

Improvement of the Protection of Mild Steel in Acid using Nicotinic acid N-oxide and Isonicotinic Acid N-oxide as Inhibitors

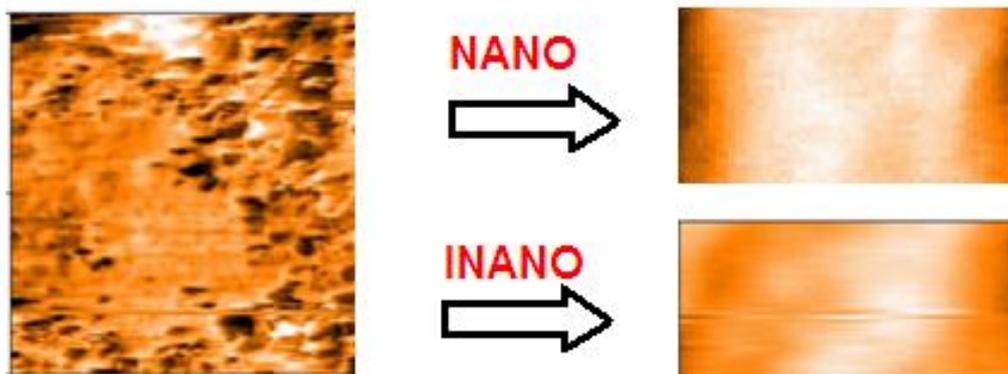
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GRAPHICAL ABSTRACT



Nicotinic acid N-oxide (NANO) and isonicotinic acid N-oxide (INANO) both compounds act as cathodic inhibitors. The better performance of INANO compared to NANO as corrosion inhibitors on mild steel

Abstract

Inhibition of mild steel in 2N sulphuric acid has been tried with green inhibitors of nicotinic acid N-oxide and isonicotinic acid N-oxide. The corrosion inhibitors were synthesised and tested by UV-Vis, FTIR, NMR, weight loss, Tafel, Impedance, SEM and AFM. The studies were made with 2N sulphuric acid with the inhibitor concentration in the range of 50-350ppm. Among the four concentrations of both inhibitors 250 ppm is produced higher inhibitor efficiency. The results reveal that the selected inhibitors performed better in the medium and function by adsorption mechanism on to the metal surface.

Keywords: Mild steel; Corrosion inhibition; Nicotinic Acid N-Oxide; Isonicotinic Acid N-Oxide; AFM

1. INTRODUCTION

The corrosion inhibitors in acidic solution can interact with metal and affect the corrosion reaction in a number of ways. Most of well known acid inhibitors are organic compounds containing heteroatom like, nitrogen, sulphur and oxygen atoms¹. Nicotinic acid as a non-toxic corrosion inhibitor for hot dipped Zn and Zn-Al alloys coating on steel in hydrochloric acid medium. The corrosion inhibition of these coatings was discussed in terms of blocking the electrode reaction by adsorption of the molecules at the active centers on the electrode surface. It was found that the adsorption of nicotinic acid on coating surface followed Langmuir adsorption isotherm with single molecular layer, and nicotinic acid adsorbed on the coating surface probably by chemisorptions². Many synthetic and green inhibitors³⁻¹³ have been tried with the corrosion of mild steel in sulphuric acid medium. Each inhibitor has its own merits and demerits. Synthetic inhibitors pose the problem of high cost because of the costlier raw materials and toxicity exhibited by them. Natural plant extracts have been tried as green inhibitors so that they resemble non-toxic and easy to extract compared to the synthetic methods. Corrosion inhibitors are either synthesised or extracted from the plants by refluxing with water or alcohol or a suitable solvent miscible with water. The extract concentrate is too difficult to be assessed in terms of concentration of green inhibitors. The inhibitive action of synthesized benzimidazole derivatives, namely: 2-(1-(morpholinomethyl)-1*H*-benzo[*d*]imidazol-2-yl)phenol, 2-(1-((piperazine-1-yl)methyl)-1*H*-benzo[*d*]imidazol-2-yl)phenol and 2-(1-((piperidine-1-yl)methyl)-1*H*-benzo[*d*]imidazol-2-yl)phenol on corrosion of N80 steel in 15% HCl solution has been studied¹⁴. Corrosion inhibition effect of 2-(2-(2-phenyl)-3-(isonicotinamido)-4-oxothiazolidin-5-yl) acetic acid and N-(2-phenyl-4-oxothiazolidin-3-yl)-isonicotinamide on mild steel corrosion in 15% HCl solution was studied¹⁵.

The present study focuses on the synthesis of nicotinic acid N-oxide and isonicotinic acid N-oxide and testing the synthesised compounds for their corrosion inhibition behaviour. Synthesised compounds are tested with FTIR spectra for their structural confirmation. They are then suitably diluted to get the desired concentration and tested with weight loss studies, polarization studies and electrochemical impedance spectra (EIS) techniques.

2. EXPERIMENTAL

2.1 Synthesis of inhibitors

Inhibitors of nicotinic acid N-oxide and isonicotinic acid N-oxide were synthesised through reported method ¹⁶. To a mixture of nicotinic acid (200 mg), molecular sieve catalyst (10 mg) and water (10 ml) at a temperature of 60°C, and 30% dilute hydrogen peroxide (1:2 or 1:4 molar ratio) was added drop wise. The course of the reaction was monitored by thin layer chromatography (TLC). After the completion of the reaction, the catalyst was separated by filtration. The excess solvent was removed at reduced pressure and the product was separated by column chromatography using silica gel column packed with 15% methanol in chloroform.

The same procedure was repeated with isonicotinic acid as the raw material to get the compound INANO were subjected to the analysis by FTIR spectroscopy using the instrument, NICOLET iS5 model, Thermo scientific instrument, USA. UV-Vis absorption spectra, a computer controlled JascoV-500 model spectrophotometer was used. The Bruker 500 MHz FT-NMR spectrometer was used to record the NMR spectra.

2.2 Weight loss studies

2.2.1 Preparation of specimens

Mild steel specimens were cut into rectangular (2x5cm²) strips with typical dimensions of 1.0 cm² area. The chemical composition of mild steel used was Cu-0.14%, Mn-0.35%, Si-0.17%, S-0.025, P-0.03%, and the remainder iron¹⁷. These specimens were polished with different grade emery papers, washed with acetone and distilled water, dried and weighed to get constant weight.

2.2.2 Weight Loss Measurements

The weighed mild steel specimens were suspended by means of glass hooks in beakers containing 2N sulphuric acid with various concentrations of inhibitors. After the specified periods of immersion (1, 2, 6 and 24 hours) the specimens were taken

out, washed in running water, dried and weighed. The inhibition efficiency and the corrosion rate were calculated from the observed weight losses using the following relationship^{18, 19}.

Corrosion inhibition efficiency (IE) was then calculated using the equation.

$$\% \text{ IE} = 100[1 - W_2/W_1] \%$$

Where W_1 =weight loss in the absence of the inhibitor and W_2 = weight loss in the presence of the inhibitor.

Corrosion rate

$$= \frac{\text{Loss in weight(mg)}}{\text{Surface area of the specimen (dm}^2\text{)} \times \text{Period of immersion(days)}}$$

2.3 Polarization studies

Polarization studies for the mild steel specimens were carried out with a computer controlled CHI electrochemical workstation, Model 650C, USA and a three electrode cell assembly was used. The working electrode was mild steel specimen of 1.0 cm² area. A saturated calomel electrode (SCE) was used as the reference electrode and platinum strip was used as the counter electrode for the polarization study. The corrosion parameters of the working electrode (mild steel) such as corrosion potential (E_{corr}), corrosion current (I_{corr}) and Tafel slopes (b_a and b_c) were calculated, for the cases of electrolytes with optimal concentrations of the inhibitors.

2.4 AC impedance measurements

CHI electrochemical workstation, Model 650C, USA, was used to record AC impedance measurements. Here also a three electrode cell assembly was used. The working electrode was a rectangular specimen of mild steel with one face of the electrode of constant 1.0 cm² area exposed. A rectangular foil of platinum was used as the counter electrode. The three electrode assembly was placed in a glass cell provided with provisions for enclosing the electrolyte. The corroding medium (2N H₂SO₄) was filled in the glass cell with or without inhibitors (at their optimal concentration). A time interval of 5 to 10 min was given for the system to acquire the open circuit potential. Then a steady state potential of 10 mv was superimposed as the electrode under test. The AC frequency was varied from 0.1 to 10⁵ HZ same retention was allowed for the electrode to attain its equilibrium potential (open circuit potential). The potentials were then scanned at a particular sweep as either direction anodic and cathodic to its equilibrium potential. The results were plotted as strip-chart recorder of polarization unit, and of the cell impedance were measure in ohm for various

frequencies. The R_t (charge transfer resistance) and C_{dl} (double layer capacitance) values were calculated. The double layer capacitance (C_{dl}) were calculated using the following relationship²⁰.

$$C_{dl} = \frac{1}{2 \times 3.14 \times f_{max}}$$

2.5 Scanning Electron Microscopy

The surface morphology of mild steel specimen in the absence and presence of inhibitor species were carried out using a digital scanning electron microscope model Hitachi, Japan Model SN 3000. Make with an accelerating voltage of 5 KV between the scan speeds and micrographs were taken at the magnitudes ranging from 350 X to 2000 X.

3. RESULTS AND DISCUSSION

3.1 Spectral studies

UV-VIS absorption spectrum of prepared organic compound of nicotinic acid N-oxide was studied in the range from 800 nm to 200 nm. The UV-VIS spectrum exhibits two bands around 272 nm and 355nm as shown in Fig. 1a. The first peak about at 272 nm is attributed to the π - π^* transition for the benzene ring, the band at 355 nm represents for π^* -n transition due to the compound. Isonicotinic acid N-oxide also shows two peaks but broader than nicotinic acid N-oxide as presented in figure 1b. Band appearing around at 260 nm is attributed for π - π^* transition of the benzen ring and 350 nm for π^* -n transition in to the compound

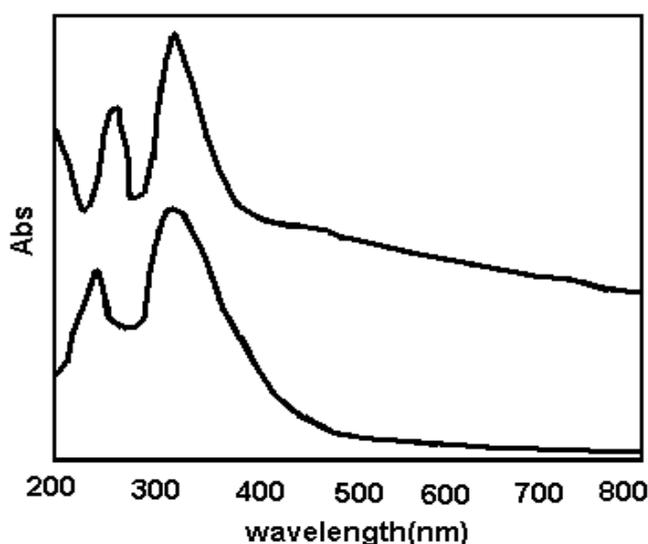


Fig. 1. UV-VIS spectrum of (a) Nicotinic acid N-oxide (b) Isonicotinic acid N-oxide

The FTIR Spectra of the synthesised inhibitors NANO and INANO are shown in figure 2. In the spectrum absorption at 1800-800 cm^{-1} region NANO and INANO exhibits characteristic $\nu(\text{C}=\text{O})$ (1720-1708 cm^{-1}), $\nu(\text{C}-\text{O})$ (1439 cm^{-1}), $\nu(\text{N}-\text{O})$ (1290 cm^{-1}) and $\nu(\text{N}-\text{O})$ (867-852 cm^{-1}) absorptions. In this study significant absorption bands in the infrared spectrum are observed at 3450 cm^{-1} , 1705, 1437 cm^{-1} , 1287 cm^{-1} and 868-851 cm^{-1} and assigned $\nu(\text{OH})$, $\nu(\text{C}=\text{O})$, $\nu(\text{C}-\text{O})$, $\nu(\text{N}-\text{O})$ and $\nu(\text{N}-\text{O})$, respectively. The presence of characteristic IR absorption at 3400 cm^{-1} , 3000 cm^{-1} respectively confirm the heterocyclic six-member ring with hetero atom and the carboxylic acid substituent at the meta and para (3,4) position ($\approx 1200 \text{ cm}^{-1}$) with respect to the hetero atom of nitrogen. It is evident that the $\text{N} \rightarrow \text{O}$ group and the aromatic ring exhibit characteristic peaks at 1150 cm^{-1} and 3000 cm^{-1} respectively. It is to be noted that the $\text{N} \rightarrow \text{O}$ Coordination is more ionised in the INANO isomer compared to the NANO isomer.

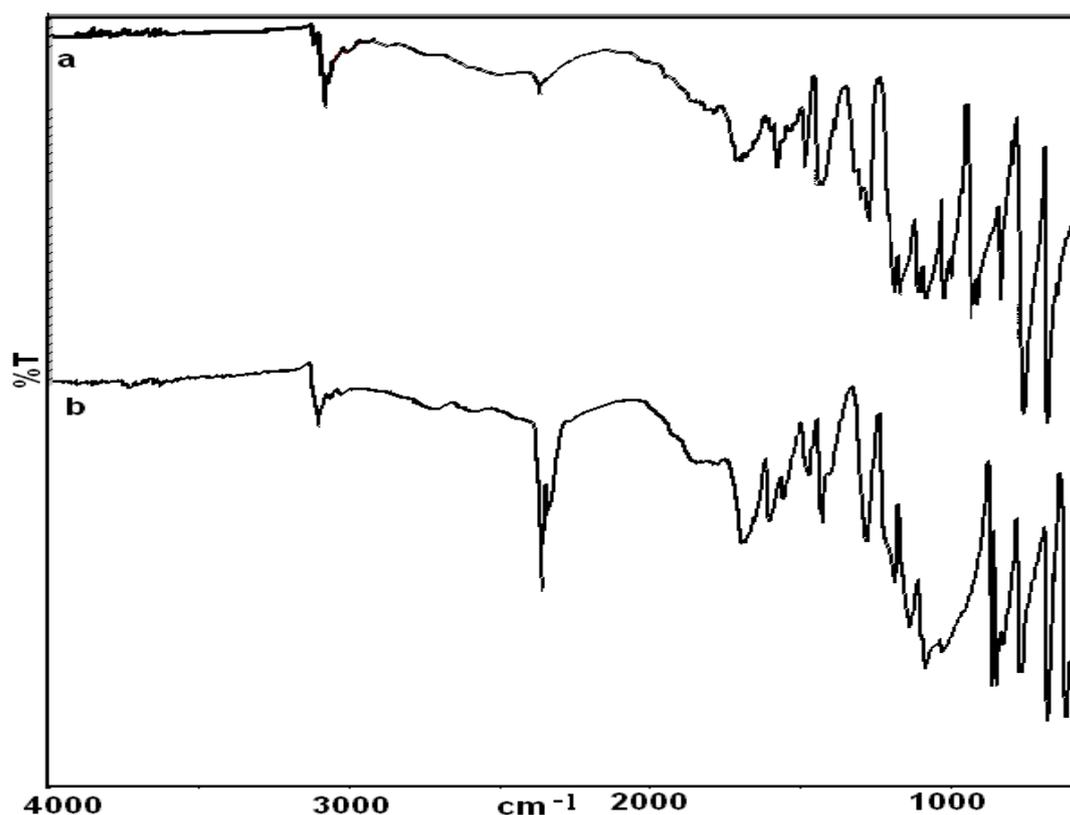
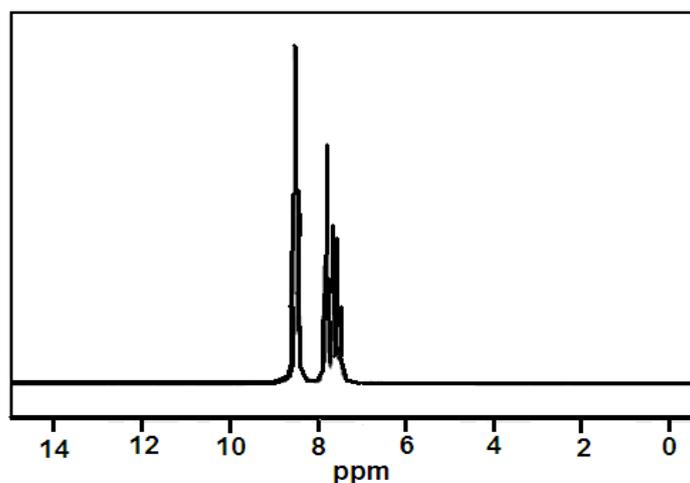
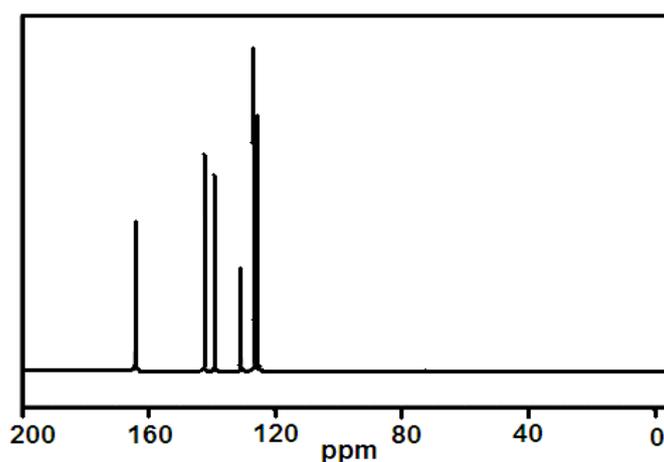


Fig .2. FTIR Spectra of (a) NANO (b) INANO

Fig. 3. ^1H NMR for Nicotinic acid N-oxideFig. 4. ^{13}C NMR for Nicotinic acid N-oxide

^1H and ^{13}C NMR spectra of nicotinic acid N-oxide are shown in figure .3 and 4. The ^1H NMR (200 Hz,) isotropic chemical shifts (δ) are observed at 7.5-7.7 (m); 8.4-8.5(m); molecule has six different carbon atoms, therefore six chemical shifts are observed in ^{13}C NMR spectrum. Commonly the fact that the range of ^{13}C NMR chemical shifts for a typical organic molecule usually are higher than 100 ppm the accuracy ensures reliable of spectroscopic parameters. ^{13}C NMR chemical shifts in the ring for the compound are presented at 125.3, 126.5, 130.99, 138.7, 142.3 and 164.6 ppm. The results of NMR are good agreement for formation of compounds. Isonicotinic acid N-oxide also exhibits three ^1H NMR signals at 7.8, 8.3, 13.7 ppm. The ^{13}C NMR of same compound shows three peaks at 126, 139, 165 ppm.

3.2 Weight loss measurements

Figure 5&6 shows the inhibition efficiencies of the inhibitors under the various concentrations, at different times of immersion in 2N H₂SO₄. Among the two inhibitors (NANO & INANO) INANO performs better than NANO as, corrosion inhibitor for mild steel in 2N H₂SO₄.

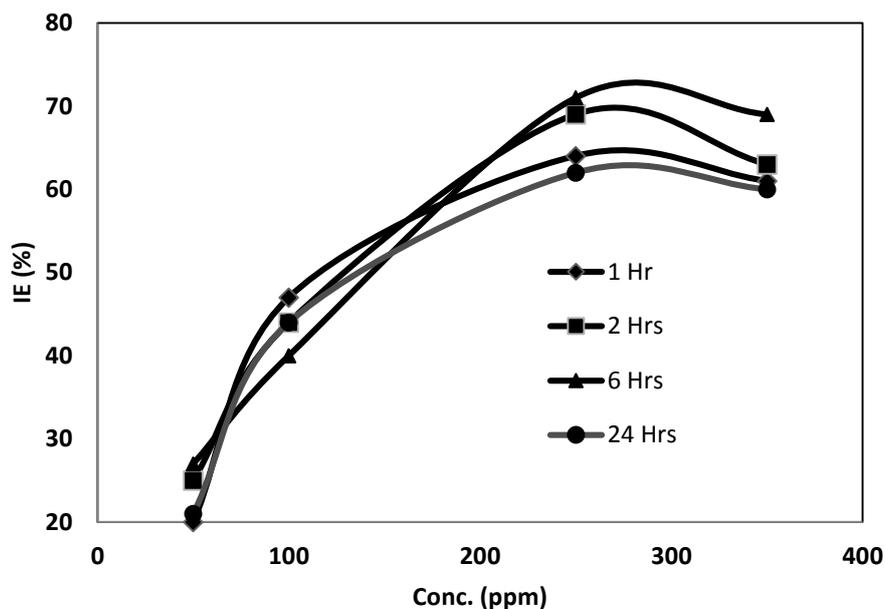


Fig. 5. Inhibitor efficiency of NANO at different time interval in 2N H₂SO₄

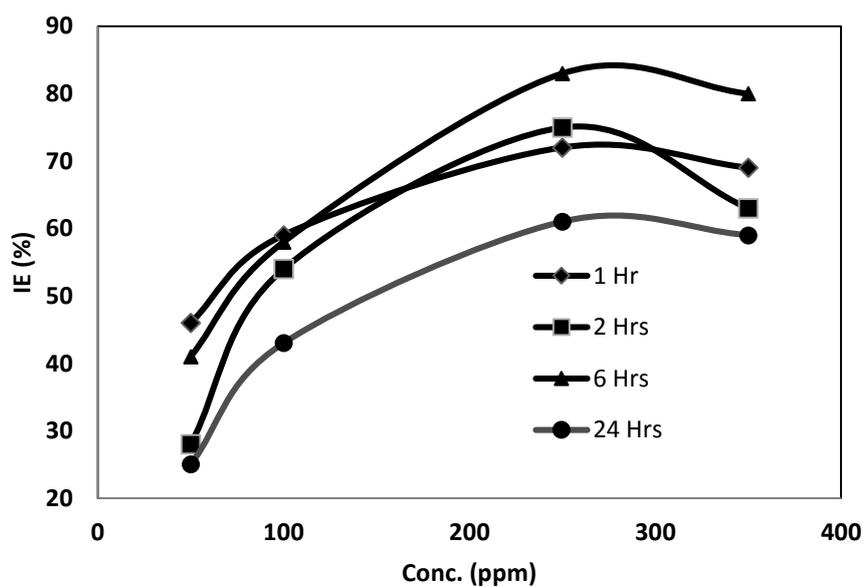


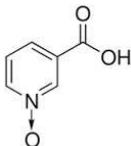
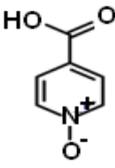
Fig. 6. Inhibitor efficiency of INANO at different time interval in 2N H₂SO₄

The better performance of INANO compared to NANO as corrosion inhibitors may be attributed to the better interactive forces between the Fe^{2+} ions formed initially due to the slight onset of corrosion of mild steel and O^- of the INANO compound, compared to the coordinated oxygen of the NANO Isomer. These observations are supported by the increasing corrosion rates of the specimen with the NANO Isomer, compared to those with the INANO Isomer. As a concentration increases the inhibiting efficiency also was increased upto 250 ppm at all time intervals but further increasing the concentration of inhibitors the efficiency was slightly decreased in both inhibitors. Since the higher concentration of inhibitors also may be act as corrosive medium. The corrosion rate of both inhibitors also shows good in 2N sulphuric acid with 250 ppm.

3.3 Polarization studies

The DC polarization curves of mild steel in 2N sulphuric acid without and with NANO and INANO in concentration of 250 ppm presented in Figure 7. This figure shows that NANO and INANO shift corrosion potential toward more negative potentials. The cathodic current versus potential gives rise to Tafel lines indicating that the hydrogen evolution reaction is activation controlled the results are given in table 1. It is evident from the figure 7 that the corrosion potential is not influenced much but the corrosion current is reduced in the presence of inhibitors. It indicates that the rate of corrosion is reduced in the presence of inhibitors, proportional to their concentrations.

Table 1 Physical properties of inhibitors

Name of the inhibitor	Molecular formula	Structure	Molecular weight
Nicotinic Acid N-Oxide (NANO)	$\text{C}_6\text{H}_5\text{NO}_3$		139
Isonicotinic Acid N-Oxide (INANO)	$\text{C}_6\text{H}_5\text{NO}_3$		139

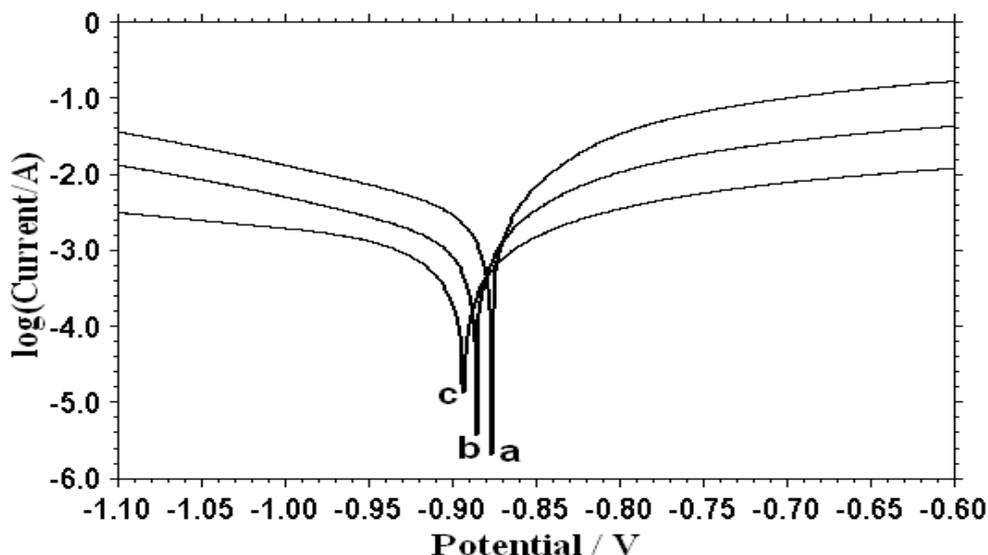


Fig.7. Polarization Curves of mild steel immersed in 2N H₂SO₄ + Inhibitors

a) NANO b) INANO

Thus the inhibitors function by adsorption on to the metal surface. The extent of adsorption is improved due to electrostatic forces established between the Fe²⁺ ions formed on the mild steel surface, due to the initial, slight but unavoidable corrosion of the mild steel specimen, whereas the adsorptive forces are stabilised between the lone pair of electrons of Oxygen atom of NANO Isomer (Nitrogen coordinated to Oxygen and hence no ion formation) and the metal atom of the mild steel surface.

Thus the film formation by the NANO inhibitor is supported by the increased Linear polarization resistance compared to that with the blank corrodent medium and hence a reduced corrosion current with the inhibited corroding medium. A linear region with apparent Tafel was observed. The cathodic reaction was activation-controlled and the addition of the compounds tested decreased the current densities in large cathodic domains of potential. This result indicated that the compounds studied acted as cathodic type inhibitors. Generally, the addition of inhibitors in solution does change corrosion potential significantly because they inhibit cathodic reactions. The large changes in potentials can be a result of the competition of the cathodic inhibiting reactions, and of the metal surface condition.

3.4 Electrochemical impedance studies

The charge-transfer resistance (R_t) values are calculated from the difference in impedance at low and high frequencies. The double layer capacitance (C_{dl}) was obtained, at the frequency at which the imaginary component of the impedance is maximum ($-Z_{im.}$), using the equation

$$C_{dl} = \frac{1}{2\pi f_m \times R_t}$$

The impedance diagrams in figure 8, corresponds to the blank, shows perfect semi-circle indicating a charge transfer process mainly controlling the corrosion of steel. The addition of tetrazolic compounds enhances the value of the transfer resistance in acidic solution. EIS study confirms that the tested compounds are efficient inhibitors.

The higher values of charge transfer resistance (R_{ct}) and lower value of double layer capacitance (C_{dl}) observed with the inhibited corroding medium preferably the INANO isomer is attributed to the film formation by the inhibitor- INANO at the metal surface leading to the inhibition of corrosion the values are presented in table 2.

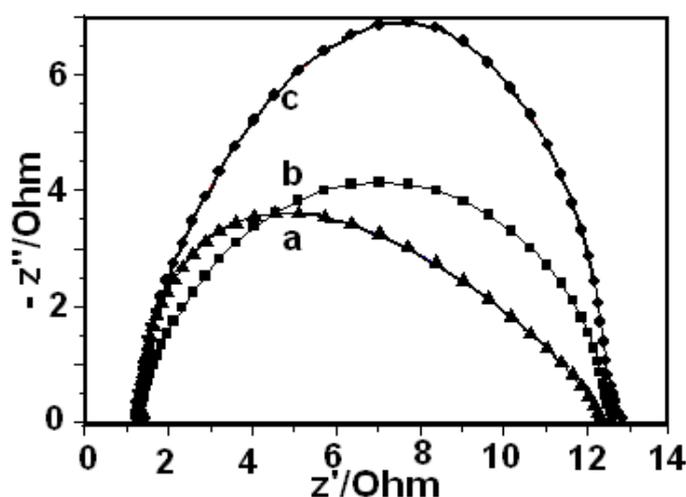


Fig.8. Nyquist plots for mild steel immersed in (a) 2N H₂SO₄ b) 2N H₂SO₄ + NANO c) 2N H₂SO₄ + INANO

Table 2: Potentiodynamic polarization data on mild steel

Medium (Blank/Inhibitor)	E _{corr} V vs SCE	b _a mV/dec	b _c mV/dec	LPR Ohm	I _{corr} mA	Efficiency %	Rate Mil/year
Blank(2N H ₂ SO ₄)	-0.8774	5.627	3.364	24	2.038	--	170.8
Nicotinic acid N-oxide	-0.8866	6.465	5.225	15	2.516	77.15	49.2
Isonicotinic acid N-oxide	-0.8946	5.502	2.841	33	1.577	61.67	57.1

3.5 Adsorption isotherm studies

Adsorption isotherms are often shown to demonstrate the performance of organic adsorbent type inhibitors and important in determining the mechanisms of organic electrochemical reactions. The most frequently used adsorption isotherms are Langmuir, Temkin and Frumkin isotherms which describes relation between surface coverage(θ)and bulk concentration. The values of θ were calculated by dividing the percentage inhibition efficiency value obtained from weight loss measurements by 100 ppm. Among the three isotherms assessed Langmuir isotherm is found to provide best (Table 3) description of the adsorption behaviour of the investigated NANO and INANO as evident from straight line plot ²¹ obtained when C/θ is plotted against C as shown in figure 9&10.

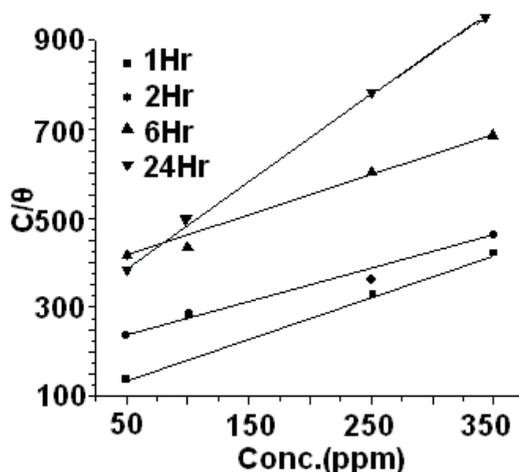


Fig.9. Langmuir adsorption isotherm of NANO on mild steel in 2N H₂SO₄.

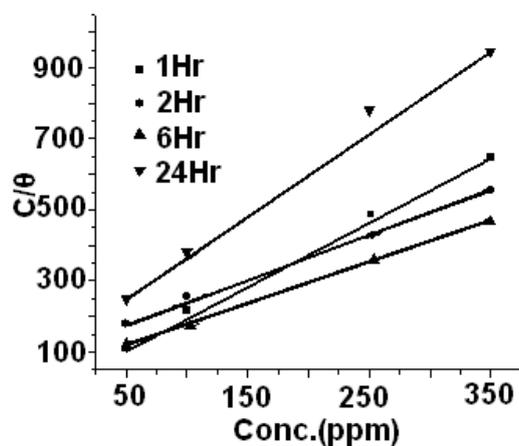


Fig.10. Langmuir adsorption isotherm of INANO on mild steel in 2N H₂SO₄.

Table 3: Electrochemical impedance spectral data on mild steel

System(Blank/Inhibitor)	R _{ct} (Ω)	C _{dl} (Farad)	ImpedanceΩ
Blank(2N H ₂ SO ₄)	30	0.03185	0.68
Nicotinic acid N- oxide	18.75	0.0279	1.38
Isonicotinic acid N- oxide	36.25	0.0306	1.24

A form of Langmuir adsorption isotherm can be expressed as

$$C_{(inh)} / \theta = 1 / K_{(ads)} + C_{(inh)}$$

where, C_{inh} is inhibitor concentration and K_{ads} is an equilibrium constant for adsorption-desorption equilibrium process.

The standard free energy of adsorption of inhibitor (ΔG°_{ads}) on mild steel surface can be evaluated using the equation.

$$\Delta G^{\circ}_{ads} = - RT \ln (55.5x K)$$

3.6 Scanning Electron Microscopy (SEM)

The surface morphology of stainless steel studied by scanning electron microscopy (SEM), surface was observed after 12 hrs of immersion in 2N H₂SO₄ with 250 ppm of inhibitors at room temperature before and after addition of inhibitor corrosion. Fig 11a shows the polished surface of stainless steel after exposed to the testing environment, it was observed as a uneven surface along with the presence of dark spots. From the figures 11(b) and 11(c), it is evident that the specimen surface is most damaged in the absence of the inhibitor and reasonably less damaged and even appears to be more polished in the presence of inhibitors. It reveals that an absorptive film of inhibitor is formed on the mild steel surface, which suppresses further corrosion and hence serves the inhibition function.

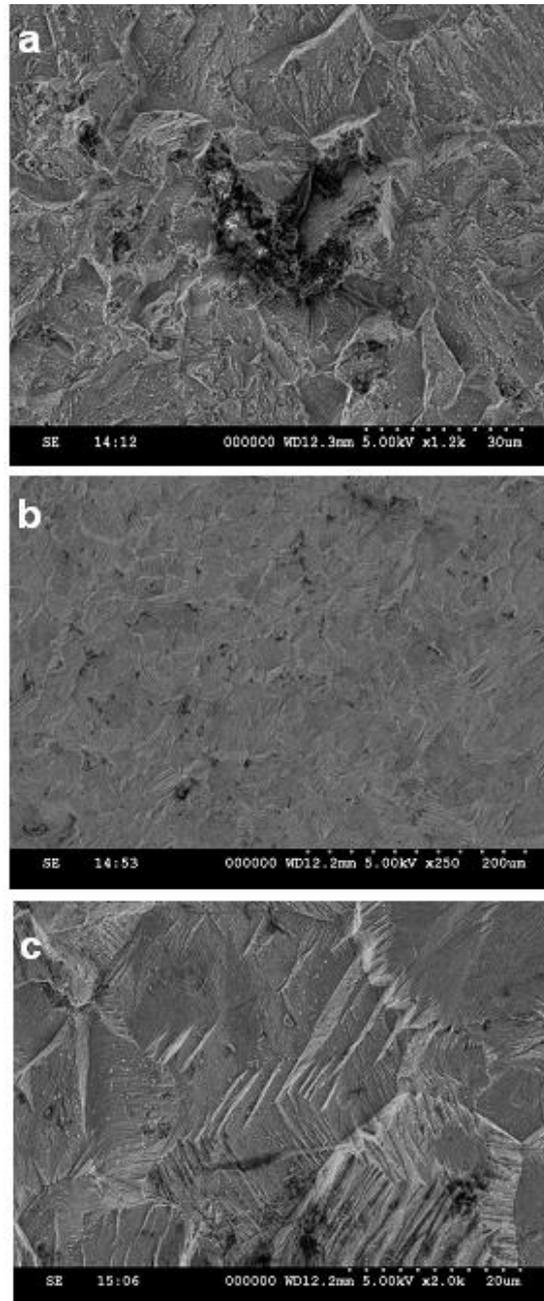


Fig.11. SEM image of mild steel specimen after immersed in 2N H₂SO₄ (a) without inhibitor (b) inhibitor NANO (c) inhibitor INANO

3.7 AFM studies

Fig. 12 appearance of two, three-dimensional AFM image and particle distribution graphs of mild steel surface immersed in 2N H₂SO₄ solution without and with addition of NANO and INANO for 12 h. From figure it can be seen that the corrosion pattern of mild steel in uninhibited 2N H₂SO₄ solution appears ununiform structure.

These data suggest that carbon steel surface immersed in acid has a greater surface roughness than the polished metal surface, which shows that the unprotected carbon steel surface is rougher and is due to the corrosion of the carbon steel in acid environment. By contrast, the corrosion morphology of mild steel in 2N H_2SO_4 solution containing the inhibitor was very smooth. Which may be adsorption of inhibitors on steel surface and the formation protective film, thus effectively protects mild steel from corrosion. The inhibitors were demonstrated good smooth surface and the roughness parameters of Sa, Sq, Sy, Sp, Sv and Sm values are presented in table 4.

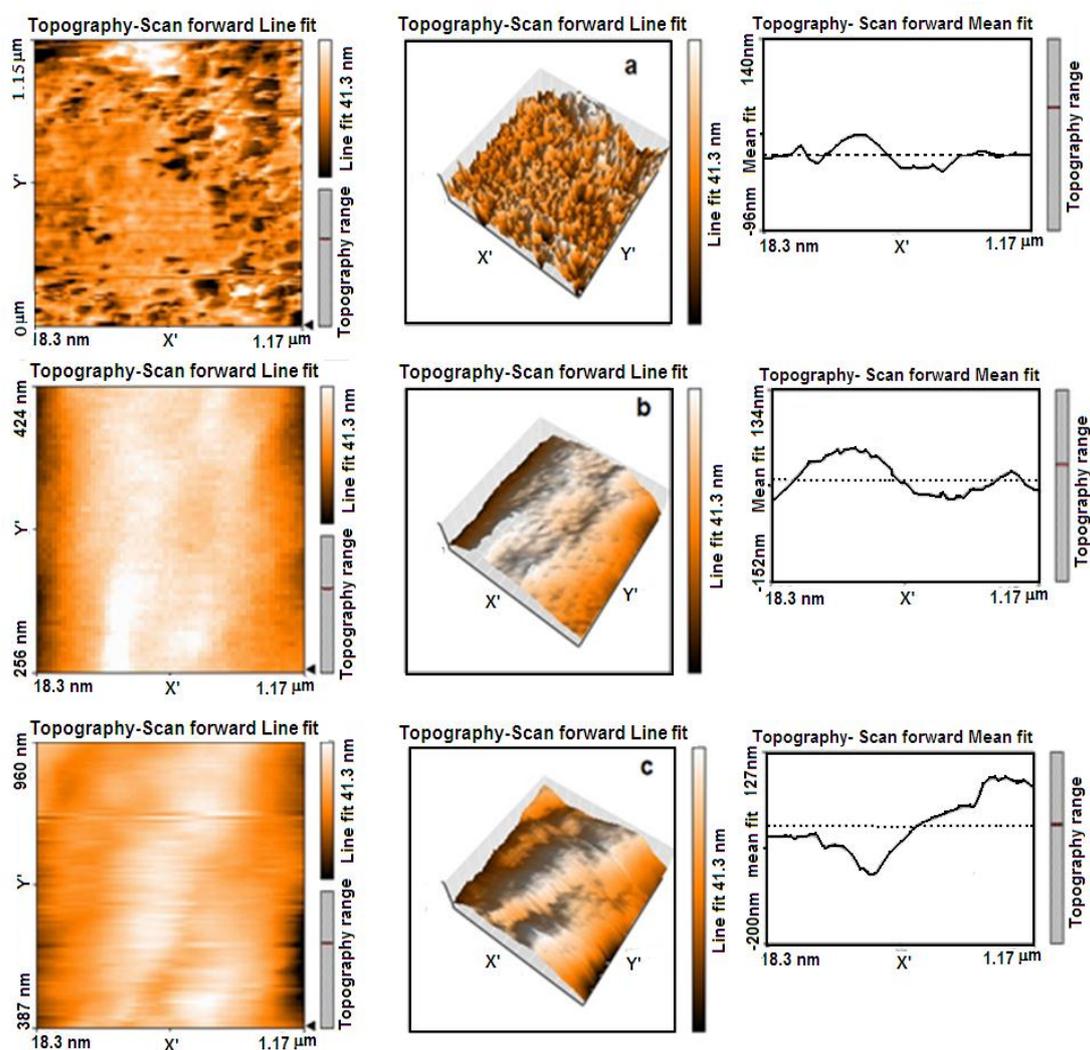


Fig.12. AFM image of mild steel specimen after immersed in 2N H_2SO_4 (a) without inhibitor (b) inhibitor NANO (c) inhibitor INANO

Table 4: Data of Langmuir parameters at 303 K temperature on different time duration

Inhibitors	Slope	R ₂	log K	- ΔG _{ads} (kJ/mol)
Nicotinic acid N- oxide	0.916	0.985	0.09831	11.732
	0.838	0.976	0.037297	10.281
	0.731	0.985	0.24708	11.321
	0.994	0.979	0.022452	10.238
Isonicotinic acid N- oxide	0.838	0.982	0.16244	10.885
	0.857	0.989	0.020997	10.236
	0.950	0.998	0.135839	10.845
	0.861	0.997	0.101033	10.648

Table 5: Roughness parameters data of without inhibitor and with inhibitor

Roughness parameters	with out inhibitor	NANO	INANO
Area (pm) ²	1.341	1.341	1.341
Average (Sa) nm	6.1486	4.854	3.564
Root mean square(Sq) nm	8.0813	6.241	5.922
Peak valley height (Sy)nm	83.747	33.26	28.533
Peak height (Sp)nm	39.062	27.231	23.551
Valley depth (Sv) nm	-44.685	-12.03	10.982
Mean (Sm) fm	-20.5	-11.471	-09.429

4. CONCLUSIONS

1. The synthesised compounds - NANO and INANO function better towards the inhibition of corrosion of mild steel in 2N H₂SO₄.
2. The Inhibition efficiency of the substances is examined through weight loss method. As increases the concentration of inhibitors upto 250 ppm shows higher efficiency in 2N sulphuric acid.
3. Among the two isomeric nitrones tested, INANO performs better as the corrosion inhibitor than the NANO which may be attributed to the better adsorption by the ionic form of the nitrone (N⁺ - O⁻) to the Fe²⁺ ions formed initially on the mild steel surface due to the slight onset of corrosion.
4. Both compounds act as cathodic inhibitors. This is proved from polarization studies.
5. Electrochemical impedance spectroscopy also confirm good inhibiting efficiency for INANO

6. Surface morphology also shows good aggregation for anticorrosion behaviour of steel on inhibitors in 2N sulphuric acid medium.

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