IRA-International Journal of Technology & Engineering
ISSN 2455-4480; Vol.03, Issue 03 (2016)
Institute of Research Advances
http://research-advances.org/index.php/IRAJTE

Anti Corrosion Ability and Biocidal Efficiency of Surfactants on Mild Steel in Aqueous Environment

K. N. Manonmani,
Research Scholar in Chemistry, Department of chemistry, Mother Teresa Women's University, Kodaikanal-624 102, Tamil Nadu, India.

P. Manjula,
Associate Professor/Head, Department of Chemistry, APA College for Women, Palani, Tamil Nadu, India.

T. Vennila,
Sri Sairam College of Engineering, Chennai,-600 044.Tamil Nadu, India.

DOI: http://dx.doi.org/10.21013/jte.v3.n3.p2

How to cite this paper:
Manonmani, K., Manjula, P., & Vennila, T. (2016). Anti Corrosion Ability and Biocidal Efficiency of Surfactants on Mild Steel in Aqueous Environment. *IRA-International Journal of Technology & Engineering (ISSN 2455-4480)*, 3(3). doi:http://dx.doi.org/10.21013/jte.v3.n3.p2

ABSTRACT

The inhibition efficiency of DMBP (Diethyl-4-methyl benzyl phosphonate) along with Biocidal action of CTAB and SDS on corrosion of mild steel in neutral aqueous medium has been evaluated by mass loss method, both in absence and presence of Zn^{2+}*. It is observed that DMBP exhibits 65% IE individually at ambient temperature. Synergistic influence of* Zn^{2+} *increases IE of DMBP to 71%. Addition of surfactants CTAB (N-Cetyl-N,N,N-Trimethyl Ammonium Bromide) and SDS (Sodium dodecyl Benzene sulphonate) enhances the inhibition efficiency of DMBP from 65% to 96% and 92% respectively. The inhibitor system consisting of DMBP (200 ppm) +* Zn^{2+} *(90 ppm)+ CTAB (50 ppm) / SDS (40 ppm) offered maximum Biocidal efficiency (BE %) of 100% after killing of microbial bacteria present in aqueous system. FTIR spectra indicates the protective film consist of* Fe^{2+}*-DMBP and* $Zn(OH)_2$*.*

Key words: carbon steel, surfactant, Biocidal efficiency, synergistic effect and Zobell medium.

I. Introduction

In the field of inhibition, scientists are persistent in seeking better and more efficient ways of combating corrosion of metals (1,2). The challenge is to develop a new class of corrosion inhibitors to protect the materials, which are environment friendly under various conditions. To be effective an inhibitor must also transfer water from the metal surface, interact with anodic or cathodic reaction sites to retard the oxidation and reduction corrosion reaction, and prevent transportation of water and corrosion active species on the metal surface. Surfactant inhibitors have many advantages, for example, high inhibition efficiency, low price, low toxicity and easy protection (3). Moreover, the investigation of surfactants adsorbed on metal surface is extremely important in electrochemical studies such as corrosion inhibition, adhesion, lubrication and detergency. The effective type of corrosion inhibitors for these applications is film forming inhibitors. Nowadays, surfactants are widely used and find a very large number of applications in the petroleum industry. This is attributed to their significant capability to influence the properties of surfaces and interfaces. Mild steel widely employed in most industries due to easy fabrication of various reaction vessels such as cooling tower tanks, pipelines etc. and of low cost. In the presence anti corrosive ability of sodium salt of DMBP and the surfactants CTAB and SDS in controlling corrosion of mild steel in aqueous medium are investigated. To inhibit MIC (microbially induced corrosion) in water, Biocidal efficiency (4) of surfactant selected in this study is also determined.

II. Materials and Methods

MATERIALS

Preparation of Mild steel specimens

Mild steel specimens were chosen from the same sheet of the following composition 0.1% C, 0.026 % S, 0.06 % P, 0.4% Mn and the rest Iron and the dimensions of 1.0 x 4.0 x 0.2 cm. Prior to all measurements, the carbon steel specimens were mechanically abraded (5) with different types of emery papers (Grade 320 – 400- 600-800-1000-1200), degreased with acetone rinsed with distilled water, dried and weighed before immersion in the experimental solution. These specimens were used for gravimetric measurements

Chemical and reagents

All the chemicals and reagents used were of Analar /Sigma Aldrich Grade

Preparation of inhibitor solution Diethyl 4- Methyl Benzyl Phosphonate (DMBP)

5 ml of the DMBP is subjected to alkaline hydrolysis with sodium hydroxide. The sodium salt is dried and recrystallised using distilled water. 1g of the sodium salt is distilled in 1 litre of DD water and made upto 1000 ml. a hundred fold dilutions is exactly 10 ppm of inhibitor concentration.

Preparation of Sodium Chloride Solution

Exactly 4.9 g of sodium chloride was dissolved in double distilled water and made up to 500 ml in a standard measuring flask. A hundred fold dilution yields exactly 60 ppm of Cl^- ion concentration.

Preparation of Zinc Sulphate solution

Exactly 2.2 g of zinc sulphate was dissolved in double distilled water and made up to 500 ml in standard measuring flask. A hundred fold dilution yields exactly 10 ppm of Zn^{2+} ion concentration.

Preparation of CTAB/SDS solution

Exactly 1 g of CTAB/SDS was dissolved in double distilled water and made up to 100 ml in standard measuring flask. A hundred fold dilution yields exactly 10 ppm of CTAB/SDS concentration.

Preparation of Zobell medium

Zobell medium was prepared by dissolving 5g of peptone, 1g of yeast extract, 0.1 g of Potassium dehydrate phosphate and 15 g of agar- agar in one litre of double distilled water. The medium was sterilized by applying 15 pounds per square inch for 15 minutes in an autoclave.

METHODS

Mass loss measurements

Carbon steel specimens in triplicate were immersed in 100 ml of solutions containing various concentrations of the inhibitor in the presence and absence of Zn^{2+} for three hours. The weight of the specimens before and after immersion was determined using a Shimadzu balance, model AY62. The corrosion products were cleansed with Clarke's solution (6). The inhibition efficiency (IE) was then calculated using the equation.

$$\text{Efficiency of Inhibitors (\%)} = WL_w - WL_i / WL_w \text{ X } 100$$

Where WL_w is the weight loss without inhibitor and WL_i is the weight loss with inhibitor.

Determination of Biocidal efficiency of the system

Diethyl-4-methyl benzyl phosphonate-Zn^{2+} formulation which offered the best corrosion inhibition efficiency was selected. The biocidal efficiency of N-Cetyl-N,N,N-Trimethyl Ammonium Bromide (CTAB) and Sodium dodecyl benzene sulphonate (SDS) in the presence of this formulation and also the effect of CTAB on the corrosion inhibition efficiency of this system were determined.

Various concentration of CTAB Viz 10 ppm , 50 ppm, 100 ppm, 150 ppm and 200 ppm were added to the formulation consisting of the inhibitor system. Polished and degreased carbon steel specimen in triplicate were immersed in these environments for a period of 3 hours. After 3 hours, 1 ml each of test solutions from environments was pipetted out into sterile petridishes each containing about 20 ml of the sterilize zobell medium. The petridishes were then kept in a sterilized environment inside the laminar flow system. Flow system fabricated and supplied by CEERI-Pilani for 24 hours. The total viable heterotropic 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 CTAB and SDS were determined in the same way as discussed. Similarly the experiment was done with SDS. The materials used and methods employed in the present study have been presented.

SURFACE EXAMINATION STUDIES

Surface analysis by FTIR spectroscopic study

After the immersion period of three hours in various environments, the specimens are taken out of the test solutions and dried. The film formed on the surface is scratched carefully and it is thoroughly mixed so as to make it uniform throughout. FTIR spectrum of the powder (KBr pellet) is recorded using Perkin-Elmer 1600 FTIR spectrophotometer with a resolving power of 400 cm^{-1}.

Surface analysis by SEM

SEM micrographs were taken to using VEGA 3 TESCAN model at Department of Chemistry, Gandhi gram Rural Institute, Gandhi gram, Dindigul, Tamilnadu, India.

III. Result and Discussion

The adsorption process is influenced by the phosphonic acid groups present in the inhibitor molecule which forms the protective complex Fe-DMBP and the distribution of charge (7) in the inhibitor molecule on to the metal surface. At higher concentration of DMBP aggressive Cl^- ion enhances the corrosivity of the metal surface due to desorption of the inhibitor molecules on the surface.

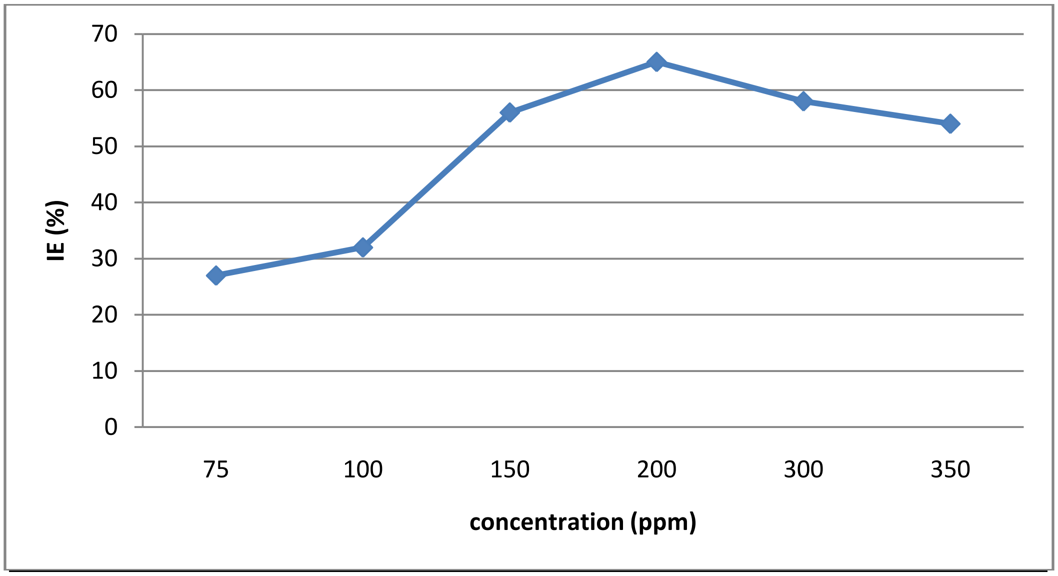
Phosphonic acids can be adsorbed on the mild steel surface by the interaction between lonepair of electron of oxygen and phosphorus atoms of the inhibitor and the metal surface. This process is facilitated by the presence of vacant orbitals of low energy in ion atoms, as observed in the transition metals.

Table 1: Corrosion rates of mild steel in neutral aqueous environment (Cl^- = 60 ppm) in the presence and absence of inhibitor and the inhibition efficiencies obtained by the weight-loss method.

Inhibitor system: DMBP+ NaCl (60ppm) system
Immersion Time: 3 Hours pH=7

S.No	Conc. of DMBP (ppm)	Conc. of Zn^{2+} (ppm)	Wt.loss (g)	CR (mmpy)	Surface coverage (θ)	IE (%)
1.Blank	0	0	0.0025	0.9297	-	-
2.	75	0	0.001825	0.6787	0.2700	27
3.	100	0	0.0017	0.6322	0.3200	32
4.	150	0	0.0011	0.4090	0.5600	56
5.	200	0	0.000875	0.3254	0.6500	65
6.	300	0	0.00105	0.3904	0.5800	58
7.	350	0	0.00115	0.4276	0.5400	54

Figure 1: Plot of inhibition efficiency of carbon steel as a function of various concentration of DMBP at pH 7



3.1 Influence of Zn^{2+} ion on IE of DMBP

The inhibitive effect of Zn^{2+} and its synergistic influence on the IE of DMBP is given in Tables 2 and 3 and also graphically represented in Figure 2 and 3 respectively. It is observed from the table 2 that when the concentration of Zn^{2+} ions is increases IE also increases. After reaching optimum concentration of 90 ppm IE is found to decrease with rise in concentration. It is observed from the Table 3 that a synergistic effect is exist between DMBP and Zn^{2+}. For example 90 ppm of Zn^{2+} has 66.8%. 200 ppm of DMBP has 65% IE. Interestingly their combination has 71% IE. This suggests that a synergism exist between Zn^{2+} and DMBP.

Table 2: Corrosion rates of mild steel in neutral aqueous environment (Cl^- = 60 ppm) in the presence and absence of inhibitor and the inhibition efficiencies obtained by the weight-loss method.

Inhibitor system: NaCl (60ppm) +Zn^{2+} pH=7
Immersion time: 3 Hours

S.No	Conc. of DMBP (ppm)	Conc. of Zn^{2+} (ppm)	CR (mmpy)	Surface coverage (θ)	IE (%)
1 .Blank	0	0	0.9297	-	-
2.	0	50	0.4369	0.5300	53
3.	0	60	0.3719	0.6000	60
4.	0	70	0.3347	0.6400	64
5.	0	90	0.3086	0.6680	66.8
6.	0	100	0.3719	0.6000	60
7.	0	120	0.4183	0.5500	55

Figure 2: Plot of inhibition efficiency of carbon steel as a function of various concentration of DMBP at pH 7

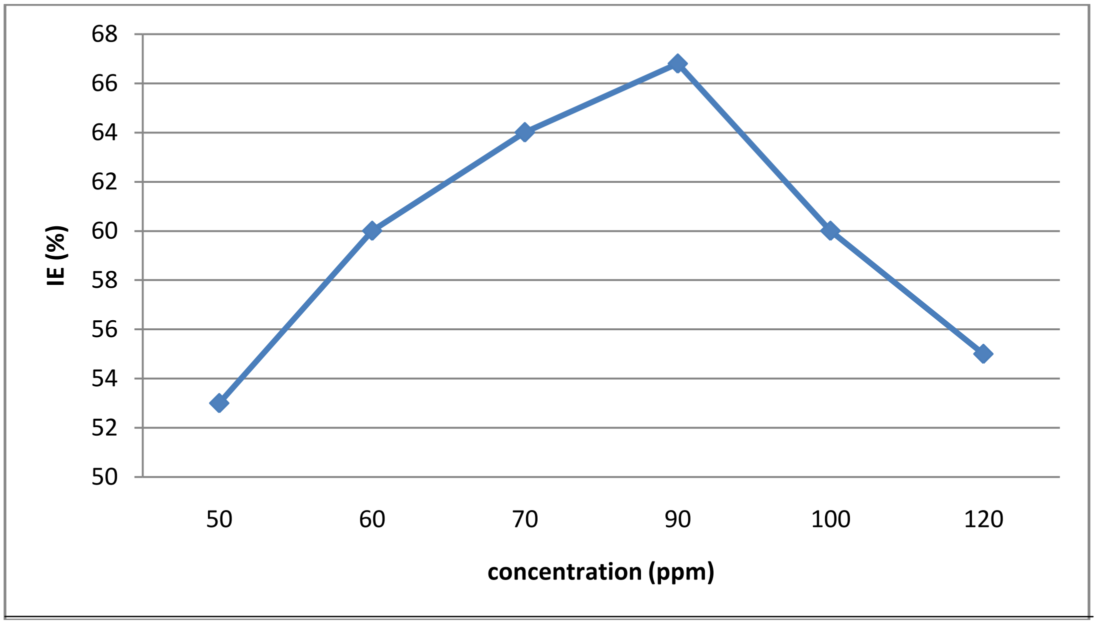


Table 3: Results of corrosion rates (CR) and inhibition efficiencies (IE) of mild steel immersed in aqueous medium containing (60 ppm Cl^- ion) in the absence and presence of inhibitors by weight loss method

Conc. Of DMBP (ppm)	Conc. Of Zn^{2+}									
	0 ppm		20 ppm		60 ppm		**90 ppm**		120 ppm	
	CR (mmpy)	IE (%)	CR (mmpy)	IE (%)	CR (mmpy)	IE (%)	CR (mmpy)	IE (%)	CR (mmpy)	IE (%)
0	0.9297	-	0.7521	19.1	0.3719	60	0.3086	66.8	0.4183	55
10	0.8553	8	0.7438	20	0.4462	52	0.3049	67.2	0.4128	55.6
20	0.7438	20	0.7252	22	0.4276	54	0.3012	67.6	0.4072	56.2
50	0.7066	24	0.6322	32	0.3904	58	0.2975	68	0.3904	58
75	0.6787	27	0.5578	40	0.3719	60	0.2949	68.3	0.3830	59
100	0.6322	32	0.4462	52	0.3254	65	0.2930	68.5	0.3719	60
150	0.4090	56	0.4183	55	0.3068	67	0.2901	68.8	0.3533	62
200	**0.3254**	**65**	**0.3904**	**58**	0.2975	68	**0.2696**	**71**	0.3347	64
300	0.3904	58	0.3719	60	0.3254	65	0.3068	67	0.3719	60
350	0.4276	54	0.3570	61.6	0.3161	66	0.2975	68	0.3719	60

Inhibitor system: DMBP +Zn^{2+} ion

Immersion Time: 3 hours

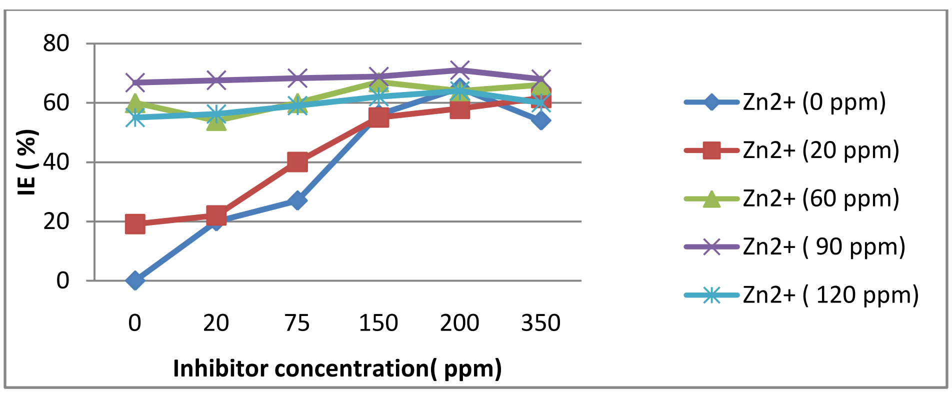


Figure 3: Plot of inhibition efficiency of carbon steel as a function of various concentration of DMBP and Zn^{2+} at pH 7

3.2 Biocidal efficiency of CTAB on binary inhibitor (DMBP-Zn^{2+}) system

The biocidal efficiency (8-12) of DMBP-Zn^{2+}-CTAB system is given in Table 6. It is found that CTAB shows very good biocidal efficiency in a wide concentration range (i.e., 10 to 250 ppm). It seems that CTAB has biocidal activity as it aggregates together. The number of bacterial colonies formed as a function of concentration of CTAB in presence of DMBP-Zn^{2+}-CTAB system is shown in Fig. 7.Mild steel widely used in the fabrication of cooling water pipes and heat exchanger tubes(13,14). Fouling and microbial corrosion causes many problems and if the water used in these systems contain 1×10^4 CFU/ml then there is no problem of microbial corrosion. When 10 ppm of CTAB along with the inhibitor DMBP-Zn^{2+} is added unacceptable value of 2.4×10^3 CFU/ml is counted. 50 ppm CTAB in the same system has shown no sign of colonies in the system. It proves that a mixture of 60 ppm Cl^-, 100 ppm DMBP, 50 ppm Zn^{2+} and 50 ppm CTAB provides 100% biocidal efficiency. The 100% biocidal efficiency as evidenced by reported results in earlier works (9-15) proved that the cationic surfactant CTAB along with other organic and inorganic inhibitors may be applied as an excellent biocide in cooling water systems.

Table 4: Corrosion rate of mild steel in neutral aqueous environment containing CTAB (Cl^- =60 ppm) in the absence of inhibitor and the inhibition efficiencies obtained by weight loss method.

Conc. of CTAB (ppm)	Corrosion rate (mmpy)	Inhibition efficiency (%)
0	0.9297	-
10	0.3719	60
20	0.3347	64
30	0.2231	76
40	0.1488	84
50	0.1116	88
80	0.2231	76
100	0.4835	48

Figure 4: Effect of CTAB on IE

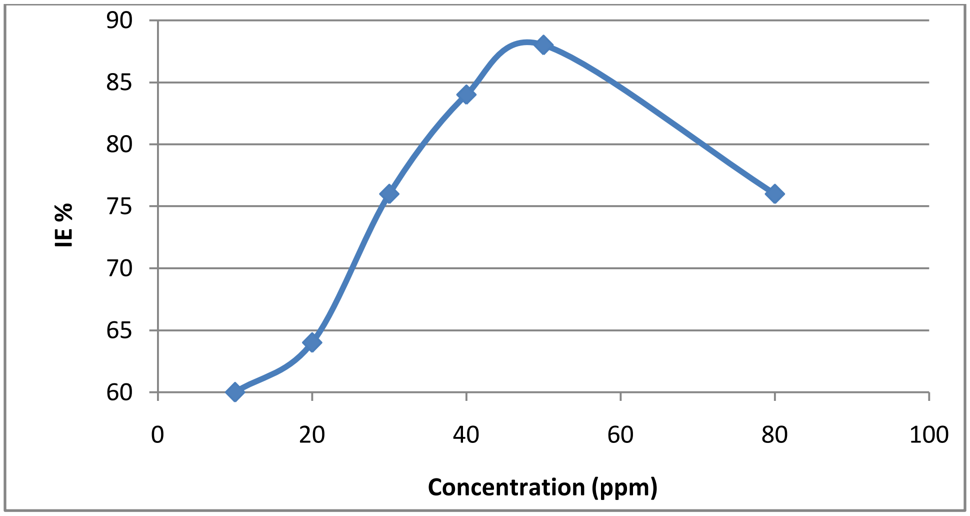


Table 5: Corrosion rate of mild steel in neutral aqueous environment (Cl^- =60 ppm) in the presence and absence of inhibitor and the inhibition efficiencies obtained by weight loss method.

Inhibitor system: DMBP+Zn^{2+} +CTAB

Conc.of DMBP (ppm)	Conc.of Zn^{2+} (ppm)	Conc. of CTAB (ppm)	Corrosion rate (mmpy)	Inhibition efficiency (%)
0	0	0	0.9297	-
0	90	0	0.3086	66.8
200	0	0	0.3254	65
200	90	0	0.2696	71
200	90	10	0.2603	72
200	90	20	0.1488	84
200	90	30	0.1118	88
200	90	40	0.0744	92
200	90	50	0.3719	96
200	90	100	0.2231	76
200	90	200	0.2975	68

Figure 5: Effect of CTAB on IE of DMBP+Zn^{2+}

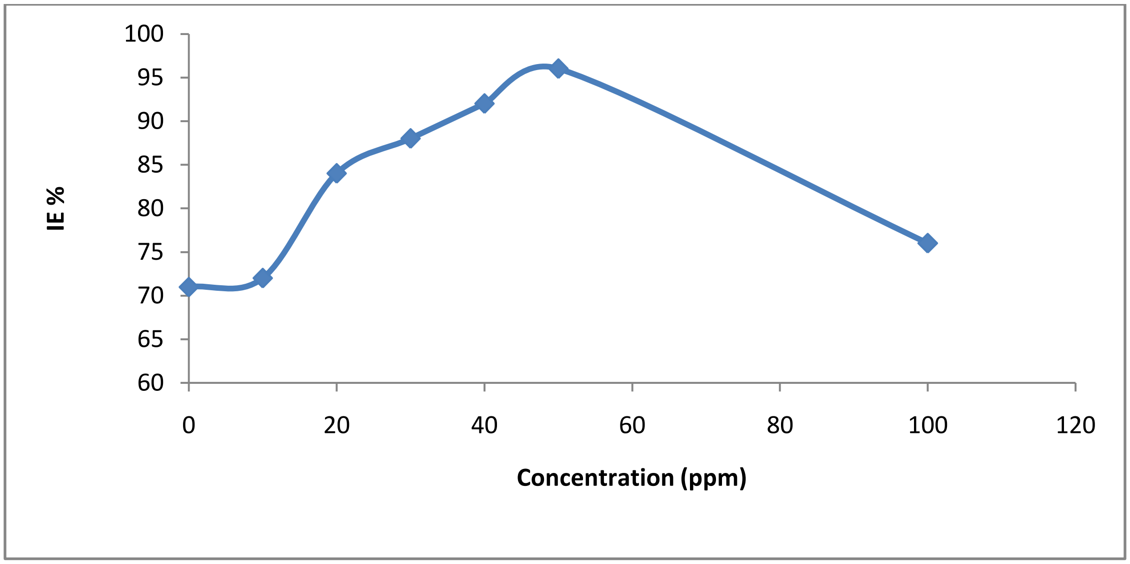


Table 6: Biocidal efficiencies of various environmental system: mild steel immersed in DMBP + Zn^{2+} +CTAB

Conc. of Cl^- ion (ppm)	Conc. of DMBP (ppm)	Conc. of Zn^{2+} (ppm)	Conc. of CTAB (ppm)	Colony forming unit (CFU/ml)	Biocidal efficiency (BE %)
60	0	0	0	1 X 10^7	-
60	200	90	0	1 X 10^6	90
60	200	90	10	2.4 X 10^3	99.99
60	200	90	50	Nil	100
60	200	90	100	Nil	100
60	200	90	150	Nil	100
60	200	90	200	Nil	100
60	200	90	250	Nil	100

Figure 6: Biocidal efficiency of CTAB as a function of number of colony forming unit in relating to concentration of biocide CTAB

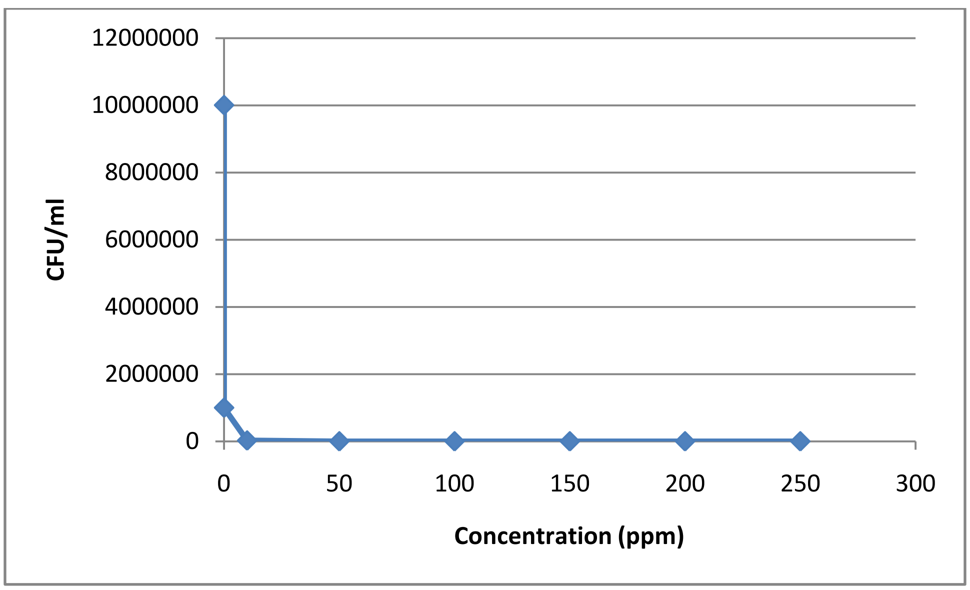
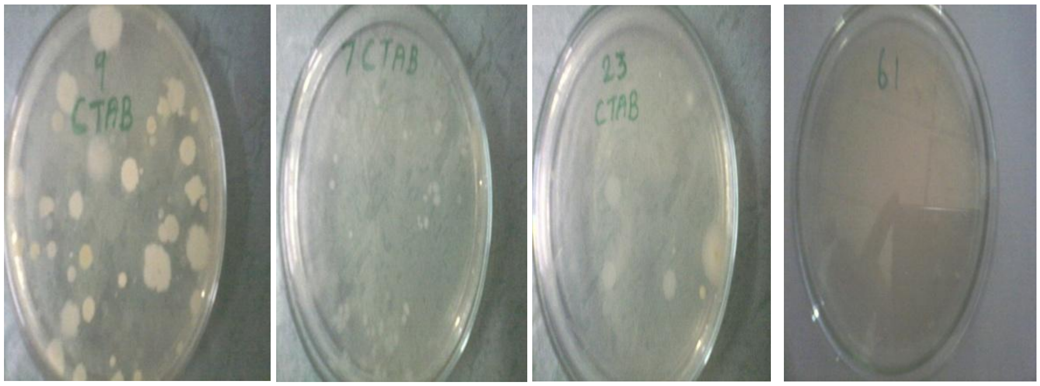


Figure 7: Schematic pictures of Bacterial colonies in petri dishes containing corrosive and inhibitor systems.

CORRODED (CFU) INHIBITED



a b c d

Figure 7a: CFU/ml in 60 ppm Cl^- + 0 ppm CTAB

Figure 7b: CFU/ml in 60 ppm Cl^- + 30 ppm CTAB

Figure 7c: CFU/ml in 60 ppm Cl^- + 40 ppm CTAB

Figure 7d: CFU/ml in 60 ppm Cl^- + 50 ppm CTAB

3.3 Biocidal efficiency of SDS on binary inhibitor (DMBP-Zn^{2+}) system

The biocidal efficiency of DMBP-Zn^{2+}-SDS system is given in Table 9. It is found that SDS has very good biocidal efficiency in a wide concentration range (i.e., 10 to 250 ppm). It seems that SDS has biocidal activity as it aggregates together. The number of bacterial colonies formed as a function of concentration of SDS in presence of DMBP-Zn^{2+}-SDS system is shown in Fig. 11. When 10 ppm of SDS along with the inhibitor DMBP-Zn^{2+} is added unacceptable value of 1.4×10^3 CFU/ml is counted. However,when 40 ppm SDS of the same system has shown no sign of colonies in the system. It proves that a mixture of 60 ppm Cl^-, 200 ppm DMBP-90 ppm Zn^{2+}-40 ppm SDS, provides 100% biocidal efficiency. The 100% biocidal efficiency is evidenced by reported results in earlier works (12, 16) proved that the cationic surfactant SDS along with other organic and inorganic inhibitors may be used as an excellent biocide in cooling water systems.

Table 7: Corrosion rate of mild steel in neutral aqueous environment containing SDS (Cl^- =60 ppm) in the absence of inhibitor and the inhibition efficiencies obtained by weight loss method.

Conc. of SDS (ppm)	Corrosion rate (mmpy)	Inhibition efficiency (%)
0	0.9297	-
5	0.4463	52
10	0.2603	72
20	0.1960	80
30	0.1488	84
40	0.0744	92
60	0.2603	72
70	0.2975	68

Figure 8: Effect of SDS on IE

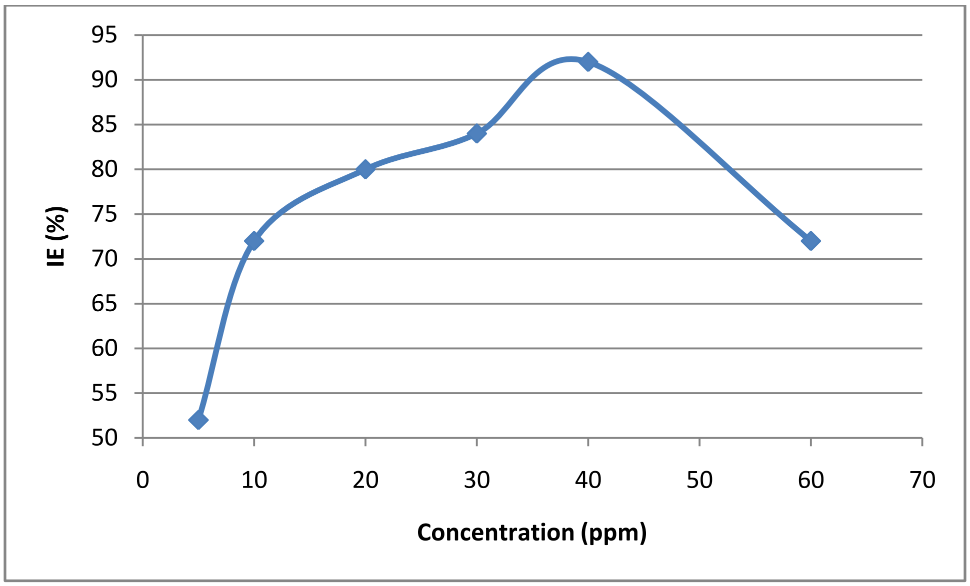


Table 8: Corrosion rate of mild steel in neutral aqueous environment (Cl^- =60 ppm) in the presence and absence of inhibitor and the inhibition efficiencies obtained by weight loss method.

Inhibitor system: DMBP+Zn^{2+} +SDS

Conc.of DMBP (ppm)	Conc.of Zn^{2+} (ppm)	Conc. of SDS	Corrosion rate (mmpy)	Inhibition efficiency
0	0	0	0.9297	-
0	90	0	0.3086	66.8
200	0	0	0.3254	65
200	90	0	0.2696	71
200	90	5	0.2231	76
200	90	10	0.1859	80
200	90	20	0.1488	84

200	90	30	0.1116	88
200	90	40	0.0372	96
200	90	70	0.1488	84
200	90	150	0.2603	72
200	90	200	0.3347	64

Figure 9: Effect of SDS on IE of DMBP+Zn^{2+}

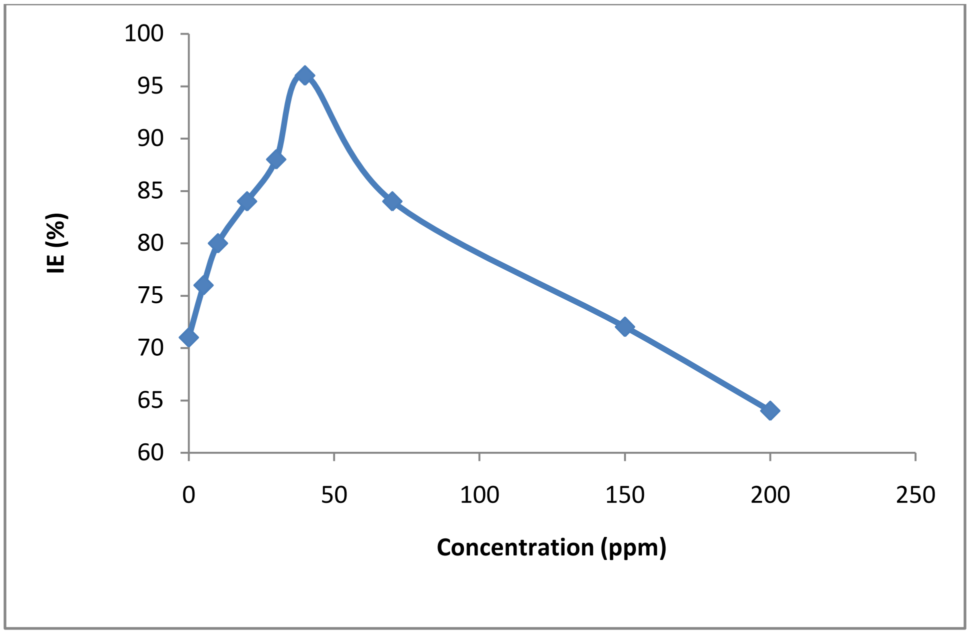


Table 9: Biocidal efficiencies of various environmental system: mild steel immersed in DMBP+ Zn^{2+} + SDS

Conc. of Cl^- ion (ppm)	Conc. of DMBP (ppm)	Conc. of Zn^{2+} (ppm)	Conc. of SDS (ppm)	Colony forming unit (CFU/ml)	Biocidal efficiency (BE %)
60	0	0	0	1 X 10^7	-
60	200	90	0	1 X 10^6	90
60	200	90	10	1.4 X 10^3	99.99
60	200	90	40	Nil	100
60	200	90	100	Nil	100
60	200	90	150	Nil	100
60	200	90	200	Nil	100
60	200	90	250	Nil	100

Figure 10: Biocidal efficiency of SDS as a function of number of colony forming unit in relating to concentration of biocide SDS

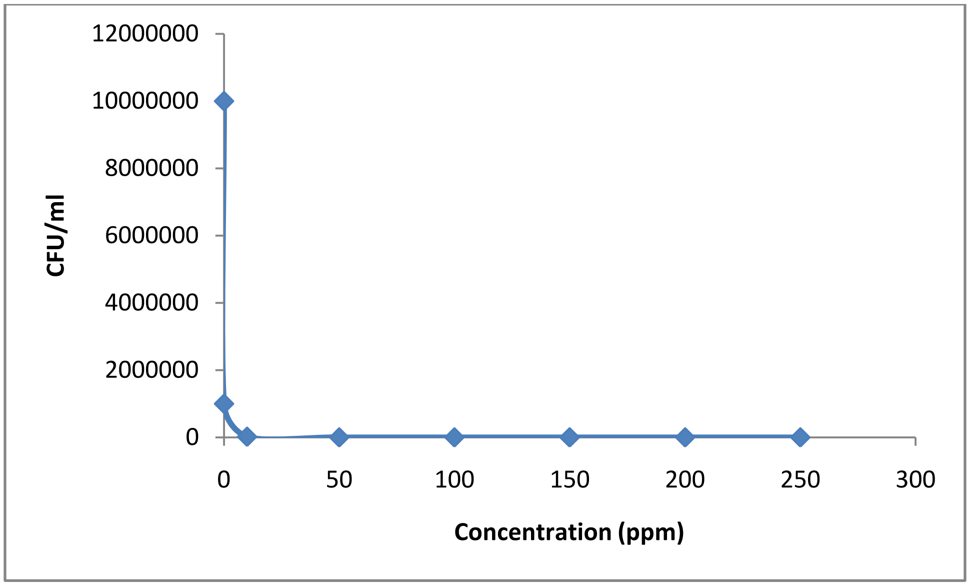


Figure 11: Schematic pictures of Bacterial colonies

CORRODED (CFU) INHIBITED

a b c d

Figure 11a: CFU/ml in 60 ppm Cl^- + 0 ppm SDS

Figure 11b: CFU/ml in 60 ppm Cl^- + 30 ppm SDS

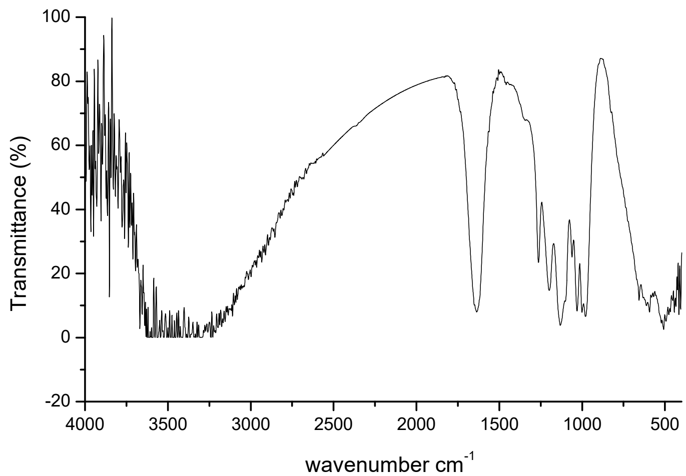
Figure 11c: CFU/ml in 60 ppm Cl^- + 40 ppm SDS

Figure 11d: CFU/ml in 60 ppm Cl^- + 50 ppm SDS

3.4 FT-IR Spectral Study

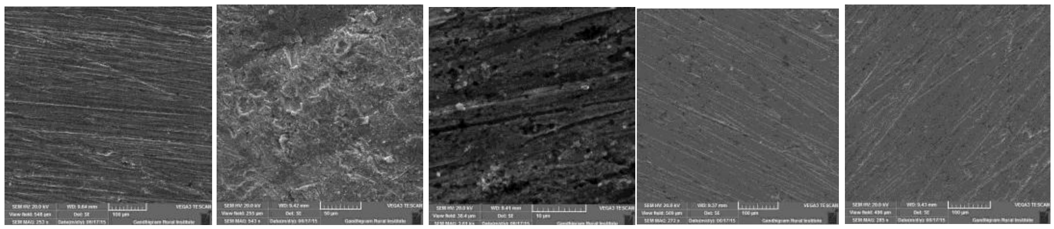
In pure DMBP the P-O stretching frequency appears at 1058 cm^{-1}. The P-OH group causes absorption at 1036 cm^{-1} and P (O) (OH) group at 3460 cm^{-1}. The absorption band at 1058 cm^{-1} represents P-O stretching frequency. The absorption at 1299 cm^{-1} represents P=O stretching. The absorption band due to the bending of O-P-O appears at 496 cm^{-1}, 569 cm^{-1} and 642 cm^{-1}. The increase in P=O stretching frequency of the ester from 1299 cm^{-1} to 1314 cm^{-1} relative to the oxides, results from the electro negativity of the attached alkoxy groups. Thus FTIR spectrum of DMBP with molecular formula $C_{12}H_{19}O_3P$ is characterized by the FTIR spectrum.

The FTIR spectrum of the film formed on the surface of the carbon steel after immersion in the test solution containing DMBP-Zn^{2+} and Cl^- is shown in Figure 12.The shift P-O frequency from 1058cm^{-1} to 1044 cm^{-1} indicates that the oxygen atom of the P-O group has coordinated with Fe^{2+} resulting in the formation of Fe^{2+}- DMBP complex formed on the anodic sites of the metal surface. The P=O stretching frequency increased from 1299cm^{-1} to 1335 cm^{-1} it also conforms the complex formation between the inhibitor and Fe^{2+}. The peak at 3343 cm^{-1} due to OH stretching. The band due to Zn-O stretch appears at 1314 cm^{-1}. These results is confirmed the presence of $Zn(OH)_2$ deposited on the cathodic sites of the metal surface (17).



3.5 **Surface morphology study**

The surface morphologies of carbon steel specimen in aqueous solution containing 60 ppm Cl^- ion in the presence and absence of inhibitor after 3 hours of immersion period are examined using a Scanning Electronic Microscopy (SEM) is displayed in fig 12-a – 12d. In the absence of inhibitor Fig 12(a) a very smooth surface is obtained due to the absence of corrosive products on the surface. In the presence of 60 ppm Cl^- ion (corrosive medium) (Fig 12(b)) a very rough surface is observed due to rapid corrosion attack of carbon steel by chloride anions. There are few pits surrounded by iron oxide layer which almost fully covers the carbon steel surface, revealing that pit formation under these conditions occurs continuously during the exposure period while iron oxide builts up over the surface in the presence of the inhibitor (fig 12(c)) the pits and roughness are reduced, very smooth surface is observed indicating the formation of the protective film. Figure 12 (d) and 12 (e) shown that CTAB, SDS and inhibitor combination act as good system for bacterial control as well as corrosion control.



SEM photograph of
Figure 12(a):polished mild steel
Figure 12(b):mild steel specimen immersed in aqueous medium containing 60 ppm Cl-ion
Figure 12(c): mild steel exposed aqueous medium containing 60 ppm Cl-ion and inhibitor solutions (200 ppm DMBP + 90 ppm Zn^{2+} ion)
Figure 12(d): mild steel exposed aqueous medium containing 60 ppm Cl-ion and inhibitor solutions (200 ppm DMBP + 90 ppm Zn^{2+} ion + 50 ppm CTAB).
Figure 12(e): mild steel exposed aqueous medium containing 60 ppm Cl-ion and inhibitor solutions (200 ppm DMBP + 90 ppm Zn^{2+} ion + 40 ppm SDS).

4. Conclusion

The following conclusion may be drawn from the study

- Results obtained from the experimental data shows that DMBP act as a effective inhibitor on mild steel in aqueous medium
- The corrosion process was inhibited by adsorption of the DMBP on the metal surface.
- The formulation (60 ppm+200 ppm +90 ppm+50 ppm) Cl^- +DMBP+Zn^{2+} + CTAB had 96% corrosion inhibition efficiency
- The formulation (60 ppm+200 ppm +90 ppm+40 ppm) Cl^- +DMBP+Zn^{2+} + SDS had 96% corrosion inhibition efficiency
- The CTAB and SDS are enhanced the microbial corrosion inhibition on the mild steel surface in the aqueous medium.
- SEM conform the presence of a protective film on the mild steel surface. The bacterial enumeration has been reduced by the addition of CTAB and SDS to the inhibitor system.

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