

## Surface Analysis of Inhibitor Film Formed by Butane Sulphonic Acid-Zn<sup>2+</sup> System on Carbon Steel in Aqueous Medium

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The inhibition effect of butane sulphonic acid as its sodium salt (SBS) in combination with zinc ion (Zn<sup>2+</sup>) on the corrosion of carbon steel in a dam water has been studied using weight-loss and potentiodynamic polarization methods. Results of weight loss method indicated inhibition efficiency increased with increasing inhibitor concentration. The influence of sodium potassium tartrate (SPT) on the inhibition efficiency of the SBS-Zn<sup>2+</sup> system has been evaluated. Polarization study reveals that SBS-Zn<sup>2+</sup> system controls the cathodic reaction predominantly. The protective film is found to be UV fluorescent. The nature of the metal surface has been analyzed by scanning electron microscope (SEM) and energy dispersive X-ray detector (EDAX).

**Key Words:** Carbon steel, Corrosion, Synergistic effect, SEM, EDAX.

### INTRODUCTION

Water is the most commonly used cooling fluid to remove unwanted heat from heat transfer surfaces. At the present there is an urgent need for better utilization of the limited water supplies due to population growth and increasing development. Due to this, open re-circulating cooling water systems that reuse cooling water are frequently used at large central utility stations, at chemical, petrochemical and petroleum refining plants, in steel and paper mills and at all types of processing plants<sup>1</sup>. Open re-circulating cooling water systems continuously reuse the water that passes through the heat transfer equipment. Water contains dissolved and suspended solids, dissolved and suspended organic matters and dissolved gases<sup>2</sup>. Finally, the open re-circulating system, with longer holding times at higher temperatures in the presence of higher dissolved solids concentrations, produces more severe corrosion and scale<sup>2</sup>.

Organic compounds have been used successfully as corrosion inhibitors, in several industries during the pickling of metals, cleaning of boilers and acidification of oil wells. Detailed studies have been carried out on the use of organic compounds containing oxygen, sulfur or nitrogen to reduce corrosion attack on steel<sup>3-6</sup>. Most organic inhibitors are believed

to work by adsorbing onto the metal surface, displacing water molecules and forming a compact barrier film<sup>7</sup>. The availability of lone pairs and  $\pi$  electrons in the inhibitor molecules facilitates the electron transfer from the inhibitor to the metal, forming a coordinate covalent bond<sup>8</sup>. The strength of the adsorption bond depends on the electron density, on the donor atom of the functional group and also on the polarizability of the group. Sulfur compounds are very effective corrosion inhibitors, because sulfur enhances adsorption to the metal surface<sup>9</sup>. A survey of the literature reveals that the corrosion inhibition of 2-naphthalenesulfonic acid, 2,7-naphthalenedisulfonic acid and 2-naphthol-3, 6-disulfonic acid on armco-iron electrode in sulfuric acid has been investigated by Vracar and Drazic<sup>10</sup>. The inhibition efficiency, changes with the number of functional groups substituted on benzene ring and increases with concentration<sup>10</sup>. The inhibition action of 2-mercaptobenzoxazol, 2-mercapto benzimidazole, N-acetyl pyridinium bromide and propargyl benzene sulphonate on the corrosion of carbon steel in acid media have also been studied by Prakash *et al.*<sup>11</sup>. The corrosion inhibitors are used to reduce corrosion damage in sub-surface equipments in oil well field. The corrosion inhibition activity was studied by gravimetric and potentiostatic polarization methods in presence of 20 % HCl<sup>11</sup>. Manickavasagam *et al.*<sup>12</sup> have reported the corrosion inhibition of poly(styrene

sulphonic acid)-doped poly aniline on carbon steel in acid media. The polymer acts as an anodic inhibitor. The adsorption of the compound on the metal surface obeys Temkin's adsorption isotherm. Aliev<sup>13</sup> has described the influence of salts of alkyl phenol sulphonic acid on the corrosion of ST3 steel. The protective effect increases with temperature. The investigated compounds inhibit corrosion of ST3 steel as a result of chemical adsorption. Shakkthivel and Vasudevan<sup>14</sup> have studied the effect of acrylic acid-diphenylamine sulphonic acid copolymer threshold inhibitor for sulphate and carbonate scales in cooling water systems. The results show that the polymer acts as a very good antiscaling inhibitor both in the carbonate and sulphate brines. Copolymer of acrylic acid-diphenyl amine sulphonic acid can be used safely in cooling water systems<sup>14</sup>.

Perusal of several literature reveal that there is no information regarding the use of butane sulphonic acid (SBS) in combination with zinc ion ( $Zn^{2+}$ ) as corrosion inhibitor. This paper focuses on the inhibition efficiency (IE) of butane sulphonic acid as its sodium salt (SBS) in controlling corrosion of carbon steel immersed in dam water in the absence and presence of  $Zn^{2+}$ . The investigation is performed using weight loss method and polarization technique. The influence of sodium potassium tartrate (SPT) on the inhibition efficiency of the SBS- $Zn^{2+}$  system has also been studied. The morphology of the protective film was examined by fluorescence spectra, scanning electron microscopy (SEM) and energy dispersive X-ray detector (EDAX).

The medium which is used in the present study is dam water collected from Sothuparai dam in Tamil Nadu, constructed across the Vaigai River, the water from which is used by the industries located downstream for cooling systems.

## EXPERIMENTAL

The chemicals used in this study, sodium butane sulfonate (inhibitor),  $ZnSO_4 \cdot 7H_2O$  ( $Zn^{2+}$  ions) co inhibitor and sodium potassium tartrate (SPT) were AR grade.

**Preparation of the specimen:** Carbon steel specimens of size 1.0 cm  $\times$  4.0 cm  $\times$  0.2 cm and chemical composition 0.026 % sulphur, 0.06 % phosphorous, 0.4 % manganese, 0.1 % carbon and the rest iron (specimen density 7.87 g/cm<sup>3</sup>), were polished to a mirror finish and degreased with trichloroethylene and used for the weight loss method and surface examination studies.

**Weight-loss method:** Carbon steel specimens were immersed in 100 mL of the dam water containing various concentrations of the inhibitor (sodium butane sulphonate) 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 AUY 220 Shimadzu). The corrosion products were cleaned with Clarke's solution prepared by dissolving 20 g of  $Sb_2O_3$  and 50 g of  $SnCl_2$  in 1 L of conc. HCl of specific gravity (1.9)<sup>15</sup>. The corrosion inhibition efficiency was then calculated using the equation

$$IE = 100 \left[ 1 - \left( \frac{W_2}{W_1} \right) \right] \% \quad (1)$$

where  $W_1$  is the weight loss value in the absence of inhibitor and  $W_2$  is the weight loss value in the presence of inhibitor. Corrosion rate was calculated using the formula<sup>16</sup>

$$\text{Mils per year (mpy)} = \frac{534W}{DAT} \quad (2)$$

$W$  = weight loss in milligrams,  $D$  = density of specimen g/cm<sup>3</sup>,  $A$  = area of specimen in square inches,  $T$  = exposure time in hours. (Where mpy is the rate of penetration in milli inches per year which is the customary unit for corrosion rate).

**Potentiodynamic polarization:** Polarization studies were carried out in a CHI-electrochemical work station with impedance model 660 A. It was provided with iR compensation facility. A three electrodes cell assembly was used. The working electrode was carbon steel. Saturated calomel electrode was the reference electrode. Platinum was the counter electrode. From polarization study, corrosion parameters such as corrosion potential ( $E_{corr}$ ), corrosion current ( $I_{corr}$ ), Tafel slopes anodic =  $b_a$  and cathodic =  $b_c$  were calculated and linear polarization study (LPR) was done. The scan rate (V/S) was 0.01. Hold time at  $E_f$  (s) was zero and quiet time (s) was two.

**UV visible absorbance spectra and fluorescence spectra:** The instrument UV Spetord S-100 Analytic Jena was used for recording UV visible absorbance spectra. Fluorescence spectra were recorded in a Glaxo F-6300 Spectro fluorimeter

**Scanning electron microscopic studies (SEM):** The carbon steel immersed in blank solution and in the inhibitor solution for a period of one day was removed, rinsed with double distilled water, dried and observed in a scanning electron microscope to examine the surface morphology. The surface morphology measurements of carbon steel were examined using Jeol MODEL6390 computer controlled scanning electron microscope. The elemental analysis of the carbon steel surface at the same condition was carried out using an energy dispersive X-ray analyzer unit attached to the SEM machine.

## RESULTS AND DISCUSSION

**Weight-loss study:** The physicochemical parameters of dam water are given in Table-1.

Parameters	Result
Appearance	Brownish
Total dissolved solids	100 ppm
Electrical conductivity	140 $\mu$ S/cm
pH	8.25
Total hardness as $CaCO_3$	50 ppm
Calcium	10 ppm
Magnesium	06 ppm
Iron	1.2 ppm
Nitrate	10 ppm
Chloride	10 ppm
Sulphate	02 ppm

**Inhibition efficiency (IE) and the corrosion rates (CR) in mils per year (mpy) of carbon steel immersed in dam water in the presence of SBS- $Zn^{2+}$  system:** The corrosion inhibition efficiencies and the corresponding corrosion rates (mils per year) of sodium butane sulphonate (SBS)- $Zn^{2+}$  systems are given in Table-2.

It is found that the inhibition efficiency increases as the concentration of sodium butane sulfonate increases. As the

TABLE-2  
INHIBITION EFFICIENCIES (IE) AND THE CORROSION RATES MILS PER YEAR (mpy) OF CARBON STEEL IMMERSSED IN DAM WATER OFFERED BY SBS-Zn<sup>2+</sup> SYSTEM

Inhibitor SBS (ppm)	Zn <sup>2+</sup> (ppm)					
	0		15		30	
	IE (%)	CR (mpy)	IE (%)	CR (mpy)	IE (%)	CR (mpy)
0	-	4.4384	12	3.9058	21	2.5063
50	-23	5.4592	14	3.8170	42	2.5743
100	-17	5.1929	17	3.6839	49	2.2636
150	-09	4.8379	20	3.5507	53	2.0860
200	02	4.3496	22	3.4620	58	1.8641
250	05	4.2165	25	3.3288	61	1.3759

concentration of Zn<sup>2+</sup> increases, inhibition efficiency also increases. A synergistic effect exists between sodium butane sulfonate and Zn<sup>2+</sup>. For example, 250 ppm of sodium butane sulfonate has 5 % inhibition efficiency. 30 ppm of Zn<sup>2+</sup> has 21 % inhibition efficiency. However, interestingly, the formulation consisting of 250 ppm of sodium butane sulfonate and 30 ppm of Zn<sup>2+</sup> has 61 % inhibition efficiency. That is, mixture of inhibitors shows better inhibition efficiency than the individual inhibitors<sup>17</sup>.

**Synergism parameter (S<sub>I</sub>):** Synergism parameters (S<sub>I</sub>) are indications of synergistic effect existing between inhibitors. When synergism parameter value is greater than one, synergistic effect exists between the inhibitors<sup>18,19</sup>. Synergism parameter value is found to be greater than one indicating synergistic effect exists between Zn<sup>2+</sup> of concentrations 15 ppm and 30 ppm with various concentrations of sodium butane sulfonate. The results are given in Table-3.

TABLE-3  
SYNERGISM PARAMETER (S<sub>I</sub>)

SBS (ppm)	I <sub>1</sub>	SBS-Zn <sup>2+</sup> (15ppm) I <sub>(1+2)</sub>	S <sub>I</sub>	SBS-Zn <sup>2+</sup> (30ppm) I <sub>(1+2)</sub>	S <sub>I</sub>
50	-0.23	0.14	1.2586	0.42	1.6753
100	-0.17	0.17	1.2405	0.49	1.8124
150	-0.09	0.20	1.1990	0.53	1.8321
200	0.02	0.22	1.1056	0.58	1.8433
250	0.05	0.25	1.1147	0.61	1.9244

$$S_I = \frac{1 - I_{1+2}}{1 - I'_{1+2}} \quad [3]$$

where I<sub>1+2</sub> = (I<sub>1</sub> + I<sub>2</sub>) - (I<sub>1</sub>, I<sub>2</sub>), I<sub>1</sub> = surface coverage of inhibitor (sodium butane sulfonate), I<sub>2</sub> = surface coverage of inhibitor (Zn<sup>2+</sup>), I'\_{1+2} = combined surface coverage of inhibitors (sodium butane sulfonate) and (Zn<sup>2+</sup>), surface coverage = inhibition efficiency %/100. I<sub>2</sub> for Zn<sup>2+</sup> (15 ppm) = 0.12 and I<sub>2</sub> for Zn<sup>2+</sup> (30 ppm) = 0.21.

**Influence of sodium potassium tartrate (SPT) on the inhibition efficiency of sodium butane sulfonate (50 ppm)-Zn<sup>2+</sup> (15 ppm) system:** The inhibition efficiency and corrosion rate of carbon steel immersed in solutions containing sodium butane sulfonate (50 ppm) - Zn<sup>2+</sup> (15 ppm) and various concentrations of sodium potassium tartrate are tabulated in Table-4.

It is noted that for sodium potassium tartrate, inhibition efficiency increases up to the concentration 150 ppm and then it decreases. As the concentration of inhibitor (sodium potassium

TABLE-4  
INFLUENCE OF SODIUM POTASSIUM TARTRATE (SPT) ON THE IE OF SBS (50 ppm)-Zn<sup>2+</sup> (15 ppm) SYSTEM

SBS (ppm)	Zn <sup>2+</sup> (ppm)	SPT(ppm)	CR (mpy)	IE (%)
0	0	0	4.4384	-
50	15	0	3.8170	14
50	15	50	0.7989	82
50	15	100	0.4882	89
50	15	150	0.2663	94
50	15	200	0.6214	86
50	15	250	0.6214	86

tartrate) increases more of the inhibitor is transported towards the metal surface through the formation of Zn<sup>2+</sup>-inhibitor complex. On the metal surface Zn<sup>2+</sup>-inhibitor complex is converted into Fe<sup>2+</sup>-inhibitor complex. This complex is insoluble, stable and more compact. So, inhibition efficiency increases up to 150 ppm concentration. On further increasing the concentration of the inhibitor, the complex formed on the metal surface goes into solution. So, inhibition efficiency decreases gradually. Another reason may be that when more inhibitor is added, the Zn<sup>2+</sup>-inhibitor complex formed is precipitated in the bulk of the solution. Also the inhibitor is not transported towards the metal surface. Hence inhibition efficiency decreases. Similar observations have been made by Rajendran *et al.*, while studying the inhibition efficiency of Henna extract<sup>20</sup> and also by Arockia Selvi *et al.*<sup>21</sup> while studying the inhibition efficiency of diethylene-triaminepentamethylene phosphonic acid -Zn<sup>2+</sup> system.

**Analysis of polarization curves:** The potentiodynamic polarization curves of carbon steel immersed in various test solutions are shown in Fig. 1. The corrosion parameters are given in Table-5.

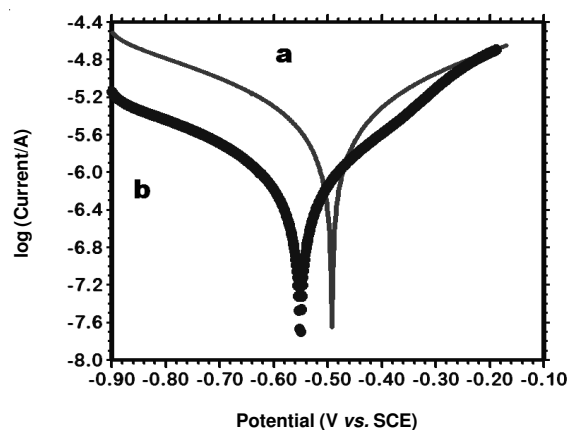


Fig. 1. Polarisation curves of carbon steel immersed in various test solutions a) dam water b) dam water containing 250 ppm of sodium butane sulfonate and 30 ppm of Zn<sup>2+</sup>

When carbon steel is immersed in dam water, the corrosion potential is -494 mV *versus* SCE. The formulation consisting of 250 ppm sodium butane sulfonate + 30 ppm Zn<sup>2+</sup> shifts the corrosion potential to -552 mV *versus* SCE. This suggests that the cathodic reaction is controlled predominantly. This is further confirmed from the data that the shift in the cathodic slope (from 203-158 mV/dec) is higher than the shift in the anodic slope (from 166-176 mV/dec). Hence, it can be said that the inhibitor system predominantly controls the cathodic reaction<sup>22</sup>.

TABLE-5  
CORROSION PARAMETERS OF CARBON STEEL IMMERSED IN DAM WATER IN  
THE PRESENCE AND ABSENCE OF INHIBITOR OBTAINED BY POLARIZATION METHOD

Inhibitor SBS (ppm)	Zn <sup>2+</sup> (ppm)	E <sub>corr</sub> mV vs SCE	I <sub>corr</sub> (A/cm <sup>2</sup> )	b <sub>a</sub> (mV/dec)	b <sub>c</sub> (mV/dec)	LPR (Ω cm <sup>2</sup> )
0	0	-494	2.66 × 10 <sup>-6</sup>	166	203	2.053 × 10 <sup>4</sup>
250	30	-552	5.50 × 10 <sup>-7</sup>	176	158	7.377 × 10 <sup>4</sup>

The corrosion current value and LPR value for dam water are 2.660 × 10<sup>-6</sup> A/cm<sup>2</sup> and 2.053 × 10<sup>4</sup> Ω cm<sup>2</sup>.

For the formulation of sodium butane sulfonate (250 ppm) + Zn<sup>2+</sup> (30 ppm), the corrosion current value has decreased to 5.500 × 10<sup>-7</sup> A/cm<sup>2</sup> and the LPR value has increased to 7.377 × 10<sup>4</sup> Ω cm<sup>2</sup>. This indicates that a protective film is formed on the metal surface. When a protective film is formed on the metal surface LPR value increases and corrosion current value decreases. The fact that the LPR value increases with decrease in corrosion current indicates that inhibition of corrosion takes place by the formation of protective film. Similar observations have been reported by Rathish *et al.*<sup>23</sup>, while studying the corrosion behaviour of metals in artificial sweat. Thangam *et al.*<sup>24</sup> while studying inhibition of corrosion of carbon steel in a dam water by sodium molybdate-Zn<sup>2+</sup> system and also by Kanimozhi and Rajendran while studying the corrosion inhibition sodium tungstate-Zn<sup>2+</sup>-ATMP system<sup>25</sup>.

#### UV-Visible absorbance spectra and fluorescence spectra:

UV-Visible absorption spectra and fluorescence spectra can be used to confirm the protective film formed on the metal surface<sup>26,27</sup>.

UV-Visible absorption spectrum of an aqueous solution of sodium butane sulphonate (SBS) is shown in Fig. 2 curve (a). Peak appears at 282 (2.98) nm. (The peak intensities are given in parentheses). The corresponding emission spectrum (λ<sub>ex</sub> = 282 nm) is shown in Fig. 3 curve (a). Peaks appear at 388.5 (2.22), 508 (0.99) and 564 (0.61) nm. Fe<sup>2+</sup>-sodium butane sulfonate complex in solution was prepared by mixing sodium butane sulfonate solution and Fe<sup>2+</sup> ion (as FeSO<sub>4</sub>·7H<sub>2</sub>O + H<sub>2</sub>O freshly prepared). Its UV-visible absorption spectrum is shown in Fig. 2 curve (b). Peaks appear at 260 (3.37) and 280 (3.01) nm. The corresponding emission spectrum (λ<sub>ex</sub> = 260 nm) is shown in Fig. 3 curve (b). Peaks appear at 398.5 (4.2), 450 (1.88) and 506 (2.66) nm. These peaks correspond to Fe<sup>2+</sup>-sodium butane sulfonate complex.

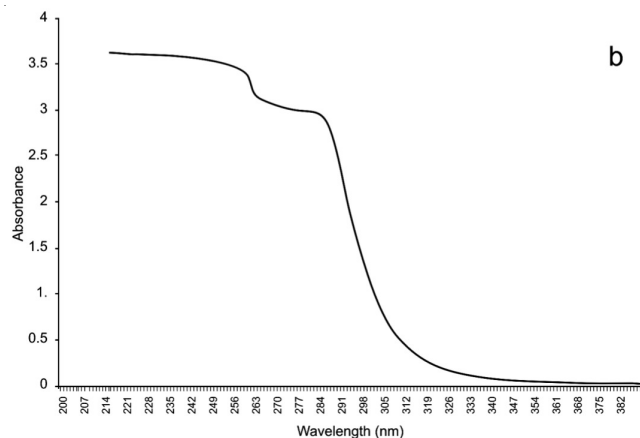
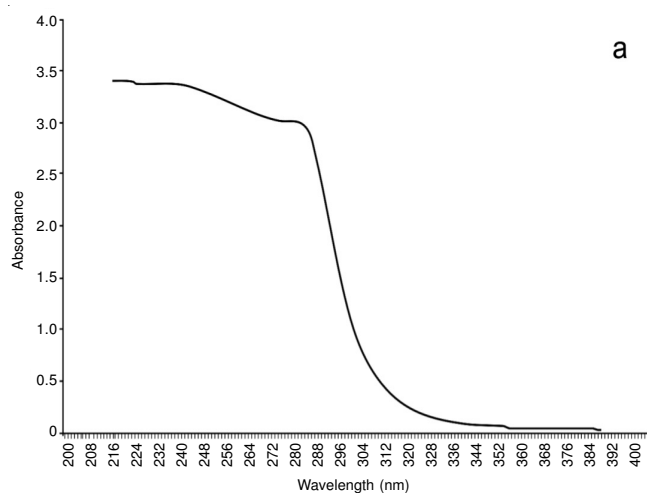


Fig. 2. UV Visible absorption spectra of solutions. (a) sodium butane sulfonate solution (b) sodium butane sulfonate + Fe<sup>2+</sup> solution

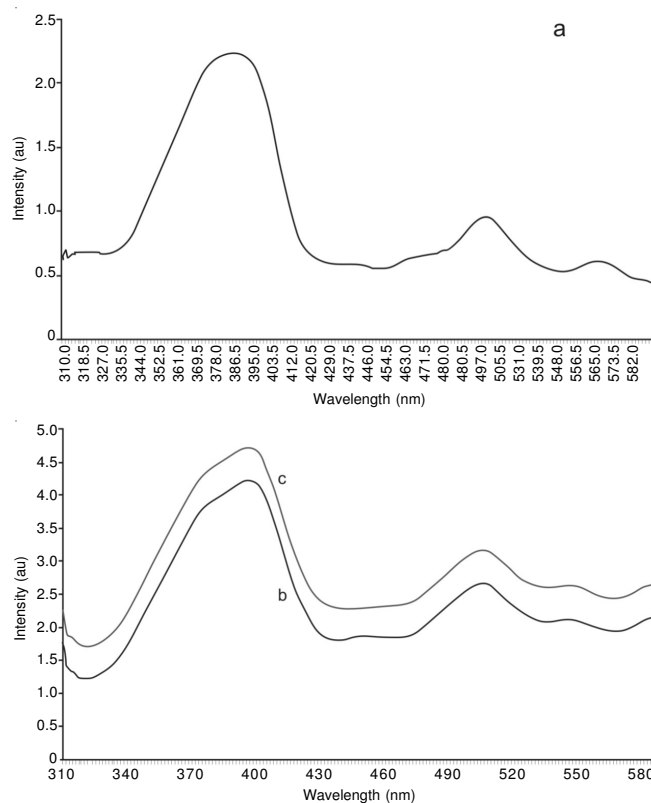
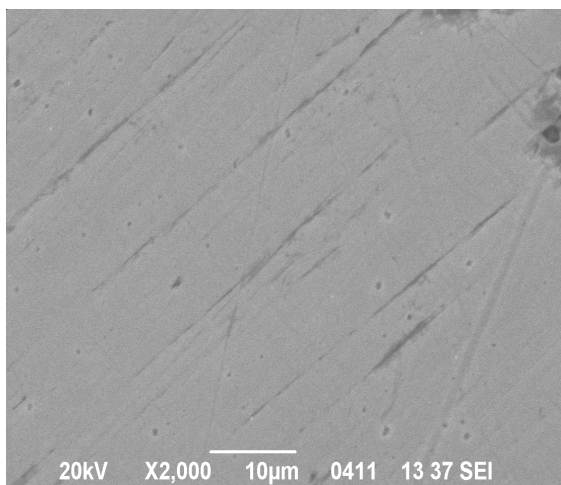


Fig. 3. Fluorescence spectra (a) sodium butane sulfonate solution (b) sodium butane sulfonate + Fe<sup>2+</sup> solution (c) film formed on metal surface after immersion in solution containing dam water + 250 ppm of sodium butane sulfonate and 30 ppm of Zn<sup>2+</sup>

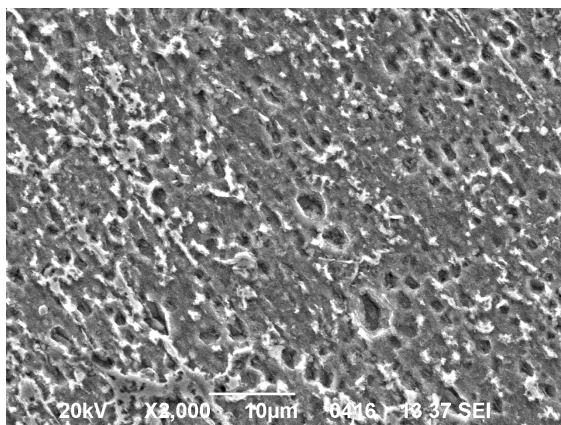
The emission spectrum (λ<sub>ex</sub> = 260 nm) of the film formed on the carbon steel metal surface after immersion in the solution containing 30 ppm of Zn<sup>2+</sup> and 250 ppm of sodium butane sulfonate is shown in Fig. 3 curve (c). Peaks appear at 397 (4.72) and 507(3.15) nm. These peaks correspond to Fe<sup>2+</sup>-sodium butane sulfonate complex. Thus fluorescence spectral

study leads to the conclusion that protective film consists of Fe<sup>2+</sup>-sodium butane sulfonate complex formed on the metal surface.

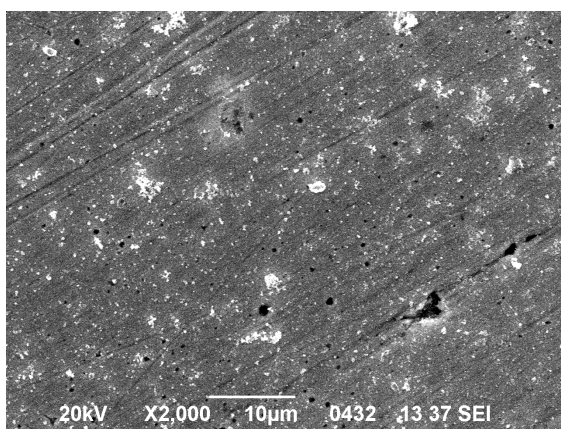
**SEM analysis of metal surface:** The SEM image of magnification (X2000) of carbon steel specimen immersed in dam water for 1 day in the absence and presence of inhibitor system are shown in Fig. 4 (b and c, respectively).



(a)



(b)



(c)

Fig. 4. SEM micrographs of (a) polished carbon steel (control)-magnification-X2000. (b) carbon steel immersed in dam water-magnification-X2000. (c) carbon steel immersed in dam water containing 250 ppm of sodium butane sulfonate and 30 ppm of Zn<sup>2+</sup>-magnification-X2000

The SEM micrographs of polished carbon steel surface (control) in Fig. 4 image (a) shows the smooth surface of the metal. This shows the absence of any corrosion products formed on the metal surface. The SEM micrographs of carbon steel surface immersed in dam water in Fig. 4 image (b) shows the roughness of the metal surface which indicates the corrosion of carbon steel in dam water. Fig. 4 image (c) indicates that in presence of 250 ppm of sodium butane sulfonate and 30 ppm of Zn<sup>2+</sup> mixture in dam water, the surface coverage increases which in turn results in the formation of insoluble complex on the surface of the metal (sodium butane sulfonate-Zn<sup>2+</sup> inhibitor complex) and the surface is covered by a thin layer of inhibitors which controls the dissolution of carbon steel. Such results have been reported earlier<sup>28</sup>.

**Surface examination by EDAX:** EDAX is used to analyze corrosion films. Fig. 5 spectrum (a) shows the EDAX analysis of carbon steel immersed in dam water. The analysis shows the presence of corrosion products elements (Fe, O and C). Fig. 5 spectrum (b) shows the EDAX analysis of carbon steel immersed in dam water containing 250 ppm of sodium butane sulfonate and 30 ppm of Zn<sup>2+</sup>. The analysis shows the presence of (Zn and S), which could be attributed to the presence of zinc and the organic molecule (sodium butane sulfonate) on the metal surface, forming a protective film<sup>29</sup>.

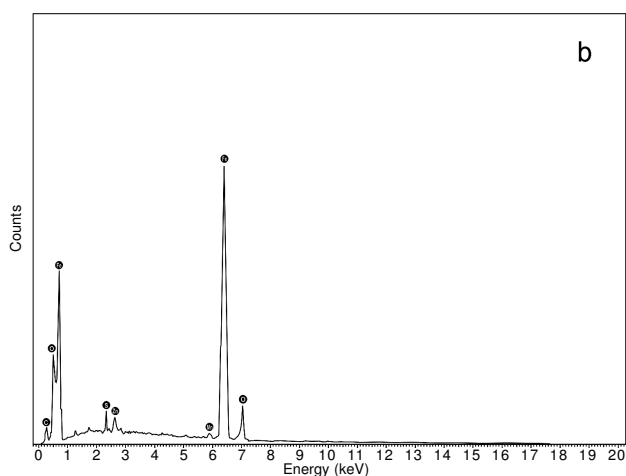
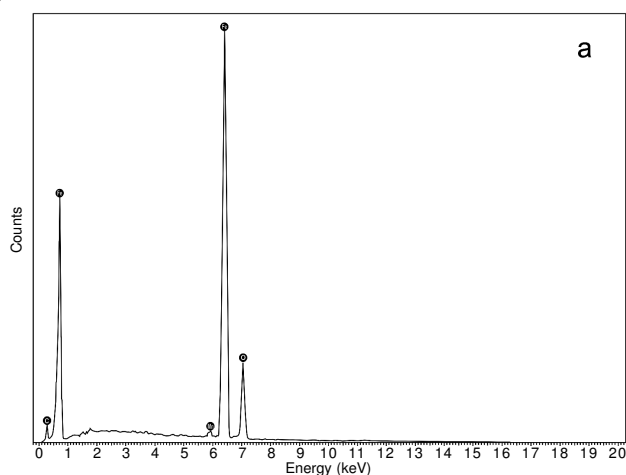


Fig. 5. EDAX analysis of (a) carbon steel immersed in dam water (b) carbon steel immersed in dam water containing 250 ppm of sodium butane sulfonate and 30 ppm of Zn<sup>2+</sup>

## Conclusion

**Present study leads to the following conclusions:** The inhibition efficiency (IE) of sodium butane sulfonate in controlling corrosion of carbon steel immersed in dam water in the absence and presence of  $Zn^{2+}$  has been evaluated by weight loss method. The formulation consisting of 250 ppm sodium butane sulfonate and 30 ppm  $Zn^{2+}$  has 61 % inhibition efficiency. Polarization study reveals that sodium butane sulfonate- $Zn^{2+}$  system controls the cathodic reaction predominantly. SEM analysis reveals that the surface coverage increases which in turn results in the formation of insoluble complex on the surface of the metal (sodium butane sulfonate- $Zn^{2+}$  inhibitor complex) and the surface is covered by a thin layer of inhibitors which controls the dissolution of carbon steel.

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