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Benzylidene Schiff base Corrosion Inhibition and Electrochemical Studies of Mild Steel In 1M HCl and 0.5m H_2SO_4 acidic Solutions

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ABSRACT

N-Benzylidene-4-Methoxyaniline (NB4MA) Schiff base was synthesized and investigated corrosion behavior of mild steel (MS) in 1M HCl and 0.5M H_2SO_4. The corrosion efficiency studied by means of mass loss and electrochemical techniques. The electrochemical studies confirm that E_{corr} displacement about 32mV confirms anodic and cathodic type of inhibitor. The positive direction of corrosion potential curves confirmed that adsorption efficiency on metal surface. Langmuir adsorption isotherm was observed. Electrochemical impedance studies (EIS) exposed that inhibition effectiveness enlarged with even increasing concentration of Schiff base and found inhibition capacity 90-95%. Furthermore, thermodynamic parameters of adsorption were found which elaborates that increasing ΔG^0_{ads} parameter since favorable interaction behavior between mild steel and inhibitor causes efficient adsorption. The morphology studies revealed that synthesized Schiff base has strong affinity to adhere on mild steel and improve corrosion efficiency against acidic media. The quantum chemical parameter also confirmed that electrons distribution and efficiency of Schiff base.

Keywords: Corrosion, Inhibition, Schiff base, Impedance studies, Quantum chemical parameter.

INTRODUCTION

Metal corrosion process happens frequently which mainly ensue and directly affect the industrial equipment. The mild steel is extensively utilized for build materials reason in many industries such as petroleum production industry, power generation plants and cooling tower. Normally, Hydrochloric acid and sulphuric acid solutions specifically has been used for cleaning purpose when acidizing functions cause possibility to happen corrosion in the steel. The inhibitor used to protect corrosion for that intention prepare organic compound contains hetero atoms utilized for excellent corrosion inhibitor of steel present in acidic media. The inhibitor is adsorbed on metal surface by the means of the electron donating N, S, P and O atoms as well as double/ triple bonds or aromatic ring[1].

Recently, derivatives of Benzylidene amine Schiff base molecules expressed by the formula C_6H_5-CH=N-C_6H_5 are considered a significant inhibitor behavior because of presence azomethine group –CH=N and aromatic ring pi-electron. These molecules are thin hence their products are reducing rate of corrosion since slowing anodic reaction[2]. Ece altunbas sahin *et al.,*[3] reported that corrosion effect in 1 N HCl of using synthesized 4 amino N-benzylidene-benzamide Schiff base suggested that Langmuir adsorption isotherm for adsorption process and protective film was formed homogeneously on the metal steel develops inhibition capacity due to presence of amine and aldehydes involving in inhibition action.

El Hassane Anouar *et al.,*[4] investigated that substituted benzylidene Schiff base corrosion effect on mild steel immersed in 1M HCl. It suggested that benzene ring C=C and C=N involving in chemisorptions pi-pi interaction and suggested functional group C=O and C=N and heteroatom O, N, S increasing protecting ability of inhibitors is strengthen by molecular structure such as electro negativity of hetero atom and aromatic electron clouds. M. A. Bedair reported benzidine based Schiff base compound inhibition efficiency found in carbon steel of 1.0M HCl further concludes that benzidine derivative efficient corrosion inhibition followed chemisorptions, Langmuir adsorption especially aromatic ring, imine group and lone pair electron of hetero atoms leads inhibition efficiency[5]. Abdelghani Madani *et al.,*[6] Suggested that synthesis of benzidine based Schiff base and demonstrated from SEM and DFT quantum studies that is function groups are responsible for corrosion inhibition performance. Corrosion inhibition sites such as hetero atom, aromatic ring, azomethine linkage group are inevitable groups those contribute major role for the purpose of adsorption between metal surfaces and inhibitor then improves efficiency of inhibitor in acidic solution 1.0 N HCl along with mild steel.

Recently, Caio Machado Fernandes *et al.,*[7] reported that mild steel present in HCl using green synthesized benzylidene derivative as inhibitor and suggested that electrochemical behavior indicates corrosion process increasing due to reducing anodic and cathodic corrosion reaction. AFM, SEM depicted smooth surface in the presence of organic molecules die to formation of protective layer, covalent bond formation with steel act as corrosion inhibition confirmed by DFTB. Hulya keles *et al.,*[8] reported that benzylidene compound inhibition behavior in 1M HCl which exposed that the immersion time extends corrosion inhibition, hetero atom and aromatic ring pi- electrons were found the possible interaction site on the inhibition surface.

In this study, synthesized a Schiff base N-benzylidene-4 methoxyaniline by using precursor p- anisidine and benzaldehyde. Especially, chosen for above Schiff base since presence of –CH=N– and $–OCH_3$ these groups consist donor hetero atoms induces adhere metal surface of mild steel creates effective corrosion inhibition efficiency. Further the synthesized inhibitor corrosion effectiveness on mild steel surface has been investigated in the presence of acidic solutions 1M HCl and 0.5M H_2SO_4. The corrosion effect has been examined by the means of weight loss method, polarization plots, electrochemical impedance study, scanning electron microscope (SEM). The density functional theory (DFT) at B3LYP/ 6-31G level has been used to determined quantum chemical calculation of the inhibitor molecules.

MATERIALS AND METHODS

p-anisidine [4-(CH_3O) $C_6H_4NH_2$ (assay 98%)], benzaldehyde [C_6H_5CHO (assay 99%)], ethanol [Assay 99.9%]. 1 M HCl (assay 37%) and 0.5M H_2SO_4 (95-97%) chemicals were purchased from Merck. Then 1 M Hydrochloric acid, 0.5M H_2SO_4 solutions were prepared by using distilled water.

Synthesis and Characterization

The Schiff base N-Benzylidene-4-Methoxy aniline was synthesized by adding p-anisidine and benzaldehyde are dissolved in minimum amount of ethanolic solution followed a round bottom flask used to mix above solutions. Then refluxed 45^{o}C maintained six hours finally transfer into ice cold water, grey color crystals separated out. This solid product was recrystallized with ethanol. The above synthesized crystalline corrosion inhibitor (melting point 112^{o}C)[9] denoted as NB4MA.

Mild steel has been used for examine corrosion effectiveness denoted MS. The reaction scheme is shown in Figure 1.

Weight loss method

Corrosion inhibition efficiency found using following equation,

Inhibition efficiency (η) = W_0-W/W_0

Where, W0-MS weight loss without inhibitor, W-weight loss of MS by means of synthesized inhibitor. The various concentration of Schiff base inhibitor was used to examine inhibition efficiency of MS in acidic solutions.

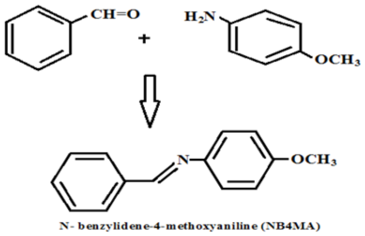

Fig. 1. Schematic diagram of synthesis of Schiff base NB4MA

Electrochemical Measurements

Electrochemical behavior investigated CH1660°C workstation. MS sheet as the working electrode (WE), platinum electrode counter electrode (CE), and saturated calomel electrode (Hg_2Cl_2/sat, KCl) as the reference electrode (RE). Current density of corrosion (I_{corr}) values was obtained in the electrochemical measurements. Tafel plot was used to establish Inhibition efficiency (ηp%) by formula[10].

$$\text{Inhibition efficiency } (\eta \text{ pol}) = \frac{I^0_{corr} - I'_{corr}}{I^0_{corr}}$$

Where, corrosion current densities I^0_{corr} and I'_{corr} were blank and presence of inhibitors respectively. The adsorption isotherm has been used to calculate surface coverage (θ) which can be calculated following equation[11].

$$\theta = \eta pol/100$$

Quantum chemical calculation methods

Density functional theory (DFT) was applied calculate HOMO- LUMO, Electro negativity (χ), and other quantum chemical parameter values in level basis set B3LYP and 6-31G. Gaussian 09W software has been used to conclude the molecule geometry and structure optimization of prepared Schiff base NB4MA inhibitor.

RESULTS AND DISCUSSION

The formation of Schiff base N-Benzylidene-4-Methoxyaniline has been confirmed by following spectroscopic studies and shown in Figure 2, 3 and 4.

The 1048 (C-O aliphatic), 1171 (C-N), 1268 (aromatic C-O), 1514 (C=C), 1654 (C=N), 3032 (C-H). Benzaldehyde IR region C=O 1696 cm^{-1} this peak has been shifted to 1707 cm^{-1} for synthesized schiff base N-Benzylidene-4-Methoxyaniline[12]. ^{1}H NMR: δ 7. 3ppm (Ar-H), signal at 8.35ppm assigned for azomethine proton, δ 3.7ppm ($O-CH_3$)[13].

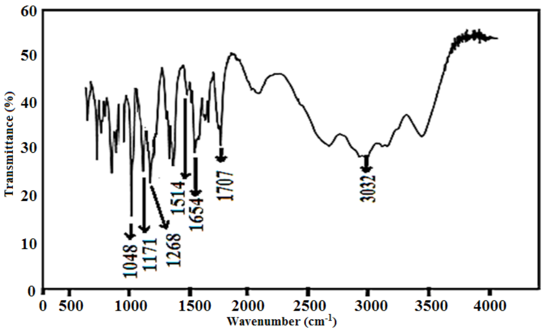

Fig. 2. FT-IR spectra of Schiff base NB4MA

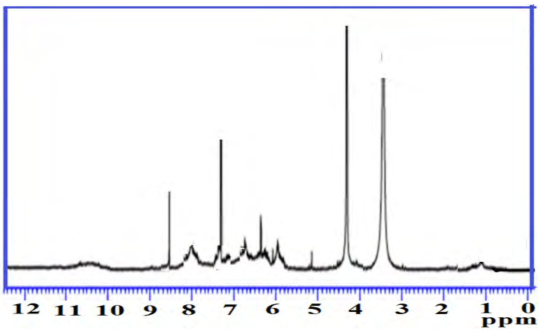

Fig. 3. ^{1}H NMR spectra of Schiff base NB4MA

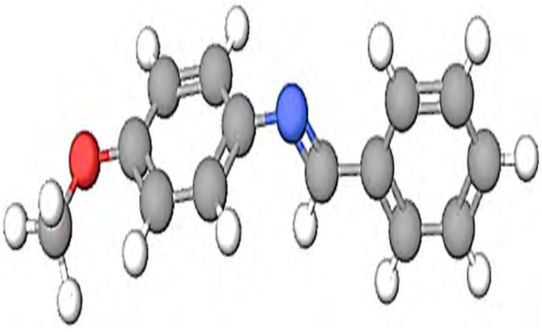

Fig. 4. Molecular structure of schiff base NB4MA

Weight loss measurements

Corrosion inhibition efficiencies examined for molecules NB4MA on MS of 0.1M HCl and 0.5M H_2SO_4, 24 h at 28°C shown in Fig. 5 and inhibition efficiency depicted in Table 1.

Table 1: Weight loss method variation of corrosion inhibition efficiency at various concentrations of NB4MA

Medium	Concentration (ppm)	Inhibitor efficiency (I.E%)
	Blank	---
	50	68.9
1.0 M HCl	100	74.4
	200	78.9
	400	83.4
	600	93.1
	Blank	---
	50	64.5
0.5 M H_2SO_4	100	72.2
	200	75.9
	400	80.2
	600	89.8

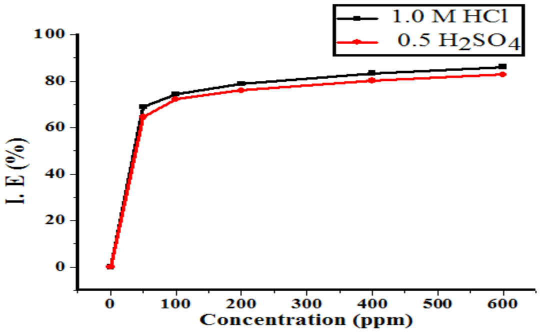

Fig. 5. Corrosion inhibition effectiveness of NB4MA

Corrosion inhibition efficiency was increased with inhibitor concentration and found 93.1% and 89.8% for 1M HCl and 0.5M H_2SO_4 correspondingly. The molecules displayed efficiency on mild steel because of presence of –C=N group and hetero atom attached. Moreover, the azomethine linkage and aromatic ring increases inhibition efficiency at room temperature. The metal surface has been protected since inhibitor was adsorbed causes protective layer formed on the surface of MS corrosion prevention occurs. The concentration of inhibitor increased with inhibition efficiency[14].

Electrochemical parameter

Potentiodynamic polarization studies were done by Tafel extrapolation shown in Fig. 6. It is useful to determination of corrosion potential (E_{corr}), corrosion current density (I_{corr}) and inhibition efficiency (ηpol%). Tafel data is one of polarization studies in between -0.2 to -0.8V with sweep rate of 1 mVs^{-1}. The acidic solutions 1M HCl and 0.5M H_2SO_4 inhibition effectiveness found to be 94.64% and 90% respectively. The maximum efficiency found at concentration of inhibitor as 600ppm.

The electrochemical measurement analyses were shown in Table 2. The results indicated that anodic and cathodic current decreasing with increasing inhibitor concentration because of adsorption of inhibition molecules in mild steel surface. The results from polarization curve βa and βc parameters were decreased since anodic and cathodic process decline. Initially the inhibitor molecules adsorbed on the MS surface make corrosion inhibition in this way of mechanism cathodic hydrogen reaction and blocking active sites on the mild steel surface. Schiff base $-OCH_3$ group contained inhibitor results E_{corr} displacement exceeds about 32 mV which confirmed that studied compound is mixed type inhibitor. The 1M HCl and 0.5M H_2SO_4 acidic solutions in the presence inhibitor the efficiency found to be 85.2% and 81.8% respectively. The maximum efficiency found at concentration of inhibitor as 600ppm[15].

Table 2: Polarization parameters of MS in Schiff base NB4MA presence of acidic solutions

Medium θ	Inhibitor (ppm)	$-E_{corr}$(mV/SCE)	I_{corr}	-βc	βa	η(%)	
	Blank	450	1.17	257	110	-	-
	50	432	0.383	251	111	70.61	0.71
1M HCl	100	448	0.318	249	118	83.75	0.84
	200	460	0.263	397	156	87.64	0.88
	400	459	0.209	235	105	88.19	0.88
	600	466	0.171	176	71.2	94.64	0.95
	Blank	455	2.930	245	143.9	---	---
	50	451	1.116	124	81.9	75.98	0.76
	100	448	0.841	138	69.3	82.05	0.82
0.5 H_2SO_4	200	442	0.729	122	69.5	87.43	0.87
	400	453	0.638	130	75.9	88.37	0.88
	600	450	0.533	124	77.7	90.00	0.90

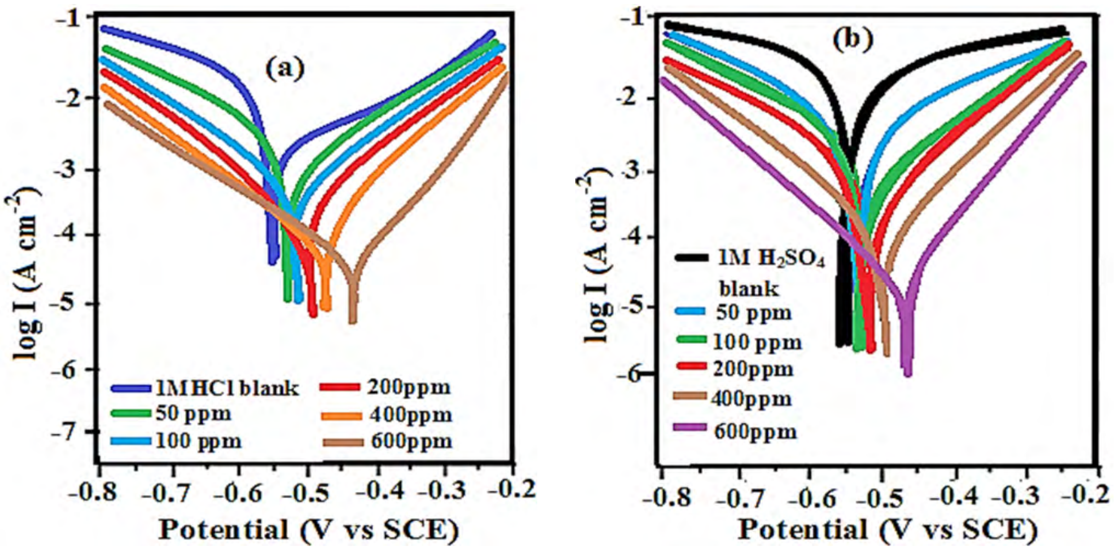

Fig. 6. Tafel plots of MS in NB4MA (a) 1M HCl (b) 0.5M H_2SO_4

Impedance studies

Nyquist plots shown in Fig. 7 and provide the impedance parameter such as charger resistance (R_{ct}), double layer capacitance (C_{dl}) and Inhibition efficiency (%) were specified in Table 3. The gradual decreases of C_{dl} with increasing inhibitor concentration of Schiff base. This was happened since adsorbed inhibitor molecules on surface of MS. These adsorption processes useful for protect MS roughness increases by acid 1M HCl and 0.5M H_2SO_4. The protective layer thickness prolonged those are confirmed by semicircles diameter increase with concentration of inhibitor. These impedance behaviors conclude that the frequency dispersion ascribed roughness and homogeneities of solid surface[16].

Influence of temperatures on inhibition:

The inhibition efficiency at various temperatures range 303-333K were found by using impedance studies shown in Fig. 8 and listed in Table 4. It depicted that efficiency decreased since inhibited molecules desorption takes place. These are confirmed by the electrochemical impedance studies which elaborated charge transfer resistance (R_{ct}) decreased with increasing temperature.

Adsorption Isotherm

Inhibitor adsorption surface of metal has been investigated by Langmuir's adsorption isotherm shown in Fig. 9 (a) and (b) for 1M HCl and 0.5M H_2SO_4 acidic solutions respectively. Schiff base involving chemical reaction on metal surface and transferred atom coverage the surface of MS. Langmuir's adsorption isotherm model has been used under following equation[17].

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C$$

Where, C-Concentration of inhibitor, θ-Fractional surface coverage and K_{ads}-Adsorption equilibrium constant.

Table 3: Impedance parameters of MS in inhibitor NB4MA presence of acidic solutions

Medium	Concentration of the Schiff base (ppm)	R_{ct}(Ohm cm^2)	n	C_{dl}(μF cm^{-2})	η(%)
1.0 M HCl	Blank	19.1	0.912	173	-
	50	60.7	0.926	97	68.5
	100	72.3	0.933	73	73.6
	200	88.8	0.927	48	78.5
	400	110.4	0.973	32	82.7
	600	134.5	0.971	25	85.8
0.5 M H_2SO_4	Blank	12.5	0.909	207	-
	50	34.5	0.917	112	63.8
	100	44.4	0.955	87	71.9
	200	51.6	0.961	55	75.8
	400	61.6	0.947	42	79.7
	600	71.1	0.958	31	82.4

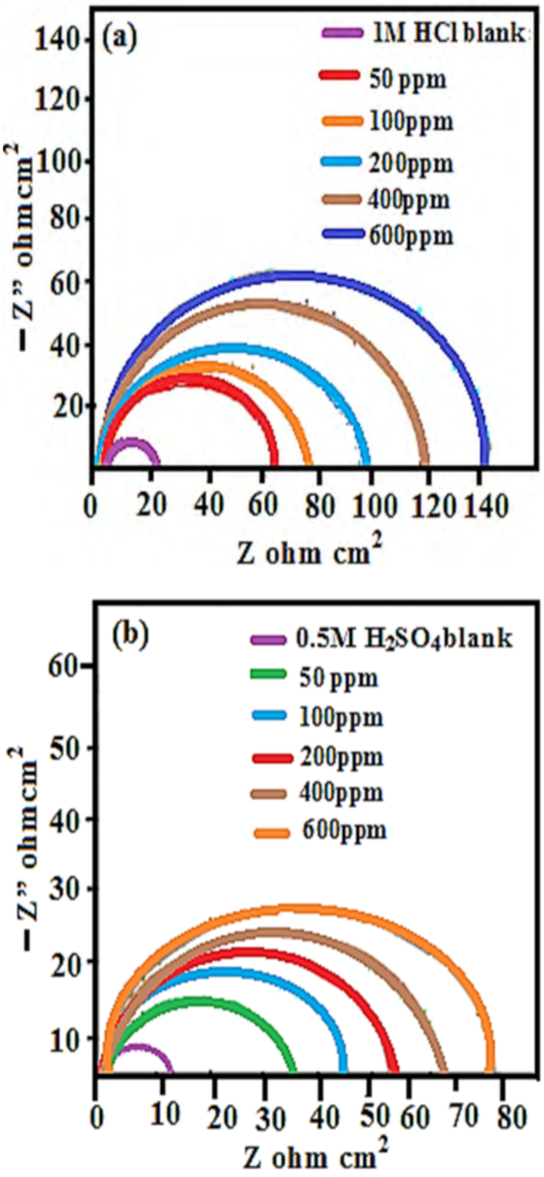

Fig. 7. Nyquist plots of MS in NB4MA (a) 1M HCl (b) 0.5M H_2SO_4

Table 4: Temperature effect of inhibitor NB4MA in mild steel

Temperature (K)	Charge transfer resistance (Ohm cm²) 1M HCl + 600ppm inhibitor	0.5M H_2SO_4 + 600ppm inhibitor
303	134.4	71.10
308	96.60	51.60
313	70.20	37.90
318	51.50	28.10
323	38.10	21.10

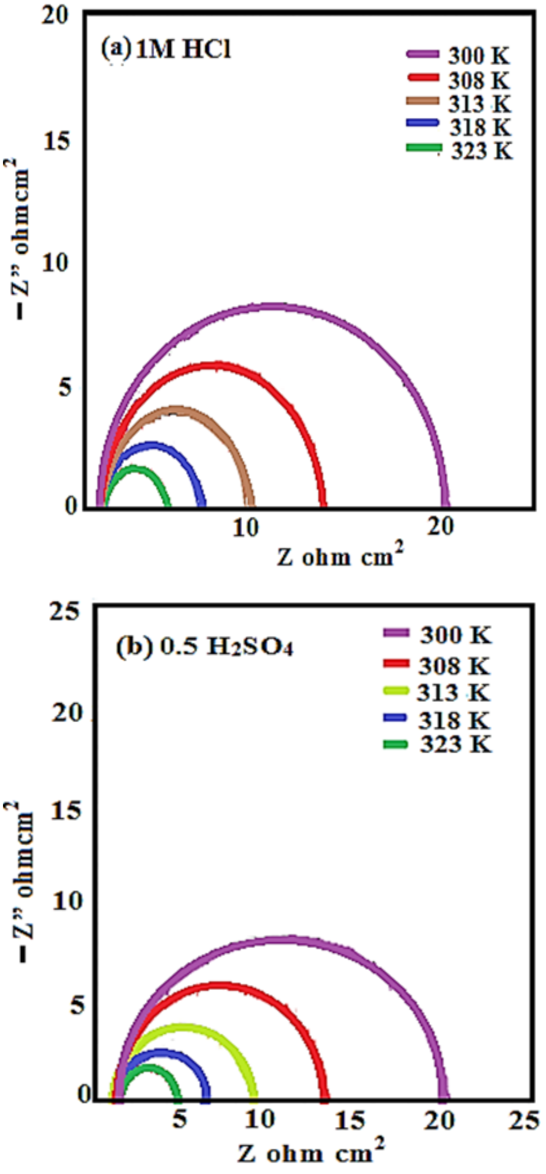

Fig. 8. Nyquist plots of MS in NB4MA of (a) 1M HCl (b) 0.5M H_2SO_4

The linear regression plot of C/θ against C gives correlation co-efficient (R^2) and slope closer to 1 which confirmed that inhibitor and MS and solutions interface follow this type of adsorption isotherm. K_{ads} value calculated by using intercept in straight line of isotherm graph these values further used to calculate energy of adsorption (ΔG_{ads}).

Thermodynamic parameters

Free energy (ΔG^0_{ads})

Thermodynamic parameters Free energy

(ΔG^0_{ads}) has been calculated by.

$$\Delta G^0_{ads} = -RT \ln [55.5 K_{ads}]$$

Where, R-Gas constant, T-absolute temperature, Value 55.5 exposed concentration of water expressed in M.

Activation energy (E_a)

Corrosion activation energy (E_a) exposed using Arrhenius plot as,

$$k = A \exp(-E_a/RT)$$

Where, k-Corrosion rate, E_a-apparent activation energy of the corrosion reaction, R-gas constant and T-absolute temperature and A-Arrhenius pre-exponential factor.

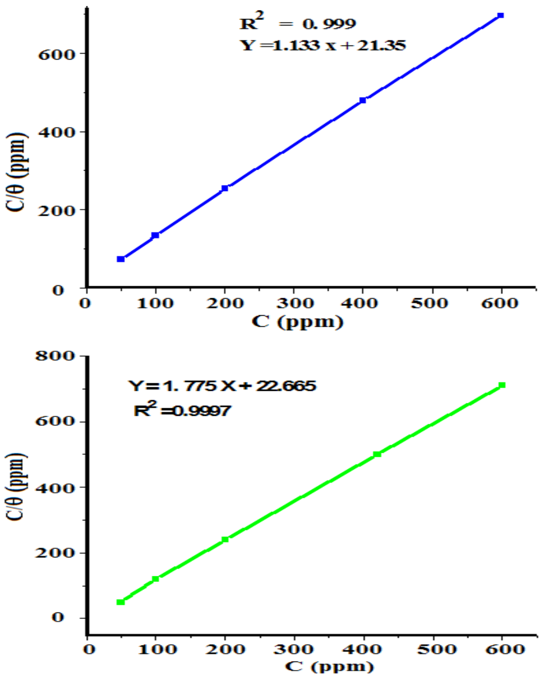

Fig. 9. Langmuir's isotherm of MS with NB4MA (a) 1M HCl (b) 0.5M H_2SO_4

Thermodynamics parameter listed Table 5. The free energy and activation energy parameter revealed that the strong interaction behavior of schiff base confirmed by ΔG^0_{ads} values. The negative sign of ΔG^0_{ads} specify strong interaction behavior between schiff base and MS surface that is high efficient adsorption. Usually, ΔG^0_{ads} value-20kJmol^{-1} or lesser attributed physisorption since electrostatic interaction occurs charged molecules were attracted on metal surface. Meanwhile, ΔG^0_{ads} value shows more negative values than -40kJmol^{-1} since charge transfer forms co-ordinate bond facilitates chemisorption takes place due to electron transfer from molecules to the metal surface.

Table 5: Thermodynamic parameters of MS in NB4MA presence of acidic solutions

Sample	Ea(kJ/mol)	$-\Delta G^0_{ads}$ (kJ/mol)
1.0 M HCl blank	69.13	-
0.5 M H_2SO_4 blank	61.23	-
1.0 M HCl+600 ppm of inhibitor	51.28	-27.09
0.5 M H_2SO_4+600ppm of inhibitor	49.45	-26.95

The synthesized Schiff base ΔG^0_{ads} found value found -27.09 and -26.95 for 1.0M HCl and 0.5M H_2SO_4 correspondingly in the presence of 600 ppm concentration of inhibitor has been used. The ΔG^0_{ads} higher than -20kJmol^{-1} and not exceeds -40 kJmol^{-1} which concludes physisorption obeyed. However, another one thermodynamic parameter activation energy (E_a) of blank solution is higher than presence of inhibitor confirms adhere chemisorption since electron transferred from inhibitor metal surface forms co-ordinate type of bond. These concludes Schiff base initially involving electrostatic attraction further formed co-ordinate type of bond hence synthesized Schiff base adsorption mechanism on mild steel surface might be equally physisorption and chemisorption observed[18].

Quantum chemical studies

The Schiff bases HOMO and LUMO geometry are shown in Fig. 10. Quantum chemical parameters were calculated by the means of DFT method and listed in Table 6. The Frontier molecular orbital (FMO) theory concludes reactant HOMO and LUMO involving in interaction causes reaction is possible because EHOMO and ELUMO have electron donor and acceptor capability respectively. The quantum chemical parameters including ionization potential and electron affinity were determined by HOMO and LUMO behavior.

The benzylidene Schiff bases has been synthesized and investigated corrosion behavior in 1M HCl reported by U. J. Naik *et al.*,[19] and suggested that electron donating ability $\Delta N<3.6$ attributed metal surface has been protected by the inhibitor. The inhibition efficiency enhanced since aromatic ring has $–OCH_3$ and –OH groups present in the Schiff base.

Table 6: Quantum chemical parameters of NB4MA

Quantum chemical parameters	Values
E_{HOMO}(eV)	-8.583
E_{LUMO}(eV)	-0.752
ΔE(eV)	7.831
Ionization potential (I)	8.583
Electron affinity (A)	0.752
Hardness (η)	3.916
Softness (σ)	0.1277
Electro negativity(χ)	4.668
Electrophilicity index (ω)	2.782
Fraction of electron transferred (ΔN)	0.2978
Dipole moment (*μ*)	1.553

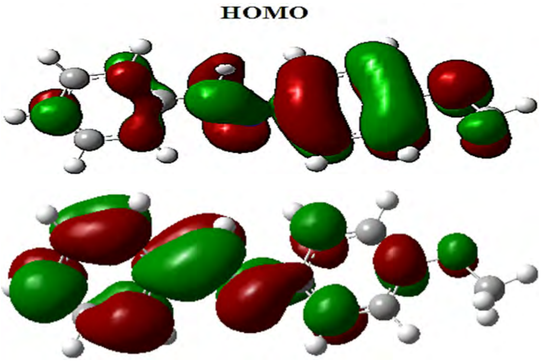

Fig. 10. HOMO and LUMO of NB4MA

In our synthesized schiff base NB4MA quantum chemical parameter electron transfer ability (ΔN) value was found 0.2978 which confirms surface electron donating ability of inhibitor increased. The molecules electronic charge density diffused completely especially electronic density present in azomethine nitrogen as well as benzene aromatic ring. For that reason, synthesized Schiff bases have enhanced electron density hence higher coverage capacity of mild steel formed enormous inhibition efficiency[20].

SEM analysis

Figure 11 (a) and (b) showed scanning electron microscopy (SEM) images of MS present inside 1M HCl and 0.5M H_2SO_4 correspondingly. These exposed that the surface of mild steel scratched or damaged in the presence of acidic solutions. Meanwhile, Fig. 11 (c) and (d) shows morphology of MS presence of HCl and H_2SO_4 of 600 ppm concentration of inhibitor NB4MA respectively. It revealed that mild steel dissolution efficiency reduced and smooth surface shown since adsorption of inhibitor forms protective film on the metal surface.

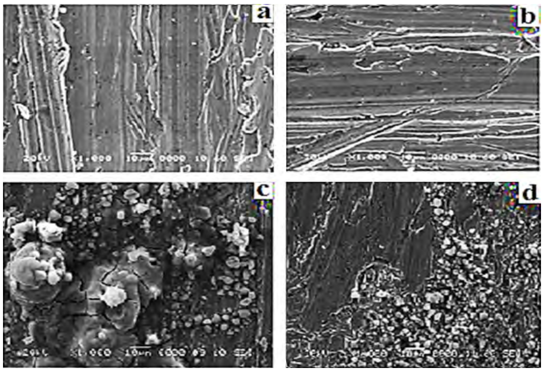

Fig. 11. SEM of (a) MS in HCl (b) MS in H_2SO_4 (c) MS in HCl + inhibitor (600ppm) (d) MS in H_2SO_4 + inhibitor (600ppm)

CONCLUSION

N-Benzylidene-4-Methoxyaniline Schiff base has been synthesized and studied their inhibition performance of mild steel. Further inhibition capacity investigated in existence of acidic solutions 1M HCl and 0.5 M H_2SO_4 with a various range concentration of inhibitor NB4MA. Weight loss method confirmed that inhibition efficiency has been enhanced with concentration of the inhibitors. The polarization and impedance studies confirm that both anodic as well as cathodic type of mixed inhibitor and inhibition capacity decreased with enhancing temperature. Thermodynamic parameter reveals that possible for both physisorption and chemisorptions process since-ΔG^0_{ads} about 20kJmol^{-1} and activation energy decreased after adding inhibitor respectively. Quantum chemical parameter studies attributed that electronic charge density dispersing in the molecule causes induced corrosion efficiency for protect mild steel. SEM images shows smooth surface appeared in the presence Schiff base inhibitor because of protective layer formed surface of mild steel.

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