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Water Soluble CdO-PANI Composite as Corrosion Resistive Delegate in Acidic Abode for Mild Steel

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Abstract

Deployment of ammonium peroxydisulphate as oxidant and sodium salt of dodecylbenzene sulphonic acid as dopant as well as surfactant at ±5 ºC causes oxidative polymerization of aniline. Inclusion of CdO nanoparticles during oxidative polymerization yielded water soluble CdO-PANI composite. Formation of composite material was confirmed by comparing the results obtained in the FTIR, XRD and SEM spectral analysis with the reported values. Weight loss measurements by gravimetric method reveal the resisting adeptness of composite, 89% even after eight hours, against acidic environment. Open circuit potential measurement for 120 minutes exposes that raise in the concentration of composite shift the potential value to positive side. Potentiodynamic polarization and electrochemical impedance spectroscopy measurements also discloses the impervious nature of the synthesized material. Impregnable working environment can be provided by the prepared composite during industrial maintenance process.

Keywords: CdO-PANI; Mild steel; OCP; Potentiodynamic polarization; EIS.

1.Introduction

Durability of machinery parts are getting shortened on exposure to corrosive environment. Providing environmentally friendly atmosphere in industries is a challenging task. Loss of energy and inferior quality of products associated with the usage of corroded equipments which in turn lead to loss of economy. Acidity maintained, flow of reaction mixtures, heat of reaction and total dissolved solids affect the life time of machinery parts. Selective inhibitors adsorbed on the interior surface of the machines [1] and improve their life time. Adjunct materials are employed to augment the viability and to improve cost-effective production [2-6]. Belligerent nature of cleaning agents employed during pickling, descaling and oil well acidizing diminishes the endurance of machines [7]. Deterioration of metal is prevented by polyaniline [8]. The influence of manufacturing techniques, size and shape of nanoparticles on their abnormal physical and chemical properties attracted to exploit them in industrial application [9-12]. Applications of cadmium oxide nanoparticles have been extensively studied [13-17]. High conducting capacity (10^{-2} -10^{-4} Ω cm) of CdO with band gap of ≥ 2.2eV [18,19] inspired to subsume it with polyaniline. Usage of PANI as inhibitor under various acidic conditions have been reported owing to its stability, good electrical conductivity and adherence nature [20-22].

This paper focuses on the synthesis of universal solvent soluble CdO-PANI composite by chemical oxidative polymerization method and to examine its accomplishment against deterioration of metal parts.

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2. Experimental Details

2.1. Materials

Analytical grade hydrochloric acid, cadmium acetate, sodium salt of dodecylbenzenesulphonic acid (DBSA), ammonium peroxydisulphate (APS), starch & 25% ammonia solution were purchased from Merck Ltd., and were used without purification. AR grade aniline was purified by distillation with zinc dust before it was employed for polymerization.

2.2. Instrumentation

Pellets made with CdO-PANI and KBr were subjected to FTIR analysis on a Perkin-Elmer 337 spectrometer in the frequency range of 4000 - 450 cm^{-1}. Rigaku Maniflex diffractometer (Japan) was used to record the XRD spectrum and JSM-6390 Scanning Electron Microscope was exerted to document the structural morphology of the synthesized materials. EIS and Potentiodynamic polarization studies were registered in ECLAB 10.37 model instrument and OCP measurements were processed in CHI electrochemical analyzer instrument 1200B model.

2.3. Synthesis of Cadmium Oxide-Polyaniline composite

Methodology reported in the literature was adopted to synthesis CdO nanoparticles [23]. Modified in-situ chemical oxidative polymerization method [24] was used to prepare CdO-PANI composite. A mixture containing 1ml of 0.1M aniline and 3ml of 0.1M HCl in 96 ml water was stirred for half-an-hour to get homogeneous mixture. Another solution containing the dispersion of CdO in 0.1M DBSA (prepared in a sonicator operated at 42 kHz oscillation frequency for 45 minutes) was added. Keeping the temperature at ±5˚C, 100 ml of 0.1M, APS solution was added in drops (2 hours duration) with constant stirring. The reaction mixture was set aside for twenty four hours. Green precipitate obtained was filtered, washed with distilled water, acetone for several times and dried in hot air oven at 80˚C for 24 hours and used for further analysis.

3. Results and Discussion

3.1. FTIR analysis

The peaks appeared around 2995 cm^{-1} and 1366 cm^{-1} in the FTIR spectrum of CdO (**Fig.1a**) were assigned to C-H symmetric stretching vibration and wagging vibration of CdO respectively. The peak at 1216 cm^{-1} was allotted to C-H wagging vibration. The characteristic peak in the range of 400-700 cm^{-1} was allocated to Cd-O stretching vibration [25,26].

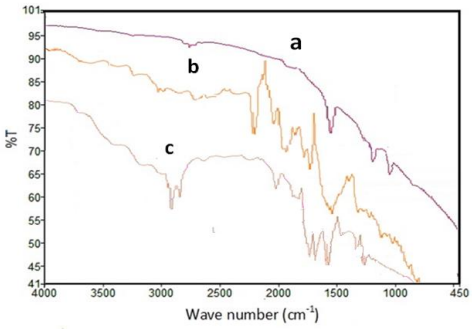


Fig.1. FTIR spectra of (a) CdO (b) PANI (c) CdO-PANI

The FTIR spectrum of PANI (**Fig.1b**) contains C=C stretching of quiniod and benzenoid rings at 1562 cm^{-1} and 1443 cm^{-1} respectively. The C-N stretching vacillation peaks are observed at 1230 cm^{-1} and 1033 cm^{-1}[27,28]. CdO-PANI composite's FTIR spectrum (**Fig.1c**) consist of N-H bending (2966cm^{-1}), C-H stretching (2921 cm^{-1} and 2852 cm^{-1}) and CH_2 bending (1497 cm^{-1}) vibrations of PANI. The metal-oxygen vibration peaks of CdO also appear around 739 cm^{-1} and 651 cm^{-1} [27]. Above observation reveals that PANI has been coated over the CdO particles.

3.2. XRD analysis of PANI and CdO-PANI composite

The peaks obtained for PANI (2θ = 31.65˚, 36.12˚ and 57.86˚) in XRD matches with the earlier report [29,30]. CdO-PANI composite XRD spectrum **(Fig.2)** contains peaks at 2θ = 44.61˚ and 50.12˚ allotted for CdO [31] and peaks related to PANI have reappeared with little change in observed 2θ values. This exposes the distribution of CdO particles in the matrix of PANI.

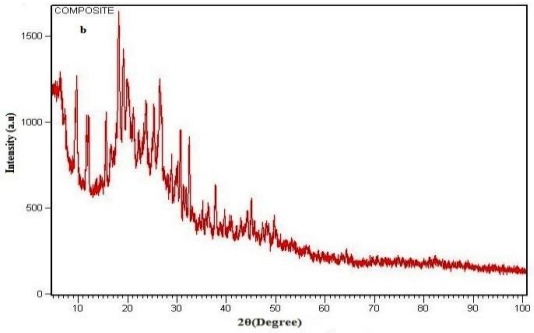


Fig. 2. XRD spectrum of CdO-PANI composite

3.3. SEM analysis of CdO and CdO-PANI composite

The SEM of CdO (**Fig.3a**) looks with granular structure having diameter ranging from 370 nm to 510 nm. The SEM of composite (**Fig.3b**) appears to have granular shape with increase in diameter ranging from 610 nm -760 nm. This matches very well with the earlier report [31].

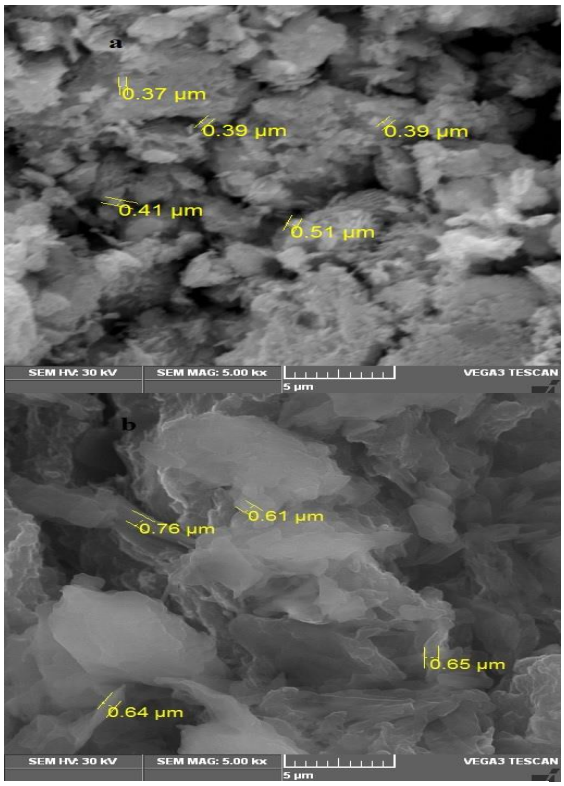


Fig. 3. SEM spectra of (a) CdO and (b) CdO-PANI composite

3.4. Preparation of Electrode materials

Plates of mild steel, having dimension 4 cm x 2 cm x 0.2 cm, comprised with C: 0.21%, Si: 0.035%, Mn: 0.25%, P: 0.082% and Fe: 99.28% were abraded with disparate grade emery papers. The abraded specimens were cleaned by distilled water, ethanol, acetone and desiccated. Gravimetric studies to measure the reticence ability were performed with freshly polished coupons.

3.5. Preparation of Hostile solutions.

Solutions (1M and 2M) used for dwindling of metal pieces were obtained by diluting the analar quality sulphuric acid. Solutions employed for inhibition measurements were prepared by adding different amount of composite (250 - 1000 ppm) to hostile solutions.

3.6. Exploration of inhibition property

3.6.1. Deterioration measurements

Effect of acidic environment on metal surfaces is estimated by assessing the loss in weight on keeping the pre weighed coupons in 250 ml of blank and test solutions. The corroded specimens were removed from the corrosion environment at two hours time interval and cleaned with bristle brush in distilled water, absolute ethanol and acetone. They were reweighed to determine inhibition efficacy (IE%) and surface coverage (θ) after drying at room temperature. The formulae reported earlier [32] were adopted to calculate the above said parameters and are presented in **Table 1** and **2.**

Table 1: Values of IE and θ deliberated from the weight loss measurements in 1m blank and test solutions

Conc. of Composite (ppm)	2-hours			4-hours			6-hours			8-hours		
	Weight loss (g)	I.E (%)	(θ)	Weight loss (g)	I.E (%)	(θ)	Weight loss (g)	I.E (%)	(θ)	Weight loss (g)	I.E (%)	(θ)
Blank	0.1351	--	--	0.2275	--	--	0.2982	--	--	0.3666	--	--
250	0.0197	85	0.854	0.0376	83	0.834	0.0526	82	0.824	0.0699	80	0.809
500	0.0171	87	0.873	0.0336	85	0.847	0.0491	83	0.830	0.0635	82	0.824
750	0.0162	88	0.881	0.8316	86	0.858	0.0468	84	0.842	0.0622	83	0.829
1000	0.0148	89	0.872	0.0277	88	0.878	0.0363	87	0.873	0.0499	86	0.863

Table 2: Values of IE and θ deliberated from the weight loss measurements in 2M blank and test solutions

Conc. of Composite (ppm)	2-hours			4-hours			6-hours			8-hours		
	Weight loss (g)	I.E (%)	(θ)	Weight loss (g)	I.E (%)	(θ)	Weight loss (g)	I.E (%)	(θ)	Weigh t loss (g)	I.E (%)	(θ)
Blank	0.2412	--	--	0.4172	--	--	0.5507	--	--	0.7046	--	--
250	0.0701	71	0.709	0.1284	69	0.693	0.1788	67	0.672	0.2390	66	0.659
500	0.0531	78	0.779	0.0998	76	0.760	0.1442	73	0.728	0.1950	72	0.723
750	0.0511	79	0.788	0.0940	77	0.771	0.1304	76	0.763	0.1756	75	0.750
1000	0.0355	85	0.852	0.0710	83	0.829	0.1130	81	0.812	0.1658	76	0.765

Data provided in the above tables reveal that the CdO-PANI composite is competent of giving protection against 1M acidic solution with eighty percent efficacy even with 250 ppm concentration of composite material **(Fig.4)**. In 2 M belligerent solutions efficiency equal to 1M dissolver solution is obtained after adding 1000 ppm of composite **(Fig.5).** Above results betrayed that CdO-PANI composite can be added with cleaning agents having acidic concentration less than or equal to 1 M.

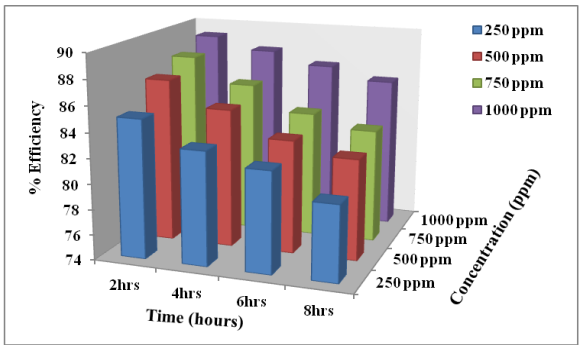


Fig. 4. Inhibition efficiency portrayal by bar chart for 1 M test solution

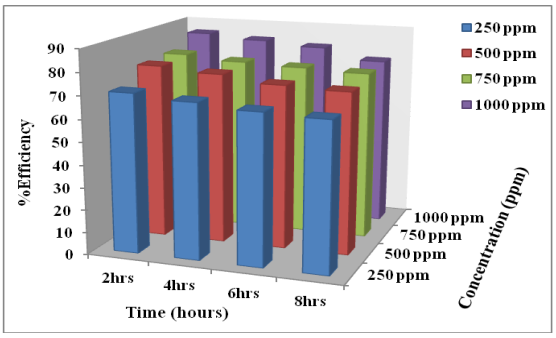


Fig. 5. Inhibition efficiency portrayal by bar chart for 2 M test solution

Combined representation of data given in **Tables 1 & 2** by bar chart (**Fig. 6**) informs that slight desorption of inhibitor is occurring after four hours exposure time which implies that the composite is stable up to 4 hours.

3.6.2. Open Circuit Potential Measurement and Inhibition Efficiency

Three compartment cell comprised of 1cm^2 area of mild steel as working electrode, saturated calomel electrode as reference electrode and platinum electrode as counter electrode was incorporated with CHI electrochemical analyzer instrument model 1200B to document the OCP data up to 120 minutes. The upshot of above studies are provided in **Figures 7** and **8**.

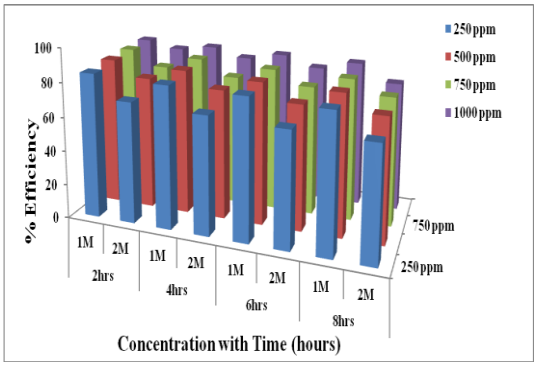


Fig. 6. Combined bar chart for 1M and 2M test solution

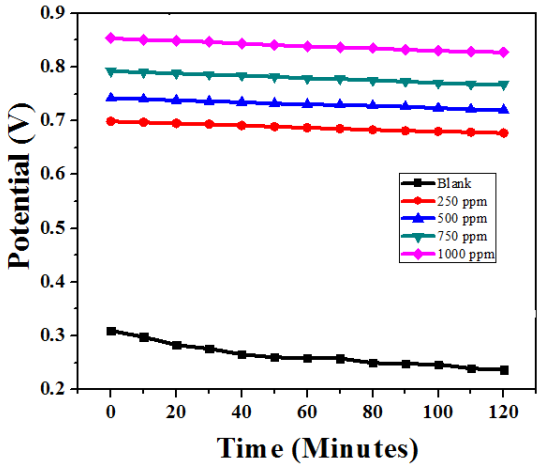


Fig. 7. Plots of OCP in 1 M H_2SO_4 and test solutions

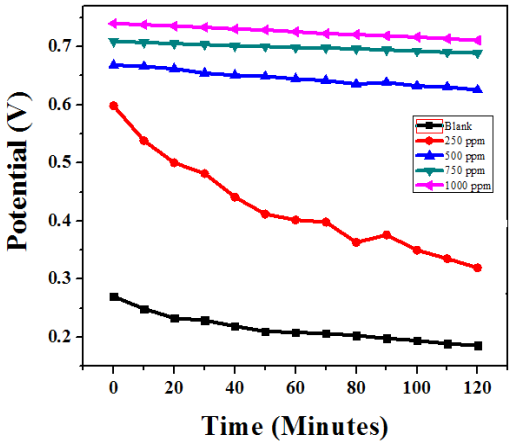


Fig. 8. Plots of OCP in 2 M H_2SO_4 and test solutions

Sudden fall in the OCP values were noticed for antagonistic solution (blank solution). Addition of CdO-PANI composite to the aggressive solution shift the open circuit potential value to positive side and steadiness in the graph up to two hours, ascertain the resistivity of the composite [33].

3.6.3. Quantification of electrochemical parameters

C-LAB analyzer model10.37 included with a three compartment cell having ASTM 415 mild steel specimen (1cm^2 area and remaining portion covered with araldite epoxy resin) as working electrode, standard platinum electrode as counter electrode and saturated calomel electrode as reference electrode was employed to document the Tafel plots and electrochemical impedance plots. Keeping the scan rate as 0.5 mVs^{-1}, Tafel plots were recorded for the applied potential value between -200 to +200 mV. EIS studies were performed in the frequency range of 100 kHz - 10 mHz AC sine wave possessing 10 mV amplitude.

3.6.3.1. Assessment of Potentiodynamic polarization

Factors reflecting the resistivity of CdO-PANI composite (I_{corr}, E_{corr}, b_c and b_a) eventuated from the Tafel plots (**Figure 9** and **10**) are documented in **Table 3** and **4.** Confessed formulae [34, 35] were applied to calculate the values of surface coverage (θ) and inhibition efficiency.

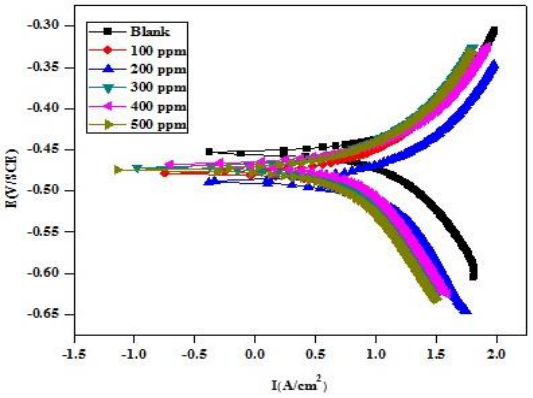


Fig. 9. Plots of potentiodynamic polarization in 1M blank

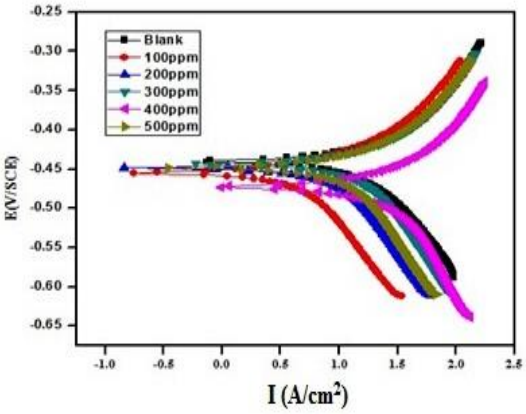


Fig. 10. Plots of potentiodynamic polarization in 2M blank and test solutions

Table 3: Corrosion resistive parameters in 1M H_2SO_4 and test solution

Conc. 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	478	32	36	844	57	0.5693
200	488	26	27	800	59	0.5918
300	471	26	28	573	71	0.7076
400	472	26	28	471	76	0.7596
500	474	20	23	398	80	0.7969

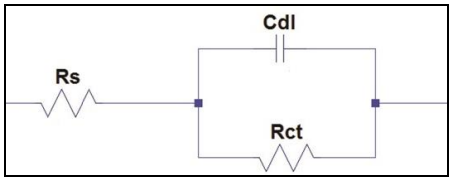
Table 4: Corrosion resistive parameters in 2M H_2SO_4 and test solutions

Conc. 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	439	44	57	2537	--	--
100	476	43	45	2010	21	0.2077
200	448	25	32	1153	55	0.5465
300	442	15	20	1022	60	0.5972
400	507	62	88	813	68	0.6795
500	447	16	17	762	70	0.6996

The decrease in the calculated I_{corr} values noticed for the successive addition of composite material throw back the efficiency of resistivity. The suggestions proposed from the weight loss studies, that CdO-PANI composite is suitable up to 1M dissolver solution is reconfirmed on comparing the I_{corr} values noticed for 1 M and 2 M solutions. Mixed type inhibition efficiency is exposed on attentive comparison of E_{corr}, b_a and b_c data [36].

2.6.3.2. Impedance quantification

Equivalent circuit diagram used for the calculation of R_{ct} and C_{dl} (Impedance parameters) values is presented below



Exemplary semi circle simulacrum of Nyquist plot (**Fig. 11** and **12**) posture out resistivity against corrosion as well as single charge transfer progression [37]. The adsorption of composite on the metal surface is exposed to increase in size of capacity loop. The relationship reported earlier [36] was used to arrive the IE values. Reduction in corrosion current as a result of protective film formation at the electrolyte/metal interface is assigned to the increase in values of R_{ct} [38, 39]. Drop in C_{dl} values have been allocated to increase in electrical double layer thickness [40].

Table 5: Impedance parameters derived for 1M blank and test solution

Conc. of Composite (ppm)	R_s (Ω)	C_{dl} (μ F cm^{-2})	R_{ct} (Ω cm^2)	Inhibition efficiency (%)	Surface Coverage(θ)
Blank	1.079	573	0.8328	--	--
100	1.154	552	1.275	35	0.3490
200	1.479	323	2.225	63	0.6269
300	1.076	511	3.108	73	0.7329
400	1.141	480	3.404	75	0.7509
500	1.492	456	3.474	76	0.7595

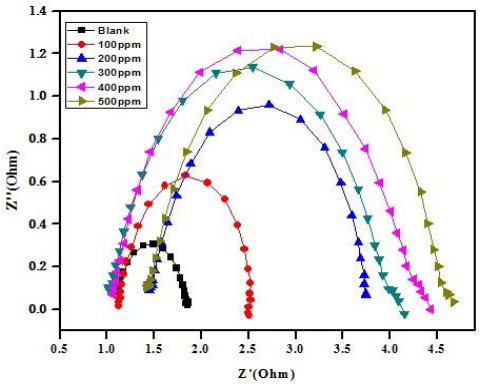


Fig. 11. EIS plots for mild steel in 1M blank and test solutions

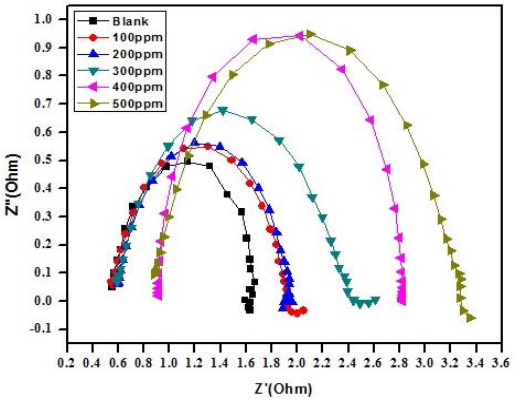


Fig. 12. EIS plots for mild steel in 2M blank and test solutions

Table 6: Impedance parameters derived for 2M blank and test solution

Conc. of Composite (ppm)	R_s (Ω)	C_{dl} (μ F cm^{-2})	R_{ct} (Ω cm^2)	Inhibition efficiency (%)	Surface Coverage(θ)
Blank	0.5549	594	1.008	--	--
100	0.5996	498	1.321	25	0.2495
200	0.5633	618	1.390	28	0.2798
300	0.6130	716	1.872	47	0.4740
400	0.6902	538	2.483	59	0.5939
500	0.9091	405	2.516	60	0.5993

4. Conclusion

PANI coated cadmium oxide composite soluble in water and possessing corrosion resistance efficiency up to 89% under 1M acidic condition for two hours, in weight loss measurement, is prepared by adopting modified in-situ chemical oxidative polymerization method. Presence of 1000 ppm of composite material prevent the corrosion, even after eight hours exposure to belligerent solution, with little changes (86% efficiency). Open circuit potential measurement, potentiodynamic polarization and impedance measurements also manifested the resistive efficiency of the CdO-PANI composite. Careful analysis of the arrived data suggest that the CdO-PANI composite could be employed as guarding agent in industrial cleaning process.

5. Conflicts of interest

There are no conflict to declare.

6. Acknowledgments

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