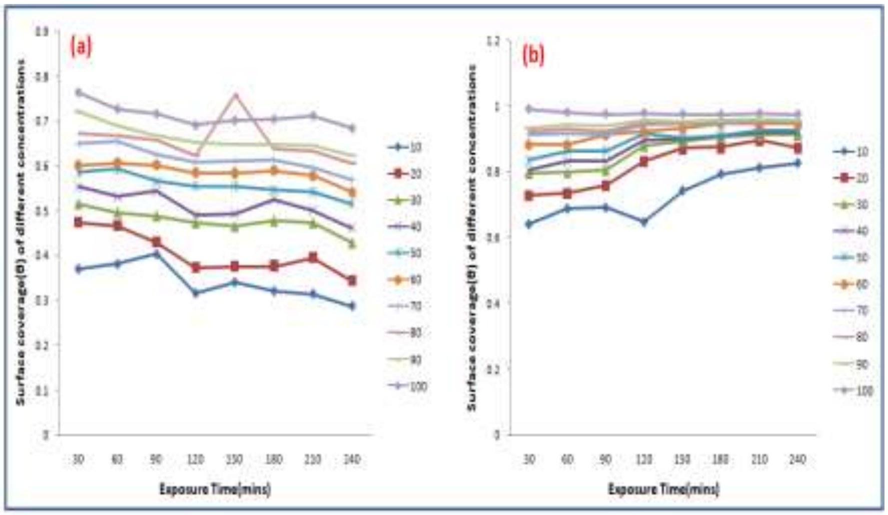

Fig 7. Surface coverage against exposure time during weight loss analysis of (a) Corn cob (b) rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature.

Corrosion Rate: A corrosion inhibitor when added in a small amount effectively reduces the corrosion rate of a metal. Therefore, by introducing either corn cob or rubber leaf extract on aluminium in a corrosive environment of 1.0 M H_2SO_4 solution corrosion rate apparently decreased. Figure 8 shows the corrosion rate of corn cob and rubber leaf extract against exposure time for weight loss analysis on aluminium in 1.0 M H_2SO_4 at room temperature. The decrease in corrosion rate goes with increase in concentration of the adsorbate with exposure time. This might be as a result of the adsorbate being readily available to adsorb on the metal surface thus reducing corrosion. A critical observation of the plots shows that the corrosion rate of aluminium in the system with corn cob extract (Figure 8a) was much higher than that of rubber leaf (Figure 8b) with a difference up to 0.0013 mm/yr on each concentration used. Therefore, rubber leaf extract has proven to be a superior corrosion inhibitor over corn cob on the mitigation of aluminium in 1.0 M H_2SO_4.

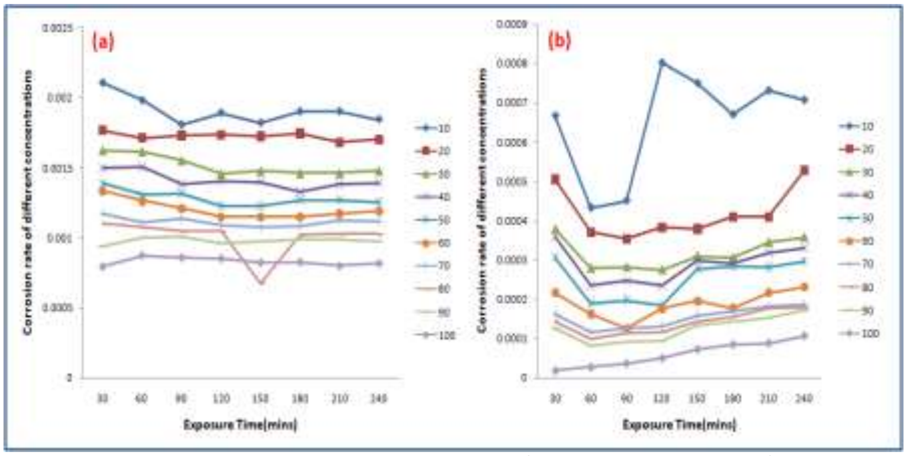

Figure 8. Corrosion rate against exposure time during weight loss analysis of (a) Corn cob (b) rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature.

Adsorption isotherm studies: Comparing the adsorption isotherm data of rubber leaf and corn cob extract, Figure 9 shows the Langmuir isotherm plots for these twoextracts on aluminium in 1.0 M H_2SO_4 at room temperature. From the Langmuir isotherm plot we observed R^2value to be 0.9864 and R^2 value > 0.70 indicates a monolayer adsorption. It was also observed that the value of the slope was greater than unity it means that one or more of each inhibitor unit occupies more than one adsorption site, Shaban et al., (2015); Saha et al., (2015); Emori *et al.*, (2020); Risti and AsepBayu (2021); therefore, there are interactions between adsorbed species on the metal surface or the adsorption heat (enthalpy) changes with increasing surface coverage. It was equally observed that the data of corn cob extract (Figure 9a) gave a less perfect fit compared to that of rubber leaf (Figure 9b) with much better straight line pattern.

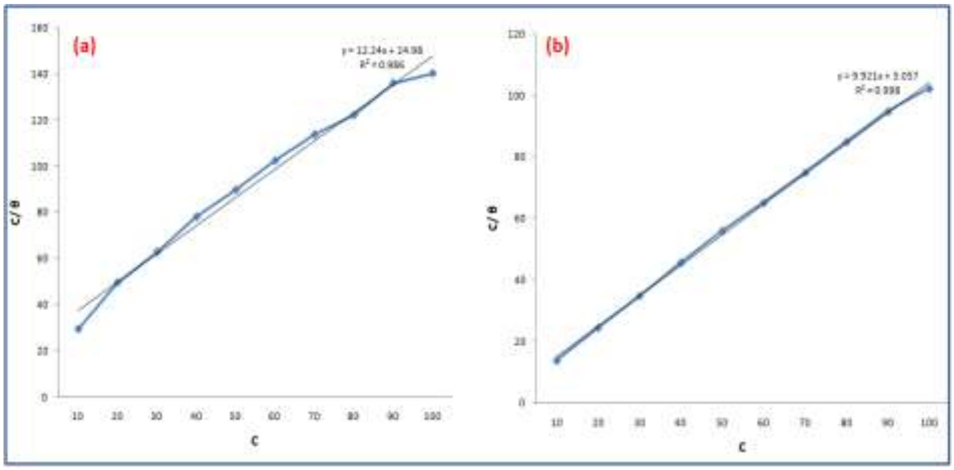

Fig 9. Langmuir Isotherm plots for (a) Corn cob (b) rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature.

Figure 10shows the Temkin isotherm plots for corn cob and rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature. From the Temkin isotherm plot the R^2 value is 0.9679 and R^2>0.70 indicates uniform distribution of adsorbate to adsorbent surface, Risti and AsepBayu (2021). Also, from the Temkin isotherm plot the calculated B_T (heat of adsorption) value is 65KJ/mol and B_T > 8 KJ/mol suggests a chemical interaction between adsorbate molecules [20]. Similarly, it was observed that the data of corn cob extract (Figure 10a) gave a less perfect fit compared to that of rubber leaf (Figure 10b) with much better straight line pattern. Therefore, the better performance of rubber leaf extract on aluminium over corn cob is buttressed for every comparison experiment.

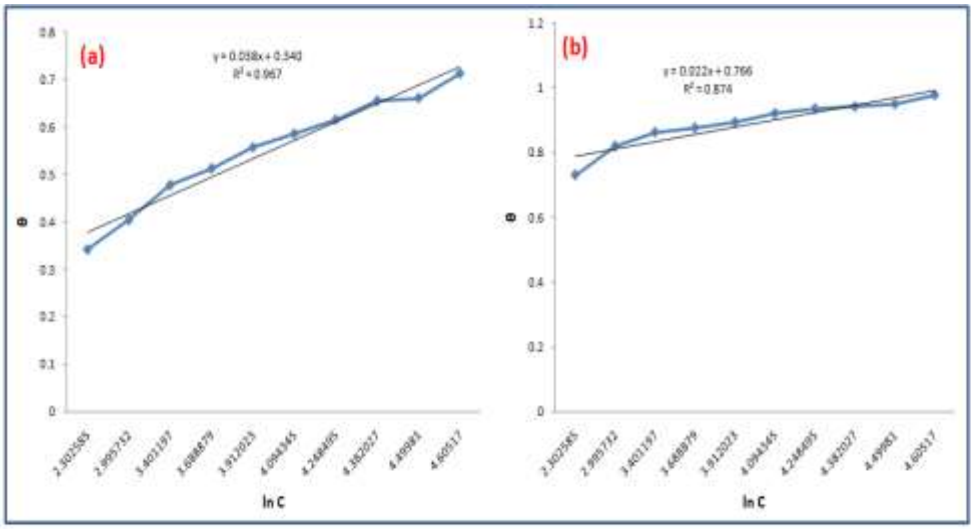

Fig 10. Temkin Isotherm plots for (a) Corn cob (b) rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature.

Figure 11shows the Freudlich isotherm plots for corn cob and rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature. The Freundlich isotherm describes physical type of adsorption, multilayer adsorption with weak Van der waal forces as well as heterogenous sites of adsorption. From the Freundlich isotherm plot the R^2 value is 0.9242 and the observed n (slope) value is 0.0742; $R^2 > 0.70$ shows a multilayer adsorption while $n < 1$ value indicates that the adsorption process is with chemical interaction between adorbate and inhibitor molecules Risti and AsepBayu (2021). Freudlich isotherm plots shows the poorest expression of the adsorption mechanisms. It was observed that both corn cob extract (Figure 11a) and rubber leaf data (Figure 11b) shows no straight line pattern, rather huge deviation was obtained. However, the high R^2 value confirms multilayer for rubber leaf extract and corn cob on aluminium.

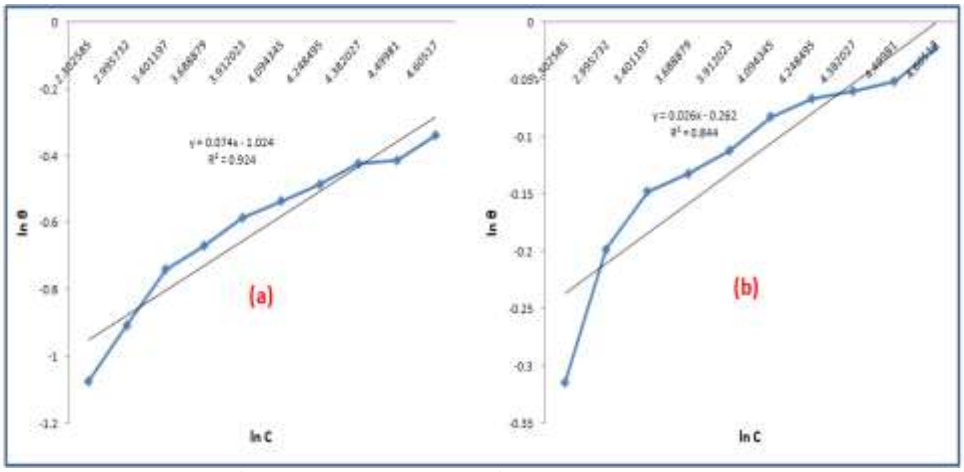

Fig 11. Freudlich isotherm plots for (a) Corn cob (b) rubber leaf extract on aluminium in 1.0 M H_2SO_4 at room temperature.

Mechanism of the corrosion process and inhibition: The corrosion inhibition mechanism of rubber leave and corn cob extract are same and can be demonstrated as follows. The anode is the active point through which metal ions move into the solution during corrosion. The corrosion process is actually the movement of electrons from the anode (donor) to an acceptor (cathode) known as the less active part of the metal. The corrosion process therefore requires the presence of an electron donor and the acceptor at the cathodic side such as oxygen, oxidizing agents, or hydrogen ions (known as the cathodic species). Thus corrosion can be retarded or stopped by changing the anodic or cathodic reaction processes, or both.

Note that the anodic process is the active metal dissolution as shown in Equation 1;

$$M \rightarrow M^{+} + e- \qquad (1)$$

This is an electron loss or donation process whereas on the other hand Equations 2; and 3 the cathodic reactions taking place simultaneously includes;

$$2H^{+} + 2e- \rightarrow H_2 \text{ (hydrogen gas evolution via reaction)} \quad (2)$$

$$O_2 + 4H^{+} + 4e - \rightarrow 2H_2O \text{ (Oxygen reduction to water)}(3)$$

When inhibitors are introduced into the corroding system (see Figure 12), they get adsorbed on the metal surface Equation 4, forming a protective barrier film, Strehblow and Titze (1980). The formation of the barrier film makes the inhibitor to interfere with the anodic and/or cathodic reaction sites by blocking the penetration of cathodic species thereby slowing down the oxidation and/orreduction processes associated with corrosion, Finšgar and Milošev (2010).

$$Inhibitor + nHads \rightarrow Inhibitor\ ads + H_2\ (4)$$

This reaction takes place by the displacement of water molecules on the metal surface. The calculated Gibb's free energy -16.432KJ/mol shows that the adsorption reaction is spontaneous.

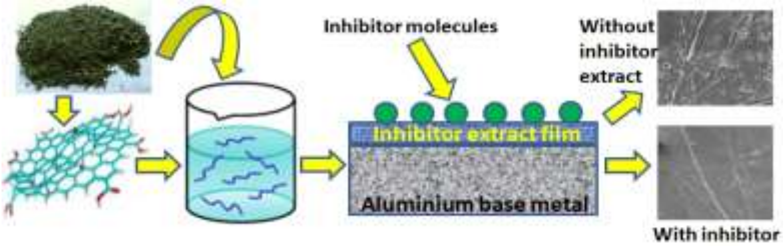

Fig 12. Schematic representation of the corrosion process and inhibitor's action on aluminium surface.

Conclusion: Plant extracts from rubber leave and corn cob has been successfully tested on aluminium and the results compared. This revealed that both extracts are effective at mitigation of aluminium corrosion however, rubber extract showed a better performance over corn cob.

The two inhibitors showed their inhibition effects by adsorbing their molecules on the aluminium surface to form a protective layer. Three isotherm models were tested; out of the three Langmuir and Temkin best explains the reaction mechanism as chemisorption.

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