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Corrosion inhibition of *Vitex negundo* extract as a green corrosion inhibitor for carbon steel in well water

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The inhibition efficiency (IE) of an aqueous extract of *Vitex negundo*, in controlling corrosion of carbon steel in well water has been evaluated by the weight loss method. The *Vitex negundo* extract shows good IE. In the presence of Zn^{2+} excellent IE is shown by the *Vitex negundo* Extract. A synergistic effect exists between the *Vitex negundo* extract and Zn^{2+}. The mechanistic aspects of corrosion inhibition have been investigated by polarization study, AC impedance spectra. Polarization study reveals that the formulation consisting of *Vitex negundo* extract and Zn^{2+} functions as mixed inhibitor with predominantly cathodic effectiveness. AC impedance spectra reveal that a protective film is formed on the metal surface. The active principle in the *Vitex negundo* extract is 5-hydroxy-2-(3-hydroxy-4-methoxyphenyl)-3, 6, 7 -trimethoxychromen-4-one. The protective film formed on the metal surface has been analyzed by FT-IR, SEM and EDAX Spectra. It is found that the protective film consist of Fe^{2+} - 5-hydroxy-2-(3-hydroxy-4-methoxyphenyl)-3,6,7-trimethoxychromen-4-one complex and $Zn(OH)_2$.

Keyword: Corrosion, Carbon steel, *Vitex negundo*, Synergistic effect, FT-IR, SEM, EDAX.

1. Introduction

Corrosion is a surface phenomenon in which the metal or alloy is attacked by air, water or soil through a chemical or electrochemical reaction to form more stable compounds. We need to use the inhibitors to avoid the wastage of metal by means of corrosion and usage of environmentally friendly corrosion inhibitors makes this approach a sustainable one. Therefore, our only hope is to depend upon plants as an inhibitor. Various parts of plants are used as inhibitors such as seeds, fruits, leaves, flowers etc. These corrosion inhibitors are biodegradable and have no heavy metals or toxic compounds. And some of them found to inhibit corrosion in acidic and alkaline medium [1-3].

For example *Propolis* extract is used to inhibit corrosion of carbon steel in aqueous solution in the presence of aluminium sulphate, chloride solution. It's delivered to be a mixed type inhibitor using potentiodynamic polarization method and the inhibition efficiency is 92% [4].

Bract extract of *musa acuminata* in HCl solution is found to inhibit corrosion upto 94.93% and the results are studied using electrochemical impedance spectra, potentiodynamic polarization and SEM techniques [5].

Vitex negundo is known to have a lot of medicinal uses such as leaves used externally for cleaning ulcers and internally for flatulence. Seeds are boiled in water and eaten or the water drunk to prevent the spread of toxin from the bites of poisonous animals. Root used as tonic, febrifuge and expectorant. And fruit used as nervine, cephalic, and emmenagogue. In this study *Vitex negundo* leaf extract is used as a corrosion inhibitor for mild steel in well water in the presence of zinc and the inhibition efficiency is evaluated with the help of weight loss method, potentiodynamic polarization techniques and electrochemical impedance spectra. The influence of zinc over the inhibitor is studied using synergistic parameter and 'F' test. And for surface analysis is done by SEM and EDAX methods adopted.

2. Materials and methods

2.1 Preparation of specimen

Composition of the carbon steel used in carbon-0.1%, sulphur-0.026%, phosphate-0.06%, manganese-0.4% and the balance iron. Carbon steel specimen of dimension 1.0 X 4.0X 0.2 cm were polished to mirror finish, degreased with trichloro ethylene and used for weight-loss and surface examination studies [6].

2.2 Preparation of the Plant extract

The leaves of the *Vitex negundo* were taken and cut into small pieces. From this, 10 g of the sample was ground with distilled water and then filtered carefully, the stock solution was prepared from the collected filtrate and making upto 100 ml. This extract is employed as a corrosion inhibitor in the present study.

2.3 Weight-loss method

2.3.1 Determination of surface area of the specimens

The length, breadth and the thickness of carbon steel specimens and the radius of the holes were determined with the help of vernier calipers of high precision and the surface areas of the specimens were calculated.

2.3.2 Weighing the specimens before and after corrosion

All the weight of the carbon steel specimens before and after corrosion was carried out using Shimadzu Balance-AY62.

2.3.3 Determination of corrosion rate

The weight specimen in duplicate were suspended by means of glass hooks in 100 ml test solutions and after 24 hour of immersion, the specimens were taken out, washed in running water dried and weighed.

$$\text{Corrosion rate (mm/y)} = \frac{\text{Loss in weigh t (mg)} \times 1000}{\text{Surface area of the specimen (dm}^2\text{)} \times \text{Period of immersion (days)}} \times \frac{(0.0365)}{\delta}$$

IE = 100[1-(W_2/W_1)] %

W_1 = Corrosion rate (mm/yr) in absence of Inhibitor.

W_2 = Corrosion rate (mm/yr)in presence of Inhibitor.

IE = Inhibition efficiency.

2.3.4 Potentiodynamic polarization study

Potentiodynamic polarization studies were carried out using CHI electrochemical impedance analyzer, model 660A. Three electrode cell assemblies were used. The working electrode was a rectangular specimen of carbon steel with one face of the electrode exposed. A saturated calomel electrode (SCE) was used as a reference electrode and a rectangular platinum foil was used as the counter electrode. Polarization curves were recorded using iR compensation. The results, such as Tafel slopes, and I_{corr}, and E_{corr}, values were calculated. During the polarization study, the scan rate (v/s) was 0.01, hold time at E_f(s) was zero, and quit time(s) was 2.

2.3.5 AC Impedance measurements

A CHI electrochemical impedance analyser (model 660A) was used for AC impedance measurements. The R_t (charge transfer resistance) and C_{dl} (double layer capacitance) values were calculated.

2.3.6 Synergism parameters: Synergism parameters are indication of synergistic effect existing between two inhibitors (1, 2)

Synergism parameters were calculated using the relation

$$S_1 = [1-\theta_{1+2}/1-\theta'_{1+2}]$$

Where,

$\theta_{1+2} = (\theta_1+\theta_2)-(\theta_1 X\ \theta_2)$

θ_1 = inhibition efficiency of substance 1

θ_2 = inhibition efficiency of substance 2

θ_{1+2} = combined inhibition efficiency of substance 1&2

θ = surface coverage = IE%/100

2.3.7 F-Test

F-Test was carried out to investigate whether the synergistic effect between *Vitex negundo* extract and Zn^{2+} was statistically significant or not.

2.3.8 FTIR spectra

These spectra were recorded with the Perkin Elmer RXI spectrophotometer. The FTIR spectrum of the protective film was recorded by carefully removing the film mixed it with KBr and making the pellet.

2.3.9 SEM study

The surface morphology measurements of the carbon steel were examined by JEOL JSM 6390 Model. All SEM micrographs of carbon steel are taken at a magnification of X=500.

3. Result and discussion

3.1 Analysis of the weight loss method

An aqueous extract was prepared by grinding 10gm of *vitex negundo* extract, filtering and making up to 100ml using distilled water. It was used in the present study. The calculated inhibition efficiencies (IE) of *vitex negundo* extract in controlling the corrosion of carbon steel immersed in well water both in the absence and presence of zinc ion have been tabulated in Table 1 and Table 2. It's observed from Table 1shows the inhibition efficiency 40% for 2ml of *Vitex negundo* extract, as the concentration of *Vitex negundo* extract increases the inhibition efficiency also increases. The calculated values indicate the ability of extract to be a good corrosion inhibitor. The inhibitor efficiency is found to be enhanced in the presence of zinc ion. The formulation consisting of 10ml of *Vitex negundo* leaf extract and 5ppm of Zn^{2+} offers 93% inhibition efficiency.

Table 1: Corrosion rates of carbon steel immersed in well water in the absence and presence of inhibitors and the inhibition efficiencies obtained by weight loss method. Inhibitor system: Vitex negundo extract +Zn^{2+} (0ppm), Period of immersion: 1 day

***Vitex negundo* extract (ml)**	**Zn^{2+} (ppm)**	**CR (mm/y)**	**IE%**
0	0	0.1529	-
2	0	0.09177	40
4	0	0.08412	45
6	0	0.05812	62
8	0	0.04129	73
10	0	0.03059	80

Table 2: Corrosion rates of carbon steel immersed in well water in the absence and presence of inhibitors and the inhibition efficiencies obtained by weight loss method. Inhibitor system: *Vitex negundo* extract $+Zn^{2+}$ (5ppm), Period of immersion: 1 day

Vitex negundo extract (ml)	Zn^{2+} (ml)	CR (mm/y)	IE%
0	0	0.1529	-
0	5	0.1345	12
2	5	0.04894	68
4	5	0.03823	75
6	5	0.03059	80
8	5	0.01988	87
10	5	0.01070	93

3.2 Synergism parameters (S_I)

Synergism parameter (S_I) have been used to know the synergistic effect existing between two inhibitors [1, 2]. Synergism parameter (S_1) can be calculated using the following relationship.

Synergism parameters

$$(S_I) = \frac{1-\theta_{1-2}}{1-\theta'_{1-2}}$$

Where,

$\theta_{1+2} = (\theta_1+\theta_2)-(\theta_1\theta_2)$

θ_1 = Surface coverage by *Vitex negundo* extract

θ_2 = Surface coverage by Zn^{2+}

θ_{1+2} = Surface coverage by both *Vitex negundo* extract and Zn^{2+}

$$\theta = \text{Surface coverage} = \frac{IE\%}{100}$$

The synergism parameters of *Vitex negundo* extract-Zn^{2+} system are given in Table 3. For different concentrations of inhibitors, S_1approaches 1 when no interaction between the inhibitor compounds exists. When $S_1>1$, it points to synergistic effects. In the case $S_1<1$, it is an indication that the synergistic effect is not significant. From Table3, it is observed that value of synergism parameters (S_1) calculated from surface coverage were found to be one and above. This indicates that the synergistic effect exist between *Vitex negundo* extract and Zn^{2+}. Thus, the enhancement of the inhibition efficiency caused by the addition of Zn^{2+} ions to *Vitex negundo* extract is due to the synergistic effect.

Table 3: Inhibition efficiencies and synergism parameters for various concentrations of *Vitex negundo* extract-Zn^{2+} (5ppm) system, when carbon steel is immersed in well water. Immersion day: 1 day

Vitex negundo extract (ml)	Inhibition efficiency IE (%)	Surface coverage θ_1	Zn^{2+}(ppm)	Inhibition efficiency IE (%)	Surface coverage (θ_2)	Combined IE%	Combined surface coverage θ'_{1+2}	Synergism parameters (S_I)
2	40	0.40	5	12	0.12	68	0.68	1.65
4	45	0.45	5	12	0.12	75	0.75	1.94
6	62	0.62	5	12	0.12	80	0.80	1.67
8	73	0.73	5	12	0.12	87	0.87	1.83
10	80	0.80	5	12	0.12	93	0.93	2.51

3.3 F-test

To know whether the synergistic effect existing between *Vitex negundo* extract and $Zn^{2+}$is statistically significant or not, F-test was used. The result are given in Table 4. It is observed that the calculated F-value 5.56 is greater than the table value 5.32 for 8 degree of freedom at 0.05 level of significance. Hence it is concluded that the synergistic effect existing between Vitex negundo extract and Zn^{2+}(5ppm) is statistically significant. Therefore, its concluded that the synergistic effect existing between *Vitex negundo* extract and Zn^{2+}(5ppm) is statistically significant.

Table 4: Distribution of F-value between the inhibition efficiency of various concentration of *Vitex negundo* extract (0ppm) of Zn^{2+}. The inhibition efficiencies of *Vitex negundo* extract in the presence of 5ppm of Zn^{2+}.

Source of variance	Sum of squares	Degree of freedom	Mean square	F-value	Level of significance
Between	220.5	1	220.5	5.56	p>0.05
Within	317	8	39.625		

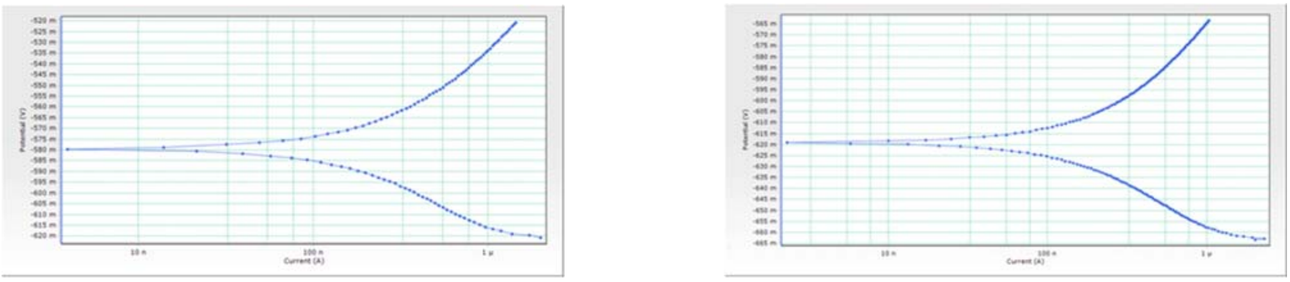
3.4 Analysis of polarization curves:

A polarization study has been used to detect the formation of protective film on the metal –surface [7-9]. When a protective film is formed on the surface of the metal, the corrosion current (I_{corr}) decreases. The potentiodynamic polarization curves of carbon steel immersed in various test solutions are shown in Fig.1 (a,b). The corrosion parameters namely, corrosion potential (E_{corr}), Tafel slopes (b_c=cathodic,b_a=anodic) and corrosion current(I_{corr})

are given in Table 5.When carbon steel is immersed in an aqueous solution containing well water, the corrosion potential is -579mV vs SCE. The formulation consisting of 10ml of *Vitex negundo* extract (VNE) and 5ppm Zn^{2+} shifts the corrosion potential to -619mV vs SCE. This suggests that a protective film is formed on the metal surface. Further the corrosion current decreases from 2.417X10^{-7}A/cm^2 to 1.95X10^{-7}A/cm^2. Thus, polarization study confirms the formation of the protective film on the metal surface.

Table 5: Potentiodynamic polarization curves of carbon steel immersed in various test solutions

System	E_{corr} mv vs SCE	b_c mv	b_a mv	I_{corr} A/cm^2
Well water	-579	58.10	72.226	2.417X10^{-7}
Vitex negundo extract(10ml)+Zn^{2+}	-619	52.61	73.445	1.95X10^{-7}



a) Well water

b) Well water + *Vitex negundo* extract (10ml) + Zn^{2+}

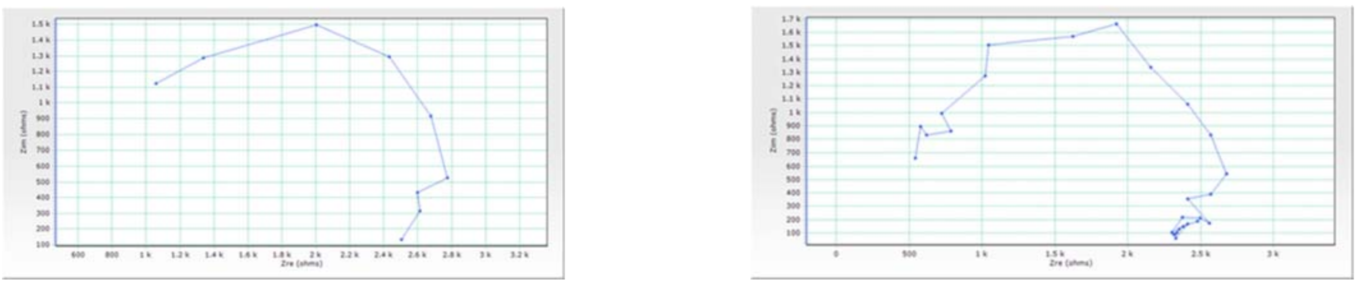
Fig 1: Polarization curves of carbon steel immersed in various test solutions
a) Well water; b) Well water + *Vitex negundo* extract (10ml) + Zn^{2+}

3.5 Analysis of AC impedance spectra

AC impedance spectra have been used to detect the formation of film on the metal surface. If a protective film is formed, the charges transfer resistance increases and double layer capacitance value decreases [10-12]. The AC impedance spectra of carbon steel immersed in various test solutions are shown in Fig.2 (a,b). The AC impedance parameter, namely charge transfer resistance (R_t) and double layer capacitance (C_{dl}) are given in Table 6. When carbon steel is immersed in aqueous solution containing well water R_t value is 1450 ohm cm^2 and C_{dl} value 3.2063X10^{-9} µF/cm^2. When *Vitex Negundo* extract and Zn^{2+} are added, R_t value increases from 1450 ohm cm^2 to 2080 ohm cm^2 and C_{dl} value decreases from 3.2063X10^{-9}µF/cm^2 to 2.2352X10^{-9} µF/cm^2. This suggests that a productive film is formed on the surface of the metal. This accounts for the very high IE of *Vitex negundo* extract-Zn^{2+}system.

Table 6: AC impedance parameters of carbon steel immersed various test solution.

SYSTEM	R_t Ohm cm^2	Cdl µF/cm^2
WELL WATER	1450	3.2063X10^{-9}
Vitex negundo extract (10ml) + Zn^{2+}(5ppm)	2080	2.2352X10^{-9}



a) Well water

b) Well water + *Vitex Negundo* extract (10ml) + Zn^{2+} (5ppm**)**

Fig 2: AC impedance spectra of carbon steel immersed in various test solutions (Nyquist plots)
a) Well water; b) Well water + *Vitex Negundo* extract 10ml) + Zn^{2+} (5ppm)

3.6 Analysis of FTIR spectra

The active principle in an aqueous extract of *Vitex negundo* is shown in Fig.4. A few drops of an aqueous extract were placed in on a glass plate and evaporated to dryness. A solid was obtained. Its FTIR spectrum is shown in fig.3 a). The C=C stretching frequency appear at 610 cm^{-1}. The C=O stretching frequency appears at 1645 cm^{-1}. The O-H stretching frequency appears at 3380 cm^{-1}. The O-C stretching frequency appear at 2925 cm^{-1}. The FTIR spectrum of the film formed on the metal surface after immersion in the solution containing well water, 10ml *Vitex Negundo* extract and 5ppm Zn^{2+} is shown in Fig. 3(b). The C=O stretching frequency has shifted from 1645 to 1589 cm^{-1}. The C=C stretching frequency has shifted from 610 to 699 cm^{-1}. The O-C stretching frequency has shifted from 2925 to 2924 cm^{-1}. The O-H stretching frequency has shifted from 3380 to 3405 cm^{-1}.

The peak at 835 cm^{-1} is due to Zn-O and the peak at 1401 cm^{-1} corresponds to $Zn(OH)_2$. This confirms $Zn(OH)_2$ is formed on the metal surface. Thus the FTIR spectrum leads to the conclusion that the protective film consists of Fe^{2+}-*Vitex negundo* extract complex, $Zn(OH)_2$.

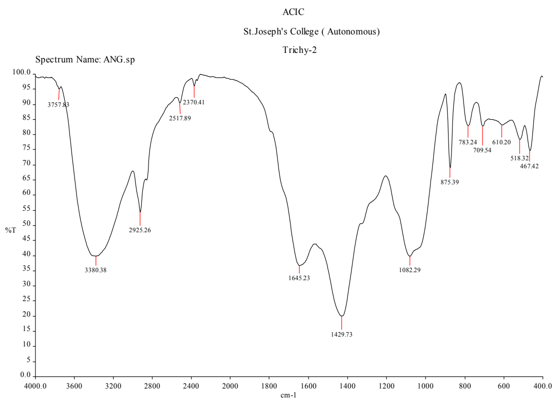


Fig 3a: FTIR spectra of pure *Vitex negundo* extract

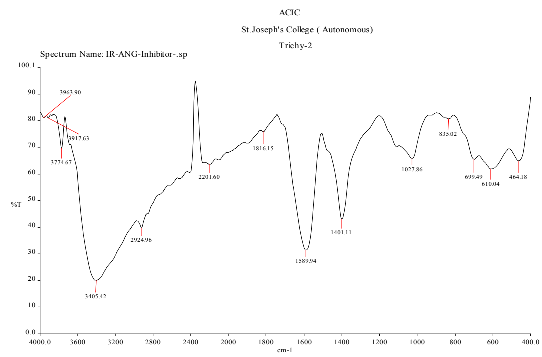
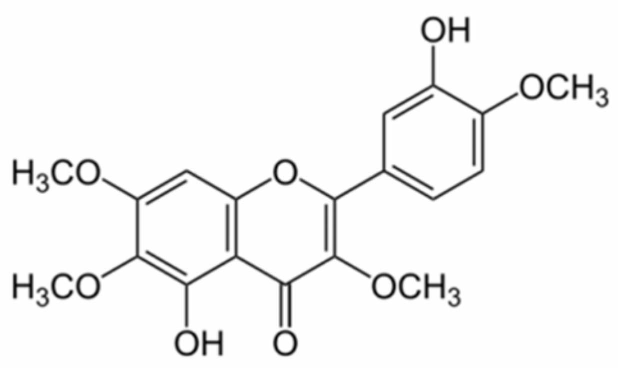


Fig 3b: FTIR spectra of film formed of the carbon steel surface after immersion in aqueous solution containing well water + *Vitex negundo* extract (10ml) + Zn^{2+} 5ppm.



5-hydroxy-2-(3-hydroxy-4-methoxyphenyl)-3,6,7-trimethoxychromen-4-one

Fig 4: Active principle of *Vitex negundo*

3.7 Analysis of SEM

SEM provides a pictorial representation of the surface. To understand the nature of the surface film in the absence and presence of inhibitors and the extent of corrosion of carbon steel, the SEM micrographs of the surface are examined [13-15].

The SEM images of different magnification (X1000) of carbon steel specimen immersed in well water for 1day in the absence and presence of inhibitor system are shown in Fig.5(a,b,c) respectively.The SEM micrographs of polished carbon steel surface in Fig.5(a). Shows the smooth surface of the metal. This shows the absence of any corrosion products (or) inhibitor complex formed on the metal surface.The SEM micrographs of carbon steel surface immersed in well water Fig.5 (b). shows the roughness of the metal surface which indicates the highly corroded area of carbon steel in well water. However Fig.5(c) indicates that in the presence of inhibitor (10ml *Vitex negundo* extract and 5ppm Zn^{2+}) the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The metal surface almost free from corrosion due to the formation of insoluble complex on the surface of the metal. In the presence of *Vitex negundo* extract and Zn^{2+}, the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of carbon steel.

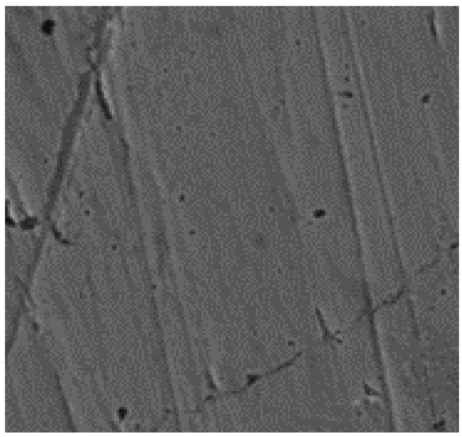


Fig 5(a): Carbon steel

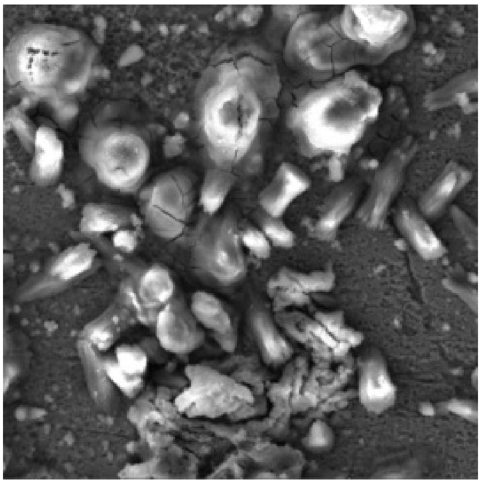


Fig 5(b): Carbon steel immersed in well water

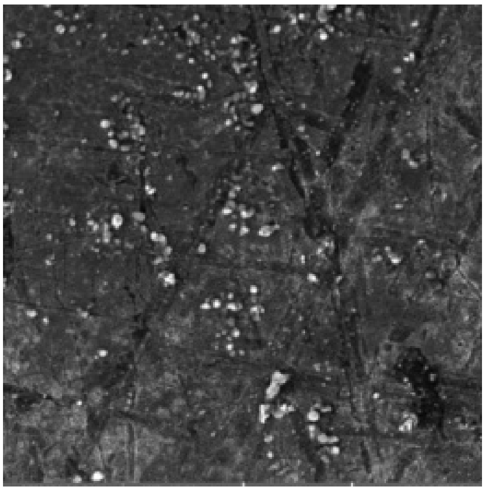


Fig 5(c): Carbon steel immersed in well water +10 ml *Vitex negundo* extract + 5 ppm Zn^{2+}.

3.8 Analysis of energy dispersive analysis of X-rays (EDAX)

The EDAX survey spectra were used to determine the elements present on the metal surface before and after exposure to the inhibitor solution[15]. The goal of this section was to confirm the results obtained from chemical and electrochemical measurements that a protective surface film of inhibitor is formed on the metal surface. To achieve this goal, EDAX examinations of the metal surface were performed in the absence and presence of inhibitors system.The EDAX spectrum of carbon steel immersed in well water is shown in Fig.6a) . It shows the characteristic peaks of some of the elements constituting the carbon steel sample. The EDAX spectrum of carbon steel immersed in well water 10ml of *Vitex negundo* extract and 5ppm of Zn^{2+} is shown Fig.6(b). It shows the additional line characteristic for the existence of Zn. In addition, the intensity of C and O signals are enhanced. The appearance of the Zn signal and the enhancement in C and O is due to the presence of inhibitor. These data show that metal surface is covered by C, O and Zn data. This layer is undoubtedly due to the inhibitor system. The Zn signal and the high contribution of C and O is not present on the metal surface exposed in well water .Fig.6(b) shows that the Fe peaks observed in the presence of inhibitor are considerably suppressed relative to these observed in well water (blank). The suppression of the Fe peaks occurs because of the overlying inhibitor film. This observation indicates the existence of an adsorbed layer of inhibitor that protects steel against corrosion. These results suggest that C and O atoms of *Vitex negundo* extract has coordinated with Fe^{2+}, resulting in the formation of Fe^{2+}-*Vitex negundo* extract complex and presence of Zn atoms are precipitated as $Zn(OH)_2$. Thus the EDAX spectral study leads to the conclusion that the protective film consist of Fe^{2+}-*Vitex negundo* complex and $Zn(OH)_2$.

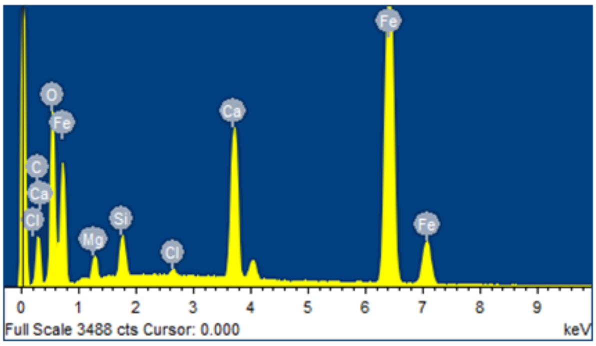


Fig 6(a): EDAX spectra of Carbon steel after immersion in well water (blank)

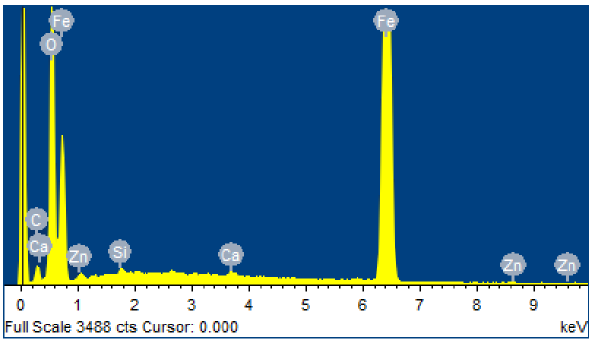


Fig 6(b): EDAX spectra of carbon steel after immersion in solution containing well water + *Vitex negundo* extract(10ml) +Zn^{2+}(5 ppm).

3.9 Mechanism of Corrosion Inhibition

The results of the weight-loss study show that the formulation consisting of 10 ml of *Vitex negundo* extract and 5ppm of Zn^{2+} has 93% Inhibition efficiency in controlling corrosion of carbon steel in well water. A synergistic effect exists between Zn^{2+} and *Vitex negundo* extract. Polarization study reveals that this formulation functions as cathodic inhibitor. AC impedance spectra reveal that the protective film is formed on the metal surface. FTIR spectra reveal that the protective film consists of Fe^{2+}-*Vitex negundo* extract and $Zn(OH)_2$.

- When the solution containing well water, 5ppm Zn^{2+} and 10ml of *Vitex negundo* extract is prepared, there is formulation of Zn^{2+}-*Vitex negundo* complex in solution.
- When carbon steel is immersed in this solution, the Zn^{2+}-*Vitex negundo* complex diffuses from the bulk of the solution towards metal surface.
- Zn^{2+}-*Vitex negundo* complex diffused from the bulk solution to the surface of the metal and is converted into a Fe^{2+}-Vitex Negundo complex, which is more stable than Zn^{2+}-*Vitex negundo* complex.
- On the metal surface Zn^{2+}-*Vitex negundo* complex is converted in to Fe^{2+}-Vitex Negundo and Zn^{2+} is released.
  Zn^{2+}-*Vitex negundo* + Fe^{2+} $\longrightarrow$ Fe^{2+}-*Vitex negundo* + Zn^{2+}
- The released Zn^{2+} combines with OH^- to form $Zn(OH)_2$
  $Zn^{2+} + 2OH^- \longrightarrow Zn(OH)_2 \downarrow$
  Thus the protective film consists of Fe^{2+}-*Vitex negundo* complex and $Zn(OH)_2$.
- The EDAX and SEM micrographs confirm the formation of protective layer on the metal surface.

4. Conclusions
Present study leads to the following conclusions.
- The formulation consisting of 10 ml of *Vitex negundo* extract and 5 ppm of Zn^{2+}offers good inhibition efficiency of 93%.
- Polarization study reveals that this formulation functions as a cathodic inhibitor.
- AC impedance spectra reveal that a protective film is formed on the metal surface.
- The FTIR spectral study leads to the conclusion that the Fe^{2+} - *Vitex negundo* complex and $Zn(OH)_2$ formed on the metal surface.
- SEM and EDAX confirm the presence of a protective film on the metal surface.

5. Acknowledgement
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