Influence of *Aloe vera* Extract on Corrosion Inhibition of Mild Steel in Well Water

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1. Introduction

Plant extracts are viewed as an incredibly rich source of naturally synthesized chemical compounds that can be extracted by simple procedures with low cost and are biodegradable in the environment.Plant extracts have become important as environmentally acceptable, readily available and renewable source for wide range of inhibitors.[1] In general, the plant extracts are inhibitors with high inhibition efficiency and of non toxicant. Natural products are nontoxic, bio-degradable and readily available. They have been used widely as inhibitors. Several plant extracts[2-6] and eco-friendly inhibitors[7,8] attracted the researchers. Natural products such as caffeine[9,10] have been used as inhibitors. Corrosion inhibition of steel by plant extracts in acidic media has been reported[11,12].

Corrosion inhibition by beet root extract has been studied[13]. Aqueous extracts of Onion[14] and Androgaphis panizulata[15] have been used as corrosion inhibitors. Opuntia extract[16] was investigated for the corrosion of Aluminium in acid medium and vanillin[17] for the corrosion of mild steel in acid media. Extracts of tobacco from twigs, stems, and leaves can protect steel and aluminium in saline solutions and strong pickling acids[18,19]. Extract of Hibiscus sabdariffa can be used as corrosion inhibitor for mild steel in 2M HCl and 1M H_2SO_4 solution[20]. Anthony et al. have studied the effect of caffeine against chloride corrosion of carbon steel.[21] Bo yong *et al*. Investigated the corrosion inhibition of mild steel in acidic media by garlic[22, 23], Eddy *et al*.[24] was studied the corrosion inhibition of ethanol extract of Aloe vera on mild steel in acid media. Sribharathy *et al.* investigated the corrosion of mild steel in sea water by *Aloe vera* extract[25]. Through these studies, it is agreed that the inhibition performance of plant extract is normally ascribed to the presence of their composition of complex organic species such as tannins, alkaloids and nitrogen bases, carbohydrates, amino acids and proteins as well as hydrolysis products. These organic compounds contain polar functions with N, S, O atoms as well as conjugated double bonds or aromatic rings in their molecular structures, which are the major adsorption centres.

Aloes have abundant organic components in which N, S, O atoms are the main constituent atoms. The present work investigated the inhibition efficiency of an aqueous extract of plant material, *Aloe vera* (L) Burm f. (Liliaceae) extract, in controlling corrosion of carbon steel immersed in well water in the absence and presence of inhibitor, using weight loss method, analyzed the protective film by

Fourier transform infrared (FTIR) spectroscopy and proposed a suitable mechanism of corrosion inhibition, based on the results of the above studies.

2. Experimental

2.1 Preparation of plant extract and specimens

An aqueous extract was prepared by grinding 10 g of fresh extract of aleo vera gel, filtering and making up to 100 ml using double distilled water. Carbon steel specimens (0.0267% S, 0.06% P, 0.4% Mn, 0.1% C and the rest iron) of dimensions 1.0 cm × 4.0 cm × 0.2 cm were polished to a mirror finish and degreased with trichloroethylene.

2.2 Weight loss method

Carbon steel specimens were immersed in 100 ml of the well water, containing various concentrations of the inhibitor in the absence and presence of Zn^{2+} for 3 days. The weights of the specimens before and after immersion were determined using a Digital Balance Model AY 62 SHIMADZU. The corrosion products were cleaned with Clarke's solution. It can be prepared by dissolving 20 g of Sb_2O_3 and 50 g of $SnCl_2$ in one litre of conc. HCl of specific gravity (1.9)[26]. The corrosion IE was then calculated using the equation.

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

where W_1 is the corrosion rate in the absence of inhibitor and W_2 is the corrosion rate in the presence of inhibitor. Corrosion rate was calculated using the formula:

$$\text{Millimetre per year} = 87.6\ W / DAT$$

W = Weight loss in milligrams
D = Density of specimen g/cm^3 = 7.87 gm/cm^3
A = Area of specimen = 10 cm^2 and
T = Exposure in hours = 72 hr

2.3 Synergism Parameter

The synergism parameter can be calculated by using the equation indicates the synergistic effect existing between the inhibitors[27-29]. S_I value is found to be greater than one suggesting that the synergistic effect between the inhibitors is $S_I=1-I_1+2\ /1-I'_{1+2}$. where I_1 = inhibition efficiency of substance 1, I_2 = inhibition efficiency of substance 2,I'_{1+2} = combined inhibition efficiency of substance 1 and 2. If synergistic effect exists between the inhibitors, S_1 value will be greater than one.

2.4 Analysis of Variance (F-Test)

An F-test was carried out to investigate whether the synergistic effect existing between inhibitor systems is statistically significant[30]. If F-value is greater than 5.32 for 1, 8 degrees of freedom, the synergistic effect proves to be statistically significant. If it is less than 5.32 for 1, 8 degrees of freedom, it was statistically insignificant at a 0.05 level of significance.

2.5 Determination of the biocidal efficiency

The biocidal efficiency of the system was determined using Zobell medium and calculating the numbers of colony forming units per ml using a bacterial colony counter. The biocidal efficiency of sodium dodecyl sulphate (SDS) and SM-Zn^{2+} - *Aloe vera* system was determined. Various concentrations of SDS such as 50 ppm, 100 ppm, 150 ppm, 200 ppm and 250 ppm were added to the formulation consisting of the inhibitor system. Polished and degreased mild steel specimens in triplicate were immersed in these environments for a period of 3 days.

After 3 days, 1 ml each of test solutions from environments was pipetted out into sterile petri dishes each containing about 20 ml of the sterilized Zobell medium. The petri dishes were then kept in a sterilized environment inside the laminar flow system fabricated for 48 hours. The total viable hydrotropic bacterial colonies were counted using a bacterial colony counter. The corrosion inhibition efficiencies of the formulation consisting of the inhibitor in the presence of various concentrations of SDS were also determined.

2.6 Surface Examination Study

The carbon steel specimens were immersed in various test solutions for a period of one day, taken out and dried. The nature of the film formed on the surface of the metal specimen was analyzed by FTIR spectroscopic study.

These spectra were recorded in a Perkin-Elmer-1600 spectrophotometer using KBr pellet. The FTIR spectrum of the protective film was recorded by carefully removing the film, mixing it with KBr.

3. Result and Discussion

3.1 Analysis of Result of the weight loss method

The physicochemical parameters of well water are given in Table 1.

In order to examine the role of *Aloe vera* in the ternary inhibitor formulation, experiments were conducted with *Aloe vera* alone, with SM and with SM-Zn^{2+} in a wide concentration range. The inhibition efficiency (IE) of *Aloe vera* in controlling corrosion of carbon steel immersed in well water for a period of three days in the absence and the presence of Zn^{2+} and SM is given in Table 2.

It can be seen from the data that *Aloe vera* alone shows some IE. The inhibition efficiency of *Aloe vera* and Zn^{2+} are shown in Table 3.

The Zn^{2+} ion acts as one synergist and *Aloe vera* acts as the other. When *Aloe vera* is combined with Zn^{2+} ions it is found that the IE increase with concentration of *Aloe vera*. For example, 10 ml of *Aloe vera* extract has only 32% IE and 25ppm of Zn^{2+} has only 5% IE their combination shows 51% IE. This suggests a synergistic effect between the binary inhibitor formulation of *Aloe vera* and Zn^{2+} ion.

Table 1. Physico-Chemical Parameters of Well Water

Parameter	Value
Appearance	Clear
Turbidity NT units	1.4
Ph	7.73
Conductivity	5820 µmhos/cm
Chloride	1450 ppm
Sulphate	73 ppm
TDS	4034 ppm
Total hardness	880 ppm
Total Alkalinity	548 ppm
Calcium	304 ppm
Magnesium	29 ppm
Sodium	820 ppm
Potassium	40 ppm
Iron	0.30 ppm
Nitrate	2 ppm
Fluoride	0.64 ppm
Phosphate	0.08 ppm

Table 2. Corrosion Rate (CR) and IE of Carbon Steel in Well Water, in the Absence and the Presence of Inhibitors and IE Obtained by Weight-Loss Method Immersion period: 3 days

Aloe vera **ml**	**SM ppm**	**Zn^{2+} ppm**	**IE %**	**CR mmy^{-1}**
0	0	0	-	0.1174
2	0	0	15	0.0995
4	0	0	19	0.0950
6	0	0	23	0.0903
8	0	0	28	0.0845
10	0	0	32	0.0798

Table 3. Corrosion Rate (CR) and IE of Carbon Steel in Well Water, in the Absence and the Presence of Inhibitors and IE Obtained by Weight-Loss Method (Immersion period: 3 days)

Aloe vera **ml**	**SM ppm**	**Zn^{2+} ppm**	**IE %**	**CR mmy^{-1}**
0	0	25	5	0.1115
2	0	25	23	0.0903
4	0	25	29	0.0833
6	0	25	33	0.0786
8	0	25	42	0.0680
10	0	25	51	0.0575

The synergistic effect in the SM- Zn^{2+}- *Aloe vera* system is evident from the data in Table 4. The Zn^{2+} ion acts as one synergist and *Aloe vera* acts as the other. From the data in Table 4, it is seen that at relatively higher concentrations of SM, Zn^{2+} and *Aloe vera*, 87% IE was obtained. However, such efficiency is not obtained with combinations of SM and *Aloe vera*, even at relatively high concentrations. Thus, it may be concluded that Zn^{2+} is the primary synergist and *Aloe vera* is the secondary synergist and both play a significant synergistic role in inhibiting corrosion. Hence, the highest IE is obtained at such low concentrations of each of the components in the ternary inhibition formulation.

Table 4. Corrosion Rate (CR) and IE of Carbon Steel in Well Water, in the Absence and the Presence of Inhibitors and IE Obtained by Weight-Loss Method (Immersion period: 3 days)

Aloe vera **ml**	**SM ppm**	**Zn^{2+} ppm**	**IE %**	**CR mmy^{-1}**
0	100	25	23	0.0903
2	100	25	61	0.0457
4	100	25	65	0.0410
6	100	25	73	0.0316
8	100	25	83	0.0199
10	100	25	87	0.0152

3.1.1 Influence of immersion period on the SM-Zn^{2+}-Aloe vera system

The influence of immersion period on IE of SM (100 ppm)-Zn^{2+} (25 ppm)-*Aloe vera* (10 ml) is given in Table 5. It is found that as the immersion period increases, the inhibition efficiency decreases[31]. This is due to the fact as the immersion period increases the protective film is ruptured by the continuous attack of the Cl^-, present in the solution. The iron complexes of SM and *Aloe vera* film formed on metal surface is converted into iron chloride which goes into solution and hence, the IE decreases as the immersion period increases.

Table 5. Influence of immersion period on the IE of SM (100ppm)-Zn^{2+} (25 ppm) -*Aloe vera* (10ml) system. Inhibitor system: SM-Zn^{2+}-*Aloe vera*

System	**Immersion Period (Days)**			
	1	**3**	**5**	**7**
Well water (WW) CR (mmy^{-1})	0.0432	0.1174	0.1484	0.1607
WW + SM (50 ppm) Zn^{2+} (10 ppm) *Aloe vera* (10 ml) CR (mmy^{-1})	0.0058	0.0152	0.0281	0.0387
IE (%)	**95**	**87**	**76**	**67**

3.1.2 Synergism parameter

The values of synergism parameters are shown in Table 6. The values of S_I are greater than one, suggesting a synergistic effect. S_I approaches 1 when no interaction exists between the inhibitor compounds. When $S_I > 1$, this points to the synergistic effect. In the case of $S_I < 1$, the negative interaction of inhibitors prevails (i.e., corrosion rate increases).

Table 6. Synergism Parameter of Carbon Steel Immersed in Well Water in the Presence and Absence of Inhibitor

Aloe vera (I_1) IE (%)	SM+Zn^{2+} (I_2) IE (%)	SM-Zn^{2+}-Aloe vera (I'_{1+2}) IE (%)	S_I
15	23	61	1.67
19	23	65	2.80
24	23	73	3.60
28	23	83	5.68
32	23	87	7.38

3.1.3 Analysis of variance (ANOVA)

F-test is used if the synergistic effect exists between inhibitors and is statistically significant[32]. The results are given in Table 7 and 8. Influence of various concentrations of *Aloe vera* (2, 4, 6, 8 and 10 ml) on the inhibition efficiencies of SM (100ppm) -Zn^{2+} (25 ppm) is tested in Table 7. The calculated F-value is 12.94. It is statistically significant, since it is greater than the critical F-value (5.32) for 1, 8 degrees of freedom of 0.05 level of significance. Hence, it is concluded that the inhibition efficiencies of the SM-Zn^{2+}-*Aloe vera* system is statistically significant.

Table7. Distribution of F Value between the Inhibition Efficiencies of SM-Zn^{2+} and *Aloe vera* Systems

Source of variance	Sum of squares	Degrees of freedom	Mean square	F	Level of siginificance of F
Between	4984.41	1	4984.4	12.94	P > 0.05
Within	3080	8	385		

Inhibition efficiencies of SM (10ml) - Zn^{2+} (25ppm) system on the various concentrations of SM (100 ppm)-Zn^{2+} (25 ppm) - *Aloe vera* (250 ppm) systems are tested in Table 8. The calculated F-value is 10.65. It is statistically significant, since it is greater than the critical F-value (5.32) for 1, 8 degrees of freedom at 0.05 level of significance. Hence, it is concluded that the inhibition efficiencies of SM- Zn^{2+} system and SM- Zn^{2+} and *Aloe vera* system is statistically significant.

Table 8. Distribution of F Value between the Inhibition Efficiencies of SM-Zn^{2+} and SM-Zn^{2+}- *Aloe vera* Systems

Source of variance	Sum of squares	Degrees of freedom	Mean square	F	Level of siginificance of F
Between	3630.48	1	3630.48	10.65	P > 0.05
Within	2726.5	8	340.75		

3.1.4 Effect of sodium dodecyl sulphate (SDS) on the inhibition efficiency of SM-Zn^{2+} - *Aloe vera*

The biocidal efficiency of SM-Zn^{2+} in the absence of SDS was found to be 23 percent. The number of colony forming units/ml was 6 x 10^3 this is objectionable. When 150ppm of SDS was added, nil CFμ/ml is obtained. The biocidal efficiency is 100%. Hence the optimum concentration of SDS is 150ppm. Thus it is seen in Table 9, that the formulation consisting of 100ppm of SM, 25ppm of Zn^{2+} and 10ml of *Aloe vera* extract and 150 ppm of SDS has 97% of corrosion inhibition efficiency and 100% of biocidal efficiency.

Table 9. Corrosion Rates of Carbon Steel in Well Water in the Presence and Absence of Inhibitors and the Corrosion Inhibition Efficiencies, Biocidal Efficiencies of Various Environments Obtained by the Weight-Loss Method

SM ppm	Zn^{2+} ppm	AV ml	SDS ppm	CR mmy^{-1}	IE %	Colony forming units/ml	Biocidal Efficiency (%)
0	0	0	0	0.1174	-	8×10^3	-
100	25	0	0	0.0903	23	6×10^3	25
100	25	10	0	0.0152	87	5×10^3	38
100	25	10	50	0.0140	88	3×10^3	63
100	25	10	100	0.0117	90	2×10^3	75
100	25	10	150	0.0035	97	Nil	100
100	25	10	200	0.0082	93	Nil	100
100	25	10	250	0.0117	90	Nil	100

3.2. FTIR spectra

FTIR spectra have been used to analyze the protective film formed on the metal surface[33,34]. FTIR spectrum of pure sodium molybdate is given in Figure 2a. The Mo-O stretching frequency appears at 824 cm^{-1}. The active principle in an aqueous extract of *Aloe vera* is shown in Figure 1. It contains phenolic-OH group and carboxyl (C=O) group.

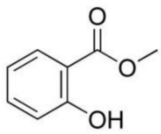

Fig 1. Aloe vera

A few drops of an aqueous extract of *Aloe vera* was dried on a glass plate. A solid mass was obtained. Its spectrum is shown in Figure 2b. The hydroxyl (-OH) group appears at 3312 cm^{-1} and carboxyl group (C=O) appears at 1626 cm^{-1}.

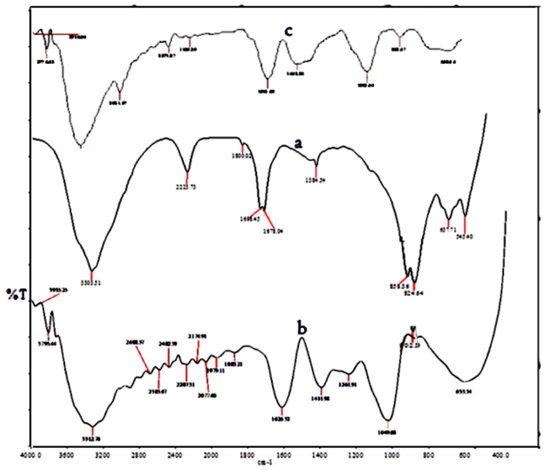

Fig. 2. FTIR Spectrum (a) Pure sodium molybdate (b) Pure *Aloe vera* (c) Film formed on metal surface after the immersion in well water containing 100 ppm SM - 25 ppm of Zn^{2+} and 10 ml *Aloe vera*

The FTIR spectrum of the protective film formed on the metal surface after immersion in the solution containing 10 ml of *Aloe vera*, 100 ppm of SM and 25 ppm of Zn^{2+} is shown in the Figure 2c. The MoO^{4-} stretching frequency of SM shifted from 824cm^{-1} to 853 cm^{-1}. This suggests that MoO^{4-} of SM is coordinated with Fe^{2+} on the anodic sites of the metal surface, also resulting in the formation of Fe^{2+}- MoO^{4-} complex.

The phenolic -OH stretch shifted from 3312 cm^{-1} to 3385 cm^{-1}. The C=O stretching shifted from 1626 cm^{-1} to 1613 cm^{-1}. These shifts confirm the formation of Fe^{2+}-*Aloe vera* complex on the anodic sites of the metal surface[35, 36]. The Zn-O stretching frequency appears at 569 cm^{-1} and the stretching frequency due to -OH appears at 3385 cm^{-1}. Therefore, it is concluded that $Zn(OH)_2$ is formed on cathodic sites of the metal surface[37].

4. Mechanism

In order to explain the experimental results, the following mechanism of corrosion inhibition is proposed. The mechanistic aspect of the inhibition of carbon steel in well water by SM-Zn^{2+} and *Aloe vera* can be explained in terms of complexation and adsorption.

- Before immersion of carbon steel in well water environment, SM, Zn^{2+} and *Aloe vera*- Zn^{2+} form complexes, *viz.*, Zn^{2+}-SM, Zn^{2+}-*Aloe vera*, and Zn^{2+}-SM-*Aloe vera*. These complexes are in equilibrium in the solution with free Zn^{2+}, SM, and *Aloe vera* ions.
- During the dissolution of iron, the pH increases at the metal/electrolyte interface due to oxygen reduction. Thus, $Zn(OH)_2$ precipitate may take place at cathodic sites[38,39], thus decreasing the rate of further oxygen reduction.
- Addition of *Aloe vera* reduces metal dissolution; this may be due to adsorption and complex formation at the surface with the combined application of Zn^{2+} and *Aloe vera*. The corresponding anodic and cathodic reactions of the metal can be generalized as follows. Zn^{2+} inhibits the local cathodic region and the local anodic region was inhibited by *Aloe vera*.
- The Zn-AVcomplex diffuses from the bulk solution to the surface of the metal and is converted into a Fe-AV complex[40]. The released Zn^{2+} causes $Zn(OH)_2$ precipitation at the local cathodic sites. Thus, the protective film consists of an Fe-AV complex and $Zn(OH)_2$.
- The film formed on the metal surface of the carbon steel consists of oxides/hydroxides of iron and zinc. It is also likely to comprise complexes of Fe^{2+}/Fe^{3+} and Zn^{2+} with SM as well as with *Aloe vera*. The FTIR spectra of the surface film suggests the formation of these complexes and the presence of $Zn(OH)_2$ in the surface.

5. Conclusion

A formulation consisting of Zn^{2+}, SM, and *Aloe vera* can be used as a potent inhibitor to prevent the corrosion of carbon steel in well water. *Aloe vera* plays an excellent synergistic role in the SM- Zn^{2+}-*Aloe vera* system. The ternary system SM (100 ppm)- Zn^{2+} (25 ppm)-*Aloe vera* (10ml) is effective and has 87% IE. Significant synergism was attained by the combined application of SM-Zn^{2+}-*Aloe vera*. The concentrations of both SM and Zn^{2+} are reduced, and *Aloe vera,* which is environmentally friendly, is required only at low concentrations.

Thus, this new inhibitor formulation is more environment-friendly. Both SM and *Aloe vera* form stable complexes with metal ions in the metal surface. The protective film consists of Zn (OH) 2 and complexes of Fe2+/Fe3+ and Zn^{2+} with SM as well as with *Aloe vera*.

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