Corrosion resistance of Aluminium steel in 1N Sodium Hydroxide solution by an aqueous extract of *Thespesia populnea* plant leaves

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Abstract
Corrosion resistance effect of aluminium steel in 1N sodium hydroxide by an aqueous extract of Thespesia populnea plant leaves has been investigated by mass loss method. It is observed that as the concentration of the inhibitor increases, the inhibition efficiency increases. The mechanistic aspects of corrosion resistance have been studied by potentiodynamic polarization technique and electrochemical impedance spectroscopy. A maximum inhibition efficiency of 78.70% is achieved by this inhibitor system. Potentiodynamic polarization technique reveals that the inhibitor system functions as a mixed type of inhibitor, controlling anodic and cathodic reactions. It is noted that in the presence of inhibitor, linear polarization resistance value increases and corrosion current decreases. Electrochemical impedance studies reveal that a protective film is formed on the Al steel surface, since in the presence of inhibitor system, the charge transfer resistance value increases and double layer capacitance value decreases. This is due to adsorption of the molecules of the active ingredients of the extract on the Al steel surface. The inhibition efficiency increases due to adsorption of hetero atoms present in TP plant leaves extract on the Al steel surface. The maximum inhibition efficiency and the lower corrosion rate are obtained at high concentration 1200 ppm for TP plant leaves extract.

By further increase in inhibitor concentration above 1200 ppm, the inhibition efficiency and corrosion rate almost remained constant. So, the 1200 ppm concentration is fixed as the maximum inhibition for the investigative aqueous extract of plant leaves as corrosion inhibitor. This concentration corresponds to the attainment of a saturation value in surface coverage of Al steel. The protective film formed over the Al steel surface has been characterized by Fourier Transform infra-red spectroscopy. The surface morphology of the protective film of carbon steel immersed in sodium hydroxide in the absence and presence of inhibitor has been studied by Scanning electron microscopy. The outcome of the study can be used in pickling industry wherein sodium hydroxide is used to remove the rust on the Al steel surface.

Keywords: Corrosion inhibition, basic medium, surface morphology, EIS, Al steel, *Thespesia populnea* plant leaves, green inhibitor.

Introduction

Corrosion is destruction of materials by its chemical or electrochemical reaction with their environments. The use of corrosion inhibitors to control the corrosion is one of the most important techniques. The study of corrosion inhibition using inhibitor in alkaline media is one of the most stimulating areas in the present research due to its potential applications in industries[6,9,30].

In alkaline medium, several corrosion inhibitors have been used. The ability of a compound to serve as inhibitor is dependent on its ability to form a compact barrier film and nature of adsorption of inhibitor on metal surface. Most of the corrosion inhibitors contain hetero atoms such as oxygen, nitrogen, sulphur and multiple bonds.

The electrons on these atoms will coordinate with metal ions on the anodic sites of the metal surface and form metal inhibitor complex as protective layer. Many corrosion inhibitors such as chromates have been banned due to their toxic nature and due to the objection from environmental scientists. So, there is a need to use less toxic (or nontoxic) environmentally friendly corrosion inhibitors. The ingredients in natural products can be extracted by simple procedures with low cost.

Moreover, the extracts of the natural products are biodegradable in nature. Bijauliya et al[4] reported that S*yzygium cumini linn* plant extract possessed various chemical constituent which are responsible for pharmacological activity. This plant is reported to possess many pharmacological activities like antidiabetic activity, antioxidant, antiinflammatory, antidiarrheal activity, antiviral, antifertility activity, gastroprotective, antipyretic, anti-histaminic, antimicrobial and antiplaque. Natural corrosion inhibition and adsorption characteristics of *Tribulusterrestris* plant extract on aluminium (Al) in hydrochloric acid environment have been investigated[5].

Potentiodynamic polarization (PDP) measurements indicated that the nature of inhibitor is a mixed type. Impedance studies supported the formation of a protective layer of inhibitor on a metal surface. Scanning electron microscopy (SEM) images confirmed the creation of a protective film over the metal surface.

Mofidabadi et al[23] have investigated steel-alloy surface protection against saline attacks via the development of Zn (II)-metal-organic networks using *Lemon verbena* leaves extract. Synergistic effect is noticed when Ce and Zn ions are added to the leaves extract. Fourier transform infrared spectroscopy (FTIR), Ultraviolet-visible (UV-Vis) and Raman spectra have been employed to analyze the protective film. Field-emission scanning electron microscopy (FESEM) and Atomic force microscopy (AFM) have been used to study the surface morphology of the protective film.

Mirinioui et al[22] have used an eco-friendly inhibitor, namely, *Dysphania ambrosioides* essential oil, for mild steel corrosion in hydrochloric and sulfuric acid medium. Thermodynamic parameters and quantum chemical parameters have been calculated and discussed. Corrosion mitigation of carbon steel in hydrochloric acid solution using grape seed extract has been studied by Marhamati et al[20].

The study revealed the formation of more compact corrosion products with improved integrity in the presence of grape seed, which confirmed electrochemical test results. Besides, water droplet contact angle, FESEM coupled with energy dispersive spectroscopy, FTIR, Raman spectroscopy, X-ray photoelectron spectroscopy (XPS) and AFM were utilized to study the surface of mild steel specimens after dipping in acidic solutions. Corrosion inhibition of mild steel by aqueous leaf extract of purple hedge plant has been studied by Mukhopadhyay et al[24].

Gas chromatography along with FTIR and Nuclear magnetic resonance (NMR) spectroscopy revealed the presence of carbohydrate molecules as major chemical constituents of the freeze-dried leaf extract. A plausible mechanism for inhibition properties was proposed on the basis of experimental and theoretical studies.

Shahini et al[34] have used chamomile flower extract as a green corrosion inhibitor for mild steel in HCl solution. 98% inhibition efficiency (IE) was obtained. The surface morphology of the protective film has been analyzed by FESEM, EDAX, AFM and contact angle examinations. The electrochemical behavior of 5083 Al alloy in an alkaline solution in the absence and presence of two green additives extracted from Menthapiperita L 'MP' and *Lawsoniainermis* has been analyzed[31]. The results of this study can open the way for future investigations to replace the synthetic anti-corrosion additives of Al air-batteries with non-toxic and biodegradable organic ones having similar corrosion inhibition performance without restricting anode discharge currents. The aqueous extract prepared using the biomass was applied to protect the surface of Al under acidic environment[25]. The influence of extract concentration, contact time and temperature on the inhibition efficiency has been studied. The biomass-based corrosion inhibitor was characterized using SEM and attenuated total reflection techniques. Fouda et al[10] have used *Feruula hermonis* (FH) plant extract as safe corrosion inhibitor for zinc in hydrochloric acid solution. The efficiency of FH plant extract, as inhibitor for zinc (Zn) corrosion in 1N HCl solution, has been tested by mass loss, electrochemical measurements (PDP), electrochemical impedance spectroscopy (EIS) and electrochemical frequency modulation (EFM) procedures, in addition to surface examination analysis. The outcomes indicated that FH extract showed good efficiency to Zn metal corrosion and displayed high inhibition efficiencies. The maximum IE approached 90.6% at 300 ppm extract.

The surface morphology of the protective film has been analysed by FTIR, AFM and SEM. The corrosion inhibitory effect of the ecofriendly addition of *Terminalia Catappa* leaf extract to soybean oil biodiesel in contact with zinc and carbon steel 1020 has been evaluated by Fernandes et al[11]. The morphology of the metal plates was analyzed by SEM/Dispersive Energy Spectroscopy, X-ray Diffraction (XRD) and the biodiesel through acidity index and FTIR spectroscopy.

The focus of the present study is to explore the corrosion resistance of Al steel in 1N sodium hydroxide solution by an aqueous extract of *Thespesia populnea* plant leaves (TPPL) by mass loss method. Another aim is to study the mechanistic aspects of corrosion inhibition by electrochemical studies such as potentiodynamic polarization (PDP) technique and electrochemical impedance spectroscopy (EIS). The surface film formed over the Al steel surface has been characterized by FTIR and SEM.

Material and Methods

The Al steel specimens were made from the same sheet of the following composition: 1.5% Pb, 0.1% Ga, 1% In and the remaining Al. Al specimen of the dimension $1.0 \times 4.0 \times 0.2$ cm were polished to mirror finish, degreased with trichloroethylene and used for mass – loss and surface examination studies.

Al steel specimen encapsulated in Teflon with an exposed cross section of $1 cm^2$ were used as the working electrode in PDP studies. The surface of the electrode was polished to mirror finish and degreased with trichloroethylene. The medium (1N NaOH) was prepared by dilution of an analytical grade sodium hydroxide with double distilled water.

Inhibitor preparation: An aqueous extract (100 ml) of *Thespesia populnea* plant leaves (TPPL) was prepared by boiling 10 g of shade dried leaves with double distilled water. The suspended impurities were removed by filtration. The solution was concentrated to 100 mL and used as corrosion inhibitor. Phyto-constituents of the aqueous extract of TPPL inhibitor are shown in scheme 1[32].

Mass loss method*:* Mass of the three polished Al steel specimens was measured before and after immersion in various test solutions (1N NaOH with different extract concentrations) for one hour. The inhibition efficiencies[28,30] were calculated from the relation as follows:

$$IE = \frac{CR_1 - CR_2}{CR_1} \times 100\ \% \quad (1)$$

where CR_1 is corrosion rate in the absence of inhibitor and CR_2 is the corrosion rate in the presence of inhibitor.

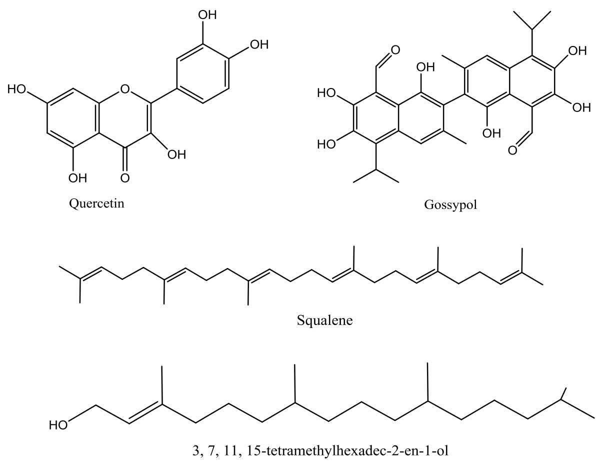
Electrochemical study*:* In the present work, corrosion inhibition of Al steel immersed in various test solutions was measured by polarization study. Electrochemical measurements were performed in a Solartron Electrochemical Analyzer, Model 1280.

Polarization study*:* Polarization studies were carried out in a three-electrode cell assembly (Figure 1). A SCE was the reference electrode. Platinum was the counter electrode. Al steel was the working electrode. From polarization study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes anodic = b_a and cathodic = b_c and LPR (linear polarisation resistance) values were measured[18,39].

AC Impedance spectra*:* The same instrument and set-up used for polarization study were used to record AC impedance spectra also. A time interval of 5 to 10 min was given for the system to attain a steady state open circuit model. The real part (Z') and imaginary part (-Z") of the cell impedance were measured in ohms at various frequencies. AC impedance spectra were recorded with initial E (V) = 0, high frequency (1-10^5 Hz), low frequency (1 Hz), amplitude (V) = 0.005 and quiet time (s) = 2. From Nyquist plot, the values of charge transfer resistance (R_t) and the double layer capacitance (C_{dl}) values were calculated[12].

FTIR spectral analysis*:* Perkin Elmer FTIR spectrophotometer was used to record the FTIR spectrum from 400 to 4000 cm^{-1}. The adsorbed plant leaves extract inhibitor on Al steel surface have been analyzed by FTIR spectra[21]. Protective film was scratched from the Al steel surface, which was immersed in 1N NaOH in the presence of the studied plant leaves extract inhibitors for one hour at room temperature.

SEM Analysis*:* The Al steel specimens immersed in various test solutions for 1 hour were taken out, rinsed with double distilled water, dried and subjected to the surface examination[33]. The surface morphology measurements of the Al steel surface were carried out by SEM using SEM instrument, JEOL MODEL JSM 6390.



Scheme 1: Constituents of the TPPL

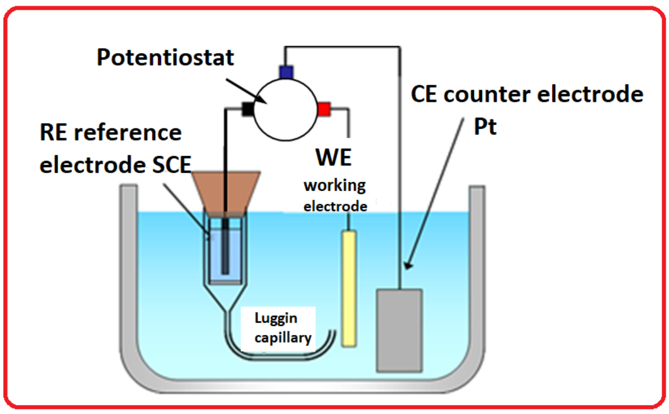


Figure 1: Three-electrode cell assembly

Results and Discussion

Mass loss method*:* Corrosion rates of Al steel in 1N NaOH and the inhibition efficiencies offered by an aqueous extract of *Thespesia populnea* (TP) plant leaves (immersion period 1 hour) are given in table 1.

It is observed that the inhibitor system offers good IE to Al steel in alkaline medium and when the concentration of the inhibitor increases, the IE increases. This is due to adsorption of the molecules of the active ingredients of the extract on the Al steel surface. A maximum IE of 78.70% is achieved by this TPPL inhibitor system[2,26,36,42].

Electrochemical studies: Electrochemical studies such as polarization study and AC impedance spectra have been employed in corrosion inhibition study[16,40]. When corrosion resistance increases because of blocking effect (Blanket effect) of adsorbed inhibitor molecules on the metal surfaces, the following observations are noted: linear polarization resistance (LPR) value increases and corrosion current (I_{corr}) decreases, charge transfer resistance increases, impedance increases and double layer capacitance decreases.

Polarization study*:* The polarization curves of Al steel immersed in 1N NaOH in the absence and presence of inhibitor are shown in figure 2 and the corrosion parameters are given in table 2. The electrodes were immersed in the test solutions for 30 minutes to reach the steady state potential[35].

At various inhibitor concentrations, leaf extract reduced corrosion current density. Inhibitors diminish both the cathodic and anodic curves. The pH-resistant TPPLE protects Al. The inhibitor concentration reduces cathodic and anodic current densities in a linear fashion. Inhibitors decreased the slopes of Tafel. Al dissolution and cathodic hydrogen production are influenced by inhibition. It suppresses both anodic and cathodic corrosion. Anodic Tafel constant (a) is more sensitive to inhibitor concentration than cathodic Tafel constant (c). The inhibitor has a greater impact on anodic dissolution than on cathodic hydrogen development. Inhibitors boost IE values.

Table 1
Inhibition efficiency of aqueous extract of *Thespesia populnea* (TP) plant leaves in controlling the corrosion of Al steel in 1N NaOH at room temperature (303K)

Concentration of inhibitor (ppm)	Corrosion rate (mmpy)	Inhibition Efficiency (%)	Surface coverage (θ)
blank	823.73	-	-
300	414.81	47.20	0.472
600	271.85	65.40	0.654
900	225.79	71.30	0.713
1200	167.61	78.70	0.787

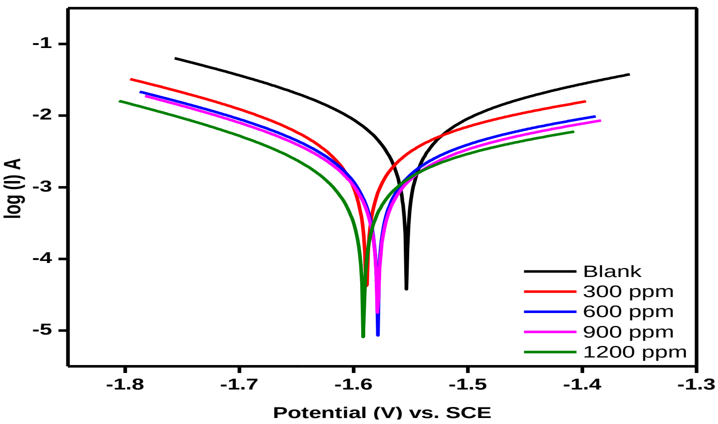


Figure 2: Potentiodynamic curves for corrosion of Al steel in 1N NaOH and inhibitor system

Table 2
PDP parameters of Al steel in 1N NaOH (90 mL) in the absence and presence of aqueous extract of TPPL

Concentration of TPPLE (ppm)	I_{corr} (mA)	E_{corr} (mV)	β_c (mV)	β_a (mV)	IE %
Blank	12.02	-1554	273	374	-
300	6.49	-1589	284	455	46.8
600	4.08	-1579	277	460	66.1
900	3.47	-1579	267	461	71.1
1200	2.59	-1591	262	462	78.5

As said, a higher quantity of inhibitor covers a larger surface area. Inhibitors augment E_{corr}, mostly anodically. A cathodic type inhibitor raises corrosion potential by almost 85 mV compared to a blank solution. TPPLE is a mixed-type inhibitor whose maximum displacement from the corrosion potential of the blank solution is less than +10mV. These observations confirm that a protective film is formed on the Al steel surface. This controls the corrosion of Al steel[1].

Analysis of results of AC impedance spectra*:* Alternating current impedance spectra (EIS) have been used to confirm the formation of protective film on the Al steel surface[7,19]. If a protective film is formed on the Al steel surface, charge transfer resistance (R_t) increases, double layer capacitance value (C_{dl}) decreases and impedance [log (Z/Ohm)] value increases. The AC impedance spectra of Al steel immersed in 1N NaOH in absence and presence of inhibitor (extract of TP lant leaves) are shown in figure 3 (Nyquist plots).

Analysis of FTIR spectra*:* FTIR characterization is a major tool that can be used to predict the nature of bonding of phytochemical constituents of the corrosion inhibitor on the carbon steel surface[15,13,41]. The absorption bands of the functional groups present in corresponding systems are given in table 4.

FTIR spectrum of aqueous extract of TPPL, dried on glass plate and then mixed with KBr is shown in figure 4. -OH stretching occurs at 3436.47 cm^{-1}. C-N stretching is 1449.77 cm^{-1}. C-H stretching is 2921.48 cm^{-1} as predicted. C-C stretching peak is at 1064.91 cm^{-1}. C=O-induced peak is at 1643.62 cm^{-1}. The N-H bond is strong as demonstrated by its 595.56 cm^{-1} frequency[29].

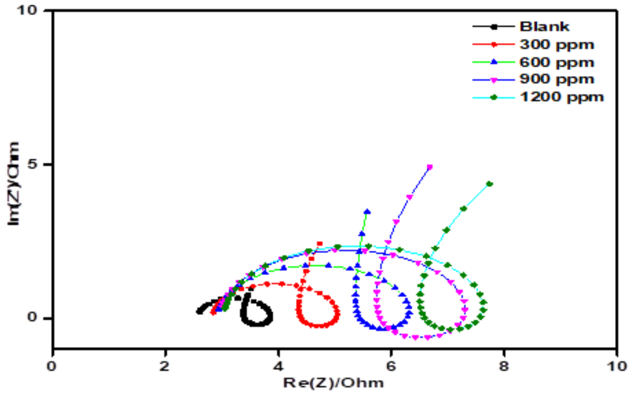


Figure 3: AC impedance spectra of Al steel immersed in 1N NaOH in the absence and presence of aqueous extract of TPPL inhibitor (Nyquist plots)

Table 3
Electrochemical impedance parameters from Nyquist plots for the corrosion of Al steel with aqueous extract of TPPL in 1N NaOH.

[TPPLE] (ppm)	Rs (Ohm)	R_{ct} (Ohm)	C_{dl} (μF)	IE (%)
Blank	2.56	0.80	7.03	-
300	2.85	1.50	3.10	46.8
600	2.95	2.32	1.63	65.8
900	3.00	2.73	1.93	70.8
1200	3.05	3.47	2.61	77.1

Table 4
FTIR spectral data for the crude extract of TPPL and the protective film from Al steel surface after dipping in 1N NaOH with inhibitor system.

IR bands of crude aqueous extract of TPPL (cm^{-1})	IR bands of protective layer from Al steel surface (cm^{-1})	Frequency assignment to functional groups
3436.47	3447.40	-OH
2921.48	2922.70	C-H stretching
1643.62	1642.42	C=O stretching
1064.91	1032.83	C-C stretching
1449.77	1453.26	C-N
595.56	780.61	N-H
-	472.78	Y-Al_20_3

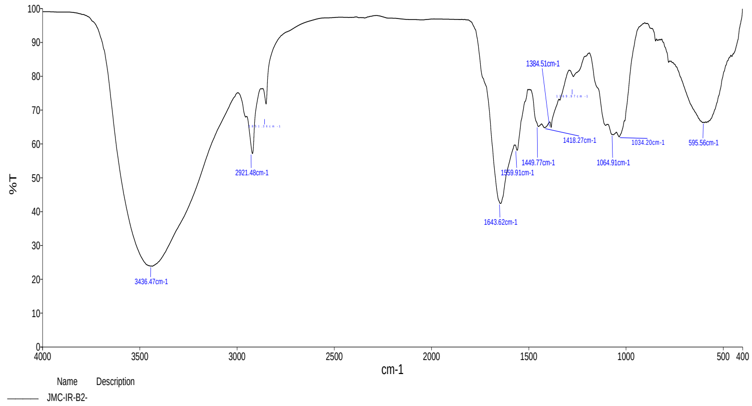


Figure 4: FTIR spectrum of aqueous of extract of TPPL

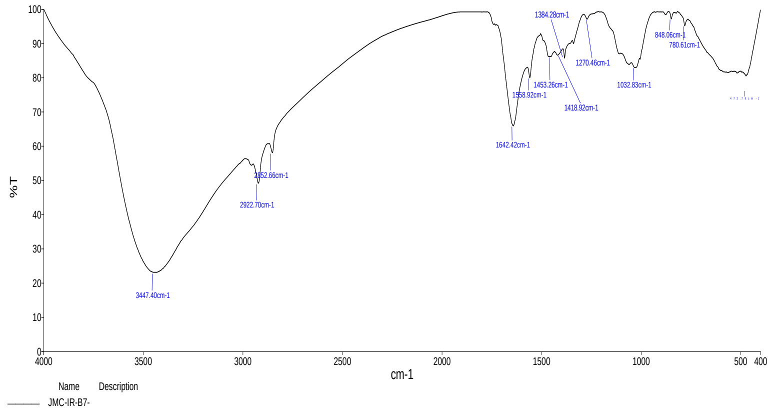


Figure 5: FTIR spectrum of protective film formed on the Al steel surface after dipping in 1N NaOH with 10% aqueous extract of TPPL

A protective film is formed on the surface of the Al steel immersed in 1N NaOH with 1200 ppm of aqueous extract of TPPL. The FTIR spectrum of the film (KBr) is shown in figure 5. Molecular adsorption may occur[17,27] via O-H if the stretching frequency increases from 3436.47 to 3447.40 cm^{-1}. C-N stretching frequency is 1453.26 cm^{-1}, up from 1449.77 cm^{-1}. The C-H group in the pure IR spectra of TPPL aqueous extract and the protective layer generated over the Al steel surface shifts from 2921.48 to 2922.70 cm^{-1}. C=-O group frequency shifts from 1643.62 to 1642.42 cm^{-1}. N-H frequency changes from 5985.56 cm^{-1} to 780.61 cm^{-1}. The C-C stretching peak's apparent wavelength changed from 1064.91 to 10323.83 cm^{-1} due to a frequency shift. The Fe-complex presumably generates the 472.76 cm^{-1} band[13].

The protective layer over Al steel matches the band peaks in the aqueous extract of TPPL, demonstrating that the inhibitor compounds are phytochemicals. All the bands on Al steel have created a complex. Almost all the band peaks appeared in aqueous extract of TPPL and defensive layer developed on the Al steel is nearby same and suggests that the phytochemical constituents of inhibitor molecules adsorbed on surface of Al steel. All bands evidently represent the formation of a complex on the Al steel surface.

Analysis of SEM*:* The SEM images of various surfaces are shown in figure 6. The SEM image of polished Al steel is shown in figure 6(a). The SEM image of polished Al steel immersed in 1N NaOH (corrosive medium) is shown in figure 6(b). The SEM image of the polished Al steel immersed in 1N NaOH (corrosive medium) and inhibitor system is shown in figure 6(c). The SEM image of polished Al steel is smooth. The SEM image of the Al steel in corrosive medium is found to be rough and the pits are visible on the Al steel surface. The SEM image of the Al steel in corrosive medium and inhibitor system is smooth due to the formation of protective film[8,14,29,38]. Thus SEM study is useful to know the smoothness of the protective film.

Conclusion

In this present study, the anti-corrosion properties of an aqueous extract of *Thespesia populnea plant* have been tested against the Al steel in 1N NaOH (corrosive medium). Increase in IE and decrease in CR of Al steel in 1N NaOH medium were observed with the increase in concentration of extract of TPPL. The maximum IE of 78.70% was achieved for 1200 ppm of TPPL as corrosion inhibitor. The PDP studies conclude that TPPL was performed as a mixed type of inhibitor. According to EIS, there was an increase in polarization resistance (R_t) and decrease in double layer capacitance (C_{dl}). This behavior was found to be due to the formation of dense protective layer on the metal/electrolyte surface. The formation of protective film on the surface of Al steel has been characterized by FTIR.

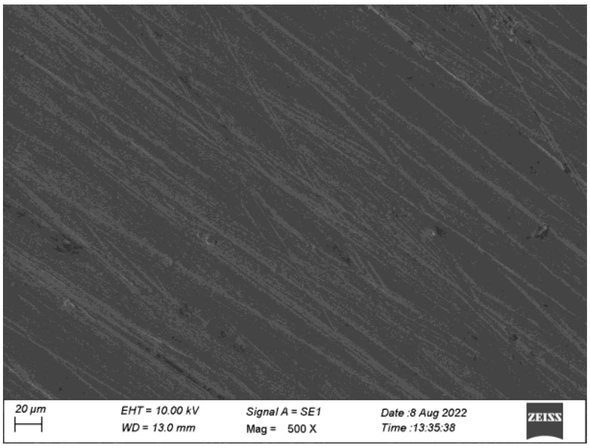


Figure 6a: SEM image of polished Al steel coupon before immersion in 1N NaOH (control)

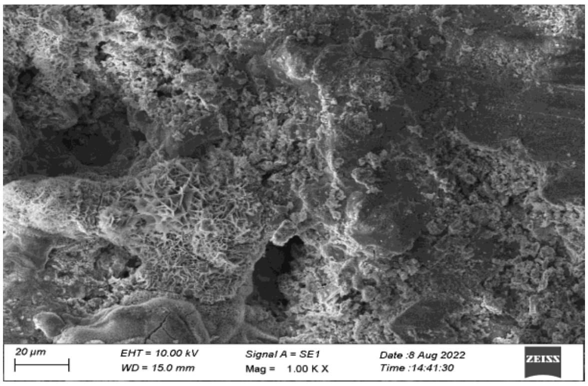


Figure 6b: SEM image of Al steel coupon after immersion in 1N NaOH (blank)

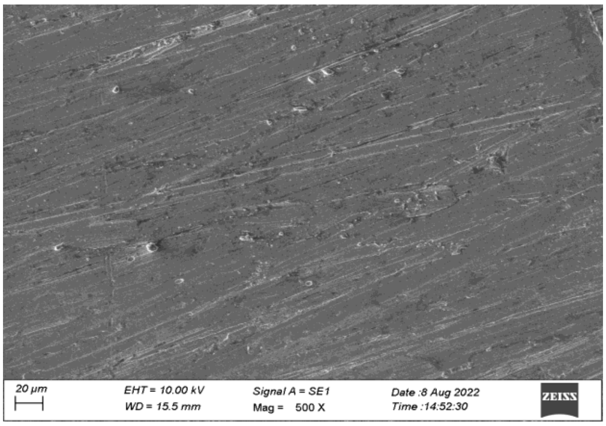


Figure 6c: SEM image of polished Al steel coupon after immersion in 1N NaOH in the presence of 1200 ppm of aqueous extract of TPPL

Furthermore, microscopic studies such as SEM have indicated the presence of smooth surface in case of inhibited Al steel when compared to the uninhibited samples. Aqueous extract of *Thespesia populnea* plant leaves has been identified to be a highly efficient corrosive inhibitor of Al steel in 1N NaOH medium. The outcome of the study may find applications in cotton fabric processing, metal cleaning and processing, oxide coating, electroplating and electrolytic extraction.

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