Investigation of Corrosion Inhibitive Property of Morus Nigra L. + Mn^{2+} System in Neutral Medium

P. Leema Sophie, Noreen Anthony, and X. Prisil Naveentha

Abstract— This research work is concerned with the development of new ecofriendly inhibitor system which functions efficiently for the corrosion control of carbon steel in aqueous environment containing 180 ppm of Cl^- ions. The inhibition efficiency of corrosion of carbon steel in neutral, aqueous environment containing 180 ppm of Cl^- ions in DD water by leaf extract of plant *Morus nigra L.* which contains active constituents that exhibit synergistic property with Mn^{2+} ions has been investigated. 300 ppm of the leaf extract worked in conjunction with 75 ppm of Mn^{2+} ions in controlling the corrosion of carbon steel immersed in chloride ion solution. A film is formed on the surface of the carbon steel immersed in the inhibitor system containing the extracts of the plant *Morus nigra L.* and Mn^{2+} ions. FTIR spectra shows that a strong interaction has taken place between the molecules present in the plant extract and the metal. In order to investigate the nature of the protective film UV-visible spectroscopy, scanning electron microscopy (SEM), FTIR spectroscopy and electrochemical studies have been used in the present study. Polarisation techniques have been used to determine the nature of inhibitor. Based on the results obtained from the mass-loss method, polarisaion studies and surface analysis studies a suitable mechanism for the corrosion inhibition has been proposed.

Key Words—Carbon Steel, Inhibitor, Inhibition Efficiency, Plant Extract, Polarisation Studies, SEM, Spectroscopic Studies

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1 Introduction

THE word corrosion is as old as earth, but it has been known by different names[1]. Corrosion is commonly known as rust, an undesirable phenomenon which destroys the beauty and luster of the objects and shortens their lives. Corrosion is a natural phenomenon and it is due to the high energy content of elements in the metallic form. Hence when exposed to environment it tends to release this stored energy through the process of corrosion. Over the years, considerable efforts have been deployed to find suitable corrosion inhibitors of organic origin in various corrosive media. The role of inhibitors is to form a barrier of one or several molecular layers against acid attack. This protective action is often associated with chemical and physical adsorption involving a variation in the charge of the adsorbed substance and transfer of charge from one phase to the other. Sulphur and nitrogen-containing heterocyclic compounds with various substituents are considered to be effective corrosion inhibitors[16]. These inhibitors decrease or prevent the reaction of the metal with the media. Of these, only very few are actually used in practice, owing to several factors including cost and amount, easy availability and the safety to the environment and its species need to be considered when choosing an inhibitor. The present study aims to develop a suitable corrosion control method using the leaves **extracts of *Morus Nigra L.*[**12] which are readily and locally available for cooling water system with a view of their application in industrial plants. The use of natural products as corrosion inhibitors can be traced back to the 1930's when plant extracts of *Chelidoniummajus* (Celandine) and other plants were used for the first time in H_2SO_4 pickling baths. Abdel-Gaber et al[8] have studied the effect of extracts of chamomile (Chamaemelum mixture L.), Halfabar (Cymbopogonproximus) and kidney bean plants on the corrosion of steel in aqueous 1 M sulphuric acid by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization techniques.

James and Akaranta[9] have carried out the study of the inhibition of the corrosion of zinc by acetone extract of red onion skin in hydrochloric acid solutions by weight loss method. The inhibition efficiency of alcoholic leaf extract of Jatropha curcas on Brass (Cu-40Zn) in 1 N hydrochloric acid has been studied by Deepa Rani and Selvaraj[15] using weight loss measurements. The inhibition efficiency increased with an increase of inhibitor concentration but decreased with rise in temperature and exposure time. The present study aims at studying the effect of *Morus Nigra L.* plant extract to prevent/minimize corrosion, and scale formation.

2 Methodology

- The inhibitor chosen for the present study is the leaf extracts of the plant mulberry. The scientific name of mulberry is *Morus Nigra.L*, a genus belonging to the Moraceae family of the Urticales subclass. Mulberry is usually associated with sericulture, the production of silk through the silkworm
- The objective of the present work is to study the inhibition efficiency of corrosion of carbon steel in neutral, aqueous environment containing 180 ppm of Cl^- ions in DD water by leaf extract of *Morus Nigra L.* which contains active constituents that exhibit synergistic property with Mn^{2+} ions

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The environment chosen is neutral aqueous medium. All the experiments are carried out at room temperature which is 30 ±1 °C. In order to investigate the nature of the protective film, UV-visible, scanning electron microscopy, FTIR spectroscopy and electrochemical methods have been used in the present study. The mechanistic aspects of corrosion inhibition are based, in a holistic way, on the results obtained from the classical mass-loss method, the electrochemical methods and different spectroscopic techniques mentioned above.

The results of literature review shows that the extract of the leaves of *Morus nigra L.* contains the most important bioactive constituents such as steroids, flavonoids, alkaloids, tannins, terpenoids, glycoside and saponins, coumarins and amino acids.

These active constituents present in the extract are responsible for the corrosion inhibition efficiency of the carbon steel immersed in 180 ppm of chloride ion solution. Application of preliminary screening of the leaf extract for the presence and absence of saturation/unsaturation, aromatic nature/aliphatic nature, elements present and the nature of the functional groups present indicate that the leaf extract contains:

S.No	Tests	Findings
1	Elements	Nitrogen
2	Unsaturation	Positive
3	Aromatics	Positive
4	Carboxylic acids	Positive
5	Carbonyl groups	Negative
6	Phenolic group	Positive
7	carbohydrates	Positive
8	Amides	Positive
9	Amines	Positive
10	Nitrogroups	Negative

3 Research Methods

3.1. Analysis of the Results of the Mass-Loss Method

The corrosion inhibition efficiencies (IE) of extract of the leaves of *Morus nigra L.* in controlling the corrosion of carbon steel in 180 ppm chloride ion solution in the absence and presence of 75 ppm of Mn^{2+} by the mass-loss method[2] are given Table-1 and in Figure-1. It is evident from the table that the corrosion rate decreases on increasing the concentration of the leaf extract from 0 to 300 ppm in the presence of 75 ppm of Mn^{2+} ions. The inhibition efficiency of the leaf extract is due to the presence of phytoconstituents[14] present in the extract. The extract of mulberry leaves contain steroids, flavonoids, alkaloids, tannins, terpenoids, glycoside and saponins, coumarins and amino acids[3]. The presence of lone pair of electrons on the hetero atoms of these compounds facilitates the formation of co-ordinate bond with the metal. Most of the effective organic inhibitors used in industry have heteroatom such as O, N, and S along with multiple bonds in their molecules through which they are adsorbed on the surface of the carbon steel.

300 ppm of the leaf extract alone offers 51.43% IE and 75 ppm of of Mn^{2+} ions offers 50.53% IE. But their combination offers 81.31% IE in 180 ppm of chloride ion solution. This is found to be maximum IE offered by the system. This shows that the Mn^{2+} exhibited synergistic effect with active constituents present in the extract of the leaves of *Morus nigra L.* The synergism is due to the formation of complex with the phytochemicals present in the extract of *Morus nigra L.* A thin film was observed on the surface of the metal. No rust or scale could be seen on the surface of the specimen piece, which shows that the inhibitor formulation acts as a rust or scale removing agent.

Table-1

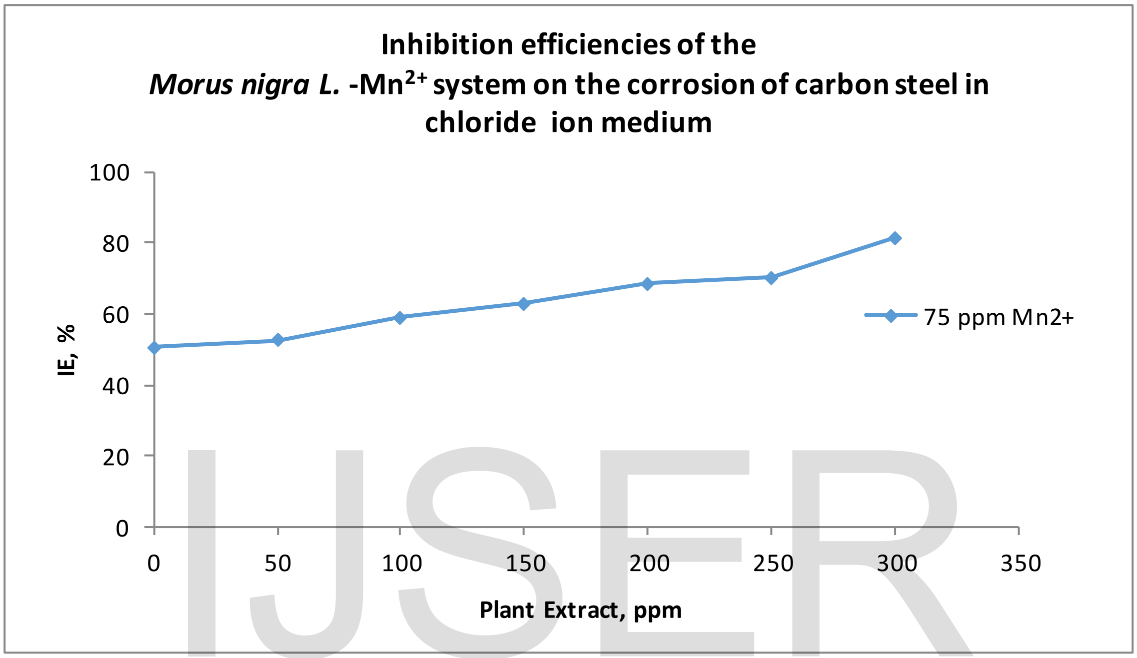
Inhibition efficiencies (IE) of carbon steel in aqueous environment (Cl^- = 180 ppm) in the presence and absence of the inhibitor system and the inhibition efficiencies obtained by the mass-loss method

Inhibitor system: *Morus nigra L.* + Mn^{2+}
Immersion period: 1 day

S.No	Leaf Extract of *Morus nigra L.*, ppm	Mn^{2+}, ppm	Corrosion rate, mdd	Inhibition Efficiency,%
1	0	0	39	-
2	0	75	19.29	50.53
3	50	75	18.49	52.38

4	100	75	16.00	58.97
5	150	75	14.47	62.89
6	200	75	12.24	68.61
7	250	75	11.38	70.28
8	300	75	7.28	81.31
9	300	0	18.94	51.43

Figure-1



3.2. Analysis of the Results of the Gasometric Method

The gasometric method measures the volume of hydrogen gas evolved from the reaction system. Table -2 presents the hydrogen gas evolved when the carbon steel is immersed in the 180 ppm of chloride ion solution in the presence and the absence of the inhibitor system and the inhibition efficiency obtained from gasometric method[13] for carbon steel in 180 ppm Cl^- ion solution containing 75 ppm of Mn^{2+} and for 300 ppm of the extract of *Morus nigra L.* It is observed that 7.5 ml of hydrogen gas is evolved with 180 ppm Cl^- ion solution without the inhibitor system and the volume of hydrogen gas decreases considerably on introducing the inhibitor system (75 ppm of Mn^{2+} and 300 ppm of the plant extract) into the aqueous medium containing 180 ppm of chloride ions. The volume of hydrogen gas evolved is found to be 1ml and the inhibition efficiency is found to be 86.66%. It is evident that the leaf extract of *Morus nigra L.* with Mn^{2+} has better ability to inhibit the corrosion of carbon steel in 180 ppm Cl^- ion solution. Thus the cathodic reaction is reduced in the carbon steel in the presence of the inhibitor system. As the concentration of plant extract increases the hydrogen evolution decreases. So, it is concluded that the cathodic reaction is decreased and hence the corrosion is prevented.

Table - 2

Volume of hydrogen evolved and Inhibition efficiency obtained from gasometric method for carbon steel in aqueous environment (Cl^-= 180 ppm) in the presence and absence of the inhibitor system.

Inhibitor system: *Morus nigra.L* + Mn^{2+}

***Morus nigra L.*, (ppm)**	**Mn^{2+},(ppm)**	**Volume of hydrogen gas evolved (ml)**	**Inhibition efficiency, (%)**

0	0	7.5	0
50	75	6.0	20
100	75	5.5	20.66
150	75	4.0	40.6
200	75	3.5	53.3
250	75	2.0	73.3
300	75	1.0	86.66

3.3. Electrochemical Studies

3.3.1. Analysis of the Results of Potentiodynamic Polarization Studies

The potentiodynamic polarization curves[17] of carbon steel immersed in 180 ppm of Cl^- ion solution in the presence and absence of Mn^{2+} and *Morus nigra L.* leaf extract are given in Figure 2. The corrosion parameters of carbon steel immersed in 180 ppm Cl^- ion solution in the presence and absence of 75 ppm of Mn^{2+} and 300 ppm of *Morus nigra L.* leaf extract are given in Table-3. When carbon steel is immersed in 180 ppm Cl^- ion solution, the corrosion potential (Ecorr) is -0.7789 V vs SCE and the corrosion current is $1.923X10^{-5}$ A/cm². When 75 ppm of Mn^{2+} and 300 ppm of *Morus nigra L.* are added to the solution containing 180 ppm of chloride ion, the corrosion potential is found to be -0.7964 V vs SCE. The corrosion current $2.097x10^{-6}$ A/cm². The decrease in the corrosion current on the addition of the inhibitor indicates that the formulation functions as a mixed inhibitor and reduces the corrosion of carbon steel in 180 ppm of chloride ion solution. The anodic slope changes from 1.459V/dec to 1.444 V/dec and the cathodic slope changes 0.385V/dec to 0.145 V/dec. The rate of corrosion decreases from 1.288mm/y to 0.1405mm/y on the addition of the inhibitor system to the 180ppm of chloride ion environment. The large difference in the cathodic slope indicates that the inhibitor controls the cathodic reactions predominantly. The inhibitor system is mixed inhibitor controlling the cathodic reactions predominantly.

Table - 3

Results of Potentiodynamic Polarization Studies

Medium: Chloride ion solution (180 ppm) in DD water

Inhibitor system: *Morus nigra L.* **(300 ppm) + Mn^{2+} (75 ppm) + 180 ppm Cl^- ion in DD water**

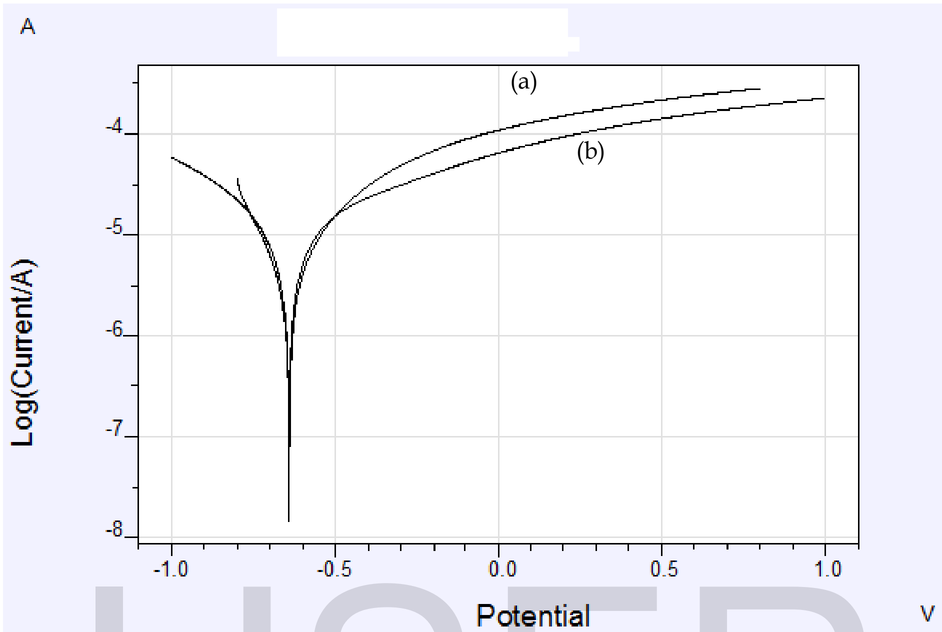
S.No	Environment in DD water	E_{corr} V	R_p in ohms	b_a(V/dec)	b_c (V/dec)	I_{corr}(A/cm²)	Corrosion rate, mm/y
1	180 ppm of Chloride ion	-0.7789	8021	1.459	0.385	$1.923X10^{-5}$	1.288
2	180 ppm of Cl^- + 300 ppm *Morus nigra L.*+ 75 ppm Mn^{2+}	-.7964	10360	1.444	0.145	$2.097x10^{-6}$	0.1405

Figure - 2

Potentiodynamic Polarisation Curves of Carbon Steel

Medium: Chloride ion solution (180 ppm) in DD water

Inhibitor system: *Morus nigra L.***(300 ppm) + Mn^{2+} (75 ppm) + 180 ppm Cl^- ion in DD water**



(a) Chloride ion solution (180 ppm) in DD water

(b) *Morus nigra L.*(300 ppm) + Mn^{2+} (75 ppm) + 180 ppm Cl^- ion in DD water

3.3.2. Analysis of the Results of AC Impedance Studies

The AC impedance spectra of carbon steel immersed in 180 ppm chloride solution containing the inhibitor formulation are shown in Figure-3. The AC impedance parameters namely charge transfer resistance and the double layer capacitance are given in Table-4. When carbon steel is immersed in 180 ppm chloride solution the R_{ct} value is found to be 233.2Ωcm². The C_{dl}value is 9.763 x10^{-6}F/cm². When 75 ppm of Mn^{2+} and 300 ppm of *Morus nigra L.* leaf extract are added, the R_{ct} value is increased to 293.3 Ωcm² and the C_{dl} value decreased to 5.328X10^{-6}F/cm². The increased R_{ct} values and decreased double layer capacitance values obtained from impedance studies justify the good performance of the extract with Mn^{2+} as an inhibitor in Chloride ions in DD water. This behavior means that the film obtained acts as a barrier to the corrosion process and proves the existence and the formation of the film.

Table - 4

Results of AC Impedance Studies

Medium: Chloride ion solution (180 ppm) in DD water

Inhibitor system: *Morus nigra L.***(300 ppm) + Mn^{2+} (75 ppm) + 180 ppm Cl^- ion in DD water**

S.No.	Environment in DD water	*Morus nigra. L* (ppm)	Mn^{2+} (ppm)	R_{ct} (Ω cm²)	C_{dl} (F/cm²)

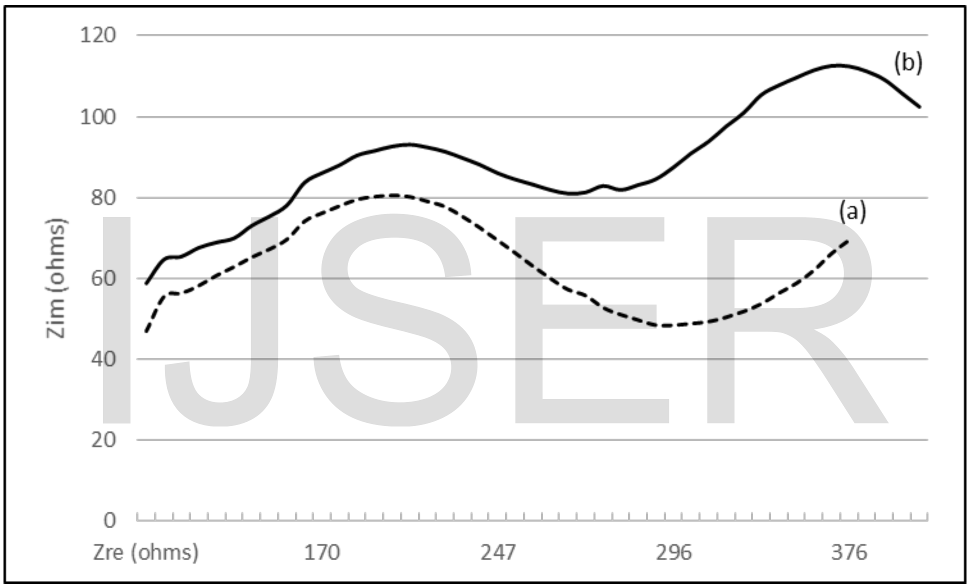
1	Chloride ion in DD water	0	0	233.2	9.763×10^{-6}
2	Cl^- + 300 ppm *Morus nigra L.* + 75 ppm Mn^{2+}	300	75	293.3	5.328×10^{-6}

Figure - 3

AC Impedance of Carbon Steel

Medium: Chloride ion solution (180 ppm) in DD water

Inhibitor system: *Morus nigra L.***(300 ppm) + Mn^{2+} (75 ppm) + 180 ppm Cl^- ion in DD water**



(a) Chloride ion solution (180 ppm) in DD water

(b) *Morus nigra L.*(300 ppm) + Mn^{2+} (75 ppm) + 180 ppm Cl^- ion in DD water

3.4. Spectroscopic Studies

3.4.1. Analysis of FTIR Spectra

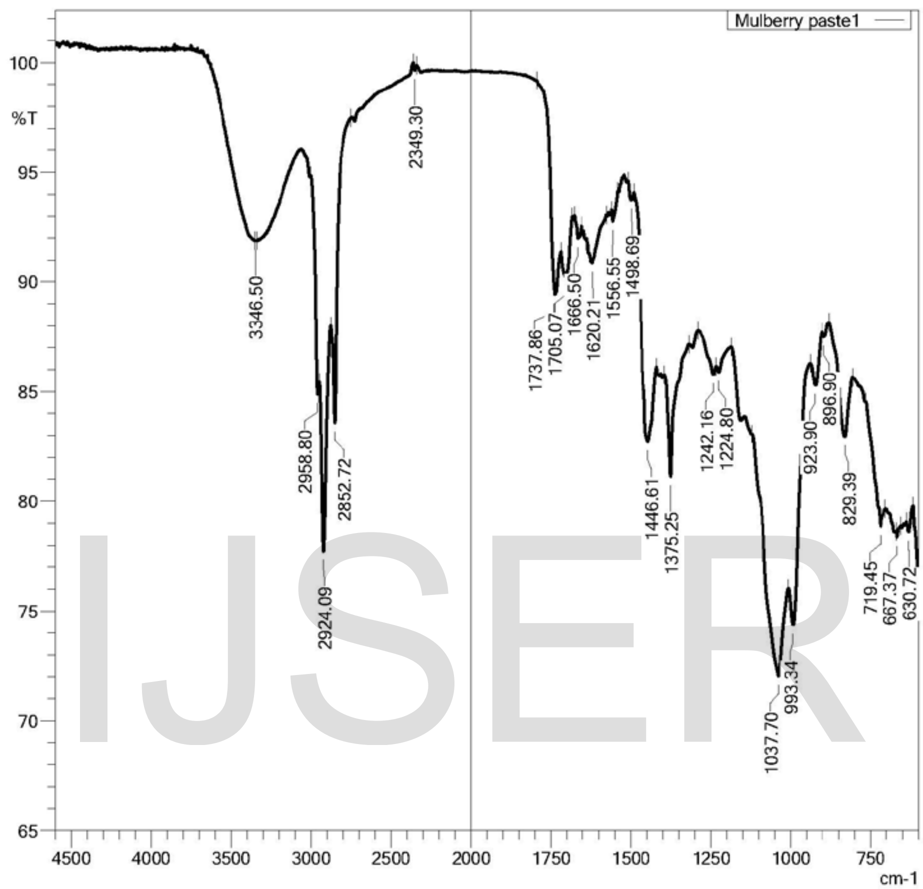
The FTIR spectrum of the pure extract of *Morus nigra L.* is shown in Figure 4a. The band at 3346.50 cm^{-1} is due to the presence of –OH. The bands at 1737.86 cm^{-1} and 1446.61 cm^{-1} are due to the coupling of -C-O stretching and -C-O-H inplane bending vibration of the carboxylate anion. The band at 1037.70 cm^{-1} is due to -C-O ring vibration.

Figure 4b shows the FTIR spectrum[19] of the thin film formed on the surface of the carbon steel immersed in 180 ppm of chloride ion solution containing the inhibitor system. The -OH frequency of the extract at 3346.50 cm^{-1} is shifted to 3381.21 cm^{-1}. The bands at 1737.86 cm^{-1} and 1446.61 cm^{-1} which are due to the coupling of -C-O stretching and -C-O-H inplane bending of the carboxylate anion are shifted to 1635.64 cm^{-1} and 1550.25 cm^{-1}. The band at 1037.70 cm^{-1} (due to the ring oxygen) is shifted to 1020.34 cm^{-1}. The shifting of the bands in the thin film indicates that the carboxylate anion and the ring oxygen present in the phyto constituents are responsible for the interaction between the phytochemicals present in the extract of the leaves of *Morus nigra L.* and iron present in the carbon steel.

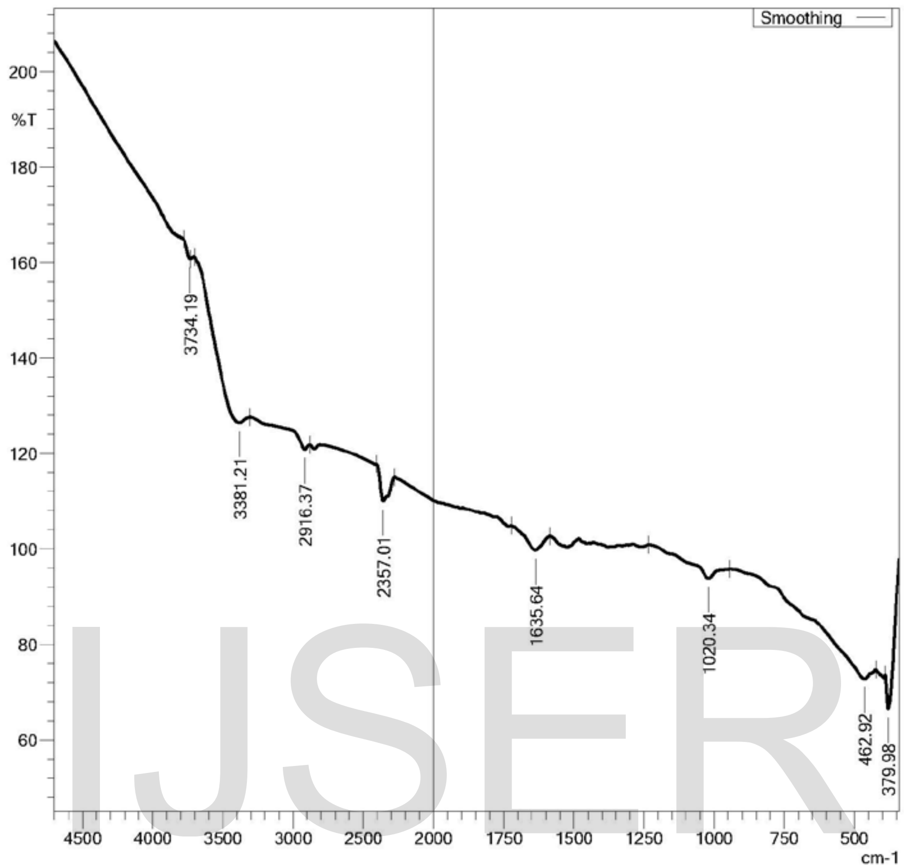
Figure – 4

FTIR Spectrum (Kbr) (a) Pure *Morus Nigra L.*And (b) Thin Film Formed On The Surface Of The Carbon Steel Immersed In 180 ppm Cl^- Ion Containing The Inhibitor System

(a)



(b)



3.4.2. UV-Visible Spectral Study

The UV-Visible spectrum of Mn^{2+}, Fe^{2+}, leaves extract of *Morus nigra L.* + Mn^{2+} in the presence of *Morus nigra L.* leaf extract, and Fe^{2+} in the presence of *Morus nigra L.* leaf extract in 180 ppm Cl^- ion solution are given in Figure 5a-e. The absorbance of leaves extract of *Morus nigra L.* at 287 nm is found to be 0.520 (Figure5b). On increasing the λ, initially the absorbance decreases and reaches 0.507 at 320 nm and at 673 nm the absorbance is found to be 0.058. The absorbance of Mn^{2+} ion is found to be 0.022 at 381nm and it increases with decrease in λ (5a).

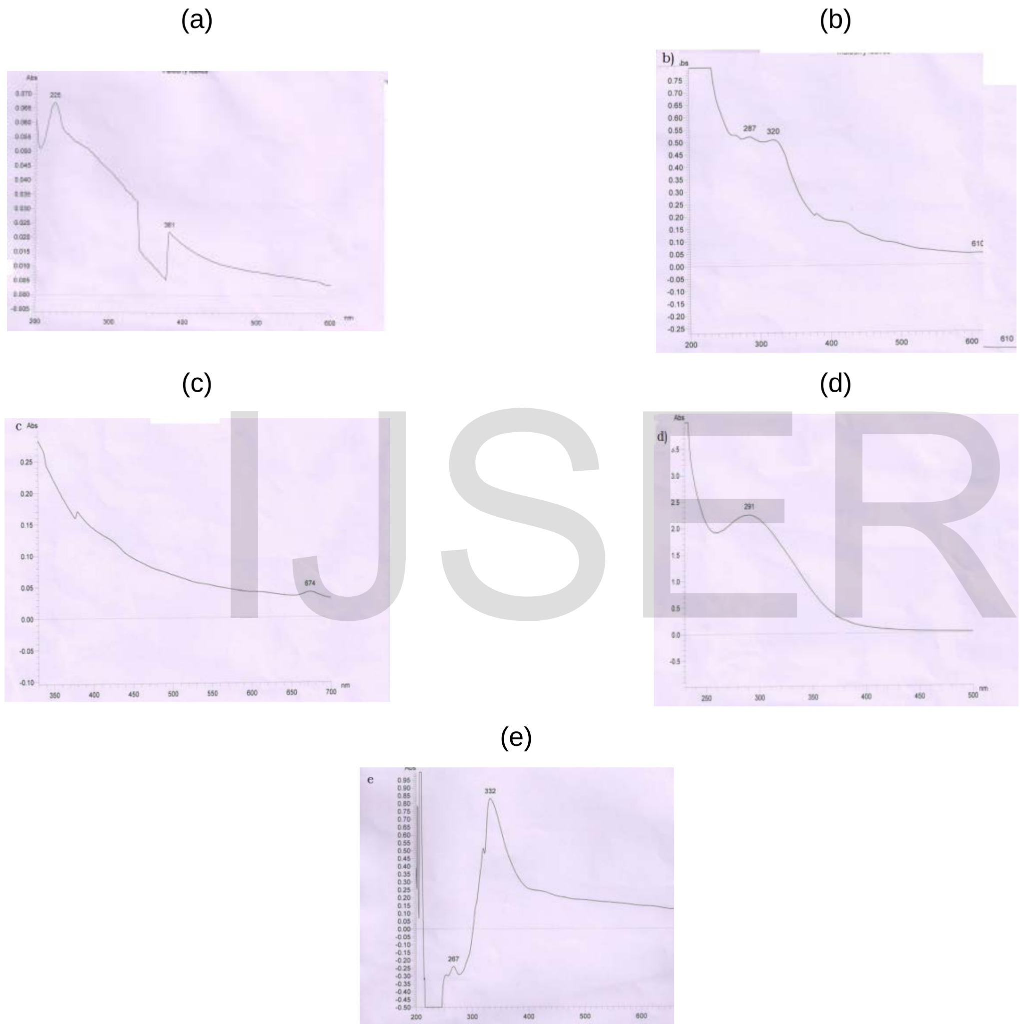
The addition of 75 ppm of Mn^{2+} to 300 ppm of *Morus nigra L.* increases the absorbance value. The absorbance is found to be 0.731 at 320nm 0.736 at 288nm. (Figure 5c). The higher value of absorbance in Mn^{2+}+ *Morus nigra L.* constituents indicates the existence of strong interaction between Mn^{2+} and phytoconstituents present in the leaf extracts of *Morus nigra L.*

UV-visible spectrum of Fe^{2+} (Figure 5d) shows that at 291 nm the solution has an absorbance of 2.249. The absorbance decreases with increase in λ. When Fe^{2+} added to *Morus nigra L.*the absorbance is found to be 0.830at 332nm (Figure 5e) then decreases gradually upto 836nm and then remains constant. This clearly proves the existence and formation of a complex between Fe^{2+} and phyto constituents present in *Morus nigra L.*

Figure – 5(a-e)

UV-Visible Absorption Spectra of the Test Solutions in 180 ppm Cl^- Ion

(a) Mn^{2+}, b) *Morus nigra L.* (c) *Morus nigra L.* + Mn^{2+} (d) Fe^{2+} ionand (e) *Morus nigra L.* + Fe^{2+}



3.5. SEM observation

The texture and pore structure of the inhibited and uninhibited surface of the carbon steel immersed in 180 ppm of chloride ion solution in neutral medium in the presence and absence of the inhibitor system are shown in Figures 6a and 6b. From Figure 6b it is confirmed that the inhibitor has formed a dense film over the metal surface in neutral medium.

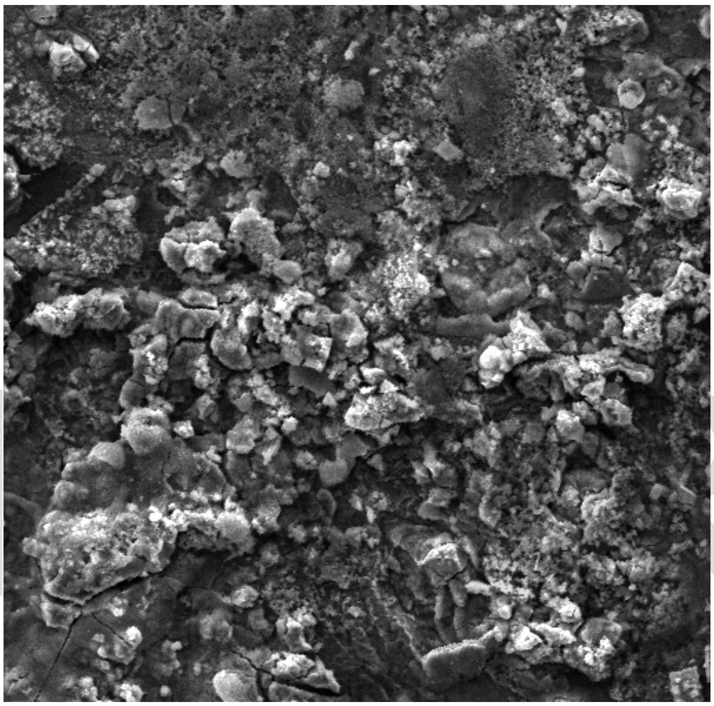
Figure - 6

SEM image of

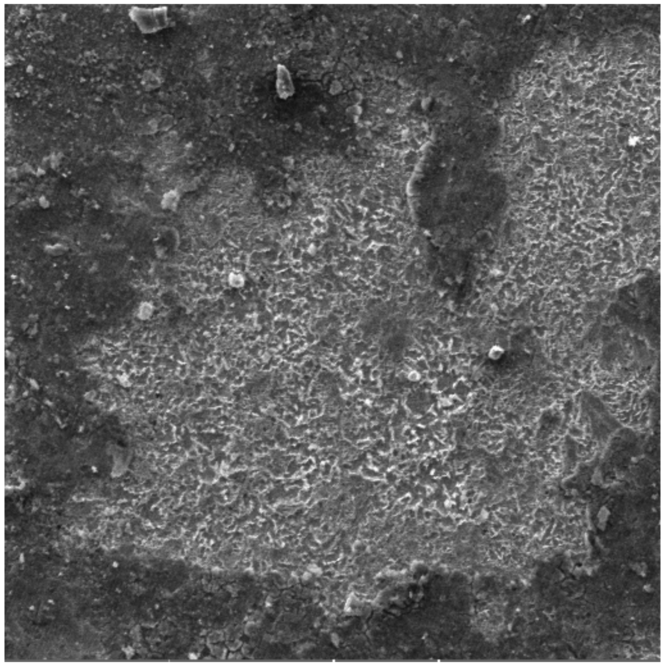
(a) Carbon steel in 180 ppm Cl^- ion and

(b) Carbon steel in 180 ppm Cl^- ion + *Morus nigra L.*+ Mn^{2+}

(a)



(b)



4 Mechanism of corrosion inhibitor

1. Analysis of the results of the mass-loss method shows that the formulation consisting of 300 ppm extract of the leaves of *Morus nigra L.* and 75 ppm Mn^{2+} has 81.31% IE in controlling the corrosion of carbon steel immersed in 180 ppm Cl^- ion solution. A synergistic effect exists between Mn^{2+} and the phytoconstituents present in the extract of *Morus nigra L.*
2. Results of polarization studies show that this inhibitor controls both anodic and cathodic reactions, but behaves predominantly as a cathodic inhibitor.
3. AC impedance spectra reveals the formation of protective film on the metal surface.
4. The FTIR spectra confirms the presence of active constituents on the metal surface and also the change in nature of the active constituents.
5. The UV-visible absorption spectra indicate the possibility of formation of a film with the extract and the iron ions.
6. The SEM micrographs confirm the formation of protective layer on the metal surface.

Based on the above facts the following mechanism is proposed

1. The inhibition of corrosion of carbon steel in neutral aqueous chloride ion solution in the presence of the inhibitor system is due to the interaction between the lone pair of electrons on oxygen atoms present in the active constituents of the extract with the positively charged metal surface.
2. The protective film consists of iron-complex
3. The IE of the inhibitor formulation depends on the ability of the inhibitor to form complex with Mn^{2+} and the ability of Fe^{2+} to react with Mn^{2+} complex to form iron complex on the surface of the specimen.

5 Summary

1. Analysis of the results of the mass-loss method shows that the formulation consisting of 300 ppm extract of the leaves of *Morus nigra L.* and 75 ppm Mn^{2+} has 81.31% IE in controlling the corrosion of carbon steel immersed in 180 ppm Cl^- ion solution. A synergistic effect exists between Mn^{2+} and the phytoconstituents present in the leaf extracts of*Morus nigra L.*
2. Results of polarization studies show that this inhibitor controls both anodic and cathodic reactions, but behaves predominantly as a cathodic inhibitor.
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4. The FTIR spectra confirms the presence of active constituents on the metal surface and also the change in nature of the active constituents.
5. The UV-visible absorption spectra indicate the possibility of formation of a film with the extract and the iron ions.
6. The SEM micrographs confirm the formation of protective layer on the metal surface.

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