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Eco–friendly impact of orange (*Citrus sinensis*) seed extract as corrosion inhibitor for aluminium in 2 M HCl solution

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Abstract: The inhibition of corrosion of aluminium in 2 M hydrochloric acid solution by orange (*Citrus sinensis*) seed extract has been studied by weight loss, thermometric and hydrogen evolution methods. The results obtained from weight loss measurements revealed that *C. sinensis* demonstrated a good corrosion inhibition performance, as it considerably reduced the corrosion rate of aluminium in hydrochloric acid solution, achieving the highest inhibition efficiency of 82.69% at 5 g/L extract concentration at 30 °C. The inhibition efficiency increased with increase in temperature from 30 °C to 40 °C. Analysis of data by the thermometric method indicate a reduction in the reaction number in the presence of the extract relative to the blank. The highest inhibition efficiency obtained was 69.9% at 5 g/L extract concentration. The hydrogen evolution data indicate a drastic reduction in the volume of hydrogen gas evolved from the corrosion process in the presence of the extract compared to the blank. The highest inhibition efficiency recorded by the method was 89.80% at 5 g/L extract concentration at 30 °C. The corrosion inhibitory properties of *C. sinensis* seed extract could be attributed to the presence of phytochemicals which adsorb on the metal surface and block it from attack by aggressive ions. Chemisorption process has been proposed for the adsorption of *C. sinensis* seed extract onto aluminium surface. The adsorption of *C. sinensis* seed extract onto aluminium surface obeyed the Langmuir adsorption isotherm.

1. Introduction

Aluminium is used in the manufacture of both domestic and industrial appliances. Due to the presence of a thin oxide film on its surface, aluminium is resistant to corrosion attack (Melian *et al.*, 2021). However, this oxide layer dissolves when it reacts with acids and alkalis (Oguzie *et al.*,2007), thereby resulting in the corrosion of the exposed aluminium surface. Consequently, in order to prolong the service life of aluminium metal or alloy exposed to aggressive environments, such as during the pickling process, the use of corrosion inhibitors is imperative.

The use of green inhibitors, having more recognition in past years, is in high demand nowadays because of their environmentally friendly characteristics (Alao *et al.*,2023; El Ouariachi *et al.*,2010). Plant extracts are widely used because they possess versatile chemical, physical, and biological features. Low cost, biodegradability, and availability are the other importance of plants being applied as the bases of corrosion inhibitors (Fernine *et al.*,2021; Abboud *et al.*,2009; Hjouji *et al.*, 2023). Plants are considered a high natural origin of chemical compounds (polyphenols, flavonoids, peptides…), which can be easily hauled out with less cost and low ecosystem pollution (Atrooz *et al.*,2009). Some seeds extract reported as potential inhibitors for aluminium corrosion in acidic medium include azwain (Patel and Patel, 2015), fennel (Ladha *et al.*, 2015), *Cyamopsis tetragonolubus* (Goswami *et al.*, 2015), cantaloupe (Emran *et al.*, 2014) and akuamma (Ezeugo *et al.*, 2019).

Citrus sinensis (orange) belongs to the family Rutaceae. The preliminary phytochemical screening of *Citrus sinensis* seeds extract reveal the presence of phlobatannins, carbohydrates, flavonoids, alkaloids and terpenoids (Reddy *et al.*, 2018; Favela-Hernández *et al.*, 2016). The richness of chemical composition of C. sinensis (Juice, zest, peels and seeds) is behind its multiple uses in medicinal cares (Olawale *et al.*, 2018; Odeh *et al.*, 2014); Also, Salem *et al.* evaluated the insecticidal activity of the methanol extracts from citrus reticulate and Citrus sinensis against larvae and adults of Tribolium castaneum (Olawale *et al.*, 2018). Bensouda et al., confirmed the anticorrosion potential of Orange zest oil of steel in HCl solution (Bensouda *et al.*, 2019). The use of *Citrus sinensis* seeds extract as corrosion inhibitor for aluminium in acidic medium has not been systematically studied (Olawale *et al.*, 2018). The aim of this work is to assess the inhibitory effect of *Citrus sinensis* seed extract on aluminium corrosion in hydrochloric acid solution.

2. Methodology

2.1 Test Materials

The aluminium sheet used for this work had the following composition (w/w %): Fe (0.09), Cu (0.03), Si (0.13), Mn (0.05), Mg (0.10) and Al (99.60). It was mechanically press - cut into 4 cm x 5 cm coupons and polished to mirror finish using different grades of silicon carbide papers. The coupons were degreased in absolute ethanol, dipped in acetone before air-drying. They were then stored in a moisture – free desiccator prior to use for corrosion studies.

2.2 Preparation of *Citrus sinensis* seed extract

Ripe *Citrus sinensis* (orange) fruits were bought from a farm in Uyo, Nigeria. They were cut open and the seeds removed. The seeds were washed with deionized water and oven-dried at 40 °C till constant weight. The hard outer cover on the seeds were removed before grinding to powder. A sample (10 g) of the powdered seed was digested in 2 L of 2 M HCl solution in a corked container for 24 hours before it was filtered to get the stock (5 g/L). Different concentrations of the extract (1 g/L, 2 g/L, 3 g/L and 4 g/L) were obtained by serial dilution.

2.3 Weight loss method

Previously cleaned and weighed aluminium coupons were suspended with the aid of glass hooks and rods and immersed in 100 cm^3 of 2 M HCl solution (blank) and in 2 M HCl solution containing 1 g/L – 5 g/L *C. sinensis* seed extract (inhibitor) in open beakers. One aluminium coupon per beaker was used in each experiment. The beakers were then placed in a thermostatic water bath maintained at 30 °C and 50 °C, respectively. The aluminium coupons were retrieved from the test solutions after 30 minutes, scrubbed with bristle brush under running water, dipped in acetone before air – drying. The coupons were then reweighed. The inhibition efficiency I_{WL}(%) was calculated using the formula in Eqn. 1 (Abakedi *et al.*, 2016; Oladunni *et al.*, 2020; Kamarska, 2023):

$$I_{WL}\% = \left(\frac{W_0 - W_1}{W_0}\right) \times 100 \qquad \textbf{Eqn. 1}$$

where W_0 and W_1 are the weight losses of the aluminium coupons in the absence and presence of *C. sinensis* seed extract, respectively, in 2 M HCl at the same temperature.

The corrosion rate (CR) of aluminium in 2 M HCl solution was evaluated using Eqn. 2 (Abakedi, 2017a):

$$CR(\text{mg cm}^{-2}\text{hr}^{-1}) = \frac{W}{At} \qquad \textbf{Eqn. 2}$$

where W is the weight loss (mg), A is the total surface area (cm^2) while t is the exposure time (hours).

2.4 Thermometric Method

The thermometric method was performed using the instrumentation and procedure of corrosion testing reported in literature (El-Etre, 2001). The corrodent concentration was 2 M HCl while the volume of test solution used was 50 cm^3. The initial temperature in all experiments was at 30.0 °C. The change in temperature with time was recorded using a digital thermometer to the nearest ± 0.1 °C. The reaction number (RN) is defined as (Oza and Sinha, 1982) and is shown in Eqn. 3

$$RN\ (°C\ \text{min}^{-1}) = \frac{T_m - T_i}{t} \qquad \textbf{Eqn. 3}$$

where T_m is the maximum temperature, T_i is the initial temperature and ‘t’ is the time (min) taken to reach the maximum temperature. The inhibition efficiency I_{TM} (%) was calculated using Eqn. 4 below:

$$I_{TM}(\%) = \left(\frac{RN_0 - RN_1}{RN_0}\right) \times 100 \qquad \textbf{Eqn. 4}$$

where RN_0 and RN_1 are the reaction numbers in the absence and presence of extract, respectively.

2.5 Hydrogen Evolution Method

The hydrogen evolution tests (via a gasometric assembly) were performed as described previously (Obot *et al.*, 2011). The reaction vessel contained 100 cm^3 of 2 M HCl solution. One 4 cm x 5 cm aluminium coupon was dropped into the 2 M HCl solution (blank) and the reaction vessel quickly corked. The volume of H_2 gas evolved from the corrosion reaction was recorded every 60 seconds for 35 minutes. The experiment was repeated using 1 g/L – 5 g/L *C. sinensis* seed extract in 2 M HCl solution. The inhibition efficiency I_{HE} (%) was calculated using Eqn. 5 (Abakedi, 2017b):

$$I_{HE}\ (\%) = \left(\frac{V_0 - V_1}{V_0}\right) \times 100 \qquad \textbf{Eqn. 5}$$

where V_0 and V_1 are the volumes of hydrogen gas evolved in the absence and presence of extract, respectively, at a specified time.

3. Results and Discussion

3.1 Effect of *Citrus sinensis* seed extract concentration on inhibition efficiency

The inhibition efficiency by the weight loss method increases with increase in *C. sinensis* seed extract concentration, at a particular temperature (Figure 1). The data obtained by the thermometric tests (Figure 2) reveals an increase in the time taken to reach the maximum temperature as well as a reduction in the maximum temperature attained in the presence of the extract relative to the blank.

Table 2 reveals that the inhibition efficiency by the thermometric method increases with increase in the extract concentration. Figure 3 indicates a reduction in the volume of hydrogen gas evolved in the presence of *C. sinensis* seed extract compared to the blank. The calculated values of inhibition efficiency by the hydrogen evolution method presented in Table 2 reveal that the inhibition efficiency increases with increase in the seed extract concentration. The data obtained by the three methods confirm that *C. sinensis* seed extract inhibited the corrosion of aluminium in 2 M HCl solution. Additionally, an increase in the inhibition efficiency with increase in *C. sinensis* seed extract concentration implies that the extract inhibited the corrosion process by possibly adsorbing on the metal surface, thereby blocking the aggressive ions in solution from attacking the metal surface

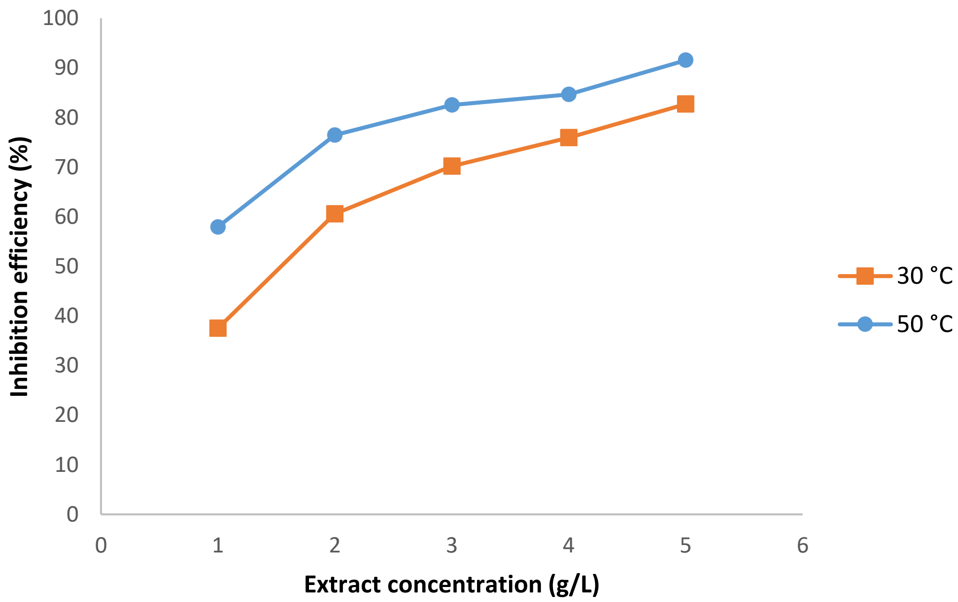


Figure. 1: Effect of *C. sinensis* seed extract concentration on the inhibition of aliminium corrosion in 2 M HCl solution at 30 °C and 5

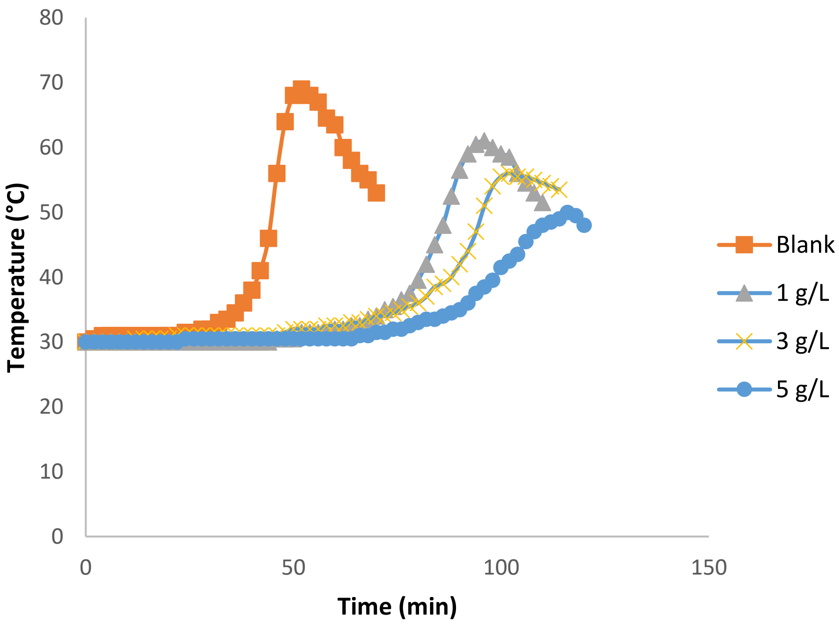


Figure. 2: Temperature – time curve obtained in the absence and presence of various concentrations of *C. sinensis* seed extract in 2 M HCl solution

Table 1: Effect of C. sinensis seed extract on inhibition efficiency of aluminium corrosion in 2 M HCl solution (Thermometric measurements)

Extract conc. (g/L)	Initial temp. T_1 (°C)	Maximum temp. T_m (°C)	Time taken to reach max. temp. t (min)	Reaction number RN (°C/min)	Inhibition efficiency I (%)
Blank	30.0	69.0	53	0.7358	-
1	30.0	61.0	96	0.3229	56.1
3	30.0	56.0	102	0.2549	65.4
5	30.0	55.5	115	0.2217	69.9

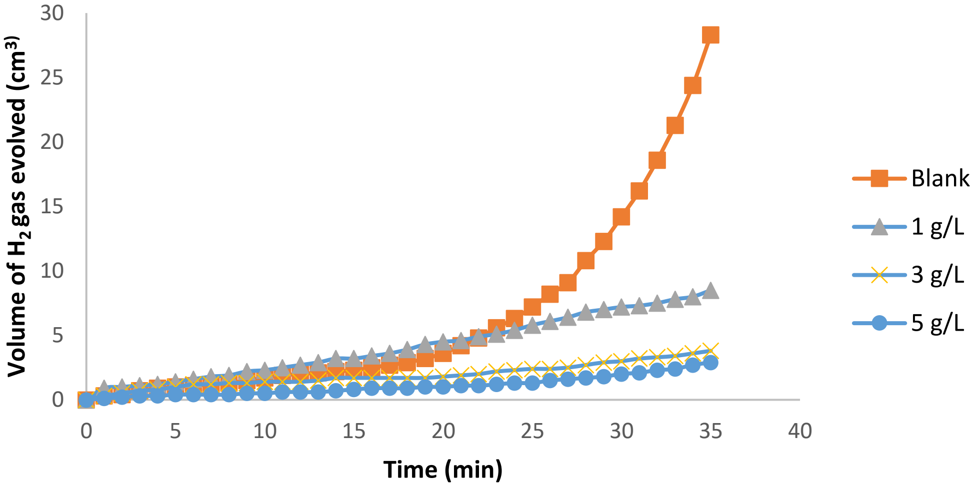


Figure 3: Volume – time curve obtained in the absence and presence of various concentrations of *C. sinensis* seed extract in 2 M HCl solution

Table 2: Effect of *C. sinensis* seeds extract on the inhibition efficiency of aluminium corrosion in 2 M HCl solution (Hydrogen evolution measurements

Extract conc. (g/L)	Volume of H_2 evolved (cm^3)	Time taken (min)	Inhibition efficiency (%)
Blank	28.3	35	-
1	8.5	35	70.00
3	3.8	35	86.60
5	2.9	35	89.80

3.2 **Effect of temperature on inhibition efficiency**

The inhibition efficiency of *C. sinensis* seed extract increases with increase in temperature (Table 3). This indicates that the extract inhibits aluminium corrosion better at higher temperature than at lower temperature. This probably signifies that the extract adsorbed onto the metal surface by a chemisorption process. The activation energies (E_a) of the corrosion of aluminium in HCl solution in the absence and presence of *C. sinensis* seeds extract were calculated using Eqn. 6, the Arrhenius equation (Ita and Abakedi, 2006):

$$\log\left(\frac{CR_2}{CR_1}\right) = \frac{E_a}{2.303R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \qquad \textbf{Eqn. 6}$$

where CR_1 and CR_2 are corrosion rates at temperatures T_1 (303 K) and T_2 (323 K), respectively, while R is the universal gas constant.

The activation energies (E_a) obtained using Eqn. 6 are presented in Table 4. Which depicts that the E_a values in the presence of *C. sinensis* seed extract were lower than that of the blank (182.46 kJ mol^{-1}). The lower E_a values in the presence of extract relative to the blank in addition to an increase in the inhibition efficiency with increase in temperature could indicate chemisorption of the extract onto the metal surface (Bentiss *et al*., 2005).

Table 3: Calculated values of corrosion rate and inhibition efficiency for aluminium corrosion in 2 M HCl solution containing *C. sinensis* seed extract (Weight loss measurements)

Extract conc.(g/L)	Corrosion rate (mg cm^{-2} hr^{-1})		Inhibition efficiency (%)	
	30 °C	50 °C	30 °C	50 °C
Blank	0.520	46.005	-	-
1	0.325	19.345	37.50	57.95
2	0.205	10.845	60.58	76.43
3	0.155	8.035	70.19	82.53
4	0.125	7.060	75.96	84.65
5	0.090	3.895	82.69	91.53

Table 4: Calculated values of activation energy (E_a) for aluminium corrosion in 2M HCl solution containing *C. sinensis* seeds extract

Extract concentration (g/L)	E_a (kJ mol^{-1})
2 M HCl (Blank)	182.46
1	166.33
2	161.53
3	160.70
4	164.19
5	153.36

3.3 Adsorption isotherm

Several adsorption isotherms were used to fit the adsorption of *C. sinensis* seed extract onto aluminium surface. The best fit for the data was found to conform to the Langmuir adsorption isotherm defined as (Iroha and Maduelosi, 2021; Satpati *et al.*, 2023; Lin *et al.*, 2024)

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad \textbf{Eqn. 7}$$

where C is the inhibitor concentration, θ is the degree of surface coverage while K_{ads} is the adsorption equilibrium constant. The obtained linear plot of C/θ vs. C (Figure 4) confirms that the adsorption of *C. sinensis* seed extract onto aluminium surface obeys the Langmuir adsorption isotherm.

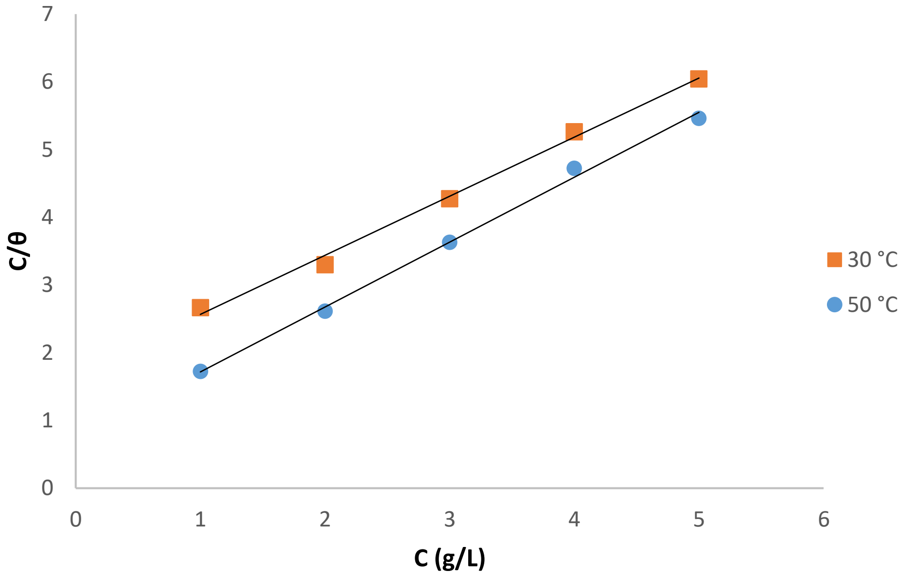


Figure. 4: Langmuir isotherm plot for aluminium corrosion in 2 M HCl solution containing *C. sinensis* seed extract

The values of K_{ads}, evaluated from the intercept of the graph, are presented in Table 5 (Nnanna *et al.*, 2011). The values of standard free energy of adsorption (ΔG_{ads}) were evaluated using the equation:

$$K_{ads} = \frac{1}{1000} \exp\left(\frac{-\Delta G^{\circ}_{ads}}{RT}\right) \qquad \textbf{Eqn. 8}$$

where R is the universal gas constant, T is the absolute temperature while 1000 is the massic concentration of water in the solution.

It's noted that the determination of ΔG°_{ads} when extract of natural plants is used as corrosion inhibitor, has no meaning because of the complexity of natural extract containing more than a known molecule as well as the concentration is expressed by g/L, not in mol/L; and consequently, ΔG°_{ads} is given in numerous papers in kJ/mol.

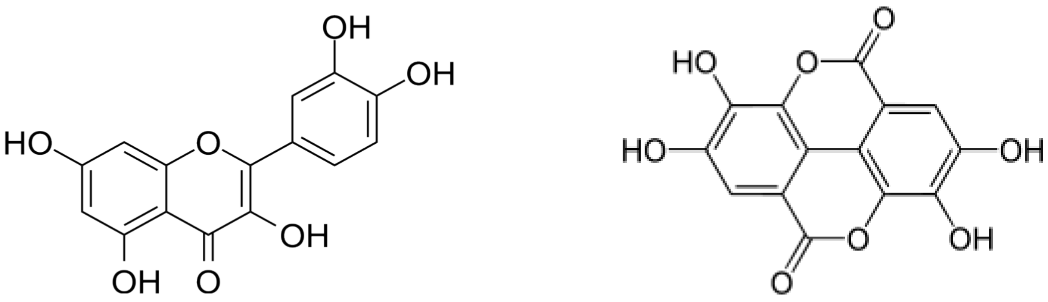
Several authors pointed out that the inhibitory process can be interpreted by the action of various components of the natural extract at different concentrations (Ezeh *et al.*, 2023; Lrhoul *et al.*, 2023; Yan *et al.*, 2019; Ali *et al.*, 2014). In other words, the synergistic combination effect of molecules that constitute extracts renders the standard adsorption free energy parameter insignificant (Lrhoul *et al.*, 2023).

Table 5: Some parameters of the linear regression of Langmuir isotherm for aluminium corrosion in 2 M HCl solution containing *C. sinensis* seed extract

Temperature	R^2	$1/K_{ads}$ (g L^{-1})	K_{ads} (g^{-1} L)
30 °C	0.9951	1.6937	0.5904
50 °C	0.9969	0.7583	1.3187

It is observed in Table 5 that the value of K_{ads} at 50 °C was higher than that at 30 °C. This indicates that the extract adsorbed more strongly onto the metal surface as the temperature increased. This viewpoint is in agreement with a previous publication (Abakedi and Asuquo, 2016).

The literature suggests that Citrus sinensis (C. *sinensis*) contains several bioactive molecules that play a crucial role in facilitating the adsorption phenomenon on metal surfaces. This process effectively creates a protective barrier against the intrusion of aggressive ions like H^+, thereby preventing metal corrosion. The adsorption mechanism primarily operates through the presence of cyclic rings, double or triple bonds, and heteroatoms such as oxygen (O), nitrogen (N), sulfur (S), and phosphorus (P). Figure 5 illustrates the major C. *sinensis* seed extract constituents (flavonoids and polyphenols). Flavonoids possess general chemical structures characterized by multiple aromatic rings fused together, often containing hydroxyl (-OH) groups and various functional groups such as carbonyl (-C=O) groups. Polyphenols, another prominent class of compounds found in C. *sinensis*, also contribute significantly to its corrosion inhibition properties. Polyphenols characterized by multiple phenolic (hydroxylated benzene) rings capable to interact with vacant orbitals of aluminium surface, thereby preventing corrosion. The rich concentration of these bioactive molecules underscores the potential of C. *sinensis* as a natural corrosion inhibitor against the corrosion of aluminium surface in 2M HCl solution. This inhibition process may be called a synergistic intermolecular effect between all components of the extract as well as intramolecular effect of various functional groups in the same molecule (Fekkar *et al.*, 2021).



General formulae of Flavonoid ***General formulae of polyphenol***

Figure. 5: Chemical Constituents of *C. sinensis* Seed Extract: Flavonoids and Polyphenols

Conclusion

Based on this work, the following conclusions could be made: Orange (*C. sinensis*) seed extract is a good inhibitor for aluminium corrosion in 2 M hydrochloric acid solution. The inhibition efficiency increases with increase in *C. sinensis* seed extract concentration and temperature. Based on an increase in the inhibition efficiency with increase in temperature coupled with a decrease in the E_a value in the presence of the extract relative to the blank, chemical adsorption process is proposed for the adsorption of the extract onto aluminium surface. The adsorption of *C. sinensis* seed extract onto aluminium surface conformed to the Langmuir adsorption isotherm. The extract adsorbed spontaneously onto aluminium surface.

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