



Inhibition Efficiency of Water Soluble Sr(ZnZr)₁Fe₁₀O₁₉-PANI Composite against Strong Acidic Condition for Mild Steel

S. SIVAKUMAR, P. KAMATCHI SELVARAJ* and S. SELVARAJ

P.G. and Research Department of Chemistry, Government Arts College for Men (AUT), Nandanam, Chennai-600 035, India

*Corresponding author: E-mail: porbal96@gmail.com

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Polymerization of aniline along with nanoparticle size of strontium zinc zirconium ferrite (mixed metal oxide) powder using ammonium persulfate as an oxidizing agent at 0 °C gives water soluble strontium zinc zirconium hexaferrite-polyaniline composite. Dodecylbenzene sulfonic acid was used as dopant and surfactant for the synthesis. Weight loss measurements, Tafel plot analysis and AC impedance measurement revealed that the synthesized material can act as good corrosion inhibitor in half molar and one molar acidic conditions at room temperature. Continuous measurement of open-circuit potential (OCP) also reflect the inhibition efficiency. Prepared composite acts as an effective pickling/corrosion inhibitor.

Keywords: Mild steel, Corrosion, Potentiodynamic polarization, Electrochemical impedance spectroscopy.

INTRODUCTION

Protection of metal from corrosion under lower pH condition during the operation such as pickling, cleaning, descaling, *etc.* is effected by the usage of inhibitors [1-3]. Addition of inhibitors reduces the loss of dissolution of metals, due to aggressive acidic environment adopted in the maintenance processes. The flow of corrosion current is minimized at the interface due to the suitable environment created upon the addition of inhibitors [4,5]. Inhibitors act as a barrier between the metal surface and its environments. The additional burden posed on industries like chemical plants, steel plants, sugar mills, power plants, oil and water pipe lines maintaining units are reduced considerably by safe guarding the machinery parts made of metals from corrosion [6,7]. Reduction in the oxidation rate of anode or reduction rate of cathode or both are reported by the usage of inhibitors [8]. Most of the potential corrosion inhibitors possess an active functional group [9] such as =NH, -N=N-, -CHO, R-OH, -NO₂, *etc.* Presence of oxygen, sulphur and especially nitrogen as heteroatom in the carbon chain reduces corrosion attack [10-12]. Conducting nature of polymers offer better corrosion inhibition efficiencies than simple organic compounds [13-16]. Some important conducting polymers like, polypyrrole, polythiophene and polyaniline are used to prevent corrosion [17-19].

Synthesis, purification and applications of polyaniline (PANI) as corrosion inhibitor has been studied extensively [20] due to its stability and ability to form protective layer. Presence of quaternary nitrogen atom, π -electron and bigger molecular size of polyaniline and its derivatives provide inhibition property [21].

The present work deals with the preparation of water soluble Sr(ZnZr)₁Fe₁₀O₁₉-PANI composite by chemical oxidative polymerization of aniline on mixed metal oxide and evaluation of its inhibition efficiency using weight loss method, open circuit potential (OCP) measurements, potentiodynamic polarization method and ESI measurements.

EXPERIMENTAL

Analytical grade HCl, Fe(NO₃)₃·9H₂O, Sr(NO₃)₂, Zn(NO₃)₂·4H₂O, Zr(NO₃)₄, glycine, ammonium persulfate (APS) and sodium salt of dodecylbenzene sulphonic acid were purchased from E. Merck (India) and used without further purification. Aniline (AR) purchased from E. Merck (India) was further purified by distillation with Zn dust before usage.

FTIR spectra of samples were recorded on a Perkin-Elmer Infrared Model 337 spectrometer in the frequency range of 4000-450 cm⁻¹ using KBr pellets. X-Ray diffraction studies were performed using Rigaku Maniflex X-ray diffractometer (Japan).

Morphology of samples was characterized by a JSM-6390 scanning electron microscope (SEM). Weight loss method (gravimetric) was used for the study of corrosion inhibition efficiency. Electrochemical impedance and potentiodynamic polarization studies were carried out using ECLAB 10.37 model software and open circuit potential (OCP) measurements were recorded using CHI Analyzer instrument 1200B model.

Synthesis of $\text{Sr}(\text{ZnZr})_1\text{Fe}_{10}\text{O}_{19}$ -PANI composite: The composite was prepared by adopting the reported method after little modification [22]. In the first step, solution (I) containing required amount of nitrate salts of Sr, Zn, Zr and Fe in 100 mL of double-distilled water was added to aqueous solution (II) containing desired amount of glycine in double distilled water. The mixture was heated in an oil bath at 110-120 °C for 4 h and evaporated to get a high viscous wet-gel. It was then ignited in air to obtain deep grey voluminous burnt ash powder. The powder was annealed at 1000 °C for 2 h to get mixed metal oxide. In the second step, mixed metal oxide powder was dispersed in double-distilled water by ultrasonic waves with oscillation frequency of 42 KHz for 40 min before polymerization. Desired amount of sodium salt of dodecylbenzene sulphonic acid was added to mixed metal oxide dispersion. Aqueous solution of aniline in HCl (1:3) was added dropwise to $\text{Sr}(\text{ZnZr})_1\text{Fe}_{10}\text{O}_{19}$ solution for a period of 1 h along with aqueous solution (100 mL) containing oxidative agent such as ammonium persulphate (APS) under constant stirring. The reaction mixture was kept under stirring (3 h) for complete polymerization. The solid particles formed were washed with double distilled water and acetone for several times sequentially. The composite was dried in vacuum oven at 60 °C for 24 h and then used for analysis.

RESULTS AND DISCUSSION

FTIR analysis: The FTIR spectra of mixed metal oxide, PANI and composite are given in (Fig. 1a-c). The more intense peaks at 600-500 cm^{-1} are corresponds to vibration of the octahedral and tetrahedral sites of $\text{Sr}(\text{ZnZr})_1\text{Fe}_{10}\text{O}_{19}$ (Fig. 1a). The characteristic peaks of PANI (Fig. 1b) occurred at various frequencies matches with the earlier report [22,23]. It is also observed that there is shift in the absorption bands of the composite (Fig. 1c) when compared to PANI. All the above observations indicate the presence of intermolecular interactions between mixed metal oxide and PANI.

XRD analysis: X-ray diffraction pattern of metal oxide (Fig. 2a) indicates a sharp intense peak at $2\theta = 21^\circ$ which is due to appearance of mixed ferrite ($\text{Sr}(\text{ZnZr})_1\text{Fe}_{10}\text{O}_{19}$). The XRD of PANI shows a broad peak centered between $2\theta = 20^\circ$ to 30° (Fig. 2b). This is in agreement with the earlier report [23]. The shift in 2θ value of mixed metal oxide from 21° to 25.50° and 2θ value of PANI from 20° - 30° to 30.37° indicates the formation of the composite [22,24].

SEM analysis: It is evidenced from the comparison of SEM images of mixed metal oxide (Fig. 3a) and composite (Fig. 3b), the diameter of hexagonal mixed metal oxide powder increases and is distributed in 270-700 nm range. This confirms that the mixed metal oxide are coated with polyaniline and form spherical coral like particles and agglomeration is also observed in the composite. The slight increase in size of composite recorded than the reported value [22] might be due to the changes adopted in preparative method.

Preparation of electrode material: Specimens with composition of C: 0.21 %, Si: 0.035 %, Mn: 0.25 %, P: 0.082 % and

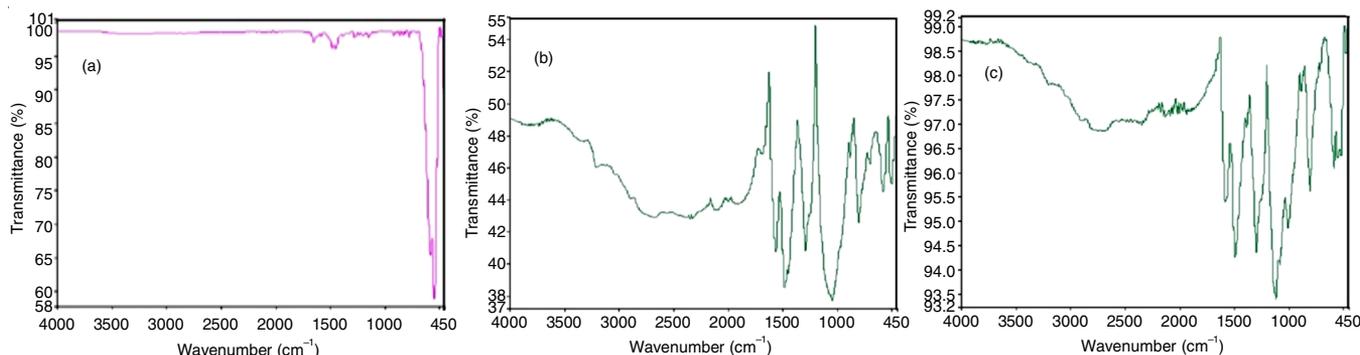


Fig. 1. FTIR spectra of (a) metal oxide (b) PANI and (c) composite

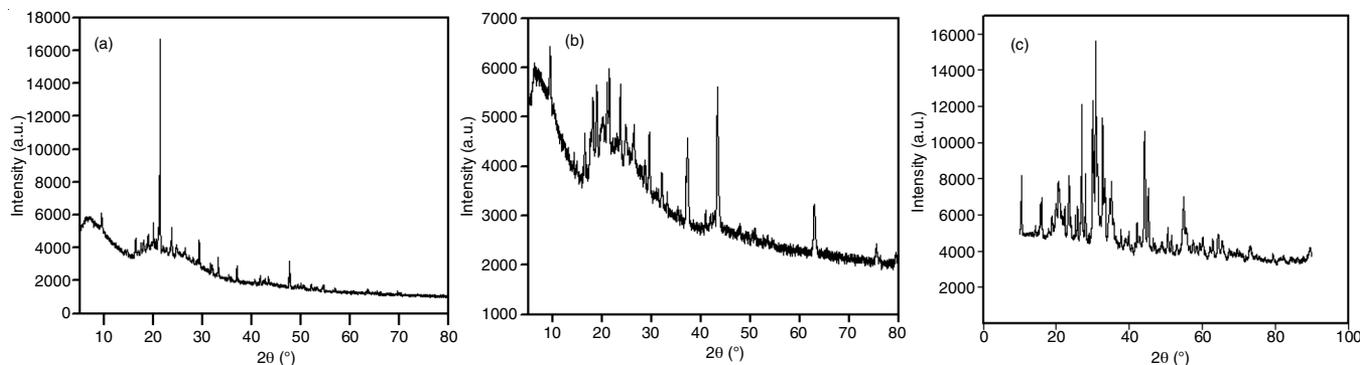


Fig. 2. XRD spectra of (a) metal oxide (b) PANI and (c) composite

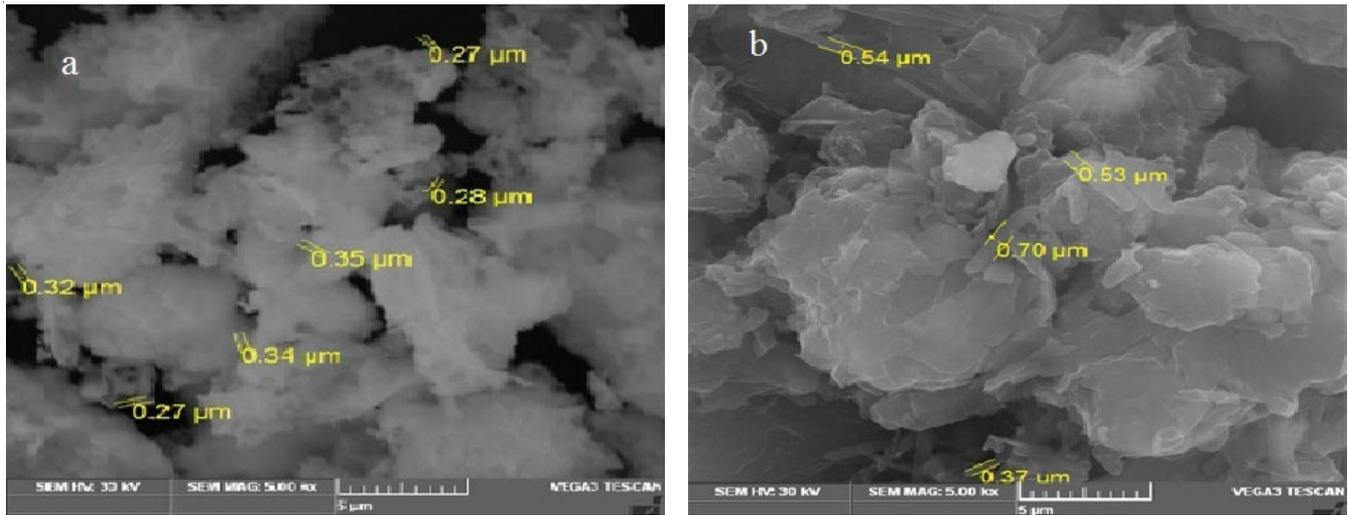


Fig. 3. SEM of (a) metal oxide (b) composite

Fe(rest) were mechanically cut into 4 cm × 2 cm × 0.2 cm dimensions and were abraded with different grade of silicon carbide abrasive papers up to 1200 grit. The abraded specimens were washed with double-distilled water, acetone and then dried at room temperature in desiccators. Freshly polished mild steel specimens were used for all test related to inhibition efficiency measurements.

Assessment of inhibition property

Weight loss method: The accessibility and consistency of the weight loss method raise it as the benchmark method of measurement in many corrosion monitoring programmes. Weight loss measurements in blank solutions and in test solutions were conducted under total immersion of mild steel coupons in 250 mL of 0.5 M and 1M H₂SO₄ at room temperature for 8 h. The mild steel coupons were weighed and suspended inside the test solutions. The coupons were retrieved at 2 h interval and washed with bristle brush in double-distilled water, acetone,

dried and reweighed. An average weight loss value of the three experiments were used for the calculation of area of surface coverage and inhibition efficiency using the formula reported earlier [25]. The observed values of percentage efficiency and surface coverage area are presented in the Tables 1 and 2.

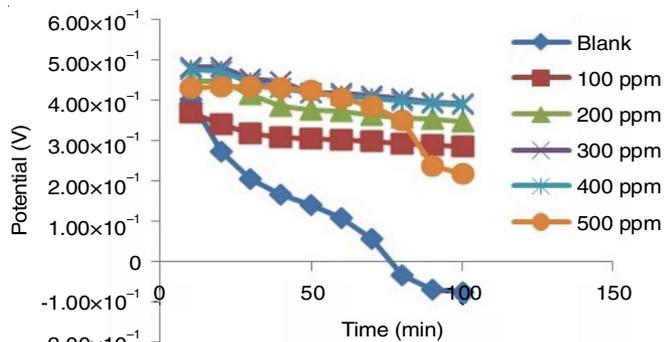
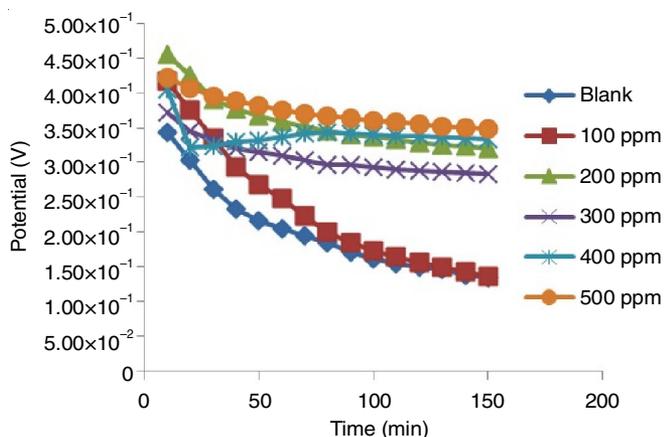
Open circuit potential measurement: The open circuit potential (OCP) measurements were performed using CHI-Electrochemical analyzer. The analyzer contains cell having working electrode, counter electrode, reference electrode made up of mild steel, platinum and calomel electrodes, respectively. The freshly polished mild steel specimens (1 cm²) were exposed to the corrosion environment with and without composites in 0.5 M and 1 M H₂SO₄ at various concentrations from (100 ppm to 500 ppm). The OCP values are measured after 20 min, and recorded continuously for 2 h to see whether the inhibitor is able to withstand the corrosion environment for atleast few hours. The results obtained are given in Figs. 4 and 5. The OCP of the mild steel samples immersed in the solutions containing

TABLE-1
CORROSION PARAMETERS DATA OBTAINED FROM WEIGHT LOSS MEASUREMENTS IN 0.5 M H₂SO₄

Conc. of composite (ppm)	2 h			4 h			6 h			8 h		
	Weight loss (%)	I.E. (%)	θ	Weight loss (%)	I.E. (%)	θ	Weight loss (%)	I.E. (%)	θ	Weight loss (%)	I.E. (%)	θ
Blank	0.0308	–	–	0.0519	–	–	0.0687	–	–	0.0859	–	–
100	0.0108	65	0.649	0.0200	62	0.614	0.0298	57	0.566	0.0404	53	0.529
200	0.0083	73	0.730	0.0143	73	0.728	0.0206	70	0.700	0.0282	67	0.671
300	0.0077	75	0.750	0.0127	76	0.757	0.0198	71	0.711	0.0290	67	0.669
400	0.0070	78	0.772	0.0127	76	0.757	0.0197	71	0.711	0.0230	73	0.732
500	0.0064	80	0.792	0.0112	79	0.784	0.0173	75	0.748	0.0214	75	0.750

TABLE-2
CORROSION PARAMETERS DATA OBTAINED FROM WEIGHT LOSS MEASUREMENTS IN 1 M H₂SO₄

Conc. of composite (ppm)	2 h			4 h			6 h			8 h		
	Weight loss (%)	θ	I.E. (%)	Weight loss (%)	θ	I.E. (%)	Weight loss (%)	θ	I.E. (%)	Weight loss (%)	θ	I.E. (%)
Blank	0.0513	–	–	0.0837	–	–	0.1111	–	–	0.1519	–	–
100	0.0209	0.593	59	0.0372	0.557	56	0.0500	0.549	55	0.0656	0.576	57
200	0.0201	0.608	61	0.0362	0.568	57	0.0487	0.566	57	0.0644	0.578	58
300	0.0168	0.672	67	0.0343	0.590	59	0.0501	0.549	55	0.0600	0.605	60
400	0.0129	0.748	75	0.0287	0.658	66	0.0431	0.612	61	0.0587	0.616	62
500	0.0120	0.766	77	0.0242	0.710	71	0.0341	0.696	70	0.0487	0.679	68

Fig. 4. Plot of open circuit potential of mild steel in 0.5 M H₂SO₄Fig. 5. Plot of open circuit potential of mild steel in 1 M H₂SO₄

composites shift towards more positive values compared to the sample immersed in the blank solution. It can be clearly seen that the OCP value decreases to little extent in presence of inhibitor, the increase of immersion time, whereas for blank solution, sharp decrease in OCP is noticed. It is therefore, understood that the composite protects the mild steel from corrosion by forming passivation layer on the mild steel surface. This is in agreement with the earlier report [26].

Electrochemical measurements: The electrochemical experiments were carried out in conventional three electrode

cell assembled to potentiostat/galvanostat EC-LAB Analyzer 10.37 Model. The mild steel ASTM 415 sample of (1 cm²) area with remaining portion fully covered with araldite epoxy resin was taken as working electrode, platinum foil was used as counter electrode and saturated calomel electrode (SCE) was used as reference electrode. The working electrode was polished with different grade emery sheet, washed with double distilled water, degreased with acetone and kept in the desiccators for dryness. EIS measurements were carried out in 100 kHz-10 mHz frequency range at steady open circuit potential (OCP), with amplitude of 10 mV AC sine wave. The polarization curves were obtained from -200 to +200 mV versus OCP with scan rate of 0.5 mV s⁻¹. The inhibition efficiency was calculated using the equation reported elsewhere [25].

Potentiodynamic polarization measurements: The Tafel polarization of anodic and cathodic behaviour of mild steel specimens in (0.5 and 1 M) H₂SO₄ with the various concentrations from 100 to 500 ppm of composite are shown (Figs. 6 and 7) and their respective electrochemical parameters, such as corrosion current density (I_{corr}), corrosion potential (E_{corr}), cathodic slope (b_c), anodic slope (b_a) and inhibition efficiency (IE) are given in Tables 3 and 4, respectively.

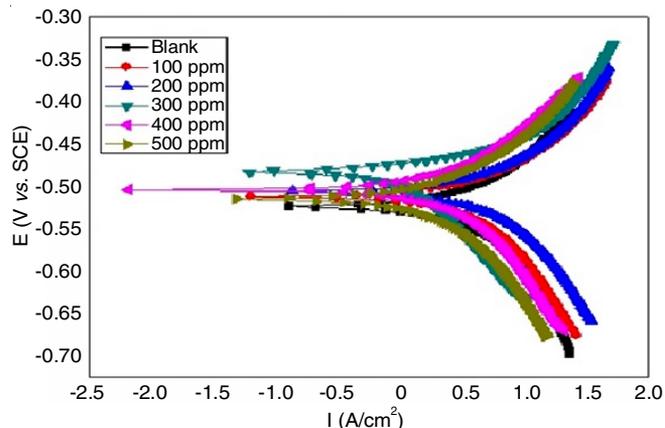
Fig. 6. Potentiodynamic polarization curve of mild steel in 0.5 M H₂SO₄ in the presence and absence of the different concentration of Sr(ZnZr)₁Fe₁₀O₁₉-PANI

TABLE-3
CORROSION KINETIC PARAMETER OF MILD STEEL IN 0.5 M H₂SO₄ IN PRESENCE OF Sr(ZnZr)₁Fe₁₀O₁₉-PANI

Concentration of the composite (ppm)	-E _{corr} (mV vs. SCE)	b _a (mV dec ⁻¹)	b _c (mV dec ⁻¹)	I _{corr} (μA cm ⁻²)	Inhibition efficiency (%)	Surface coverage (θ)
Blank	524	111	138	1454	–	–
100	510	41	71	708	51	0.5130
200	506	31	32	501	65	0.6554
300	485	31	41	221	85	0.8480
400	503	22	22	166	89	0.8868
500	515	21	20	144	90	0.9009

TABLE-4
CORROSION KINETIC PARAMETER OF MILD STEEL IN 1 M H₂SO₄ IN PRESENCE OF Sr(ZnZr)₁Fe₁₀O₁₉-PANI

Concentration of composite (ppm)	-E _{corr} (mV vs. SCE)	b _a (mV dec ⁻¹)	b _c (mV dec ⁻¹)	I _{corr} (μA cm ⁻²)	Inhibition efficiency (%)	Surface coverage (θ)
Blank	455	61	63	1960	--	--
100	500	46	51	889	55	0.5464
200	472	44	49	675	66	0.6556
300	474	41	46	525	73	0.7321
400	487	31	36	505	74	0.7423
500	468	28	34	287	85	0.8535

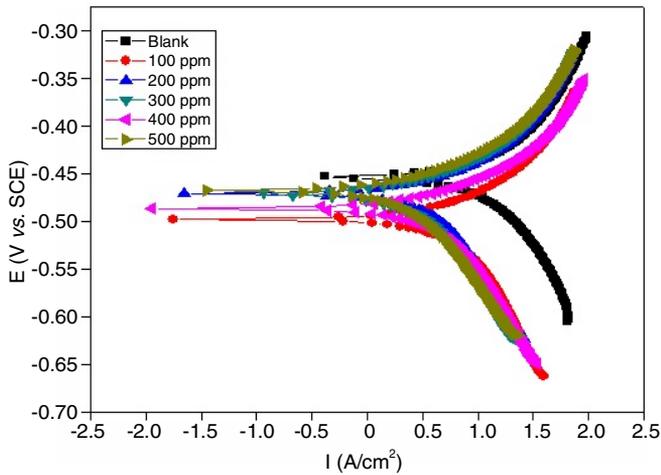


Fig. 7. Potentiodynamic polarization curve of mild steel in 1 M H₂SO₄ in the presence and absence of the different concentration of Sr(ZnZr)₁Fe₁₀O₁₉-PANI

The gradual fall in I_{corr} (Tables 3 and 4) clearly indicates that the composite is acting as an efficient inhibitor in acidic condition and a maximum inhibition efficiency of 90 % has been achieved at 500 ppm inhibitor concentration. Addition of inhibitors does not alter the values of E_{corr} , b_a and b_c . This behaviour is observed for mixed type inhibitor [15]. The results revealed that the composite can act as a highly protective layer on mild steel. Presence of π -electrons in the aromatic ring with quaternary nitrogen atom and bigger molecular size of composite are the reasons for the inhibition action [27].

Electrochemical impedance measurements: For 0.5 and 1 M H₂SO₄ solutions with and without the composite ranging from 100 to 500 ppm, the Nyquist plots obtained after 30 min of immersion time at room temperature are given in Figs. 8 and 9. The decrease in diameter of capacitive loop indicates the decrease in resistance to corrosion [28]. Appearance of single semicircle Nyquist plot reveals the single charge transfer process [29]. Further, the increase in size of capacitive loop with increase in the concentration of inhibitor also confirms the capability

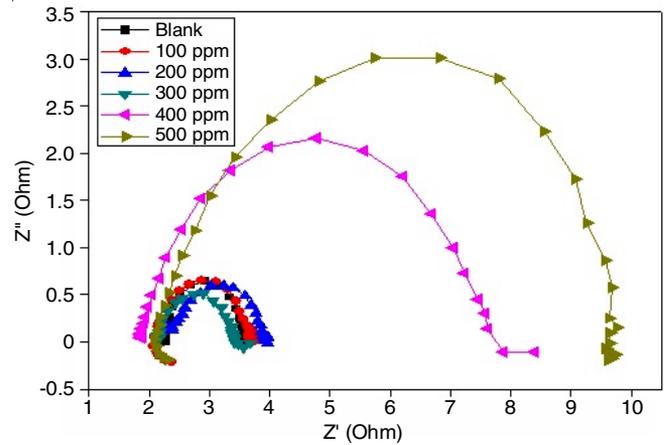


Fig. 8. Nyquist plot obtained for mild steel in 0.5 M H₂SO₄ in the presence and absence of different concentration of Sr(ZnZr)₁Fe₁₀O₁₉-PANI

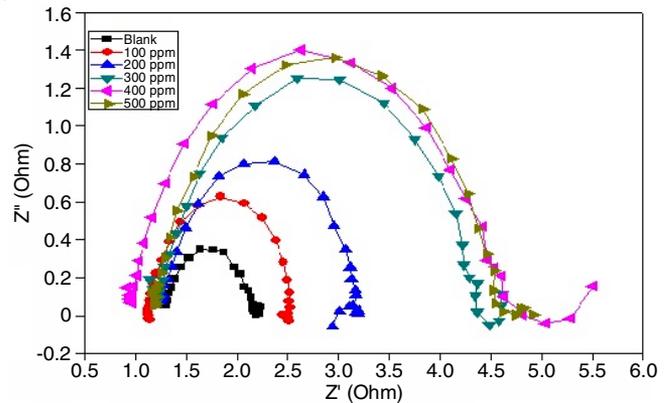


Fig. 9. Nyquist plot obtained for mild steel in 1 M H₂SO₄ in the presence and absence of different concentration of Sr(ZnZr)₁Fe₁₀O₁₉-PANI

of composite to act as an inhibitor. All the parameters values are given in Tables 5 and 6.

In Table-5, the R_{ct} values increase from 1.142 to 7.288 ohm cm² and C_{dl} values decrease from 529 to 198 $\mu\text{F cm}^{-2}$ upon increasing the concentration of composite from 100 to 500 ppm.

TABLE-5
ELECTROCHEMICAL IMPEDANCE PARAMETER FOR MILD STEEL IN 0.5 M H₂SO₄

Concentration of composite (ppm)	R_{ct} ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F cm}^{-2}$)	R_s (Ω)	Inhibition efficiency (%)	Surface coverage (θ)
Blank	1.142	529	2.215	—	—
100	1.959	400	2.241	42	0.4170
200	2.676	176	2.298	57	0.5730
300	4.160	454	2.246	72	0.7254
400	6.243	220	2.438	82	0.8170
500	7.288	198	2.758	84	0.8433

TABLE-6
ELECTROCHEMICAL IMPEDANCE PARAMETER FOR MILD STEEL IN 1 M H₂SO₄

Concentration of composite (ppm)	R_{ct} ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F cm}^{-2}$)	R_s (Ω)	Inhibition efficiency (%)	Surface coverage (θ)
Blank	0.946	555.0	1.306	—	—
100	1.274	556.0	1.155	26	0.2574
200	1.808	320.0	1.262	48	0.4823
300	3.243	349.0	1.251	71	0.7083
400	3.890	436.7	1.037	75	0.7568
500	3.999	414.0	1.269	76	0.7634

Similarly in Table-6, the R_{ct} values increase from 0.946 to 3.999 ohm cm^2 and C_{dl} values decrease from 555.0 to 414.0 $\mu F cm^2$ upon increasing the concentration of composite from 100 to 500 ppm. The formation of an adherent protective film at the metal/solution interface [30] is reflected in the change in R_{ct} values. The thickening of electrical double layer is represented by the decrease in C_{dl} values [31].

Conclusion

The water soluble composite synthesized acts as a good inhibitor for mild steel in acidic medium. The percentage efficiency of the composite obtained from, weight loss, potentiodynamic polarization and EIS measurements are in good agreement. Polarization curves demonstrated that the examined composite behaves like a mixed type of inhibitor. The uniform increase in inhibition efficiency as a function of the concentration, deals with the adsorption phenomenon of the inhibitor on the surface of mild steel and is indicated by the decrease in the double layer capacitance. Thus, the inhibition is due to the adsorption of the inhibitor on the electrode surface and results in blocking the active sites.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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