



## Characterization of the Inhibitive Potentials of the Extract of Calyx of White Sorrel (*Oxalis Acetosella*) on Mild Steel in $H_2SO_4$ and NaOH Media

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**Abstract:** The study of calyx of *Oxalis acetosella* (White sorrel) plant in tetraoxosulphate (vi) acid and sodium hydroxide media was undertaken using the Weight Loss Technique of corrosion measurement. Square mild steel rods were obtained from a local store and cut into seventy two (72) cuboidal test coupons each of dimensions (10 mm x 5 mm x 5 mm). The coupons were then subjected to standard metallurgical procedures for corrosion experimentation. Extraction of the active ingredients in the calyx of the White sorrel was done using the Reflux Technique. The required concentrations of the inhibitor (5 ml, 10 ml, 15 ml and 20 ml) were separately added to each of the beakers containing the corrosion media (0.5 M  $H_2SO_4$ , 1.0 M  $H_2SO_4$ , 0.5 M NaOH and 1.0 M NaOH). The test coupons were then tied with nylon strings and suspended inside each of the beakers at 4 coupons per beaker. The setup was allowed to stand on a laboratory table for 672 hours with one sample removed from each beaker every 168 hours and subjected to standard post-immersion procedures for corrosion characterization. Subsequently, the corrosion rates and inhibition efficiencies were calculated using known formulae. Plots of corrosion rates, inhibition efficiencies and Langmuir adsorption isotherms were made and analyzed. The results obtained showed that the corrosion rates followed the normal trends for passivating metals, which is characterized by an initial steep rise followed by a progressive decrease as exposure time increased. Comparatively, the corrosion rate was observed to decrease as the inhibitor concentration increased in both  $H_2SO_4$  and NaOH, but the inhibitive effects were more pronounced in the acid medium than the base. The effectiveness of the adsorption layer in preventing further corrosion is amply supported by Langmuir relationship that tries to examine layer thickness and uniformity on the nature and concentration of the corrosion media. A conclusion is drawn to the effect that the calyx of *Oxalis acetosella* can serve as an alternative green inhibitor to the synthetic ones in use today.

## 1. Introduction

Corrosion has been severally defined in contemporary times as the unintentional degradation of materials, particularly metals due to interaction with their environments (Callister, 2007; Fontana, 1987; Tebbji *et al.*, 2007; Ahmad, 2006). It is electrochemical in nature and starts preferentially from the surface (Askeland, 1985; Sastri *et al.*, 2007; Hammouti *et al.*, 1995). There are several types of

corrosion processes encountered in practice which includes but not limited to general corrosion, pitting corrosion, crevice corrosion, microbial corrosion and stress-corrosion-cracking (Schweitzer, 2010).

The scourge of corrosion in the industry is globally recognized as one of the most serious problems in modern societies and has much serious economic, health, safety, technological, and cultural consequences (Mejeh *et al.*, 2020). For instance, it is estimated that the annual cost of corrosion in the developed countries of the world like the United States is \$276 billion, which is about 3.1% of the GDP (Gerhardus *et al.*, 2002), in China, \$310 billion (Hou *et al.*, 2017) and in India, \$26.1 billion (Bhaskaran *et al.*, 2016) to mention but a few.

Therefore, over the years, efforts have been intensified in finding efficient and cost effective ways to mitigate this humongous corrosion cost. In this connection, the use of corrosion inhibitors is considered a major pathway to the solution used in the oil and gas production and processing industries as a defense against corrosion (Waidi *et al.*, 2020; Ouadi *et al.*, 2002). But the environmental impact, toxicity and relatively high cost of synthetic inhibitors has remained a major challenge such that in recent times, the applications of the new green inhibitors of predominantly plant origin have continued to gain interest in the technological world (Omotosho, 2016; Zarrok *et al.*, 2012). In the field of material science and corrosion control, scientists are persistent in seeking better and more efficient ways of combating the corrosion of metals (Idenyi *et al.*, 2015a; Idenyi *et al.*, 2015b; Idu *et al.*, 2015; Hmamou *et al.*, 2012; Abboudet *et al.*, 2009).

*Oxalis acetosella*, belonging to the *oxalidaceae* family of *eudicots* class in the *angiosperms* phylum of the *plantae* kingdom is a low-growing herbaceous plant with a creeping, rhizomatous growth habit. The leaves are trifoliate, meaning they are divided into three leaflets, resembling the shape of a clover leaf. Each leaflet is typically heart-shaped and measures about 1 to 2.5 cm in width. The leaf color is typically green, but it can turn reddish or purple in certain conditions. The stems are slender, often reddish or purplish in color, and they arise from the rhizomes. The plant spreads via these creeping stems. The flowers of *Oxalis acetosella* are typically white to pale pink with five petals. They are solitary and arise on slender stalks. The flowers may have faint pink or purple veins running through them. After flowering, the plant produces small, capsule-like fruits that contain tiny seeds. *Oxalis acetosella* is commonly found in temperate forests, woodlands, and shaded areas. It prefers moist, well-drained soil and is often seen in cool, damp environments. It has a circumboreal distribution, meaning it can be found in various parts of the Northern Hemisphere, including Europe, North America, and Asia. *Oxalis acetosella* has a sour taste due to the presence of oxalic acid, which gives it a tart flavor. In some regions, its leaves are edible and have been used as a source of vitamin C. However, it should be consumed in moderation because oxalic acid can be harmful in large quantities. This plant serves as a food source for various herbivores and insects in its natural habitat. The presence of *Oxalis acetosella* can indicate the health of woodland ecosystems, as it is often found in undisturbed, well-preserved forest environments (Nesom, 2009; Beaulieu, 2021; Serviss *et al.*, 2021).

*Oxalis acetosella* is known to contain phytochemicals such as ascorbic acid, tocopherols, carotenoids, chlorophyll, flavonoids, phenolic acids and total phenolics (Sircelj *et al.*, 2010; Packham, 1978; Miralrio and Vazquez, 2020). Rodenkirchen (1998) reported in Sircelj *et al* (2010) that the oxalate content for *Oxalis acetosella* is 110 – 140 mg/g dry weight. Siener *et al* (2006) also reported in Sircelj *et al* (2010) that this oxalate value compared favourably with those found in rhubarb and spinach, the two best known high oxalate-containing garden-grown plants. The medical benefits of *Oxalis acetosella* has been extensively investigated. For instance, Dashwood (1997), Dillard and German (2000), Podsedek (2007), and Meyer *et al* (1998) all stated in Sircelj *et al* (2010) that phenolics as well as carotenoids, tocopherols, chlorophyll and ascorbic acid play key role in prevention of various

diseases associated with oxidative stress, such as cardiovascular diseases, cancer, arteriosclerosis, inflammation, neurodegenerative diseases, diabetes and other chronic diseases. Selvi *et al* (2021) investigated the inhibition efficiency of *Oxalis acetosella* in HCL, however, little or no literature exists on the potentialities of *Oxalis acetosella* as a green corrosion inhibitor, particularly in tetraoxosulphate (vi) acid and sodium hydroxide media. Therefore, this work essentially focuses attention on the analysis of the corrosion inhibition performance of calyx of *Oxalis acetosella* in tetraoxosulphate (vi) acid and sodium hydroxide media as a way to broaden the frontiers of knowledge in the area of the utilization of the abundant tropical plants for corrosion prevention and control.

## 2. Methodology

### 2.1 Materials

The materials used for this study includes: mild steel, plastic containers, white sorrel calyx, tetraoxosulphate (vi) acid ( $H_2SO_4$ ), sodium hydroxide (NaOH), distilled water, acetone, emery cloth or sandpaper, cello tape, nylon thread and sieve.

The equipment and tools used include: electronic weighing balance (Tapson's Scientific Instruments Co. Model=TAPT 3000 G), beakers, measuring cylinder, hacksaw and blades, vernier caliper and measuring tape.

### 2.2 Material preparation of mild steel and leaf extracts

Fresh white sorrel's calyx was obtained from the farm behind Taraba State Polytechnic, Jalingo, Nigeria. The calyx of White sorrel were carefully sorted to remove any impurities, washed and dried separately under the shade to reduce their moisture content, after which they were grounded into fine powder. The fine powdered calyx were sieved with a sieve (150  $\mu m$  mesh size). The sieved samples were stored in different containers and labeled until needed for corrosion studies.



**Photo 1:** The leaves, dried and powder of White sorrel calyx.

Extraction of the active ingredients in the calyx of the White sorrel was done using the Reflux Technique. Here, 30 g of the powdered calyx of White sorrel was measured using an electronic balance into four round bottom flasks and each 30 g was soaked in 600 ml of boiling 1.0 M  $H_2SO_4$ , 0.5 M  $H_2SO_4$ , 1.0 M NaOH and 0.5 M NaOH for 1 hour 30 minute on a Bunsen burner. The content of each flask was filtered using a filter paper. The filtrates were used as stock solution. Square mild steel square rods were obtained from a local store. The mild steel rods were cut into seventy two test

specimens each of dimensions (10 mm x 5 mm x 5 mm). The surfaces of the test specimens were polished with abrasive paper to produce smooth surface and to remove any trace of contaminants. The test specimens were degreased in ethanol, washed thoroughly in deionized water and rinsed. The washed specimen were air dried (Ehujuo *et al.*, 2014; Eddy *et al.*, 2011) to remove any moisture that might be present on the test specimens. The required concentrations of the inhibitors (5 ml, 10 ml, 15 ml and 20 ml) were respectively added to each of the beakers containing the corrosion media (0.5 M H<sub>2</sub>SO<sub>4</sub>, 1.0 M H<sub>2</sub>SO<sub>4</sub>, 0.5 M NaOH and 1.0 M HaOH). The test coupons were then tied with nylon strings and suspended inside each of the containers at 4 coupons per container. The setup was allowed to stand on a table in the Faculty of Engineering Laboratory of Taraba State University, Jalingo, Nigeria for 672 hours with one sample removed from each container every 168 hours, washed in a distilled water, rinsed in ethanol, and air-dried before re-weighing.

### 2.3 Weight Loss Measurement

The weight loss measurement was calculated as the difference in weight before and after immersion for the periods of 168 hours, 336 hours, 504 hours and 672 hours respectively and the result was used to calculate the corrosion rate according to the following equation:

$$CR = \frac{87.6W}{\rho At} \quad \text{Eqn.1}$$

Where W = weight loss (mg);  $\rho$  = density of specimen (g/cm<sup>3</sup>), A = area of specimen (cm<sup>2</sup>) and t = period of immersion (hours).

The inhibition efficiencies (IE %) of the *Oxalis triangularis* in each of the corrosion media was calculated using the equation:

$$IE\% = \left( \frac{CR_a - CR_p}{CR_a} \right) \times 100 \quad \text{Eqn.2}$$

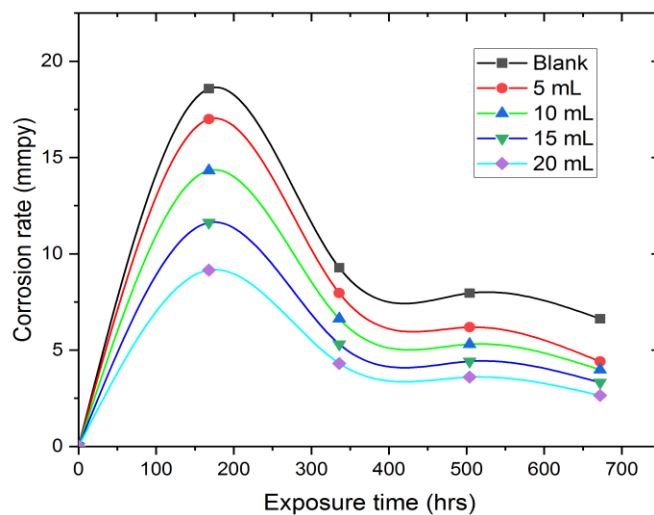
Closely associated with inhibition efficiency is the Langmuir adsorption isotherm adapted for this study. A simplistic assumption was made to the effect that the surface coverage of the adsorbed layer ( $\theta$ ) is related to inhibition efficiency as follows:

$$\theta = \left[ \frac{CR_a - CR_p}{CR_a} \right] \quad \text{Eqn.3.}$$

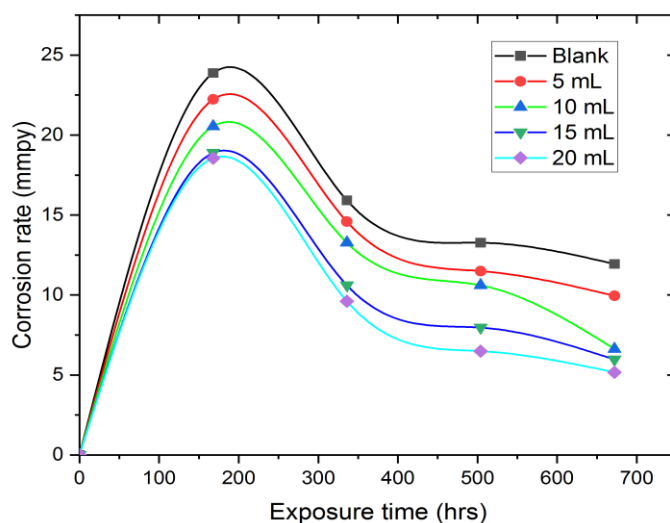
Where,  $CR_p$  = Corrosion rate in the presence of inhibitor,  $CR_a$  = Corrosion rate in the absence of the inhibitor and  $\theta$  = coverage of the adsorbate on the metal surface.

### 3. Results and Discussion

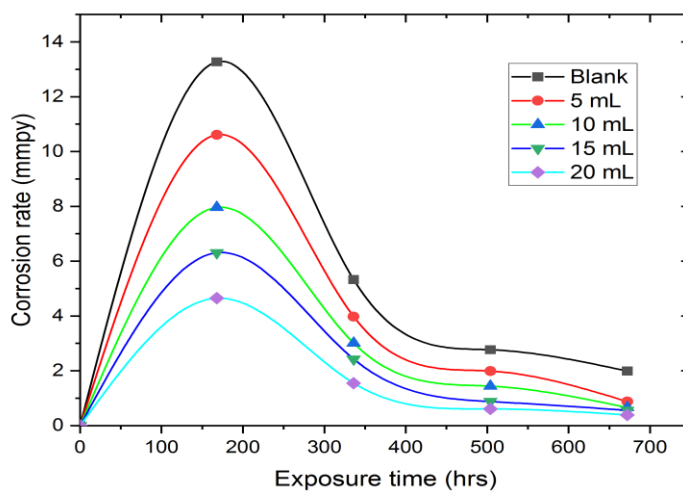
Figures 1 – 4 represent the corrosion rate against exposure time for *Oxalis acetosella* in the corrosion media, Figures 5 – 8 show the inhibition efficiency versus concentration for the extracts in the various media and Figures 9 – 12 are the Langmuir plots for the extracts in the various corrosion media.



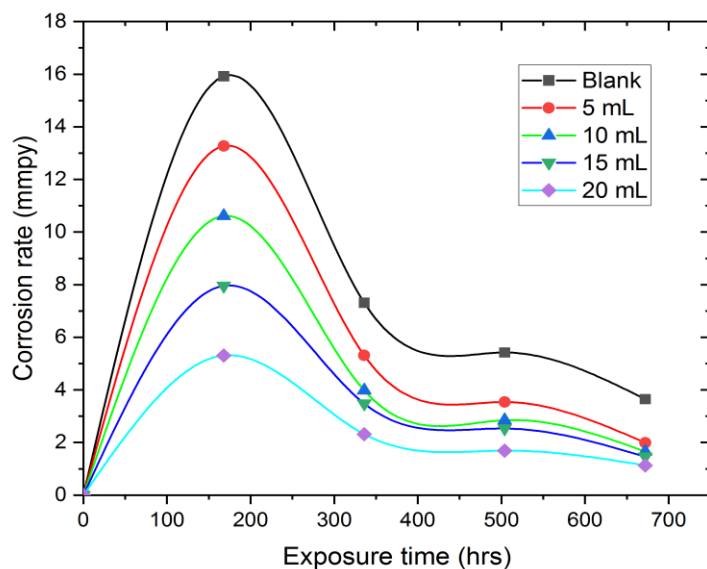
**Figure 1.** Variation of corrosion rate with exposure time for mild steel in 0.5 M H<sub>2</sub>SO<sub>4</sub>



**Figure 2.** Variation of corrosion rate with exposure time for mild steel in 1.0 M H<sub>2</sub>SO<sub>4</sub>



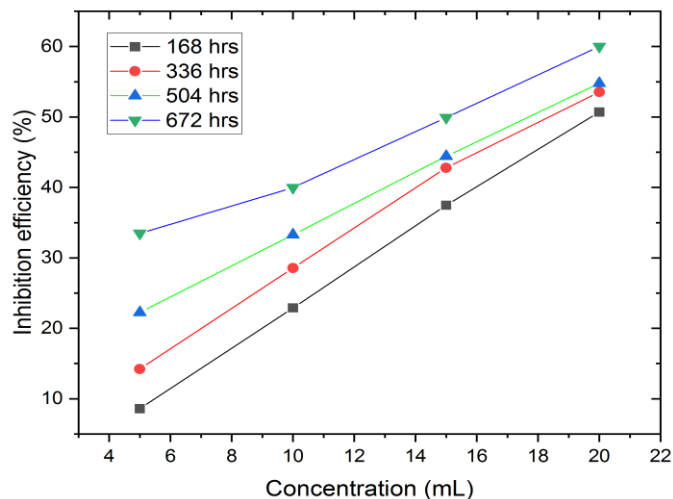
**Figure 3.** Variation of corrosion rate with exposure time for mild steel in 0.5 M NaOH



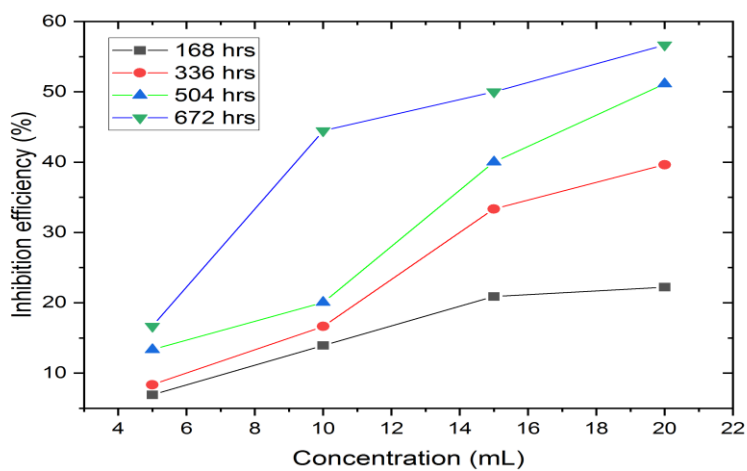
**Figure 4.** Variation of corrosion rate with exposure time for mild steel in 1.0 M NaOH

### Corrosion Rate Analysis

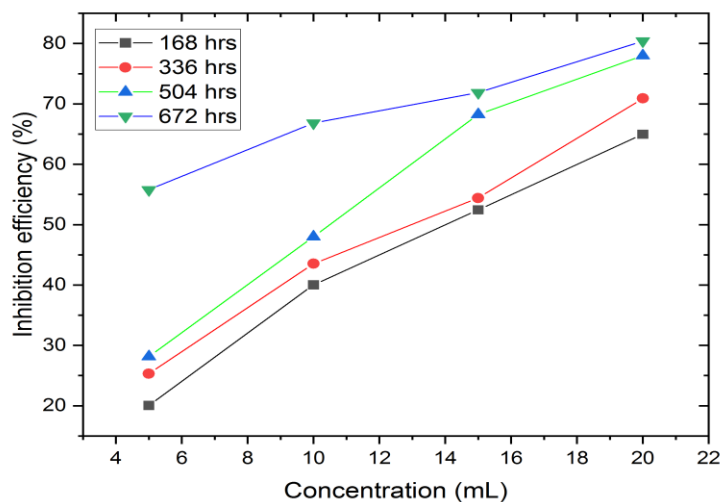
A critical look at [Figures 1 – 4](#) clearly shows that the corrosion rate profiles depict the behavior that is common with passivating metals like steel. There was an initial steep rise in corrosion rate up until the 168 hours period, which was followed by a progressive decrease as the exposure time increased to the 673 hours period. The inference drawn from this is that there was a formation of a film of corrosion product that covered the metal surface over time, thereby preventing a direct contact between the metal and the corrosion media. According to some studies, film formation is an indication of the functional groups possessed by green inhibitor, which is the basic characteristics of corrosion inhibitors ([Nwigbo et al., 2012](#); [Owate et al., 2014](#); [Okewale and Olaitan, 2017](#)), and the presence of these functional groups slow the corrosion rate, thereby protecting the mild steel from corrosion attack. This trend was somewhat however distorted in all the media where at the 336 hours period there were spikes in the corrosion rate depicted by the sudden rise in the plot again peaking at the 504 hours period before declining again to the 672 hour period. These anomalies are attributed to several factors including the decline in the potency of the extract due to deterioration, caving off of the adsorbed layer due to weakening of the adsorbent force ([Dillard and German, 2000](#); [Gopiramin et al., 2012](#); [David, 2021](#); [Kalaiselvi et al., 2010](#); [Devesh et al., 2018](#); [Nahle et al., 2010](#)) and possible agitation of the system setup by interlopers in the surrounding environment where the experiment was mounted ([Idenyi et al., 2015a,b](#); [Okafor et al., 2010](#); [Mejeh et al., 2020](#); [Kamal and Sethuraman, 2012](#)), which in this case is the general purpose laboratory in Taraba State University, Jalingo, Nigeria. In general, however, it is established that extracts of the calyx of *Oxalis acetosella* inhibited corrosion in both H<sub>2</sub>SO<sub>4</sub> and NaOH respectively. The natural extract can be used in pickling acids as well as alkaline media. The inhibition process is due by the various molecules forming the extracts at various contents, leading to a barrier against the arrival of aggressive ions as H<sup>+</sup> and OH<sup>-</sup> responsible on the degradation of metallic materials ([Nick et al., 2011](#); [Lazrak et al., 2021](#); [Fadhil et al., 2020](#); [Barouni et al., 2014](#); [Elmsellem et al., 2014](#); [Saidi et al., 2016](#)).



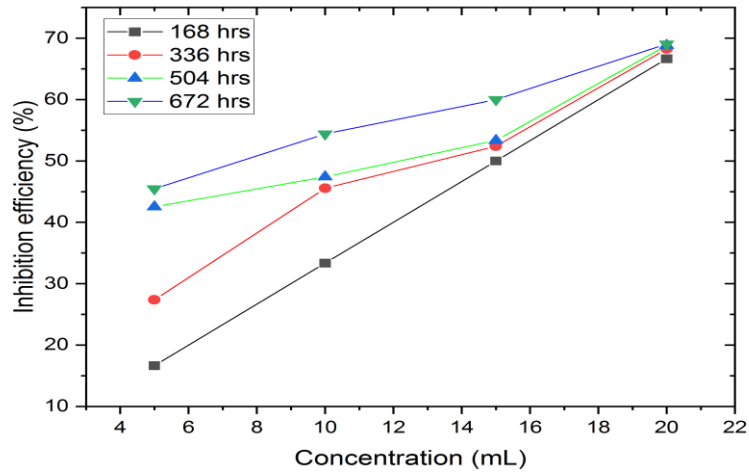
**Figure 5.** Variation of Inhibition efficiency with concentration for mild steel in 0.5 M H<sub>2</sub>SO<sub>4</sub>.



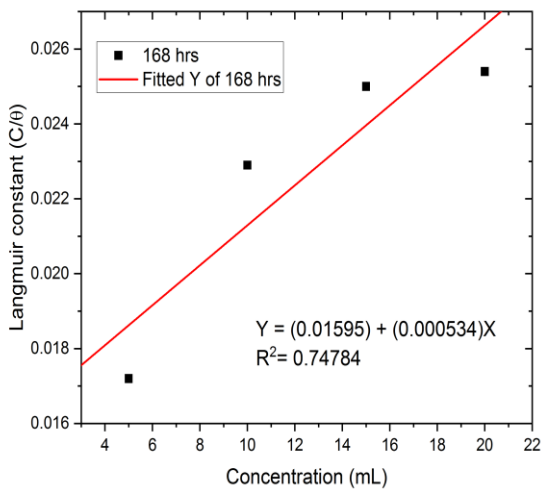
**Figure 6.** Variation of Inhibition efficiency with concentration for mild steel in 1.0 M H<sub>2</sub>SO<sub>4</sub>.



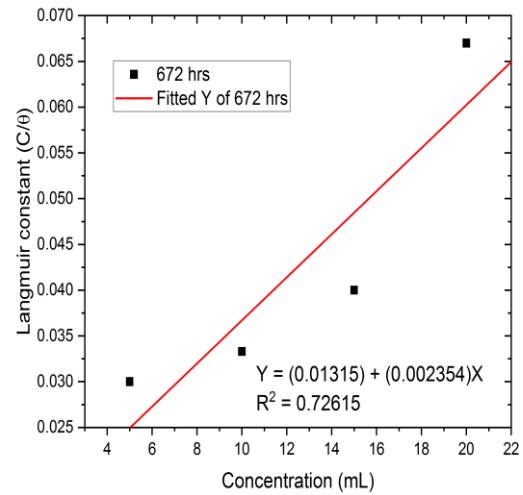
**Figure 7.** Variation of Inhibition efficiency with concentration for mild steel 0.5 M NaOH.



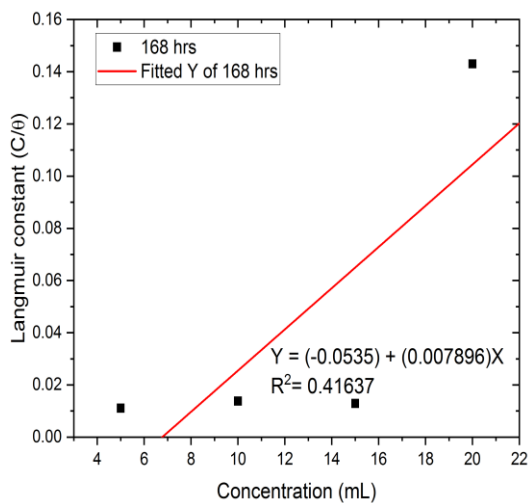
**Figure 8.** Variation of Inhibition efficiency with concentration for mild steel 1.0 M NaOH.



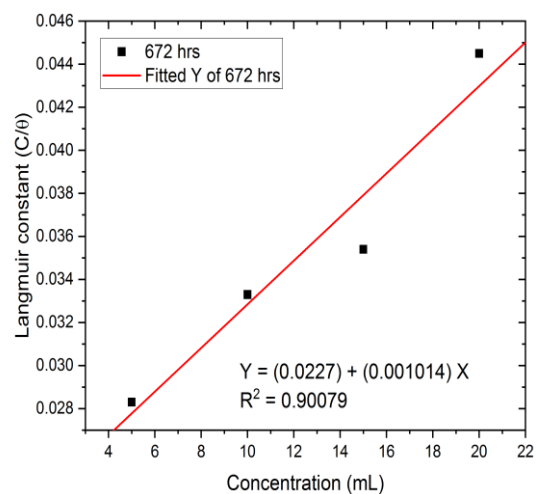
**Figure 9a.** Langmuir adsorption isotherms for white sorrel calyx at 168 hours in 0.5 M H<sub>2</sub>SO<sub>4</sub>.



**Figure 9b.** Langmuir adsorption isotherms for white sorrel calyx at 672 hours in 0.5 M H<sub>2</sub>SO<sub>4</sub>.

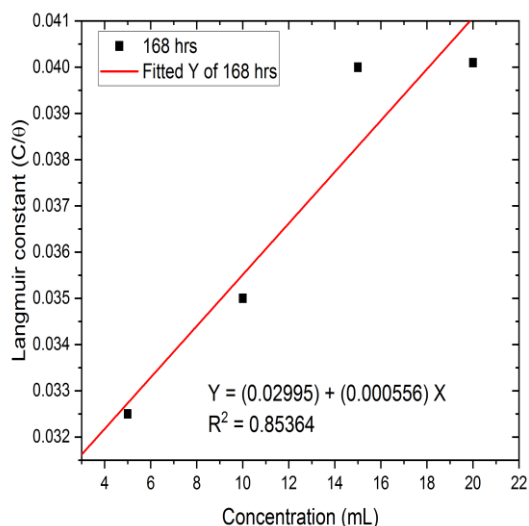


**Figure 10a.** Langmuir adsorption isotherms for white sorrel calyx at 168 hours in 1.0 M H<sub>2</sub>SO<sub>4</sub>.

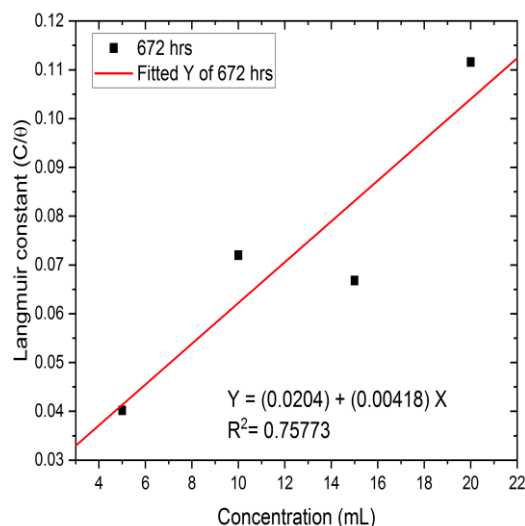


**Figure 10b.** Langmuir adsorption isotherms for white sorrel calyx at 672 hours in 1.0 M H<sub>2</sub>SO<sub>4</sub>.

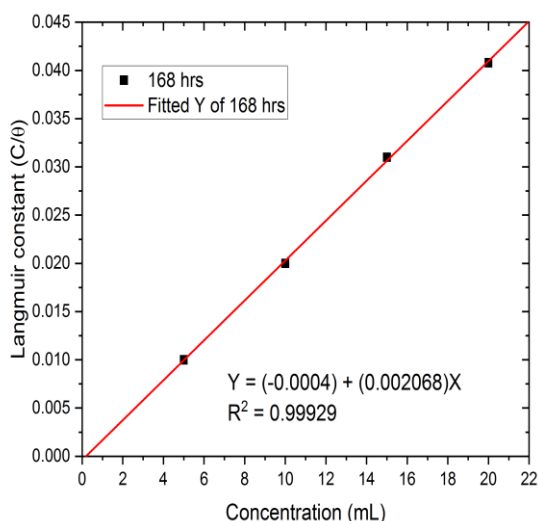




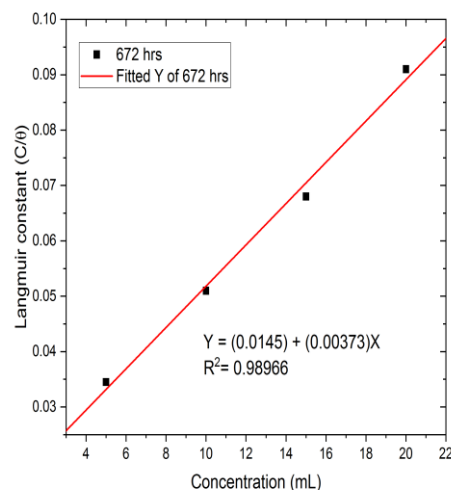
**Figure 11.** Langmuir adsorption isotherms for white sorrel calyx at 168 hours in 0.5 M NaOH



**Figure 11b.** Langmuir adsorption isotherms for white calyx at 672 hours in 0.5M NaOH



**Figure 12a.** Langmuir adsorption isotherms for white sorrel calyx at 168 hours in 1.0 M NaOH



**Figure 12b.** Langmuir adsorption isotherms for white calyx at 672 hours in 1.0M NaOH

### ***Inhibition Efficiency***

Corrosion inhibition efficiency is a measure of how well a corrosion inhibitor substance protects a metal or material from corroding in a given corrosion environment. It is generally a somewhat complex phenomenon in the sense that several factors (and not only exposure time) influence it. Initially, when the inhibitor is applied, and as exposure time increases, it can provide a strong protection against corrosion attack by helping to adsorb on the metal surface thereby creating an enabling corrosion protection film (Loto *et al.*, 2013; Fouda *et al.*, 2017). However, this adsorbed film layer may begin to cave as exposure time increases due to the desorption of the thin film from the metal surface as a result of thermodynamic kinetics and other associated variables like chemical degradation of the inhibitor material, unintended system agitation and such like factors (Odiongenyi *et al.*, 2009; Sanni *et al.*, 2019).

In the present study, the inhibition efficiency of the mild steel immersed in 0.5 M and 1.0 M ( $\text{H}_2\text{SO}_4$ ) was observed to increase with increase in concentration of the *Oxalis acetosella* extract due to the increase in the number of adsorbed molecules on the mild steel surface, thereby blocking the active sites from acid attack. These observations have equally been reported in previous studies (Loto *et al.*, 2013; Fouda *et al.*, 2017). However, increase in exposure time decreases the inhibition efficiency slightly, irrespective of inhibitor concentration. The decline in inhibition efficiency with time could be attributed to desorption of the white sorrel extract from the mild steel surface as has been observed by Odiongenyi *et al* (2009) and Sanni *et al* (2019)

### **Langmuir Adsorption Isotherm**

Langmuir plots are typically associated with adsorption phenomena, where they illustrate the relationship between the amount of substance adsorbed onto a surface and the concentration of that substance in the surrounding environment (Nwabanne & Okafor, 2012). In the context of corrosion, Langmuir plots can be used to study the relationship between the extent of corrosion and the concentration of a corrosive species (such as an electrolyte or a corrosive gas) in the environment surrounding the metal surface (Umoren *et al.*, 2006). A Langmuir plot used in the context of corrosion would involve plotting the reciprocal of the Langmuir constant ( $K^{-1}$ ),  $C/\theta$  on the y-axis and the concentration of the corrosive species,  $C$  on the x-axis. Plotting the reciprocal of the Langmuir ( $K^{-1}$ ) against concentration can offer valuable insights into the adsorption process. A linear relationship, like in these cases shown indicates that the adsorption followed the Langmuir model, showcasing a consistent equilibrium between adsorbed molecules and available surface sites (Oguzie *et al.*, 2004; Eddy *et al.*, 2008; Bouyanzer *et al.*, 2016; Da-Rocha *et al.*, 2010; Eddy, 2010). Two other important considerations are the slope and the intercept of the plot. A positive slope of the line indicates that there is maximum adsorption at equilibrium for the given adsorbate-adsorbent system (Allaoui *et al.*, 2017; Aribo *et al.*, 2017; Eddy & Ebenso, 2010), which in this case is the mild steel surface and the medium containing the extract of *Oxalis acetosella*. An intercept generally offers insights into the factors affecting adsorption. If it occurs on the y-axis, it suggests that at zero concentration, the reciprocal of the Langmuir constant ( $K^{-1}$ ) approaches infinity such that at very low concentrations, the adsorption process becomes highly favourable or approaches an infinite binding capacity between the adsorbent and the adsorbate; while on the x-axis, it suggests a high affinity of the adsorbent for the adsorbate, indicating strong binding or interaction between the two (Ekanem *et al.*, 2010; Ezemonye *et al.*, 2006; Fouda *et al.*, 2017; Kumpawat *et al.*, 2010; Odiongenyi *et al.*, 2009; Oguzie. 2008; Patela *et al.*, 2010). The examination of literature shows that the natural extract contains polyphenols, flavonoids, anthocyanins, and tannins (Peredo Pozos *et al.*, 2020; Sirag *et al.*, 2014). The presence of these kind of molecules contributes to the inhibition process via the synergistic intermolecular effect of the various molecules (Lrhoul *et al.*, 2023; El Mounsi *et al.*, 2015; El Ouariachi *et al.*, 2015).

### **Conclusion**

Based on the foregoing discussion, we conclude that the extract of *oxalis acetosella* calyx can be a veritable green inhibitors in both acidic and basic environments and can be used as a substitute for the present day synthetic inhibitors used in our oil and gas industries but which are known to be hazardous to health and the environment, including the fact that they are generally expensive as well. It is suggested that further works should focus on the development of an efficient and cost-effective extraction process for harnessing the potentialities of this inhibitor for industrial use.

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**Disclosure statement:** *Conflict of Interest:* The authors declare that there are no conflicts of interest.

*Compliance with Ethical Standards:* This article does not contain any studies involving human or animal subjects.

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