

3 **Inhibitive and synergistic properties of ethanolic extract of *Moringa oleifera***
4 **leaves on the corrosion of zinc in HCl solution**

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6
7 **ABSTRACT**

8 The corrosion inhibition potentials of ethanolic extract of *Moringa oleifera* leaves was
9 investigated by evaluating the corrosion behaviour of Zinc immersed in 0.5M, 1.0M, 1.5M and
10 2.0M HCl solutions each containing varied concentration of the extract (0.1g/L, 0.3g/L and
11 0.5g/L) and halides(0.1M, 0.3M and 0.5M each of KCl and KI) for synergism using
12 thermometric method. Maximum temperature attained, reaction time, synergism parameter and
13 adsorption characterizations were utilized to evaluate the corrosion inhibition, synergism and
14 adsorption properties of the extract. The results revealed that, the corrosion of Zinc decreases
15 with increase in concentration of ethanol extract and halides, also, increases with increase in HCl
16 concentration. The addition of KI and KCl to the extract, improve the values of inhibition
17 efficiency of the extract in the order KI > KCl. In all cases, KI exhibits least effect on the
18 corrosion of Zinc in comparison with KCl. Results obtained from adsorption studies indicated
19 that, the ethanol extract, KCl and KI were adsorbed on the surface of the Zinc and that the
20 adsorption fit excellently with the assumptions of the Langmuir adsorption isotherm model. All
21 data acquired revealed that the ethanol extract is an efficient inhibitor of corrosion in acid
22 medium due to its phytochemicals: saponins, tannins, flavonoids, glycosides, carbonhydrates,
23 reducing sugars, terpenoids, steroids and alkaloids.

24 **Key words:** *Moringa oleifera*, Corrosion Inhibition, Halides, Adsorption, Synergism

25 **1. Introduction**

26 Scientists are relentless in seeking better and more efficient ways of combating the
27 corrosion of metals. Among other methods, adding inhibitors to the corrosion environment has
28 been employed. The use of inhibitors has been well documented as an effective method of
29 protecting metallic materials from corrosion [1]. Corrosion inhibitors are substances which when

added in small concentrations to corrosive media decrease or prevent the reaction of the metal with the media. The efficiency of these inhibitors is sometimes improved by the addition of some other compounds which act in synergism. Inhibitors are added to many systems, namely, cooling systems, refinery units, chemicals, oil and gas production units, boiler, and so forth.

Most of the effective inhibitors are used to contain hetero atoms such as O, N, and S with multiple bonds in their molecules through which they are adsorbed on the metal surface. It has been observed that adsorption depends mainly on certain physicochemical properties of the inhibitor group, such as functional groups, electron density at the donor atom, π -orbital character, and the electronic structure of the molecule [2]. Many studies have been carried out using synergistic corrosion inhibitors [3-4].

Though many synthetic compounds showed good anticorrosive activity, most of them are highly toxic to both human beings and environment. The use of chemical inhibitors has been limited because of the environmental threat recently, due to environmental regulations. These inhibitors may cause reversible (temporary) or irreversible (permanent) damage to organ system, namely, kidneys or liver, or disturbing a biochemical process or disturbing an enzyme system at some sites in the body. The toxicity may be manifested either during the synthesis of the compound or during its applications[2].

In the search for more environmentally friendly and readily available inhibitors, researchers have reported the use of local plants such as *Vernonia amygdalina*[5], *Lawsonia* [6]; among others. In line with these *Nypa fruticans*' *wurmba* local plant has also been reported as inhibitor for mild steel in HCl medium, which has similar inhibition properties with 1,5-di-phenyl carbazone [7]. Orubite and Oforka, and Orubite *et al*, studied the effect of Potassium Iodide by extract of *Nypa Fruticans*' *wurmb* and showed that the mild steel is more efficiently inhibited by *Nyapafruticans*' *wurmb* in presence of KI than pure extract of *Nyapafruticans*' *wurmb* [7-8]. The inhibitive effect of the natural juice of onion bulb on the corrosion of zinc in HCl solution was studied by Abdel-Gaber using hydrogen evolution and weight loss measurements as well as potentiodynamic leav polarization technique [9]. Onion juice reduced markedly the corrosion rate of zinc in the acid solution. The inhibition efficiency increases as the juice concentration is increased. Ayuba and Mustapha investigated the inhibitive and synergistic

properties of Ethanolic Extract of *Anogeissus leiocarpus* leaves on the corrosion of Aluminium in HCl solution and found that The corrosion inhibition of aluminium in HCl in the presence of ethanol extract of *Anogeissus leiocarpus* at room temperature was studied using the thermometric technique [10]. The effect of addition of halides (KCl, KBr, KI) is also reported. The percentage inhibition efficiency (% I) increased with increase in concentration of ethanol extract of *Anogeissus leiocarpus* leaves. The addition of the halides to the plant extract increased the inhibition efficiency to a maximum of 95.18% with KI at 0.6g/L concentration. The higher the corrodent (HCl) concentration, the higher the corrosion rate, while lower inhibition efficiency was observed in the absence and presence of the inhibitor and halides.

A number of successful studies on the use of plant extract as corrosion inhibitors have been investigated and reported by several authors (Oguzie, Vijayalakshmi *et al*, Lebrini *et al*, Loto *et al*, Saratha and Vasudha, Adeyemi and Olubomehin, James and Akarata, and Nnanna *et al*) [11-18]. There are however concerns on the sustainability of the use of some of these green inhibitors when commercial scale processing is of interest, since some of the extracts can only be derived from specific plants which are still utilized for so many other applications; coupled with the fact that some of these plants are seasonal. These factors place limits to the extent most green inhibitors could be applied commercially.

The objective of this work is to focus on the inhibitive properties of some natural products particularly *Moringa oleifera* leaves for use as corrosion inhibitors for zinc in acid medium. The choice of *Moringa oleifera* leaves is significant for it has some phytochemical properties, and there was no report in literature on the use of this plant on corrosion inhibitors.

2. Materials and methods

The elemental composition of zinc sheets used for this study was determined using a spark spectrometric analyzer and summarized thus:

Table 1: Elemental Composition of Zinc sheets

Element	Weight (%)
Pb	0.001

Fe	0.002
Cd	0.001
Cu	0.003
Zn	99.993

85

86 These sheets were mechanically press-cut into 5 x 4 cm coupons of 0.11cm thickness.
87 The coupons were screened carefully to check for rough edges, which could influence the
88 corrosion monitoring process. Surface treatment of the coupons was carried out by degreasing in
89 absolute ethanol and drying in acetone. They were then stored in moisture free desiccators before
90 use. All reagents and chemicals used for the study were of Analar grade and double distilled
91 water was used for their preparation.

92 **2.1 Extraction of plant**

93 Samples of *Moringa oleifera* leaves were obtained from Ganaja Village, Lokoja, after
94 which they were identified and named at Biological Sciences Department, Federal University,
95 Lokoja, Nigeria. The Samples were washed, air dried, ground, sieved and soaked in a solution of
96 ethanol for 1 week to percolate and were filtered. The filtrates were further subjected to
97 concentration using a rotary vapour (BUCHI R110, 40°C) to obtain thick syrup which was later
98 allowed to air dry. The dry extract so obtained were used in preparing different concentrations of
99 the plant extract by dissolving 0.1g, 0.3g and 0.5g of the extract each differently in 1 dm³ of 0.5,
100 1.0, 1.5 and 2.0 M HCl respectively [19].

101 **2.2 Chemical analysis**

102 The various methods for the identification and quantification of Phytochemicals in plant
103 extract as reported by Ugwu *et al* and Eddy *et al.*, were used for the identification of the various
104 phytochemicals in ethanol extract of *Moringa oleifera* leaves [19] and [20-21].

105 **2.3 Thermometric method**

106 The reaction vessel is a three-necked round bottom flask. The procedure for determining
107 the corrosion behaviour by this method was described elsewhere by Ayuba and Mustapha
108 [10]. The flask was well lagged to prevent heat losses. In the thermometric method, the corrosive

(HCl) concentration was varied at 0.5, 1, 1.5 and 2 M, while halides (KCl and KI) were used at varying concentrations of 0.1, 0.3 and 0.5M (for synergism) respectively. The volume of the test solution used was 50cm³. The initial temperature in all the experiments was kept at room temperature. The progress of the corrosion reaction was monitored by determining the changes in temperature with time (each minute for the first five minutes, each five minutes for the next 25 minutes and each ten minutes for the last thirty minutes) using a calibrated thermometer (0 - 100°C) to the nearest $\pm 0.05^\circ\text{C}$. The data was obtained for a period of one hour for each sample solution.

From the data obtained, Reaction number was calculated as follows: From the rise in temperature of the system per minute, the reaction number (RN) was calculated using equation (1):

$$RN(\text{ }^\circ\text{C}/\text{min}) = \frac{T_m - T_i}{t} \quad (1)$$

Where T_m is the maximum temperature attained by the system, T_i is the initial temperature and t is the time required to reach the maximum temperature.

From the above, the inhibition efficiency (%IE) of the used inhibitor was computed using equation (2):

$$\%IE = \frac{RN_f - RN_i}{RN_f} \quad (2)$$

Where RN_f is the reaction number of aqueous acid in the absence of *Moringa oleifera*, and RN_i is the reaction number of aqueous acid in the presence of *Moringa oleifera* extract or the halide additives where applicable [10].

3. Results and discussion

The result of the inhibitive and synergistic properties of *Moringa oleifera* extract and halides on the corrosion of Zinc in HCl is presented as follows:

3.1 Effect of corrodent concentration only on the corrosion of zinc coupon

Figure 1 presents the effect of reaction temperature variation with time for the corrosion of Zinc in varying HCl solutions was conducted. The temperature of the reaction system was observed to increase with increasing concentration of the corrodent, HCl. Corrosion rate increases with increasing temperature. The tendencies for partial desorption of inhibitor from the metal surface and the metal dissolution increases with temperature. Therefore, an increase in concentration of the corrodent increases the rate of corrosion of Zinc [22].

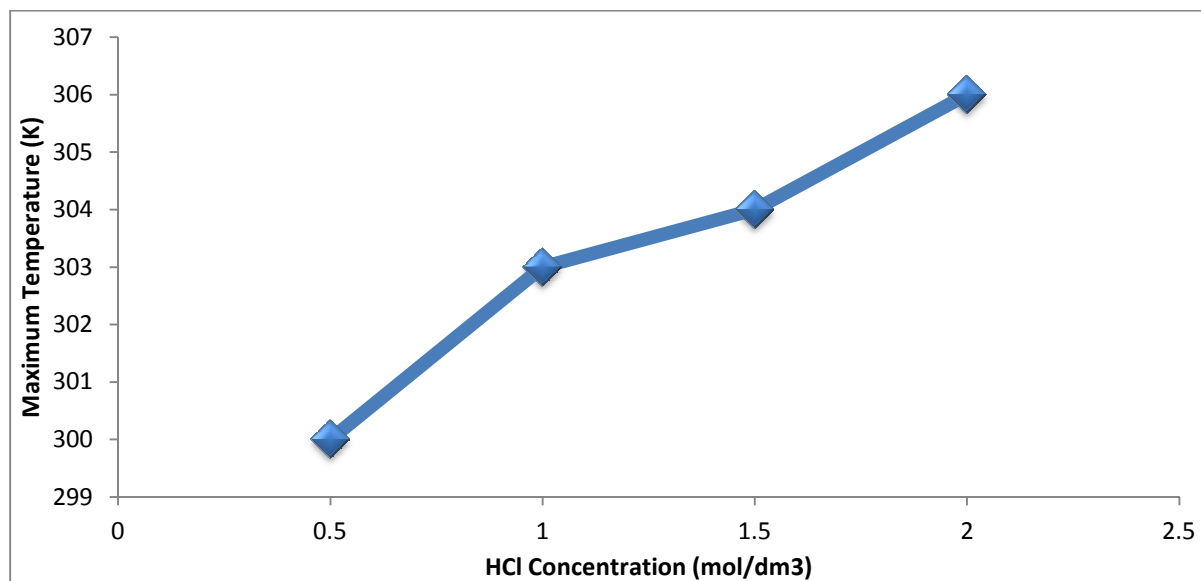


Fig 1: Variation of temperature with time for the corrosion of zinc in varying corrodent, HCl concentration

3.2 Effect of plant extract and corrodent on the corrosion of zinc coupon

The result of the effect of plant extract in different HCl solutions on the corrosion of zinc coupons was also tested. Observation showed that the presence of the plant extracts reduces the reaction temperatures and also the rate of reaction, thereby inhibiting the corrosion of Zinc. It was also observed that the higher the concentration of the plant extract the better the inhibition while the higher the corrodent concentration the higher the rate of corrosion. This may be attributed to the increase in number of adsorption active centers at higher concentration of the inhibitor. Similar results have been reported earlier by Ayuba and Mustapha, Eddy *et al* and Chauhan *et al* [10, 20-21, 23].

3.3 Effect of halide type and corrodent without plant extract on zinc corrosion

Figures 2, 3 and 4 presents the corrosion of zinc coupons in 0.1M, 0.3M and 0.5M KCl and KI respectively in varying HCl solutions. It can be observed in all the systems that KI has the least corrosion effect on Zinc in the three corrodent concentrations tested. This could be attributed to the large ionic size of iodide ion and its low electronegativity when compared to chloride. Electronegativity decreases from Cl^- to I^- ($\text{Cl}^- = 3.0$, $\text{I}^- = 2.5$), while the atomic radius also increases from Cl^- to I^- ($\text{Cl}^- = 0.90\text{\AA}$, $\text{I}^- = 1.35\text{\AA}$) [10].

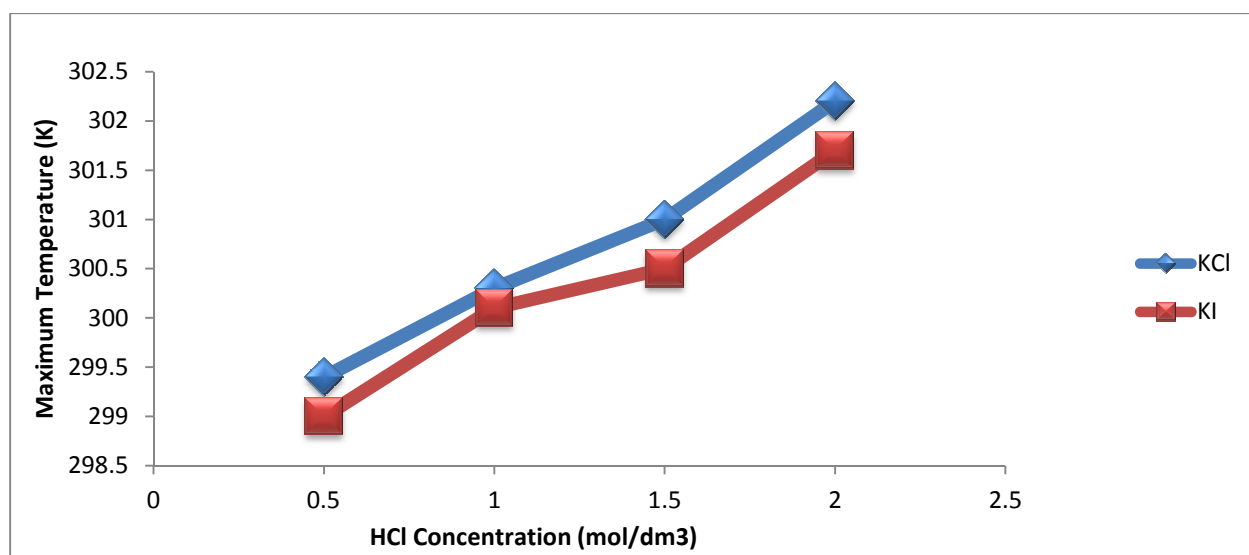


Fig 2: Variation of temperature with time for the corrosion of zinc in 0.1M KCl and KI in varying HCl solutions

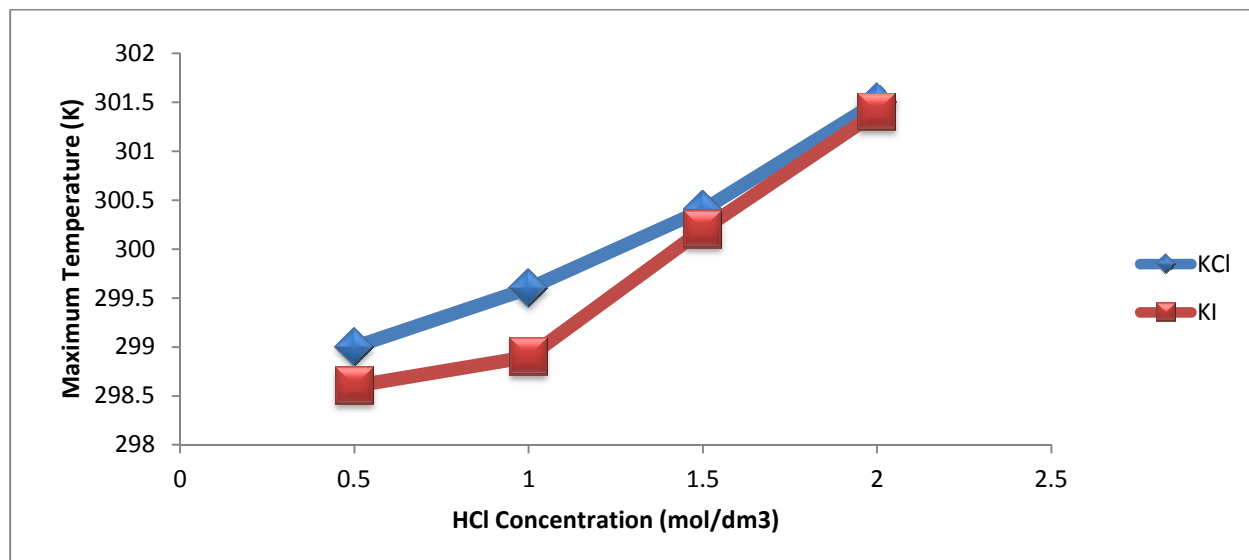


Fig 3: Variation of temperature with time for the corrosion of zinc in 0.3M KCl and KI in varying HCl solutions

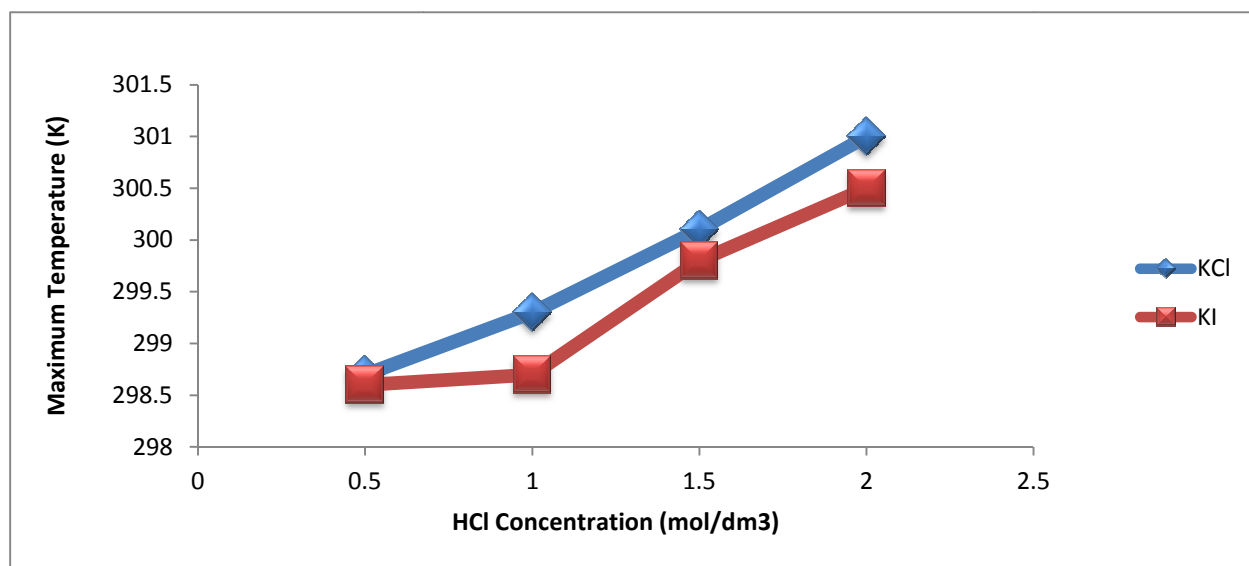


Fig 4: Variation of temperature with time for the corrosion of zinc in 0.5M KCl and KI in varying HCl solutions

3.4 Effect of Halide type with plant extract on the corrosion of zinc in HCl solution

The addition of plant extract (0.1g/L) to the corrosion solution containing HCl and the halides reduced considerably the reaction temperature and thereby reducing the rate of corrosion. Comparing the inhibitive efficiencies of such a combination, it was KI that had the best inhibitive property in combination with the plant extract. This can also be attributed to the reasons stated earlier. Similar results were obtained when the concentration of the plant extract was increased to 0.3g/L and then to 0.5g/L. This is evident in Figures 5, 6 and 7 respectively showing the variation of temperature with time for the corrosion of Zinc coupon in 0.1, 0.3 and 0.5M KCl and KI. In all cases, the inhibition increases at 0.5 and 1.0M HCl solution with a rapid decrease in inhibition at 1.5M and 2.0M HCl solution. Since phytochemicals are oxygen, nitrogen or sulphur containing organic compounds with lone pairs of electrons, they may be protonated in strong acidic solutions leading to positive charge. Zinc surface is known to have positive charge, thus it is difficult for the positively charged phytochemical to approach the zinc surface at high acidic concentration due to electrostatic repulsion and perhaps explains why these compounds exhibit decreasing inhibition efficiencies at higher HCl concentration [24]. For the halides, reason may be attributed to competition between Chloride, Iodide and plant extract on the Zinc surface at higher HCl concentration. Similar results have been reported earlier by Ayuba and Mustapha [10].

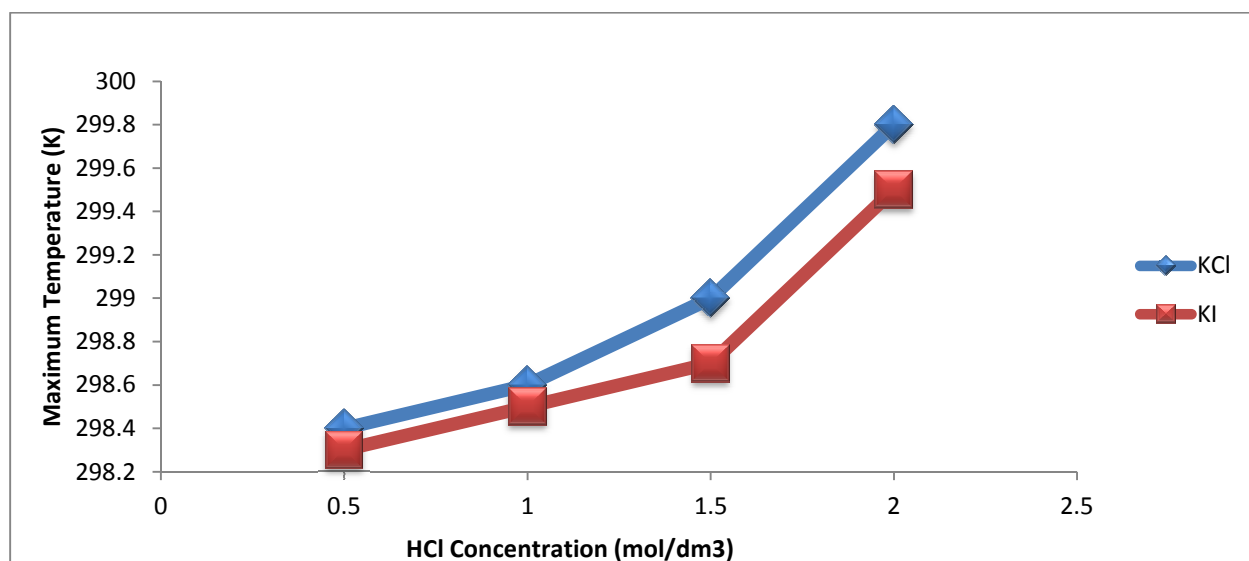
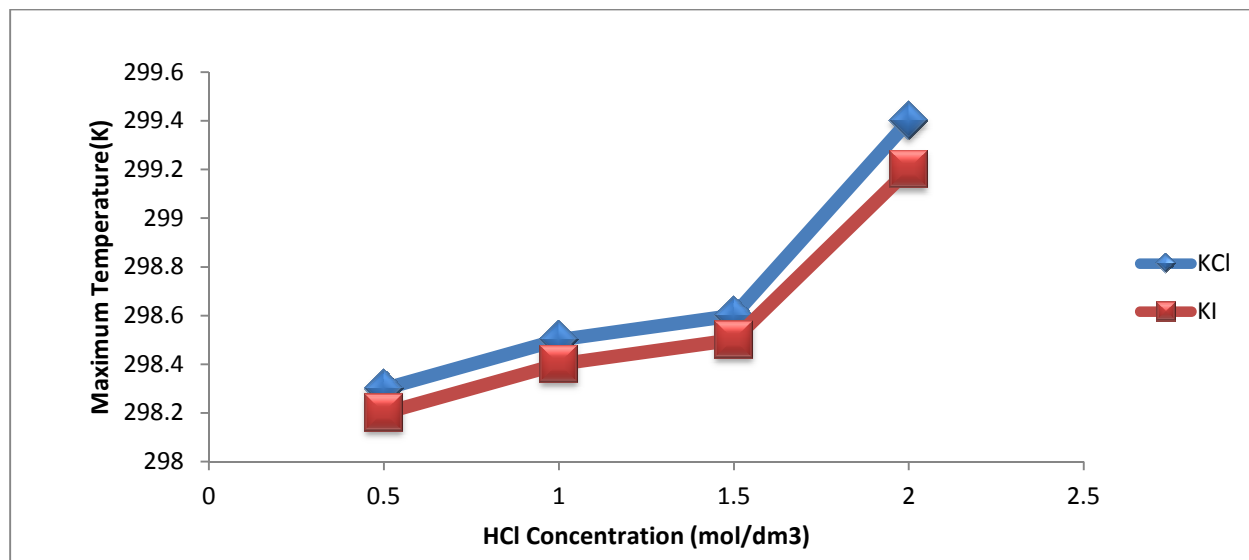


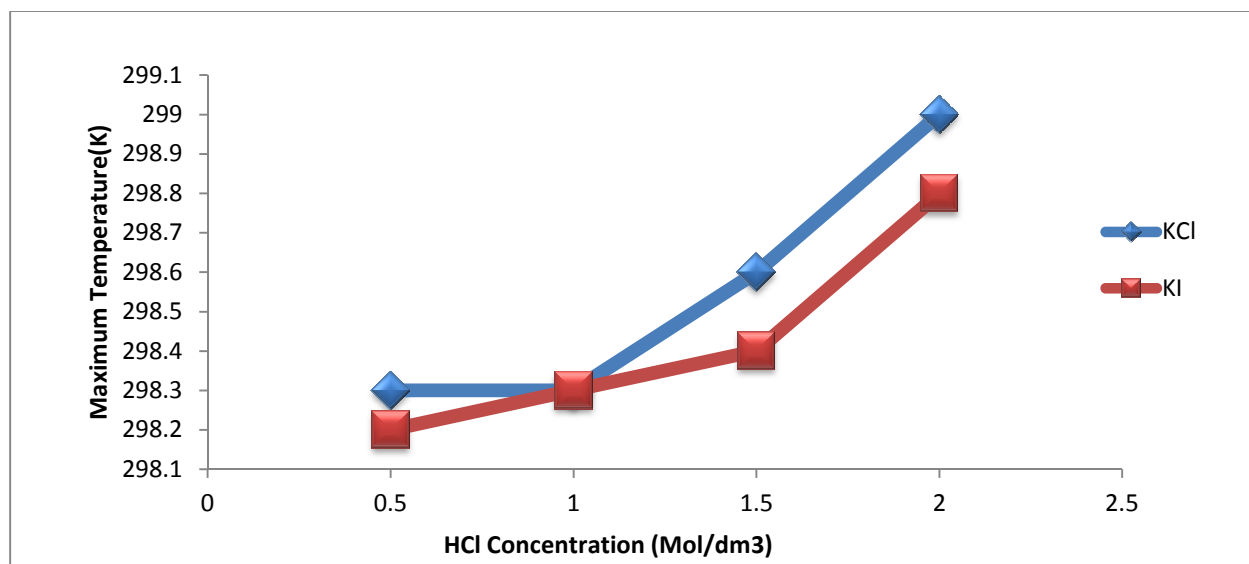
Fig 5: Variation of temperature with time for the corrosion of zinc in 0.1M KCl and KI in 0.3g/L inhibitor in varying HCl solutions

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192

193 **Fig 6: Variation of temperature with time for the corrosion of zinc in 0.3M KCl and KI in**
194 **0.3g/L inhibitor in varying HCl solutions**



195

196 **Fig 7: Variation of temperature with time for the corrosion of zinc in 0.5M KCl and KI in**
197 **0.3g/L inhibitor in varying HCl solutions**

198

Table 2 below presents the various phytochemicals detected in the plant extract with regards to their concentration.

Table 2: Phytochemical composition of ethanol extracts of *Moringa oleifera* leaves.

Phytochemicals	Results
Flavonoids	++++
Carbonhydrates	++
Alkaloids	++
Reducing Sugars	++
Steroids	++
Tannins	+
Saponins	+
Glycosides	+
Terpenoids	+
Resins	-
Proteins	-
Fats and Oil	-

Key: +++++ (High Concentration); ++ (Moderate); + (Low) ; - (Not detected)

It is therefore concluded that the inhibitive properties of *Moringa oleifera* is due to the presence of phytochemicals shown in table 2 above. Phytochemicals are Oxygen, Nitrogen or Sulphur containing organic compounds with presence of non shared electron pairs and p-electrons in triple or conjugated double bonds which are perhaps donated into available empty orbitals on the Zinc surface; hence they are expected to be good corrosion inhibitors [24].

3.5 Percentage inhibition efficiency

Table 3 and 4, presents the calculated values of inhibition efficiencies for the corrosion of Zinc in varying KCl, KI, plant extract and HCl concentrations. Table 3 shows the percentage inhibition efficiencies for the corrosion of Zinc in varying concentrations of KCl, Plant extract

214 and HCl. Observation shows that as the concentration of the plant extract increases, the
 215 inhibition efficiencies also increases. Increase in the concentration of KCl also leads to an
 216 increase in corrosion inhibition.

217 Similar observation results from the corrosion inhibition efficiencies for the corrosion of
 218 Zinc in varying KI, plant extract and HCl concentrations as shown in table 4. The only observed
 219 difference is the higher inhibition efficiencies of KI in comparison with KCl, in which the reason
 220 was stated earlier. In all cases, the inhibition efficiencies decrease with increasing corrodent, HCl
 221 concentration. Similar result was reported elsewhere by Okafor *et al* [22].

222 **Table 3: Calculated values of inhibition efficiencies (%) for zinc corrosion in varying HCl,**
 223 **plant extract and KCl concentrations**

Corrodent Conc.		0.5M HCl			1M HCl			1.5M HCl			2M HCl		
KCl Conc.	Extract conc.	KCl + P.E			KCl + P.E			KCl + P.E			KCl + P.E		
	0.0g/L	0	0	0	0	0	0	0	0	0	0	0	0
0.1M	0.1g/L	47	81.1	82.5	44.6	81.9	83.9	50	80	83.3	47.6	72.5	75
	0.3g/L	47	84.8	86	44.6	85.8	85.5	50	83.3	83.3	47.6	75	77.5
	0.5g/L	47	86	86.5	44.6	84.9	86.9	50	85	86.6	47.6	80	85
	0.0g/L	0	0	0	0	0	0	0	0	0	0	0	0
0.3M	0.1g/L	62.1	81.1	86	61.4	81.9	86.7	60	80	85.9	56.3	72.5	81.3
	0.3g/L	62.1	84.8	89.5	61.4	85.8	90	60	83.3	90	56.3	75	82.5
	0.5g/L	62.1	86	90.3	61.4	84.9	91.2	60	85	90	56.3	80	87.5
	0.0g/L	0	0	0	0	0	0	0	0	0	0	0	0
0.5M	0.1g/L	71.1	81.1	92.7	68.8	81.9	92	65	80	88.6	62.5	72.5	85
	0.3g/L	71.1	84.8	90.3	68.8	85.8	92.7	65	83.3	91.4	62.5	75	87.5
	0.5g/L	71.1	86	87	68.8	84.9	93.4	65	85	91.4	62.5	80	91.3

224 **Key: P.E (Plant extract); KCl (Potassium Chloride)**

225 **Table 4: Calculated values of inhibition efficiencies (%) for zinc corrosion in varying HCl,**
 226 **plant extract and KI concentrations**

Corrodent Conc.	0.5M HCl	1M HCl	1.5M HCl	2M HCl
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KI Conc.	Extract conc.	KI +			KI +			KI +			KI +		
		KI	P.E	P.E	KI	P.E	P.E	KI	P.E	P.E	KI	P.E	P.E
0.1M	0.0g/L	0	0	0	0	0	0	0	0	0	0	0	0
	0.1g/L	62.1	81.1	86	57.8	81.9	90	60	80	86.7	53.8	72.5	77.5
	0.3g/L	62.1	84.8	90.3	57.8	85.8	89	60	83.3	88.3	53.8	75	81.3
	0.5g/L	62.1	86	93	57.8	84.9	90	60	85	90	53.8	80	87.5
0.3M	0.0g/L	0	0	0	0	0	0	0	0	0	0	0	0
	0.1g/L	75	81.1	89.5	78.3	81.9	92	63.3	80	90	57.6	72.5	82.5
	0.3g/L	75	84.8	93.5	78.3	85.8	92	63.3	83.3	91.6	57.6	75	85
	0.5g/L	75	86	90	78.3	84.9	94	63.3	85	93.3	57.6	80	90
0.5M	0.0g/L	0	0	0	0	0	0	0	0	0	0	0	0
	0.1g/L	77	81.1	94	79	81.9	93	70	80	91.4	68.8	72.5	87.5
	0.3g/L	77	84.8	93.7	79	85.8	93.6	70	83.3	93.3	68.8	75	90
	0.5g/L	77	86	93.5	79	84.9	95.2	70	85	92.9	68.8	80	91.4

Key: P.E (Plant extract); KI (Potassium Iodide)

3.6 Synergism

Synergism is a combined action of a compound greater in total effect than the sum of individual effects. It has become one of the most important factors in inhibition process and serves as a basis for all modern corrosion inhibitor formulation. Synergism of corrosion inhibitors is either due to interaction between components of the inhibitors or due to interaction between the inhibitor and one of the ions present in aqueous solution [24, 10]. Synergistic studies were carried out on the combination of the inhibitor-plant extract (0.1, 0.3, 0.5g/L) with KCl, and KI (0.1, 0.3, 0.5M) in HCl (0.5, 1, 1.5, 2M) respectively. Synergism parameter (S) of the inhibitor due to halides was calculated using Equation (3)

$$S = \frac{1 - I_A - I_B + I_{AB}}{1 - I_{AB}} \quad (3)$$

Where I_A and I_B are inhibition efficiencies of the inhibitor and halide, respectively, and I_{AB} is the inhibition efficiency of the inhibitor, when it is combined with halide [10].

S approaches 1 when no interaction between the inhibitor compounds exists. When $S < 1$, the antagonistic interaction prevails. Values of synergism parameters as recorded in table 5 ($S < 1$ in all cases) shows that the adsorption of one compound antagonizes another which results in mass coverage and thus improve efficiency of inhibition [24]. The reason for excellent synergistic combination of halides with plant extract is not farfetched; since it is difficult for the positively charged phytochemical to approach the zinc surface at high acidic concentration due to electrostatic repulsion, presence of anions (Cl^- and I^-) improves the inhibition efficiency as the anions are first adsorbed on the zinc surface making the surface negatively charged as a result of electrostatic attraction, after which the protonated compounds can now effectively reached the Zinc surface, Hence presence of halides (KCl and KI) improves the inhibition efficiency of the plant extract in the order $\text{KI} > \text{KCl}$ [24].

Table 5: Synergism parameter (S) for plant extract, halide and plant extract-halide mixture for the corrosion of zinc in HCl solution

Corrodent									
Conc.		0.5M HCl		1M HCl		1.5M HCl		2M HCl	
Halide Conc.	Extract conc.	KCl	KI	KCl	KI	KCl	KI	KCl	KI
0.1M	0.0g/L	0	0	0	0	0	0	0	0
	0.1g/L	-45.2	-57.6	-42.5	-51.6	-47	-54.3	-45	-49.3
	0.3g/L	-45.4	-60.2	-43.8	-54.7	-49	-55.6	-45.1	-48.7
	0.5g/L	-45.7	-56.5	-42.58	-53.5	-48.1	-55.7	-43.8	-48.2
0.3M	0.0g/L	0	0	0	0	0	0	0	0
	0.1g/L	-57.6	-67	-57	-68.2	-54.9	-55.3	-49.2	-49.7
	0.3g/L	-57.9	-67	-57.5	-72	-54.6	-56.6	-50.2	-49.9
	0.5g/L	-58.2	-70.7	-56.2	-69.7	-55.7	-56.7	-50.5	-50.2
0.5M	0.0g/L	0	0	0	0	0	0	0	0
	0.1g/L	-61.2	-65.5	-60.3	-68.6	-57.7	-60.3	-52.3	-56
	0.3g/L	-65.9	-68.8	-62.7	-71.4	-58.3	-61.5	-52.6	-56.4
	0.5g/L	-69.3	-69.8	-61.6	-69.5	-59.5	-63.1	-53.8	-59.3

Key: KCl (potassium Chloride); KI (potassium Iodide)

3.7 Adsorption studies

The inhibition efficiency of *Moringa oleifera* extract or the halide additives can be related to the degree of surface coverage (θ) using equation (4) and (5):

$$\%IE = \left[1 - \frac{w_1}{w_2}\right] \times 100 \quad (4)$$

$$\theta = 1 - \frac{w_1}{w_2} \quad (5)$$

Where, w_1 and w_2 are the weight losses (grams) for zinc in the presence and absence of additives respectively in HCl solution at the same temperature [25-26].

From the surface coverage (θ) for different inhibitor concentrations the respective adsorption isotherms can be obtained. The surface coverage data fits into the Langmiur isotherm (fig. 8) represented as,

$$\frac{C}{\theta} = \frac{1}{b} + C \quad (6)$$

Where, C is concentration of additives. With either additive, it was shown that corrosion inhibition of zinc obeys the Langmiur isotherm. A plot (C/θ) against C , for all concentrations of inhibitors, a straight line relationship was obtained in all cases with a correlation coefficients (R^2) in the range $1.00 > R^2 > 0.994$. Hence the mechanism of inhibition may be due to the formation and maintenance of a protective film on the metal surface [8].

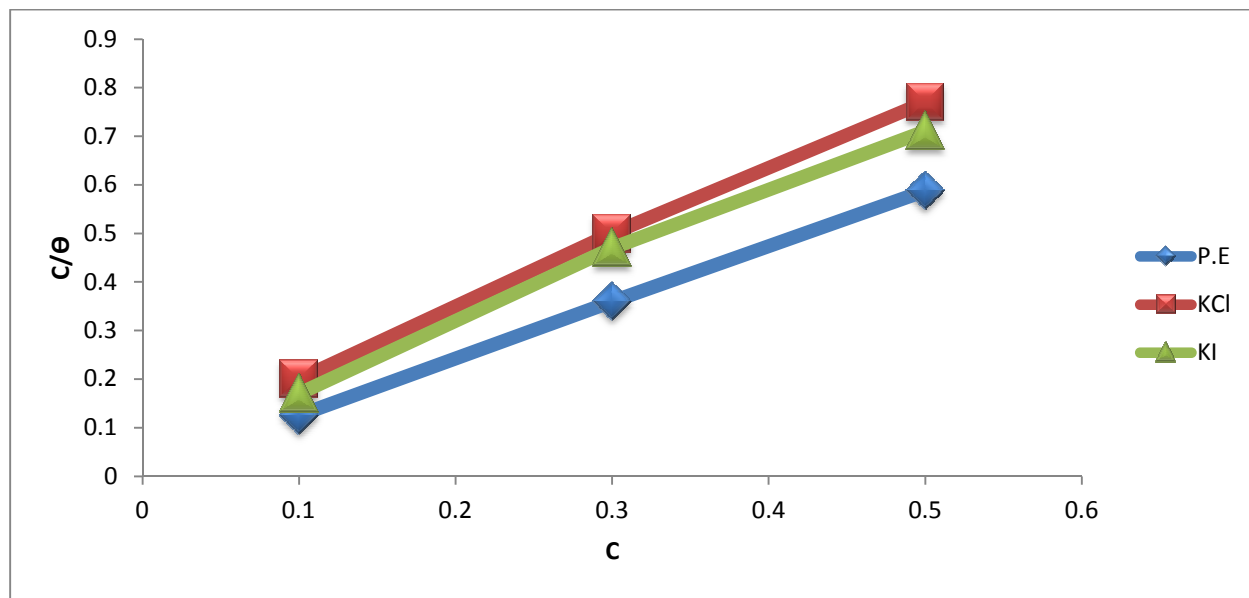


Fig. 8: Langmuir isotherm model for the corrosion of zinc in varying plant extract, KCl and KI concentration at 1.5M HCl solution

4. Conclusions

The used biologically active *Moringa oleifera* leaves extract as corrosion inhibitors for zinc in varying HCl act as an efficient inhibitor. It is concluded that the inhibition efficiency of the extract is due to the presence of saponins, tannins, flavonoids, glycosides, carbonhydrates, reducing sugars, terpenoids, steroids and alkaloids present in the plant extract. Also, the inhibition efficiency increases with the increase in inhibitor concentration and decreases with increase in corrodent concentration. Adsorption of plant extract, KCl and KI on the Zn surface from acidic solution followed Langmuir isotherm, indicating that the main inhibition process occurred via adsorption. The addition of KI and KCl to these compounds improve the values of inhibition efficiency due to synergistic effect

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