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**ORIGINAL ARTICLE**

# Corrosion inhibitory properties of elephant grass (*Pennisetum purpureum*) extract: Effect on mild steel corrosion in 1 M HCl solution



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**Abstract** Corrosion inhibition and adsorption characteristics of elephant grass extracts on mild steel in 1 M HCl solution were investigated. Mass loss, corrosion rate measurements, inhibition efficiency, atomic adsorption spectroscopy (AAS), FT-IR spectroscopy and scanning electron spectroscopic analysis were used to assess the inhibitory properties of the extract in the acid solution. The results show that the steel dissolution rate in the acidic solution was sensitive to the extract concentration as mass loss and corrosion rates were observed to decrease with increase in the extract concentration. The inhibition efficiencies were averagely above 95% at room temperature increasing with increase in concentration of the extract but decreases with increasing temperature. FTIR results showed that the inhibition was essentially by absorption through the functional groups present in the extract while the activation energies and Langmuir adsorption isotherms confirm the mechanism to be physical adsorption. The SEM images of the corroded substrates confirmed pitting as the primary corrosion mechanism which was substantially mitigated with the use of the extract. Overall, the elephant grass extract was found to be efficient for corrosion inhibition of mild steel in HCl environment.

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**1. Introduction**

Corrosion inhibitors are generally reckoned to be efficient and relatively cost effective in mitigating metal dissolution in acidified process fluids utilized in metallurgical, manufacturing, chemical and petrochemical industries [1,2]. This gives the benefits of longevity of process vessels, installations, and facilities;

reduced maintenance and replacement; and significantly reduced failure rates.

Several inorganic and organic compounds have been utilized commercially for corrosion protection with high inhibition efficiencies recorded. The downside of several of these inhibitor grades is their toxic nature which often poses health and environmental challenges [3]. The growing awareness of the health and environmental impact of industrial materials containing toxins to the environment has led to stiffer legislation and regulations on use of such materials. Consequently, efforts are being made to source for alternative materials for the development of non-toxic, environmentally friendly, cost

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effective and efficient inhibitors. In this regard, plant based inhibitors have been the most promising of the alternatives explored [4]. Plant source derived inhibitors have a number of characteristics which give reason for continued interest in its use as inhibitors. They are cheaply processed, non-toxic, and biodegradable; often constitute wastes, and are readily available [5]. They have also been investigated and observed to contain phytochemical constituents which are known to be active functional groups found in inorganic and organic compounds known to exhibit high corrosion inhibition properties [6].

Several research works on the potentials of plant based materials as corrosion inhibitors are well documented in the literature. Plants that have been reported to have inhibitory properties in acidic medium are *Tithonia divesifolia* [7]; *Murraya koenigii* [8]; *Psidium Guajava* [4]; coconut coir dust [9]; *Hunterial Umbellata* [10]; *Nicotiana tabacum* [11]; Aloe vera plant extract [12]; *Sida acuta* [13]; *Hibiscus sabdariffa calyx* [14]; among several others are plant sourced inhibitors which have recorded varied levels of success. There is still continued interest in this subject, particularly the investigation of plant sources with less competitive alternative uses such as in chemical and pharmaceutical applications [15].

In the present work, the inhibition efficiency of elephant grass extract for mitigating mild steel corrosion in HCl solution is studied. Elephant grass is widespread in most parts of tropical Africa; and mainly used as forage and pastures plant for grazing purposes. It is also utilized in some environments for erosion protection [16,17].

## 2. Materials and methods

### 2.1. Materials

Mild steel rod of composition Fe = 98.3%, C = 0.133%, P = 0.0061%, Mn = 0.82%, Cr = 0.08% was used for this study. The steel was mechanically cut to coupons of dimension 20 × 10 mm, and then polished using different grades of silicon carbide paper. Analar grade HCl and distilled water were used for the preparation of the electrolyte, while elephant grass was used as a source of corrosion inhibitor.

### 2.2. Extract preparation

Elephant grass was obtained from Akure metropolis, sun dried and pulverized. About 10 g of the pulverized leaves was weighed and soaked in 100 ml of ethanol for 48 h. The mixture was then filtered to obtain the extract. The filtrates were further subjected to evaporation in order to leave the sample free of the ethanol. The stock of the extract obtained was used in preparing different concentrations of the extract by dissolving 0.1, 0.2, 0.3, 0.4 and 0.5 g of the extract in 1 l of 1 M HCl respectively.

### 2.3. Weight loss measurement

Pre-weighed mild steel substrates were immersed in 100 ml of the blank/inhibitor solutions for 7 days (168 h). The weight loss of each coupon was determined at every 24 h interval by

retrieving the substrates from the solution, cleaning with acetone and then reweighing. The mass loss for each sample was evaluated by dividing its weight loss by the surface area of the coupon [18].

Another series of weight loss studies were carried out on pre-weighed mild steel substrates which were immersed in 250 ml capacity beakers containing 100 ml of the test solutions maintained at 303, 313, 323 and 333 K in a thermostat controlled water bath. The substrates were held in the solutions for 5 h after which they are reweighed and the difference in weight taken as weight loss. From the weight loss data collected, the corrosion rate (CR) of the substrates was determined in accordance with ASTM G31-12a standard [18] using the relation:

$$CR(\text{mmy}) = \frac{87,500W}{A\rho t} \quad (2.1)$$

where CR is corrosion rate,  $W$  is weight loss (g),  $A$  is the area of the coupon in  $\text{cm}^2$ ,  $T$  is time, and  $\rho$  is the density ( $\text{g}/\text{cm}^3$ ).

Inhibition efficiency (IE) and surface coverage ( $\Theta$ ) were also determined using Eqs. (2.2) and (2.3) respectively.

$$I.E.\% = \left(1 - \frac{CR_{\text{inh}}}{CR_{\text{blank}}}\right) \times 100 \quad (2.2)$$

where  $CR_{\text{inh}}$  and  $CR_{\text{blank}}$  correspond to the corrosion rates in the presence and absence of inhibitor, respectively; while I.E. represents the inhibition efficiency.

The surface coverage was calculated by using Eq. (2.3).

$$\Theta = \left(1 - \frac{CR_{\text{inh}}}{CR_{\text{blank}}}\right) \quad (2.3)$$

### 2.4. Atomic adsorption spectrometric analysis

Atomic adsorption analysis was performed using atomic adsorption spectrometer model bulk 200. This was required to determine the concentration of iron (II) ions in the acidic solutions after gravimetric measurements. The calibration curve of iron (II) ions was drawn before analyzing the electrolyte solution. All samples containing iron ions were diluted with distilled water to ensure that the concentrations of metal ions are within the range of the calibration curve.

### 2.5. FTIR analysis

FTIR analysis of elephant grass extract and that of the corrosion products in the presence and absence of the elephant grass extract were carried out using Perkin-Elmer-1600 Fourier transform infra-red spectrophotometer. The sample was prepared using KBr.

### 2.6. Scanning electron microscopy

The surface morphology of the mild steel substrates before and after immersion in the test solutions was examined using a JSM 7600F Jeol ultra-high resolution field emission gun scanning electron microscope (FEG-SEM) equipped with accessories for energy dispersive spectroscopy (EDS) analysis.

### 3. Results and discussion

#### 3.1. FTIR analysis

The FTIR spectra of the extract and the corrosion products are presented in Figs. 1 and 2. The spectra show the presence of hydroxyl (O—H) and unsaturated (C=C) functional groups which are known to have inhibitory properties [15]. Similar FTIR spectra have also been reported for *Tithonia Diversifolia* [7]. It is observed that there is a shift in the spectra of the extract when mild steel was immersed in it to form corrosion product. The hydroxyl (O—H) group shifted from 3379 to 3440  $\text{cm}^{-1}$  while the unsaturated (C=C) band shifted from 1635 to 1640  $\text{cm}^{-1}$ . This is an indication that there is an interaction between the extract and the mild steel substrate which resulted in inhibition. Alaneme et al. [15] reported that the shifts in the spectra suggest that the extracts (through the functional groups) interact with the mild steel surface forming a protective complex which promotes the inhibition of the metal from attack in the acidic medium.

#### 3.2. Mass loss studies

Fig. 3 presents the variation in mass loss of the mild steel substrates in the absence and presence of varying concentration of elephant grass extract as a function of exposure time. It is observed that mass loss decreases with increase in the concentration of the elephant grass extract. This infers that more of the metal surface were covered and protected from attack by the acidic medium as the concentration of the extract increases. Specifically the addition of the extract retarded the dissolution of the metal from 0.346  $\text{g}/\text{cm}^2$  in the blank solution to 0.096  $\text{g}/\text{cm}^2$  in the presence of 0.5 g/l of the extract for the exposure period utilized in the experiment. Accordingly, it is evident that elephant grass extract could act as an effective green inhibitor of corrosion on mild steel immersed in HCl solution.

The results of the atomic absorption spectroscopic analysis (Fig. 4) which reports the amount of  $\text{Fe}^{2+}$  present in the

electrolyte are noted to be completely in agreement with that from the weight loss analysis. It is observed that the concentration of the  $\text{Fe}^{2+}$  dissolved in the electrolyte decreased with increase in the concentrations of elephant grass extract in the 1M HCl solution.

#### 3.3. Effect of temperature

The results of temperature studies on the mild steel corrosion in 1 M HCl solution in the presence and absence of the elephant grass extract are presented in Table 1 and Fig. 5. From Table 1, it is observed that the corrosion rate increased with increasing temperature in both the presence and absence of the extract. Also, the inhibition efficiencies of the extracts decreased with increasing temperature, but increased with increase in the concentration of the elephant grass extract in the acidic media (Fig. 5). Temperature studies are important as it gives insight on the stability of the adsorbed inhibitor molecules on the metal surface [19]. In this regard, the results obtained showed that the adsorbed extract molecules were partially desorbed at higher temperatures. Desorption of some of the adsorbed molecules took place as the temperature of the corrosion system increases, thereby exposing the metal to the aggressive media, which resulted in decrease in inhibition efficiency. It is however noted that percent inhibition efficiency (% I.E.) of 80.6 was recorded when 0.5 g of the extract was used at 333 K. This implies that at relatively higher temperatures the extract could still function as good corrosion inhibitor. The inhibition efficiencies recorded are comparable (for similar concentrations) to results observed for plant based green inhibitors such as *Tithonia Diversifolia* [7], *Hunteria Umbellata* [10] and Rice Husk [15].

#### 3.4. Activation parameters

The corrosion rate values obtained from the weight loss studies were used to calculate the activation energy for the blank

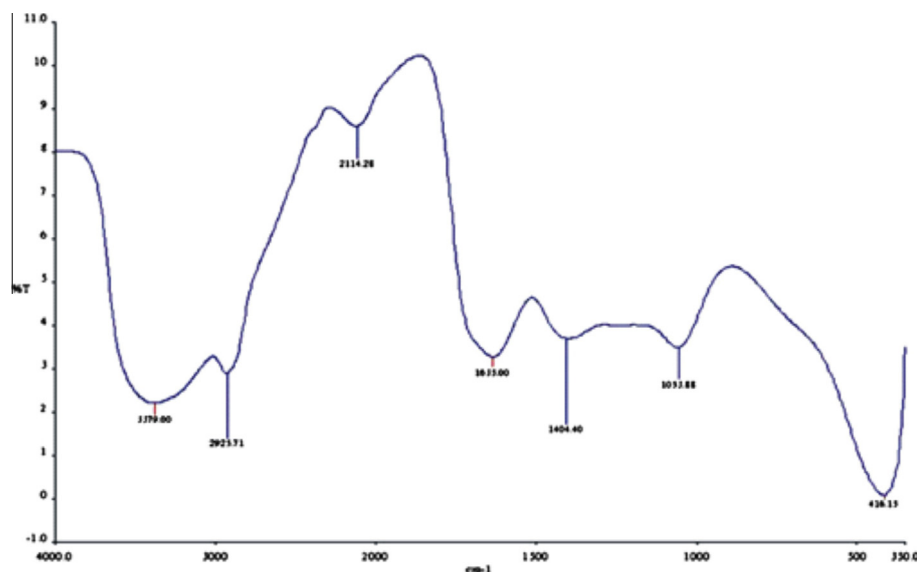


Figure 1 FT-IR spectrum of the elephant grass extract.

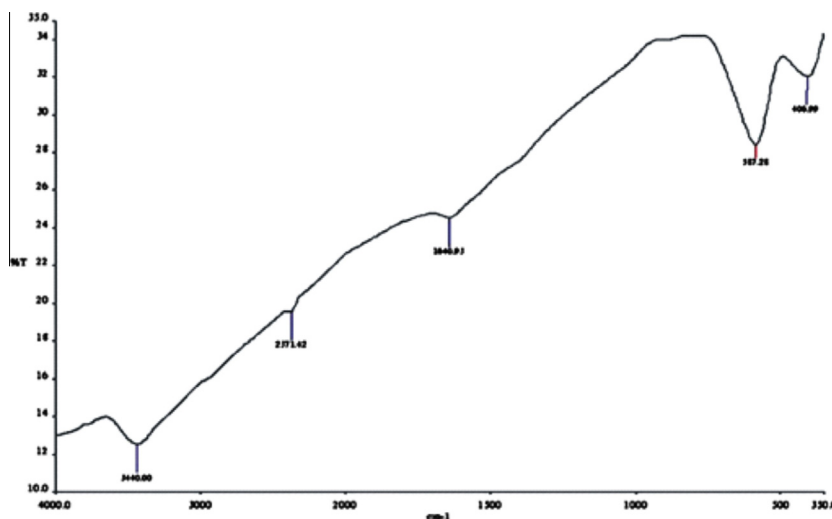


Figure 2 FT-IR spectrum of corrosion products of mild steel in the presence of elephant grass extract.

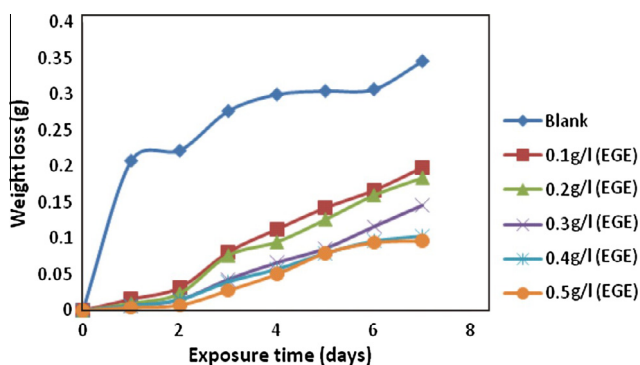


Figure 3 Plot of mass loss as a function of time for the corrosion of mild steel in 1 M HCl, in the absence and presence of elephant grass extract.

Table 1 Values of corrosion rate of mild steel in the presence and absence of elephant grass extract at temperature range 303–333 K.

Acid medium	Concentration of EGE (g/l)	Corrosion rate (mmpy)			
		303 K	313 K	323 K	333 K
1 M HCl	Blank	93.04	132.97	183.63	375.69
	0.1	10.39	14.48	35.35	256.5
	0.2	4.93	7.88	10.08	246.23
	0.3	2.69	6.41	16.67	108.24
	0.4	2.44	5.53	13.48	105.28
	0.5	1.16	4.07	15.14	72.88

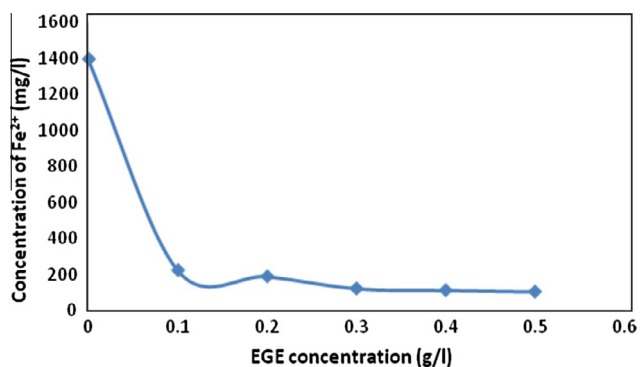


Figure 4 Plot of the concentrations of iron (II) ions in 1 M HCl acid after gravimetric measurements.

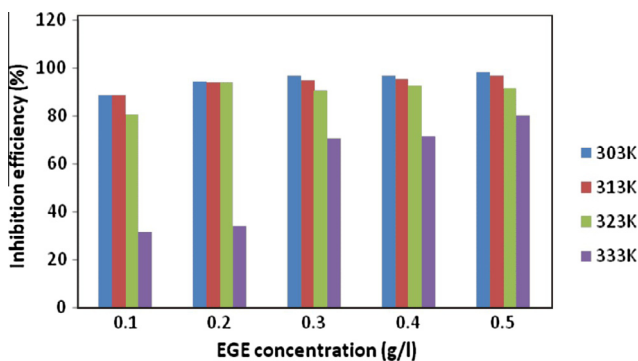
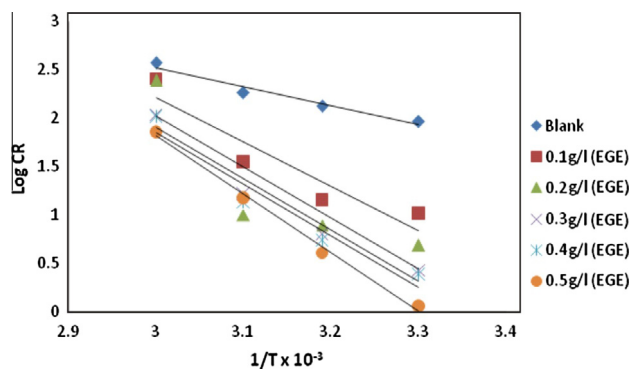


Figure 5 Variation of inhibition efficiency against the concentration of elephant grass extract for mild steel immersed in 1 M HCl solution for 5 h.

solution and the solutions containing varied concentrations of the elephant grass extract using the Arrhenius equation:

$$\log CR = \log A - \frac{E_a}{2.303RT} \tag{3.1}$$

where CR is the corrosion rate,  $E_a$  is the apparent activation energy,  $R$  is the molar gas constant,  $T$  is the absolute temperature and  $A$  is the frequency factor. A plot of  $\log CR$  vs.  $1/T$  in the absence and presence of different concentrations of the extracts gave a straight line graph as shown in Fig. 6. The



**Figure 6** Arrhenius plot for mild steel corrosion in 1 M HCl in the absence and presence of elephant grass extract.

values of activation energies ( $E_a$ ) calculated from the slopes ( $-E_a/2.303R$ ) are presented in Table 2.

The data show that the activation energy of the blank (37.79 kJ/mol) is lower than the value in the presence of the extract (115.12 kJ/mol). Comparing the results in Tables 1 and 2, increase in activation energy in the presence of the extract resulted in decrease in corrosion rates. The reduction in corrosion rates is associated with the adsorption of the extract molecules on the metal [20]. Corrosion inhibition by physical adsorption has been associated with higher activation energy values in the presence of inhibitors compared with values obtained when inhibitors are absent [21].

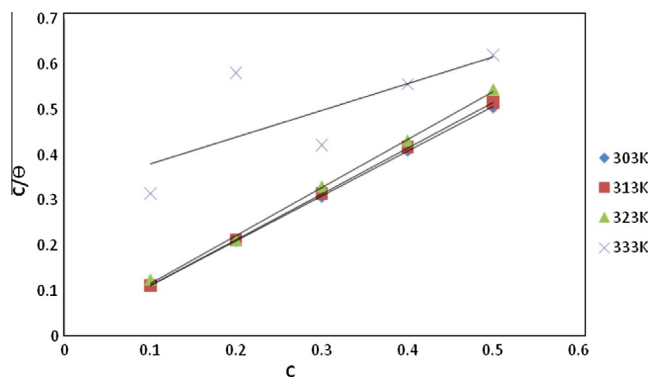
The values of Enthalpy ( $\Delta H_a$ ) and entropy ( $\Delta S_a$ ) of activation of the corrosion process were also evaluated from the results of the temperature studies using the equation:

$$\frac{\log CR}{T} = \log \left( \frac{R}{nh} \right) + \frac{\Delta S_a}{2.303R} - \frac{\Delta H_a}{2.303T} \quad (3.2)$$

where CR is the corrosion rate at temperature  $T$ ,  $R$  is the molar gas constant,  $n$  is Avogadro's constant, and  $h$  is the Planck's constant. Plots of  $\log CR/T$  vs.  $1/T$  for the different corrosion systems yielded straight line graphs. From the graphs the slope ( $-\Delta H/2.303R$ ) and intercept ( $\log(R/nh) + \Delta S/2.303R$ ) were used to compute the values of  $\Delta H_a$  and  $\Delta S_a$  (presented in Table 2). From the table it is evident that the enthalpy of activation values is all positive, which reflects

**Table 2** Values of activation energy parameters for mild steel dissolution 1 M HCl solution in the absence or presence of the elephant grass extract.

Acid medium	EGE (g/l)	Activation energy, $E_a$ (kJ mol <sup>-1</sup> )	Enthalpy of activation, $\Delta H_a$ (kJ mol <sup>-1</sup> )	Entropy of activation, $\Delta S_a$ (J mol <sup>-1</sup> k <sup>-1</sup> )
1 M HCl	Blank	37.78	35.14	-92.15
	0.1	87.59	84.95	51.27
	0.2	100.12	97.48	85.19
	0.3	100.77	98.13	84.88
	0.4	101.86	99.22	87.33
	0.5	115.11	112.47	126.32



**Figure 7** Langmuir adsorption isotherm plot for the adsorption of elephant grass extract with concentration at 303–333 K.

**Table 3** Langmuir isotherm parameters.

Temp. (K)	$K_{ads}$	$R^2$	Slope
303	72.29	0.9999	0.9871
313	90.38	0.9999	1.0118
323	102.77	0.9991	1.0622
333	3.09	0.7299	0.5852

the endothermic nature of the mild steel dissolution in the solutions.

### 3.5. Adsorption isotherm

The results discussed in Sections 3.1 and 3.2 have emphasized that the adsorption of the inhibitor molecules on the metal surface resulted in the formation of protective films which reduced the intensity of the metal dissolution process. In furtherance to understanding the adsorption behavior of the elephant grass extracts on the metal surface, the surface coverage values obtained from the data of corrosion rates as a function of concentration and temperature were plotted. The graphs from the plots were used to determine the isotherm that best fits the set of data from the corrosion inhibition study. Langmuir isotherm was found to be best suited for the experimental data from plots generated using the equation:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (3.3)$$

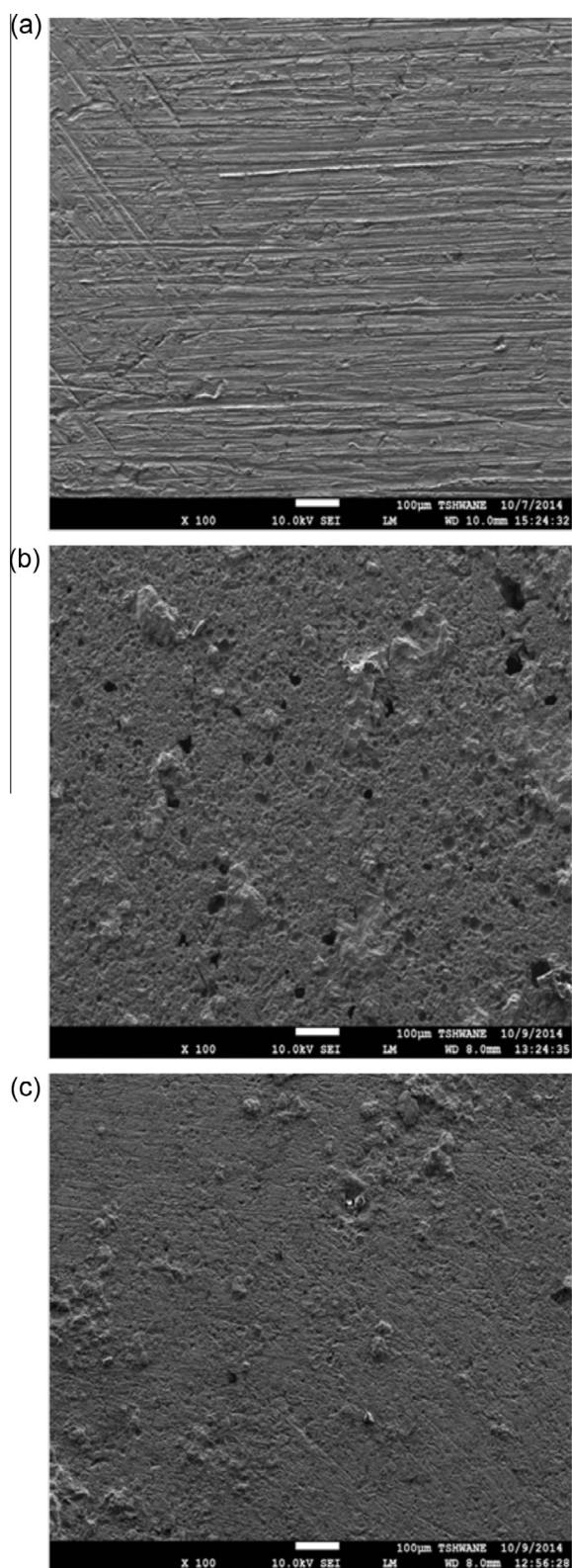
where  $C$  is the inhibitor concentration,  $\theta$  is the surface coverage and  $k_{ads}$  is the adsorption equilibrium constant. The plot of  $C/\theta$  against  $C$  yielded straight line graphs as shown in Fig. 7 with the values of the slope presented in Table 3.

The results of the slopes and  $R^2$  values listed in Table 3 are very close to unity except at 333 K. This confirms a strong adherence of the adsorption molecules of the inhibitor to Langmuir isotherm.

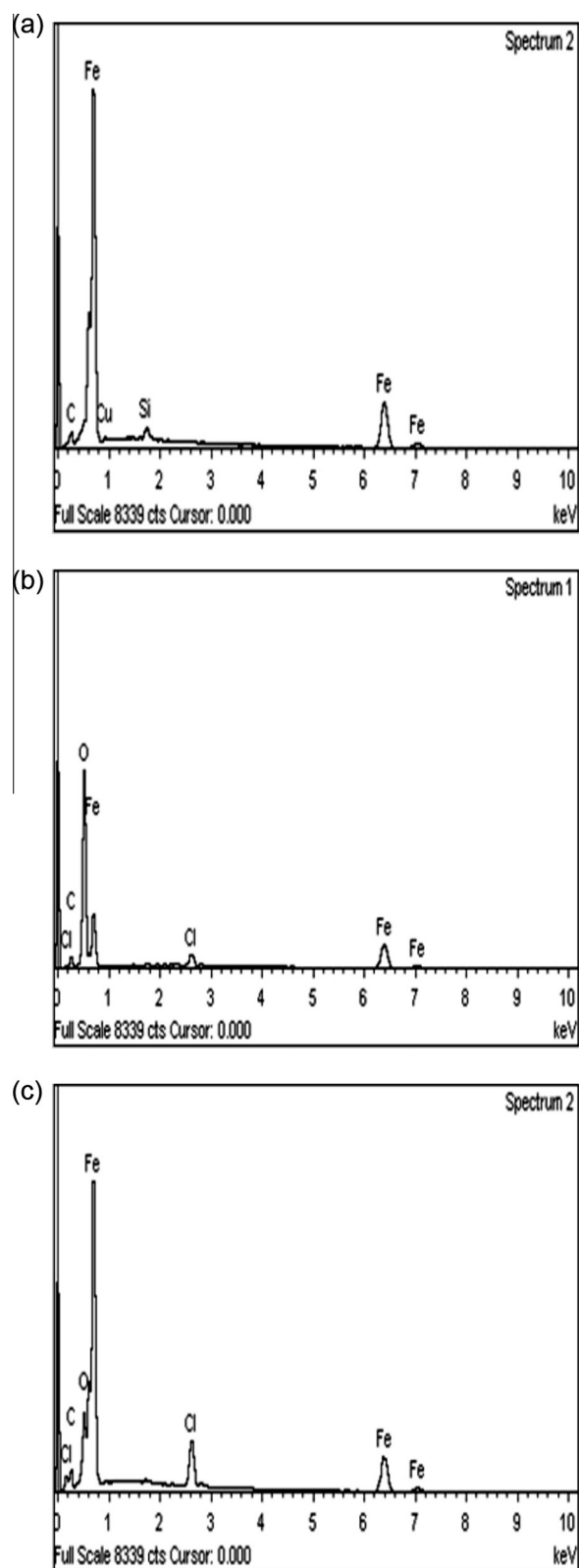
### 3.6. SEM and EDS analysis

Representative SEM (scanning electron microscopy) micrographs and EDS (Energy dispersive spectroscopy) spectra of the mild steel before and after immersion in hydrochloric acid





**Figure 8** Representative SEM micrographs of the surface morphologies of the mild steel substrates (a) before immersion in 1 M HCl solution, (b) after immersion for 7 days (168 h) in 1 M HCl solution without the elephant grass extract, and (c) after immersion for 7 days (168 h) in 1 M HCl solution containing 0.5 g/l elephant grass extract.



**Figure 9** Respective EDS spectra of the mild steel (a) before immersion in 1 M HCl solution, (b) after immersion for 7 days (168 h) in 1 M HCl solution without the elephant grass extract, and (c) after immersion for 7 days (168 h) in 1 M HCl solution containing 0.5 g/l elephant grass extract.

solutions with and without the elephant grass extracts are presented in Figs. 8 and 9, respectively.

The scanning electron micrograph of the substrate immersed in the HCl solution without the elephant grass extract revealed conspicuous pitting in the metal surface with pit depths and size substantially large (Fig. 8b compared to Fig. 8a). In the case of the mild steel substrate immersed in a representative solution containing 0.5 g/l elephant grass extract, it is without doubt that signs of pitting were scarcely evident (Fig. 8c). This attests to the effectiveness of the elephant grass extract to serve as corrosion inhibitor by reducing the intensity of the localized dissolution of the steel in HCl solution. The EDS (Energy dispersive spectroscopy) spectra of the mild steel before and after immersion in hydrochloric acid with and without inhibitor are also presented Fig. 9. The spectra for the mild steel before immersion in the HCl solution (Fig. 9a) show a high Fe peak, indicating a high amount of iron in the steel surface. This is in contrast with the EDS profile for the mild steel immersed in the 1 M HCl solution without the extract – where it is observed that the Fe peak in this case is considerably lower (Fig. 9b) compared to that of the mild steel before immersion in the acid solution (Fig. 9a). For the mild steel immersed in the HCl solution containing elephant grass extract (Fig. 9b), a high Fe peak is observable indicating a lower rate of iron dissolution in the acidic solution containing the extract. The presence of Cl ion in the EDS profiles for the mild steel immersed in the acidic solutions with and without the extract (Fig. 9b and c) confirms that interaction of the HCl and the steel does occur.

#### 4. Conclusion

Corrosion inhibition and adsorption characteristics of elephant grass extracts on mild steel in 1 M HCl solution were investigated. The results show that the steel dissolution rate in the acidic solution was sensitive to the extract concentration as mass loss and corrosion rates were observed to decrease with increase in the extract concentration. The inhibition efficiencies were averagely above 95% at room temperature increasing with increase in concentration of the extract but decreases with increasing temperature. FTIR results showed that the inhibition was essentially by absorption through the functional groups present in the extract while the activation energies and Langmuir adsorption isotherms confirm the mechanism to be physical adsorption. The SEM images of the corroded substrates confirmed pitting as the primary corrosion mechanism which was substantially mitigated with the use of the extract. Overall, the elephant grass extract was found to be efficient for corrosion inhibition of mild steel in HCl environment.

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