**ELECTROCHEMICAL AND CHARACTERIZATION OF ETHYL ACETATE EXTRACT OF *TAMARINDUS INDICA L* SHELL AS A CORROSION INHIBITOR FOR LOW CARBON STEEL IN 0.5 M HCL MEDIUM**

**Abdulmajid Abdullahia Shamsuddeen Abdullahi,a M. Hazwan Hussinb**

*aDepartment of Basic and Applied Sciences, College of Science and Technology, Hassan Usman Katsina Polytechnic, P.M.B 2052, Katsina State, Nigeria*

*bMaterials Technology Research Group (MaTReC), School of Chemical Sciences, Universiti Sains Malaysia, 11800 Minden, Penang, Malaysia.*

*Corresponding Email: captain5gmt@gmail.com*

**Abstract;**

Ethyl acetate extract of Tamarind shell (EAETS) has been examined as a corrosion inhibitor for law carbon steel (LCS) in 0.5 M HCl. Fourier-transform infrared spectroscopy (FTIR), nuclear magnetic resonance (NMR), and thermal gravimetric study (TGA), were carried out to characterize the (EAETS). The extraction yield 2.23% of condensed tannins. Also, FTIR and NMR analysis established the occurrence of tannins. Electrochemical impedance spectroscopy and potentiodynamic polarization method were applied to observe the inhibition performance of (EAETS). The inhibition efficacy of EAETS was experimental to be rising with increasing concentration of the extract. As the extract constituents settled on the low carbon substrate, inhibition efficiency also increases and lastly reached 81.11% at an extract concentration of 1000 ppm. PD curves of the extract were mixed-type inhibitors. The adsorption type of EAETS was found following Langmuir adsorption. Surface morphology analyses (SEM/EDX) displayed corrosion-inhibitive properties of the samples.

**Keywords;** Electrochemical,characterization, inhibition, corrosion, low carbon steel

**1. Introduction**

Carbon steel is efficient and abundant for structural material. Though, it is hard to shield its exposure to severe situations against deterioration 1. The use of inhibitors of corrosion is the best most useful and cost-effective methods for decreasing rates of corrosion and the protection of surfaces of metals against corrosion 2. Though various organic, inorganic and polymeric composites have accomplished sound as inhibitors of corrosion for diverse alloys and metals, according to literature numerous of the synthetic inhibitors are lethal to human and aquatic life 3. Inhibitors of natural origin are often considered nonhazardous or at least have lesser harmfulness to environmental species related with the synthetic organic counterpart 4. Hence, various scientists have dedicated on the study of ‘‘eco-friendly’’ or ‘‘green’’ inhibitors of corrosion isolated or synthesized from various medicinal plants, aromatic herbs, and spices 5. The effects of corrosion inhibition by the plant extracts and their pure constituents of various plants, such as henna 6Phaseolus vulgaris L.7 Jasminum nudiflorum Lindl 8 Aloe vera 9, Salvia officinalis 10, Ficus hispida 11bamboo 12,Aniba rosaeodora 13, Mansoa alliacea 14, Ginkgo species 15, on carbon steel, cold rolled steel, stainless steel and zinc metals were presented, with the very encouraging effectiveness. The performances of plant extracts inhibition are related closely to their chemical constituents, such as alkaloids, tannins, phenolics, proteins, and carbohydrates. These bioactive compounds typically endure functional groups or heteroatoms containing oxygen, nitrogen, or sulphur atoms, in addition to conjugated double bonds, triple bond as well as aromatic rings, which are considered to be there adsorption centres. The molecules adsorption on metal substrate was determined by the type of functional group possessions, which include electron density and steric factors 16

Several researches were carried out on corrosion inhibition of certain extracts of plant in different acidic medium and their optimal concentrations. For example, Oxandra asbecki alkaloid plant extract (OAPE) was investigated as a potential green corrosion inhibitor for C38 mild steel in 1 M HCl solution with inhibition efficacy of 92% at OAPE 100 ppm 17. various extracts of plant such as Kidney bean, Halfabar, Chamomile, and Black cumin extracts for the steel corrosion in 1M H2SO4 give rise to 83.5%, 87.1%, 90.2%, and, 87.2% of inhibition effectiveness at the optimal extract concentrations of 2.4,2.52, 7.56 and 1.14g/L, respectively 18. The maximum inhibition effectiveness showed by the extract of Pennisetum purpureum for mild steel in 1 M HCl medium was 95% 19. Water and Alcoholic extracts of Pimenta dioica (PD) leaves were described as non-toxic inhibitor of corrosion for mild steel in 0.5 M HCl acidic medium with the highest inhibition of 97% and 98%, respectively 20.

Based on literature review, there has been lack of published data on the corrosion inhibition effects of ethyl acetate extract on low carbon steel in acidic medium. Hence, the aim of this research was to study the inhibition efficiency of EAETS on low carbon steel in 0.5 M HCl. However, FT-IR, NMR and TGA spectrometry were applied to ascertain the constituents in the ethyl actate extract. Furthermore, corrosion parameters were Investigated via (PD) and (EIS) at various concentrations (1, 10, 50, 100, 500 and 1000 ppm) at 303K. Finally, Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) studies were also examine to ascertain the corrosion inhibitory properties of the samples.

**2. Experimental**

**2.1. Preparation of ethyl acetate extract of Tamarind shell**

The Tamarind shells were collected from katsina state, the shell was dried and grounded, the powdered plant samples 10 g were extracted with ethyl acetate (99.9%) 100 mL at ambient temperature. The EAETS was filtered and a rotary evaporator was used under reduced pressure to a concentrated dark green extracts. The concentrated extract was stored in refrigerator for further use.

**2.2. Characterization of ethyl acetate extract of Tamarind shell**

 The FT-IR spectra of EAETS were verified within the range of wavelength 4000 and 400 cm\_1 using a FT-IR spectrometer ((Perkin Elmer System 2000 (Waltham, MA, USA).

 The 13C NMR spectra of EAETS were studied on a Bruker Avance 500 MHz spectrometer (Fallanden, Switzerland). Tetramethylsilane (TMS) and Dimethyl sulfoxide (DMSO)-d6 were applied as solvent and internal standard for NMR analyses.

 TGA Analysis was performed using Thermogravimetric analyzer A Perkin Elmer TGA 7 (Waltham, MA, USA) verified the thermal degr**a**dation of 5 mg samples of EAETS from 30 °C to 900 °C under nitrogen atmosphere with a 10 °C min−1 heating rate.

**2.4. Electrodes and electrolytes**

The low carbon steel (LCS) substrate applied in study posses the following chemical constituents: 0.16 wt. % Si, 0.01wt.% C, 0.15 wt.% Mn, 0.01 wt.% P, 0.02 wt.% Na, 0.03 wt.% Mg, 0.06 wt.% Al and balance wt.% Fe. The electrodes were prepared by embedding carbon steel. The exposed surface area of the carbon steel was 3.142 cm2, upon the measurements, the area was manually abraded with dissimilar grades of silicon carbide papers up to the 1000 grade, and then, the electrode was rinsed with acetone and distilled water prior to immersing into the test solution. The inhibition tests were accomplished in a 0.5 M HCl solution without and with 1, 10, 50, 100, 500 and 1000 ppm concentrations of the ethyl acetate extract.

**2.5. Electrochemical measurements**

Three electrode systems were used for electrochemical measurements using Potentiostat/Galvanostat (Gamry USA Instruments) Gamry reference 600. The cell setups, includes: (a) counter electrode (CE), ie a platinum rod, (b) working electrode (WE); ie a coupon of LCS (7 cm × 3 cm × 0.1 cm) with an area of 3.142 cm2, and (c) reference electrode (RE), ie a saturated calomel electrode, hence all were placed in a 50 mL volume of electrolyte with different inhibitor concentrations, at temperature (303k).

EIS and PD analysis were carried out upon a steady-state open circuit potential (OCP), with the LCS electrodes immersed into the acidic solutions for 1 h, without and with 1, 10, 50, 100, 500 and 1000 ppm concentrations of the ethyl acetate extract at a temperature 303k.

Potentiodynamic polarization signals were acquired at 0.5 mVs-1 scanning rate; the performed experiments after 15 min were completed through immersion in the particular destructive solutions for potential stability. A ±250 mV potential range from the Eocp was scanned.

EIS tests were performed in a range of frequencies. Upon 15 min open circuit potential at frequency 10000 kHz to 0.01 Hz with 5 mV amplitude in the absence and presence of 1, 10, 50, 100, 500 and 1000 ppm samples.

**2.6. Surface characterization**

Low carbon steel surfaces were exposed to 24h immersion in 0.5 M HCl and 1000 ppm inhibited solutions were examined by a SEM (FEI Quanta FEG 650) at 500x magnification with 10 Kv beam voltage. EDX (EDX, Oxford instrument, X-Max) were carried out at an acceleration voltage of 10 kV to determine the average carbon content of the electrodes.

**3. Results and discussion**

**3.1. Chemical composition of the EAETS**

The compound structure isolated from the ethyl acetate extract of Tamarind shell was characterized by FTIR, 13C NMR, and TGA, respectively (Fig. 1). The chemical structure was established by evaluation with prior published spectroscopic data Saad et al., (2014).



Fig.1. Chemical structures of Tannin monomer

**3.2. Fourier transforms infrared spectroscopy (FT-IR) of the EAETS**

FT-IR was carried out to ascertain the functional groups presents in the extract. The signals perceived in the FT-IR bands of the extract (Fig. 2) are in agreement with the chemical structures of the isolated compound. Hence, the FTiR absorption bands O-H stretching vibration, C-H stretching vibrations, unsaturated carbonyl groups, C=C vibrations and C-O-C bonds vibrations were observed at the around 3100–3500 cm -1, 2900–3000 cm -1 , 1705 cm -1, 1400–1600 cm -1 and 1050 cm-1, correspondingly.



Fig.2.FT-IR spectrum of EAETS

**3.3. Nuclear Magnetic Resonance spectroscopy (NMR) of the EAETS**

The spectral data of the characterized compounds according to 21.are given below; 13C NMR ((DMSO)-d6): 40 ppm (C1'), 131 ppm (C2'), 118 ppm (C3'), 145 (C4'), 143 ppm (C4a), 36 ppm (C4), 30 ppm (C5), 156 ppm (C5'), 116 ppm (C6'), 118 ppm (C6), 98 ppm (C7), 155 ppm (C8a), 153 ppm and (C8), 107 ppm, as presented in (Fig 3).



Fig.3. NMR spectrum of EAETS

**3.4. Thermogravimetric analysis (TGA) of the EAETS**

The thermogravimetry analysis of EAETS carried out under nitrogen was presented in (Fig. 4), two mass losses were seen which characterize the occurrence of thermal activity. Firstly, is the thermal degradation corresponding to the absorbed moisture by the compound with a weight lost at 150-200◦C. secondly, is more prominent, which relates to tannin decomposition. EAETS decomposed at range of 400-500◦C.The excess acquired upon decomposition procedure is 20% EAETS.

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Fig.4. TGA of spectrum EAETS

**3.5. Electrochemical tests**

3.5. 1. Electrochemical impedance spectroscopy (EIS)

The Nyquist and bode plots for the low carbon steel immersed in 0.5 M HCl solutions with and without various concentrations of the EAETS after 1 h are shown in (Fig. 6). As presented, the Nyquist plots of the low carbon steel display distinct capacitive semi-circles signifying a deterioration procedure primarily controlled by charge transfer22. The increasing in concentrations of the EAETS during 1 h of immersion time with increase in semicircles capacitive diameter, described the establishment of a resilient protective film on the carbon steel surface 23. In comparism to 0.5 M HCl solution, the nature of the EIS curves achieved from solutions comprising the EAETS were preserved. The findings establish that the mechanism of corrosion inhibition was improved by the adding inhibitors 24. The deviating of the Nyquist plots from a perfect semicircle might be ascribed to dispersion of frequency and inhomogeneities of the substrate surface, subsequent in irregular charge loading among the OHP (outer Helmholtz plane) and the metal 25. Hence, a constant phase element CPE was engaged as an alternative to a pure double layer capacitor in the equivalent circuit model (Fig. 5) to attain a well fitting in the EIS analysis of results. The Rct and Rs parameters present in the equivalent circuit symbolize the charge transfer resistance and solution resistance, respectively. Also, the CPE is well-defined by the subsequent mathematical expression 26.

$Z\_{CPE}=\frac{1}{Y\_{0}(jω)^{n}}$ (1)

The CPE comprises two factors, i.e. the admittance magnitude (Y0) as well as the exponent (n)27. The exponential parameter (n) is applied to evaluation of surface irregularity as a result of surface roughness, porous layer formation and inhibitor adsorption 28.



Fig.5. Proposed equivalent electrical circuits used to fit EIS data

The electrochemical parameters Rct, Rs, n and CPE for the EIS data were fitted to some equivalent electrical circuit models using Gamry Echem analytical softwere. The inhibition efficiency (Table 1) was calculated utilizing the following equation:

IE % $=\frac{R\_{ct(i)}-R\_{ct}}{R\_{ct(i)}} ×100$ (3.1)

where Rct(i) and Rct are values of charge transfer resistance (Ω cm2) with and without inhibitor, (EAETS) respectively.



Fig.6. **N**yquist plots of 0.5 M HCl, and EAETS on carbon steel at 303 K

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Fig.7. Bode impedance diagrams; (a) Bode modulus diagrams and (b) Bode phase diagrams of carbon steel in 0.5 M HCl, and EAETS

As seen in the Table 1, the values of charge transfer resistance (Rct) increased with increasing concentrations of the EAETS. Also, the highest inhibition outcome was detected at 1000 ppm of the EAETS, the different concentrations, resulting in Rct values of 76.00 Ω cm2, 81.47 Ω cm2, 117.03 Ω cm2, 135.42 Ω cm2, 192.76 Ω cm2, and 400.60 Ω cm2, respectively. Thus, might be due to adsorption and inhibitor coverage on the substrate surface increases with increasing concentration of the inhibitor29. Furthermore, the rise in the charge resistance indicates an increase in inhibition efficiency. The inhibition efficiency value of the EAETS was 80.90 for 1000 ppm concentrations.

Based on our findings for the corrosion of low carbon steel in 0.5 M HCl in the presence of the EAETS propose that the extracts might function as effective inhibitors of corrosion. The increasing in (n), values is defined as a degree of the surface heterogeneity, related to a blank solution, may be described through establishment of a extra uniform surface due to adsorbed inhibitors 30. Based on Table 1 data, lower n values were established in EAETS inhibitor in comparism to blank 0.5 M HCl solutions. Thus, (n) values displayed uneven trends with rise in inhibitor concentrations. This could be due to the haphazard dispersion of organic molecules that were physically adsorbed on the substrate surface. In contrast to our outcomes, irregular trends in (n) values have been documented in various studies 31.

Table The Nyquist impedance parameters at different concentrations of EAETS at 303 K

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Concentrations (ppm) | Rct (Ω cm2) | CPE(μF cm-2) | n | IE (%) |
| 0.5 M HCl | 76.51 | 273 | 0.9495 | - |
| 1 ppm EAETS | 76.00 | 236 | 0.9506 | -0.67 |
| 10 ppm EAETS | 81..47 | 227 | 0.9448 | 6.10 |
| 50 ppm EAETS | 117.03 | 203 | 0.9219 | 34.63 |
| 100 ppm EAETS | 135.42 | 243 | 0.9110 | 43.50 |
| 500 ppm EAETS | 192.76 | 208 | 0.8765 | 60.31 |
| 1000 ppm EAETS | 400.60 | 189 | 0.8256 | 80.90 |

3.5.2. Potentiodynamic polarization curves

The potentiodynamic polarization signals acquired for the LCS in 0.5 M HCl and with different concentrations (1, 5, 50, 100, 500 and 1000 ppm) of the EAETS at 303 K are presented in Fig. 8. The values of Ecorr,, icorr, (bc) and (bc) attained by the linear extrapolation of Tafel parts of, anodic curve, cathodic curve and inhibition efficiency (%) were calculated using Eq. (2) are listed in Table 2.

IE % = $\frac{i\_{corr} - i\_{corr (i)} }{i\_{corr}}$ × 100 (2)

where icorr and icorr(i) denote corrosion current densities (mA cm-2) with and without EAETS inhibitor, respectively.

Where icorr and icorr(i) are the corrosion current density values in the absence and presence of the EAETS, individually., the values of corrosion current density (icorr) were reduced in the presence of all inhibitor concentrations when compared with the value of uninhibitor solution. The corrosion current density values of the EAETS decreased with increasing the concentration of the inhibitor. Thus, the inhibitors displayed well efficiency even at low concentrated solutions. This might also describe the relatively little amount of EAETS on corrosion mitigations (Table 2). As presented in Fig. 8, the current densities of the cathodic and anodic sites reduced in the existence of the EAETS which propose that the molecules establish a protective film on the substrate surface32. The occurrence of various concentrations displayed parallel cathodic curves of the EAETS and the cathodic Tafel shapes almost constant (bc), this shows that the occurrence of the inhibitor molecules does not alter the hydrogen evolution mechanism, hence, the reaction of hydrogen evolution is activation measured 33. In the presence of the EAETS, the values of corrosion potential (Ecorr) shifted irregularly as compared to the blank. If the corrosion potential displacement is fewer than 85 mV with respect to (Ecorr) blank corrosion potential, the inhibitor can then be categorized as mixed-type inhibitor 34. Thus, the EAETS can be categorized as mixed-type inhibitors.



Fig.8 Tafel curves of 0.5 M HCl solution with various concentrations EAETS at 303 K.

Table 2: Electrochemical polarization parameters for caron steel in 0.5M HCl solution in the absence and presence of EAETS at 303 K.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Concentration (ppm) |  Ecorr (mV)  | icorr(mAcm-)  | βa (mVdecade-1)  | -βc (mVdecade-1) | IE (%)  | CR (mpy) |
| 0.5 M HCl | -468  | 0.1260  | 85 | 114 | - | 57.43 |
| 1ppm EAETS | -476  | 0.0992  | 84 | 109 | 21.26 | 45.22 |
| 10ppmEAETS | -480 | 0.0910  | 76 | 98 | 27.77 | 41.47 |
| 50 ppm TEAE | -460  | 0.0743  | 64 | 87 | 41.03 | 33.86 |
| 100ppmEAETS | -483 | 0.0446  | 75 | 89 | 64.60 | 20.32 |
| 500ppmEAETS | -480 | 0.0263  | 81 | 87 | 79.12 | 12.01 |
| 1000ppmEAETS | -482  | 0.0238  | 78 | 72 | 81.11 | 10.83 |

**3.7. Adsorption isotherms studies**

An adsorption isotherm is one of the aspects of knowing the interfaces among inhibitor molecules and substrate surfaces. Isotherms such, Langmuir, Freundlich and Temkin were established in expectations on relating the behavior of adsorption of EAETS on LCS in 0.5 M HCl. Over the correlation coefficient applications (R2), the outcomes were established to best fit by Langmuir adsorption isotherm (Fig. 9):

Langmuir equation; $\frac{C}{θ}= \frac{1}{K\_{ads}}+C$ (3)

In Eq. (3), where C denotes inhibitor concentration, θ indicates the surface coverage, and Kads refers to the equilibrium constant associated with the adsorption process. Langmuir adsorption isotherms were used to fit surface coverage (θ) values obtained from potentiodynamic polarization measurement at varying concentrations of EAETS inhibitor. The of Langmuir isotherm linear curves model for the EAETS samples are shown in (Fig. 10). The equilibrium constant of adsorption $K\_{ads}$ was calculated from the straight lines intercepts and $K\_{ads}$ was besides correlated to the standard free energy of adsorption ∆Gads by the below equation 35.

∆Gads = -RT ln ($K\_{ads} ×A) $ (4)

In Eq. (4), T indicates the Kelvin temperature, R refers to the universal gas constant, and 𝐴 refers to the concentration of water (55.5 M or 1000 g L-1). Table 3 displayed that the EAETS samples intensely adsorbed on the LCS surface. Equally a common rule, ∆Gads values of less than -20kJ mol-1 designate physical adsorption, which is as a results of electrostatic interactions between the charged metal surface and the inhibitor molecules, although values more negative than -40kJ mol-1 designate transfer or sharing of electrons from molecules of inhibitor to the substrate surface, thus present the establishment of chemisorption ie co-ordinate bonds 36. The values of ∆Gads obtained for the sample -19.45 kJmol-1 representing that the interface among the inhibitor molecules and the LCS surface involves physisorption37.



Fig.9. Adsorption isotherm plots using Langmuir equations upon potentiodynamic polarization measurements at different concentrations of EAETS at 303K

Table 3: The adsorption parameters upon potentiodynamic polarization measurement at different concentrations of EAETS at 303 K

|  |  |  |  |
| --- | --- | --- | --- |
| Concentrations (ppm) | θ | Kads(L g-1) | $∆$Gads(kJ mol-1) |
| 0.5 M HCl | - | - | - |
| 1 ppm EAETS | 0.2126 | 40.65 | -19.45 |
| 10 ppm EAETS | 0.2777 |  |  |
| 50 ppm EAETS | 0.4103 |  |  |
| 100 ppm EAETS | 0.6460 |  |  |
| 500 ppm EAETS | 0.7812 |  |  |
| 1000 ppm EAETS | 0.8111 |  |  |

**3.8. Scanning Electron Microscope (SEM) and Energy Dispersive x-ray (EDX)**

The SEM effects of the LCS after 24 h of immersion are displayed in (Fig. 11). Coarse, rough external establishments and corrosion yields were detected in (Fig. 11a), subsequent from extreme metallic dissolution in the aggressive medium. Though, the SEM picture (Fig. 11b) of the LCS in solution comprising 1000 ppm of the EAETS showed smooth exteriors as a result of a protective film formation on the substrate surface. However, investigation of the SEM pictures of the LCS cured in the existence of the EAETS, offered a substrate deprived of noticeable corrosion defects, cracks and pits. However, EDX spectra were utilized to study the elements present on the LCS surface after 24 h of dipping in 0.5 M HCl and inhibited solutions. The elemental configurations acquired via EDX are presented too in (Fig. 11a and 11b). The EDX figures gained from LCS dipped in the 0.5 M HCl solution displayed that the substrate exterior contained extra oxygen as the result of iron oxides formation. Chlorine is present additionally. Also, lesser percentage of iron was seen present on the LCS exposed to 0.5 M HCl is an indication of austere corrosion destruction. Though, the percentage of oxygen was reduced significantly by the occurrence of the EAETS, (Table 4) even though there was no Cl discovered on the substrate surface cured in the occurrence of the EAETS. Thus, suggested the great efficacy of the EAETS and also attributed to tannin contents. In agreement with these results, SEM and EDX established that EAETS contribute to the formation of a protecting film on the LCS.



Fig.10. SEM micrographs and EDX spectra of mild steel after 24 h immersion in 0.5 M HCl solution (a) bare mild (b)1000 ppm EAETS at 500x magnification

Table 4: The EDX analysis of the percentage atomic content of carbon steel in 0.5 M HCl

|  |  |
| --- | --- |
| Mild steel  | Element (%) |
| Fe | C | O | Cl |
| 0.5M HCl treated  | 31.52 | 4,46 | 61.69 | 2.33 |
| 1000 ppm EAETS | 70.29 | 21.49 | 8.54 |  - |

**4. Conclusion**

In the present study, EIS results show that the EAETS are active inhibitors of the LCS corrosion. The maximum inhibition efficiency acquired from the EIS results was 81.11%. The adsorption of the EAETS on the LCS was established to follow the Langmuir adsorption model. Polarization by anodic and cathodic shapes designated that the extracts are mixed-type inhibitors. The results of the adsorption isotherm studies confirmed that the mechanism of adsorption is predominantly physisorption exists among the EAETS and LCS. According to these results, the EAETS, containing the high tannin contents, can be used as active inhibitor against the deterioration of LCS.

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