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Inhibition effect of self assembled films formed by adipic acid molecules on carbon steel surface

Well-ordered SAMs using adipic acid were formed on the oxide of iron carbon steel surface by immersion method. This leads to ordered, robust monolayers bound to the surface in a tetra dentate manner. Monolayer formation takes place when carbon steel is immersed in an aqueous solution containing 60ppm of Cl⁻ and 50ppm of adipic acid for 5 minutes, and rinsing the physisorbed molecules in distilled water and heating in a hot air oven. The adipic acid monolayers on iron oxide steel carbon can withstand rinsing with water, concentrated acid and base exposure. Additionally, these monolayers are stable over the course of at least one week. The formation of monolayers were confirmed by AFM study and FTIR spectra. The SAMs were tested by polarization and EIS electrochemical analysis. The results of this study show that adipic acid monolayers adsorbed on metal surface can reduce electrochemical activity on the surface, often the first step in corrosion.

Key words: *Self-assembled films, iron oxide surface, polarization study, electrochemical impedance spectroscopy, AFM, FTIR, corrosion, deposition method.*

INTRODUCTION

Nanometer-sized particles are attracting considerable attention because of their unique properties, including optical, electrical, electrochemical, photo-electrochemical and magnetic properties [1-3]. Noble metals, such as gold and silver, are hot spots of the research because they can be easily prepared and exist steadily. Moreover, we can manipulate these nanoparticles according to our will. Much effort has been done in exploring self-assembled (SA) nanoparticles into ordered structures [4]. Specific structures can provide controlled fabrication of nanometer-sized building blocks with distinctive and useful properties [5]. Polymer-stabilized nanoparticles are usually self-assembled into two-dimensional arrays on the substrate [6]. Yiwei Tan et al. [7] had reported the self-organization of wire-pattern arrays of Ag nanoparticles. Byeong-Hyeok Sohn [5] had demonstrated a directed self-assembly of two different kinds of nanoparticles on a block copolymer micellar template. These two particles are gold and iron oxide. The self-assembling process has been investigated to improve corrosion inhibition of metals because self-assembled substances are able to react spontaneously on the metal surface and form compact and stable films

[8]. These films can protect metal from corrosion successfully. Iron is a widely used metal with extensive industrial application and the study of its corrosion inhibition has attracted much attention [9, 10]. The first work to use self-assembled films on iron for corrosion protection was self-assembling alkanethiols [11]. But the application of thiol-compounds is limited due to their toxicity [12]. Ilona Felhosi et al. [12] studied the formation of self-assembled films of alkane monophosphonic acids on iron surface and explained it mechanism of corrosion protection.

Organic acids such as phosphonic [13, 14-18], carboxylic [19, 20-23], hydroxamic [24], and sulfuric [16] acids are capable for forming SAMs by chemisorption on stainless steel [25, 19, 26], aluminium [20-22, 27], titanium [13, 14, 17, 18], and copper oxide [28] surfaces.

In the present work, SAMs of oxide / carbon steel surface using adipic acid (AA) were formed on carbon steel surface by immersion coating (immersing metal surface in an aqueous solution containing 60 ppm of Cl⁻, in the absence and presence of 50ppm of adipic acid). The modified samples were characterized by FTIR, and atomic force microscopy (AFM). The formation, uniformity, ordering and bonding of the monolayers accomplished by immersion method have been evaluated. Furthermore, the electrochemical properties of the unmodified and modified carbon steel surfaces were characterized by polarization study and Electrochemical impedance spectroscopy (EIS) analysis to test the ability of the monolayer to reduce the corrosion of the surface.

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2. EXPERIMENTAL SECTION

2.1. Material

Very pure adipic acid of analytical reagent grade was used in the present study.

2.2. Preparation of the substrates

Carbon steel substrates (Composition : wt % : 0.026% S, 0.06% P, 0.4% Mn, 0.1% C and the rest Fe) of dimensions 1 x 1 x 0.2 cm were polished to a mirror finish and degreased with trichloroethylene and stored in oven at 100^oC for 1 h or more.

2.3. Formation of monolayers

2.3.1. Immersion deposition monolayers

The cleaned substrates were placed in the oven for 24 h. They were then immersed in an aqueous solution containing 60ppm Cl^- in the absence and presence of 50ppm of adipic acid for 5 mts and placed in an oven at 100^oC for at least 1 h.

2.4. Characterization of the monolayers

2.4.1. The substrates were studied using a Perkin Elmer 1600 FTIR spectrophotomer. FTIR spectrophotometer was used to analyze the nature of interaction between the molecules of adipic acid (AA) and the substrate and the alkyl chain ordering the vCH_2 peaks as the reference. The film was removed carefully, mixed with KBr and made into pellets. FTIR spectra were recorded.

2.4.2 Atomic force microscopy

The carbon steel specimen immersed in blank and in the inhibitor solution for a period of 5 mts was removed, rinsed with double distilled water, dried and subjected to the surface examination. Atomic force microscopy (Veeco innova model) was used to observe the samples' surface in tapping mode, using cantilever with linear tips. The scanning area in the images was 5 μm x 5 μm and the scan rate was 0.6 HZ / second.

2.4.3. Potentiondynamic Polarization Study

Polarization studies were carried out in a CHI – Electrochemical work station with impedance, model 660 A. It was provided with iR compensation facility. A three electrode cell assembly was used. The working electrode was used carbon steel. A saturated calomel electrode (SCE) used the reference electrode; platinum was the counter electrode.

From polarization study, corrosion parameters such as corrosion potential parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes (b_c and b_a) and linear polarization resistance (LPR) were calculated. During the polarization study, the scan rate (v/s) was 0.01; Hold time at E_f (s) was zero and quiet time (s) was 2.

2.4.4. Electrochemical Impedance Spectroscopy (EIS) measurements

The instrument used for polarization study was used to record electrochemical impedance spectra also. The cell set up was also the same. The real part ($Z^{'}$) and imaginary part (Z'') of the cell impedance were measured in ohms at various frequencies. Value of charge transfer resistance (R_t), double layer capacitance (C_{dl}) and impedance, log (Z/ohm) were derived from Nyquist and Bode plots. Electrochemical impedance spectra were recorded with initial E(v) = 0; High frequency (Hz = 1 x 10^5); Low frequency (Hz = 1); Amplitude (v) = 0.005 and Quiet Time (s) = 2.

3. RESULTS

3.1. Immersion depositon method

3.1.1. FTIR Spectra

A simple procedure of immersing mild steel into an aqueous solution containing 60 ppm of Cl^- and 50 ppm of adipic acid for 5 minutes led to spontaneous film formation on the iron oxide surface, produced by the interaction of chloride iron present in the medium with iron. There is also possibility of formation of iron-adipic acid film, in the absence of iron oxide, if there is less corrosion or no corrosion of carbon steel. The samples were characterised by FTIR spectra to determine alkyl chain conformation and head group-substrate bonding [25, 17]. Position of the CH_2 stretching in the infrared spectrum can be used to determine the ordering of the alkyl chain within a film [22, 29-31].

For disordered chains, the frequency of the CH_2 stretching is close to that of a liquid alkane (ν CH_2 asym ~ 2924 cm^{-1}) due to the presence of a gauche conformation in the alkyl chains [30, 32]. For well-ordered alkyl chains the frequency is shifted to lower wave numbers and is close to that of a crystalline alkyl (ν CH_2 asym ~ 2914 – 2918 cm^{-1}) which is considered to have a high degree of order, with all-trans conformation throughout the alkyl chain. The

FTIR spectrum of pure adipic acid (AA) is shown in Fig 1a. The peaks due to ν CH$_2$ asym and ν CH$_2$ sym appear at 2810 and 2724 cm^{-1}. The FTIR spectrum of iron oxide film formed on metal surface after immersion in 60ppm Cl$^-$ is shown in Fig 1b. The peaks due to iron oxide appear at γ FeOOH = 1045, 1632 cm^{-1}; α FeOOH = 602 cm^{-1}. The peaks due to Fe$_3$O$_4$ are absent. [32a].

The position of the peaks corresponding to ν CH$_2$ asym and ν CH$_2$sym after immersion in an aqueous solution containing 60ppm of Cl$^-$ and 50ppm of adipic acid for 5 minutes and after rinsing in distilled water to remove any physisorbed material or multilayers, were 2811cm^{-1} and 2726 cm^{-1}, respectively (Fig 1c). This indicates that the film in stable, well ordered and strongly bound to the surface.

The IR spectra further indicate that the organic molecules are bound to the surface in a tetradentate manner as determined from the shifting of ν C = O and ν − OH stretching of the two carboxyl groups of adipic acid specifically the ν C = O has shifted from 1715 to 15 97cm^{-1} and νOH has shifted from 3417 to 3414 cm^{-1}. Similar bidentate interaction has been reported for alkyl phosphonic acid on nickel oxide surface [33] and other organic molecules on other oxides [25, 19, 17].

While FTIR spectroscopy can be used to characterize alkyl chain ordering and binding of the molecules to the surface, it cannot determine film thickness and integrity. Therefore, AFM imaging was used to examine the monolayer at nanometer resolution and to verify that the deposition method produces uniform monolayers.
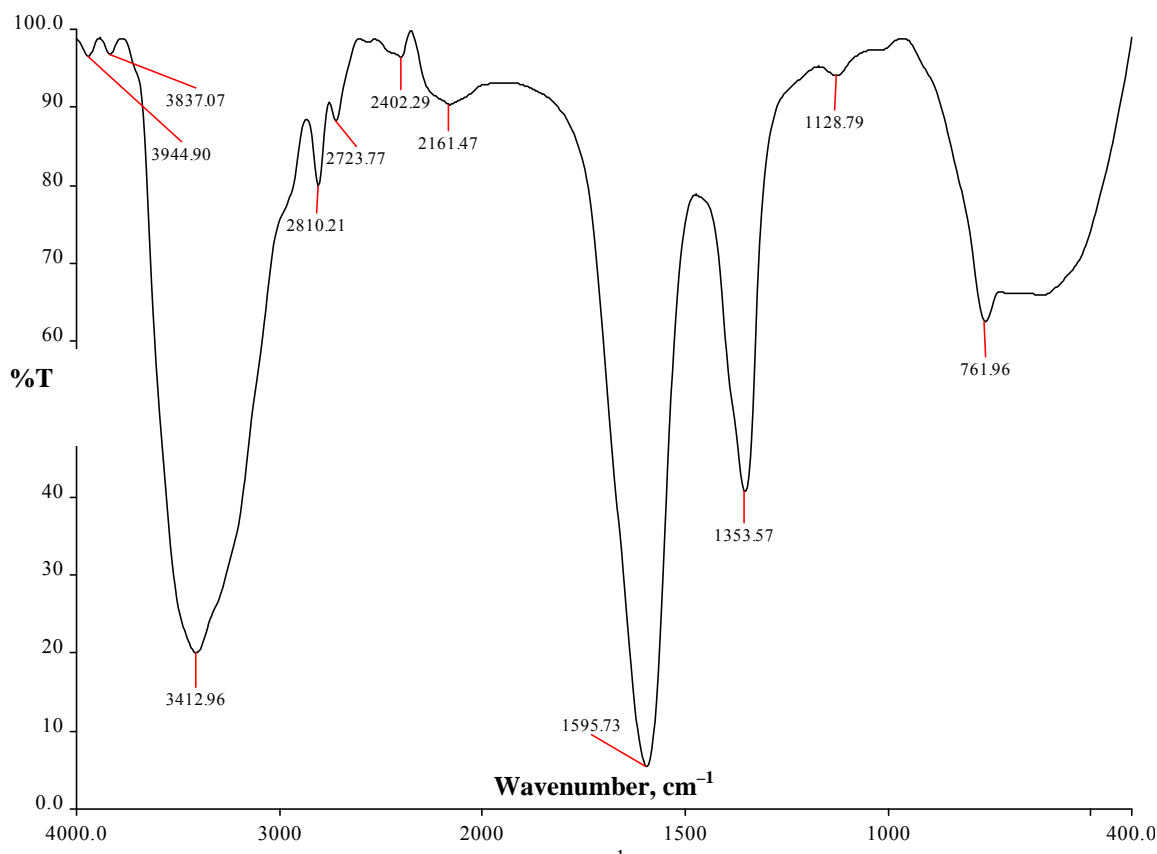

Fig1: FTIR spectra (KBr) of films formed on carbon steel surface after immerse on in various test solution: a) pure adipic acid (AA)
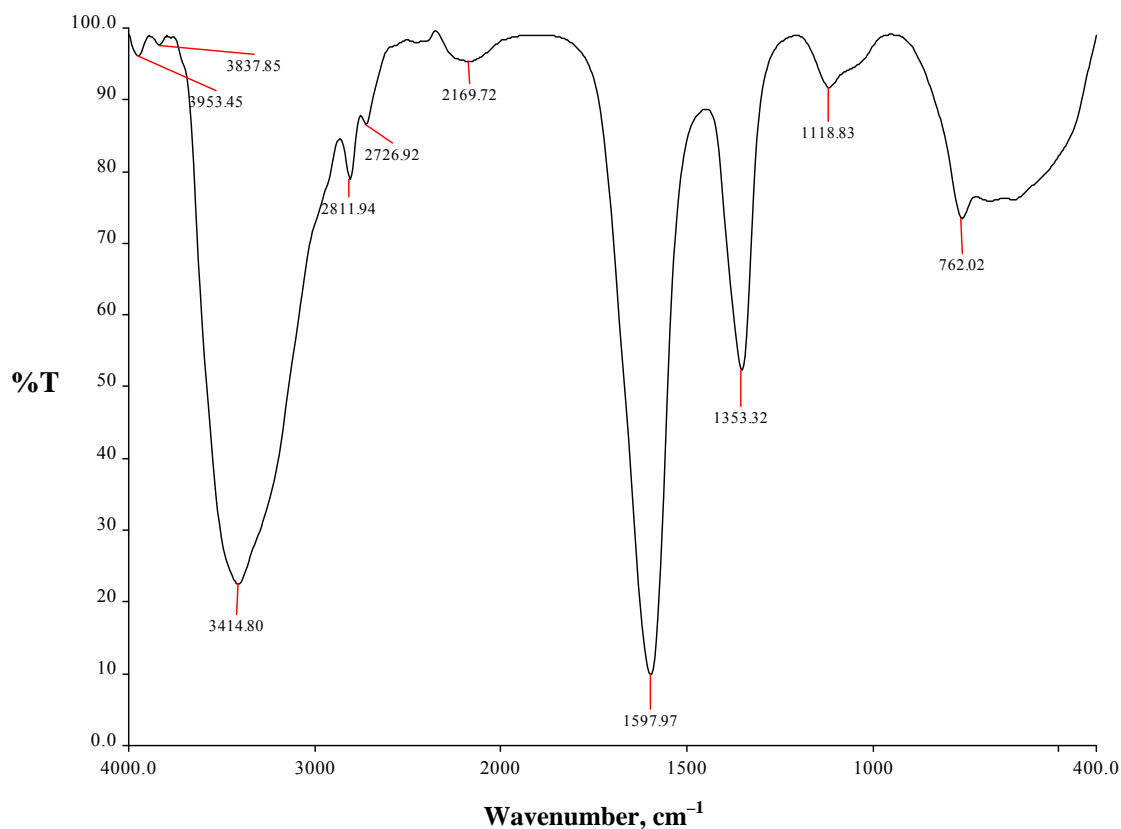
Fig 1: FTIR spectra (KBr) of films formed on carbon steel surface after immerse on in various test solution: b) Adipic acid 50ppm + Cl⁻ 60ppm
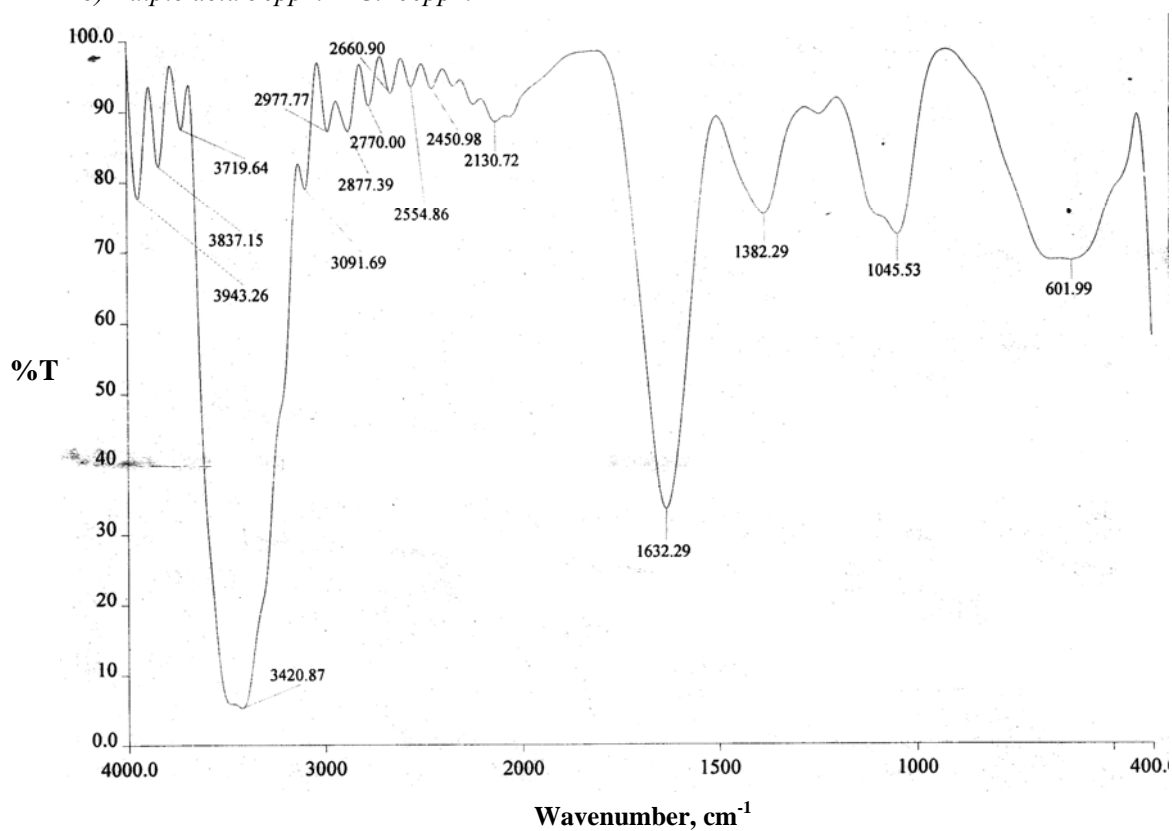

Fig 1c: Iron oxides formed on metal surface after immersion in solution confining: 60 ppm of Cl[1].

3.1.2. AFM imaging

The comparison of the root mean square (rms) roughness of the unmodified substrate to the modified samples is an indicator of the film uniformity. The rms roughness parameter is a measure of the deviations in the surface from the mean plane within the sampling area [34].
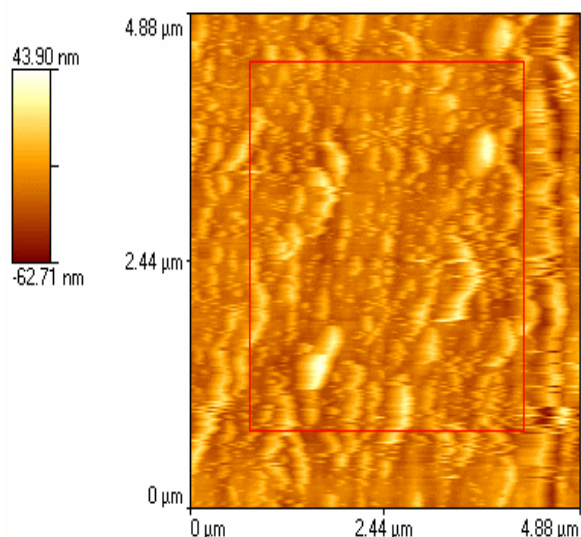

Fig 2: AFM to pography images of films formed on carbon steel surface after immerse on in various test solution: a) Cl⁻ 60ppm
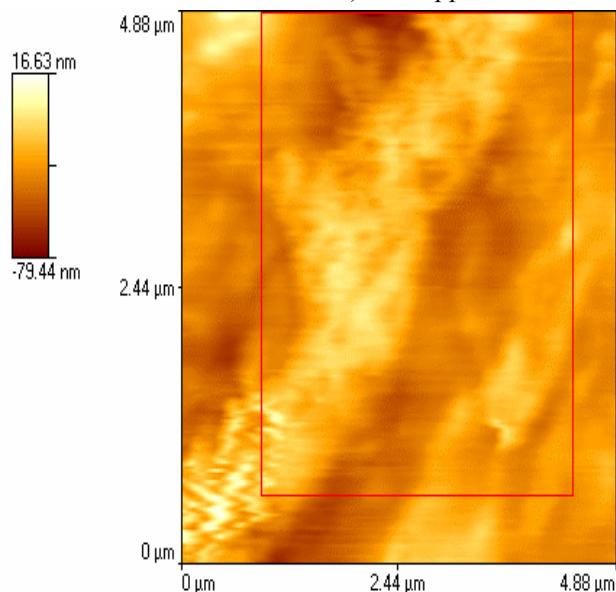

Fig 2: AFM to pography images of films formed on carbon steel surface after immerse on in various test solution: b) Cl⁻ 60ppm + Adipic acid 50ppm

Modified surfaces with a rms roughness similar to the control surface are considered to be films of monolayer thickness that follow the contour of the

surface, while modified surfaces that have much larger rms roughness than the control are multilayer or non-uniform films [35, 36].

The iron oxide control samples had an average rms roughness of 12.06 nm (Fig 2a). The control sample of iron oxide is rough in comparison to model surfaces such as gold and silicon [37, 38]. Modified samples formed by the immersion method (immersed in 60 ppm of Cl^{-1} had an rms roughness of 11.88 nm (Fig 2b). The rms roughness of the substrate did not change very much after the adsorption of adipic acid molecules suggesting uniform deposition of the organic molecules on the surface without aggregate or micelle formation. Therefore it is concluded that within the scope of the analysis, the film formed is a single layer of molecules that follows the contour of the underlying substrate.

The rms roughness values of the films (SAMs) formed on iron oxide / metal surface after immersion in various test solutions are given in Table 1.

Table 1 - The rms roughness values of the films (SAMs) formed on iron oxide / metal surface immersed in various test solutions.

Test solution	rms roughness of the films (SAMs), nm
Cl⁻ 60ppm	12.0558
Cl⁻ 60ppm + AA 50ppm	11.8833

The rms roughness 12.0558 nm is due to oxides of iron, confirmed by FTIR spectrum Fig 1b which are formed when carbon steel is immersed in 60ppm Cl$^-$ environment. When 50ppm of adipic acid (AA) is added the rms value decreases to 11.8833 nm. This indicates that the protective film (SAMs) is formed on the metal surface itself, and not on the iron oxide surface. Had it formed on the iron oxide surface the rms value would have been greater than 12.0558. So it is concluded that SAMs are formed not on the iron oxide surface but on the metal itself, even before the corrosion process starts, leading to the formation of iron oxide.

3.1.3. Stability of monolayer films

The monolayers formed by deposition method were analyzed for stability. After rinsing in water, the samples were left under atmospheric conditions for one day and analyzed. The monolayers remained ordered and bound to the surface as indicated by the lack of change in the FTIR spectra over one day. Stability to acid and base exposure was tested by rinsing modified substrates 1 M HCl or 1 M NaOH. FTIR spectra taken after these treatments remained unchanged.

Film stability and chain ordering can be dependent on alkyl chain length. In general, stability and order increase as the chain length increases [39, 40-43]. Therefore, a long chain length (> 11 carbons) is commonly used in the formation of SAMs. Substantial disorder is generally found in films formed by short chain molecules, although ordered monolayers of short chain phosphonic acids have been formed on nitinol, titanium and stainless steel oxide [25, 17, 44]. In the present study there are only 6 carbons in the chain. However, the monolayer is found to be stable. Since adipic acid is a dicarboxylic acid tetradentate bonding is expected, and this leads to the stability of the monolayer. It is obvious from the proposed structure of the monolayers, there is some strain in the CH_2 chain, as per Baeyer's strain theory. There are even number of (four) CH_2 groups in adipic acid. Therefore there is strain in the CH_2 chain. Interestingly, if the number of CH_2 groups is odd number (1, 3, 5) as in the case of malonic acid, glutaric acid and pimelic acid, more stability is expected, in the SAMs after coordination to the metal surface in a tetradentate fashion. Further research in this line will confirm the preposed concepts.

This will also be reflected in the more corrosion resistance offered by these SAMs, during electro-chemical studies such as polarization study and AC impedance spectra.

These conclusisons are confirmed by visual observations also. In the blank experiment (60ppm Cl⁻ only) brown film was observed on the metal. In the presence of 50 ppm of AA brown film was absent and thin interference films (VIBGYOR colour) were noticed.

3.1.4. Potentiodynamic Polarization Study

The potentiodynamic polarization curves of carbon steel immersed in an aqueous solution containing 60ppm of Cl⁻ in the absence and presence of adipic acid (AA) are shown in Fig 3. The corrosion parameters such as corrosion potential (E_{corr}), Tafel slopes (b_c, b_a), linear polarization resistance (LPR) and corrosion current (I_{corr}) are given in Table 2.

Table 2 - Corrosion parameters of carbon steel immersed in aqueous solution containing 60ppm of Cl⁻

AA ppm	E_{corr} m V vs SCE	b_c m V / decade	b_a mv / decade	LPR ohm Cm²	I_{corr} A / cm²
0	-609	183	189	23319.1	1.735 x 10⁻⁶
50	-715	166	157	26664.4	1.315 x 10⁻⁶

Carbon steel electrode was immersed is various test solutions for 5 minutes to attain the steady state potential. Then polarization curves were recorded. When carbon steel is immersed in an aqueous solution containing 60ppm of Cl⁻, oxides of iron are formed on the metal surface (FTIR spectra, Fig 1b). The corrosion potential is -609 mV vs SCE. The LPR is 23319.1 ohm cm² and the corrosion current 1.735 x 10⁻⁶ A/cm² (Fig 3a). When 50ppm of AA is added to the above environment, and allowed to stand for 5 minutes, self assembling monolayers (SAMs) of AA are formed on the metal surface. Now the LPR value increases from 23319.1 ohm cm² to 26664.4 ohm cm² (Fig 3b). The corrosion current decreases from 1.735x10⁻⁶ A/cm² to 1.315x10⁻⁶ A/cm². This reveals that the SAMs formed on the metal surface have corrosion resistance property. The attack of Cl⁻ ions, present in the bulk of the solution, on the metal surface, is prevented by the SAMs of adipic acid.
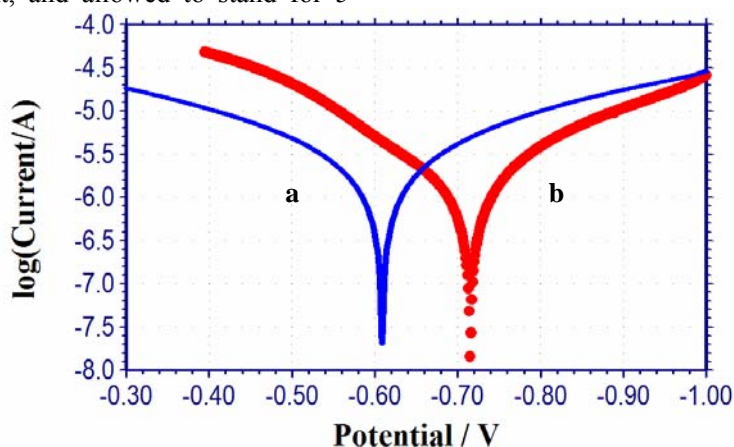

Fig 3: Polarization curves of carbon steel immersed in various test solutions: a) Cl⁻ 60ppm, b) Cl⁻ 60ppm + Adipic acid 50ppm

3.1.5. Electrochemical impedance spectra

The electrochemical impedance spectra of carbon steel immersed in various test solutions are shown in Fig 4. The equivalent electrical circuit for the model is shown in Fig 5. The impedance parameters such as charge transfer resistance (R_t), double layer capacitance (C_{dl}) and impedance value, long (Z/ohm), are given in Table 3.

When carbon steel is immersed in an aqueous solution containing 60 ppm of Cl⁻, and left for 5mts, oxides of iron are formed on the metal surface. The R_t value is 1225 ohm cm². The C_{dl} value is 4.1632×10^{-9} F/cm². The impedance, log (Z/ohm) is 3.119 (Fig 4c).

Table 3 - Impedance parameters of carbon steel immersed in an aqueous solution containing 60ppm of Cl⁻

AA ppm	R_t ohm cm²	C_{dl} F/cm²	Impedance log (Z/ohm)
0	1225	4.1632×10^{-9}	3.119
50	2249	2.2677×10^{-9}	3.493

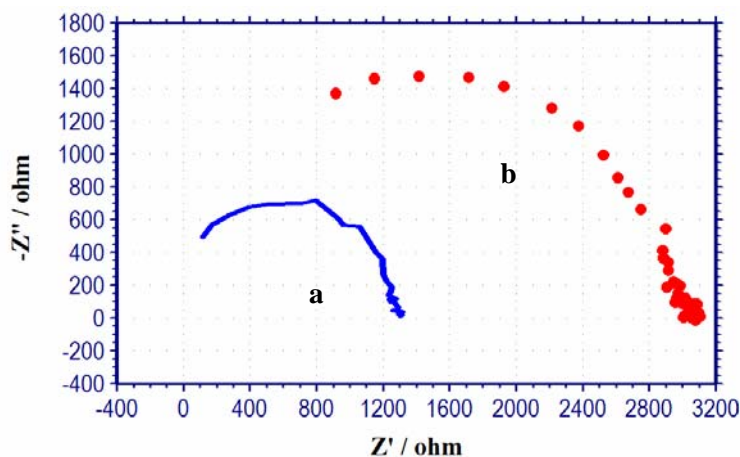*Fig 4: Electrochemical Impedance spectra of carbon steel immersed in various test solution (Nyquist Plots): a Cl⁻ 60ppm, b) Cl⁻ 60ppm + Adipic acid 50ppm*
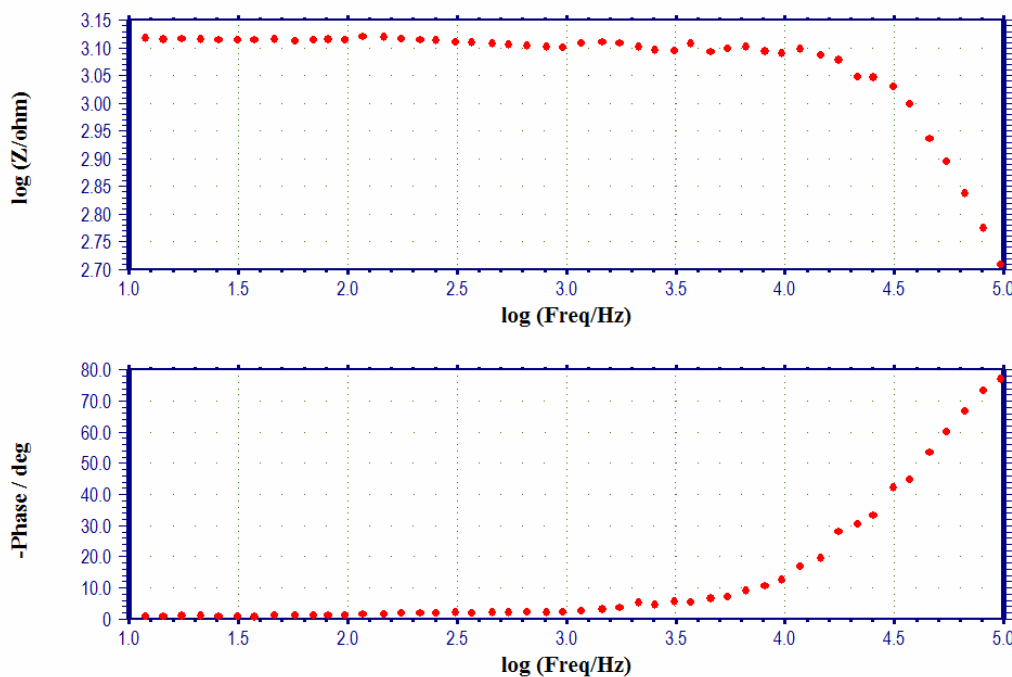

Fig. 4c: AC impedance spectra (Bode Plots) of carbon steel immersed in solution containing 60ppm of Cl⁻.
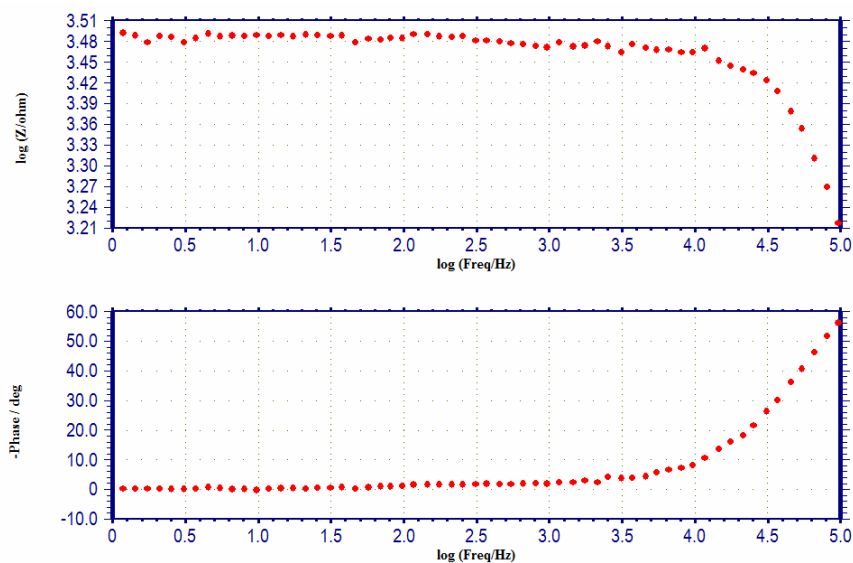
Fig. 4d: AC impedance spectra (Bode Plots) of carbon steel immersed in solution containing 60ppm of Cl⁻ and 50ppm of adipic acid.
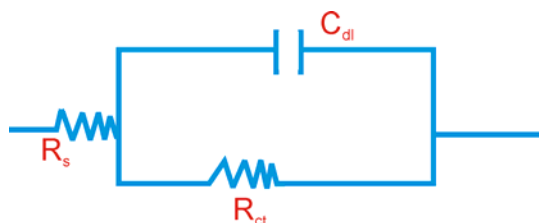

Fig 5: Equivalent electrical circuit diagram: R_s = Solution resistance, R_{ct} = Charge transfer resistance, C_{dl} = Double layer capacitance
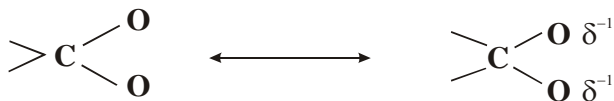

Fig 6: Electron density on oxygen atoms of carboxyl group.

When carbon steel is immersed in an aqueous solution containing 60 ppm of Cl⁻ and 50 ppm of AA, the R_t value increases from 1225 ohm cm² to 2249 ohm cm²; the C_{dl} value decreases from 4.1632 x 10⁻⁹ F/ cm² to 2.2677 x 10⁻⁹ F/cm²; the impedance value increases 3.119 to 3.493 (Fig 4d). These results suggest that SAMs formed on the metal surface have corrosion resistance property. This results in increase in R_t value and decrease in C_{dl} value.

4. DISCUSSION

Ordered, complete monolayers of adipic acid were formed on the iron oxide / iron surface. These monolayers were formed using an immersion deposition process. To accomplish monolayer by immersion, a counteraction of 50 ppm of adipic acid, solution exposure time of 5 minutes and setting temperature of keeping the metal specimens in the oven at 100°C for 1 hr.

The process of monolayer formation using immersion coating has been described as a sequential process of nucleation, growth and coalescence of densely packed two-dimensional islands, finally covering the entire substrate surface or a large fraction of it [45, 46-49]. The average surface coverage for adipic acid films on iron surface is found to increase monotonically with solution concentration, consistent with a quarter surface density of adipic acid molecules. It is proposed that the initial islands are nucleated through the adsorption of individual adipic acid mole from solution [45, 46, 47]. Therefore a higher concentration of the monomer (50 ppm) in solution leads to more collisions between monomers and subsequent island nucleation becomes more probable leading to a large number of islands on the surface [46, 50]. For the immersed substrates, increasing the solution concentration leads to the formation of a complete monolayer. When the substrates were heated the tetradentate increased, the molecules become thermally mobile and therefore more available for nucleation and / or growth [51].

The molecules in the monolayers formed by immersion method were bound to the iron oxide / iron surface in a tetradentate manner. This bonding motif was persistent from deposition through solvent, acid and base rinsing and exposure to atmosphere for one day. A bidentate or monodentate bonding motif is seen between phosphonic acids and various metal oxides such as stainless steel 316L and Zircorium [25, 17, 44, 52-55]. In the present case, adipic acid is bonded to iron oxide/ iron surface in tetradentate manner, since adipic acid is a dicarboxylic acid and coordination can take place through oxygen atoms of carboxyl groups.

The cause of the differences in bonding motifs has not been determined but may be a function of several factors including oxygen-metal-oxygen distance on the surface, hydroxyl content of the native oxide surface and the coordination sites available on the adsorbate molecules.

5. SUMMARY

Well-ordered SAMs using adipic acid were formed on the oxide of iron carbon steel surface by immersion method. This leads to ordered, robust monolayers bound to the surface in a tetra dentate manner. Monolayer formation takes place when carbon steel is immersed in an aqueous solution containing 60ppm of Cl^- and 50ppm of adipic acid for 5 minutes, and rinsing the physisorbed molecules in distilled water and heating in a hot air oven. The adipic acid monolayers on iron oxide steel carbon can withstand rinsing with water, concentrated acid and base exposure. Additionally, these monolayers are stable over the course of at least one week.

The formation of monolayers were confirmed by AFM study and FTIR spectra. The SAMs were tested by polarization and EIS electrochemical analysis. The results of this study show that adipic acid monolayers adsorbed on metal surface can reduce electrochemical activity on the surface, often the first step in corrosion. Monolayer formation on the iron oxide carbon steel surface is a significant advance in surface modification of iron oxide and can be used as a building block for future applications in corrosion barriers and electronics.

6. Acknowledgement

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IZVOD

EFEKAT INHIBICIJE NA FILMOVE FORMIRANE OD STRANE MOLEKULA MASNIH KISELINA NA POVRŠINI UGLJENIČNOG CELIKA

Dobro uređene SAMs slojevi su formirane na površini oksida ugljeničnih celika pomoću masnih kiselina putem metode potapanja. To dovodi do stvaranja monosloja vezanog za površinu tetraedra. Monosloj se stvara kada se ugljenični čelik uroni pet minuta u vodeni rastvor koji sadrži 60ppm Cl⁻ i 50ppm masnih kiselina a zatim se ispira i suši toplim vazduhom. Dobijeni monosloj masnih kiselina na površini ugljeničnog čelika može da izdrži ispiranje vodom i izlaganje koncentrovanih kiselina i baza. Pored toga, ovi monoslojevi su stabilni najmanje tokom jedne nedelje. Formiranje monoslojeva potvrđeno je pomoću AFM studije i FTIR spektra. SAMs su testirane na polarizaciju i putem EIS elektrohemijske analize. Rezultati ovih ispitivanja pokazuju da monoslojevi masnih kiselina adsorbovani na metalne površine mogu da smanje elektrohemijsku aktivnost na površini, koja je često prvi korak za početak korozije.

Ključne reči: samoformirani filmovi, površina oksidnog gvožđa, polarizaciona studija, elektrohemijska impedansa spektroskopija, AFM, FTIR, korozija, taloženje