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# **ORIGINAL ARTICLES**

# Corrosion inhibition properties of the synergistic effect of 4-hydroxy-3-methoxybenzaldehyde and hexadecyltrimethylammoniumbromide on mild steel in dilute acid solutions



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# KEYWORDS

Inhibitor; Acid; Corrosion; Mild steel Abstract The synergistic effect of the corrosion inhibition behaviour of 4-hydroxy-3-methoxybenzaldehyde and hexadecyltrimethylammoniumbromide on mild steel in  $0.5\,\mathrm{M}$  H<sub>2</sub>SO<sub>4</sub> and HCl acid test solutions was studied through weight loss measurement and potentiodynamic polarization test. Results show the admixture performed effectively in both acids with inhibition efficiency above 90% in H<sub>2</sub>SO<sub>4</sub> and 80% in HCl acid at all inhibitor concentrations from both tests. Thermodynamic calculations showed the compound chemically adsorbed onto the steel surface forming a protective film. Molecular adsorption followed the Langmuir and Frumkin isotherm models.

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# 1. Introduction

The consequences of corrosion are a major problem of international importance. Corrosion is responsible for plant shutdowns and accidents, costly design and maintenance, product contamination and wastage of valuable resources

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due to the aggressive nature of industrial environments on metallic surfaces of equipment and structures such as in acid pickling of steel, chemical processing plants, oil well acidification etc. due to the natural tendency of refined metals such as mild steel to return to their natural state of existence (Mars; Introduction to Corrosion; Li et al., 2009; Zaafarany, 2013). Mild steel is the most available form of steel due to its low cost and it exhibits metallurgical characteristics required for many applications (Knowles, 1987; Classification of Carbon and Low-Alloy Steels). Most pipelines worldwide are constructed with mild steel; however the steel easily deteriorates in corrosive environments. This is noticeable in the scale formation in steam boilers made of mild steels (Preboiler and boiler corrosion; Boiler/Feeder guidelines; Ludwig), in acid pickling and petrochemical processes. It is important to apply chemical

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compounds which significantly reduce the corrosion rate of the metallic alloys. This invariably helps extend their life span and enable it perform its desired function. Research has shown that organic chemical compounds with active electronegative groups and  $\pi$  electrons are very effective in minimizing the corrosion rate of mild steels (Ju et al., 2008; Yurt et al., 2004; Ahamad et al., 2010). The corrosion inhibition behaviour of the chemical compounds is the product of electrochemical reactions with the metal surface through adsorption. Significant scientific studies have been conducted on corrosion inhibiting compounds for mild steel in aggressive environments (Abbasova et al., 2013; Athareh and Fatemeh, 2011), however enhancing the inhibition efficiency of the compounds at the lowest volumetric concentration is possible. Previous research on 4-hydroxy-3-methoxy-benzaldehyde and hexadecyltrimethylammonium bromide separately without combining them gave above average results. Niamien et al. (2011) studied the corrosion inhibition properties of hexadecyltrimethylammonium bromide on aluminium alloy in dilute hydrochloric acid at 25–55 °C. Result showed that the compound performed effectively, adsorbing onto the aluminium alloy according to the Langmuir isotherm model. The inhibition efficiency as observed to decrease with increasing temperature. The synergistic effect of hexadecyl trimethyl ammonium bromide and sodium bromide on the corrosion inhibition of cold rolled steel in dilute H<sub>2</sub>SO<sub>4</sub> was investigated through weight loss measurement and polarization technique. Results showed that the combined admixture effectively inhibited the corrosion of the steel with inhibition efficiency increasing with increase in concentration of the compound. Polarization study shows that the inhibition type is mixed (Tang et al., 2006). Lia et al. (2010) studied the adsorption and corrosion inhibition effect of 4hydroxy-3-methoxy-benzaldehyde on cold rolled steel dilute H<sub>3</sub>PO<sub>4</sub> medium through electrochemical methods. Results show that inhibition efficiency increases with the inhibitor concentration, but decreases with temperature. Adsorption was determined to obey the Temkin adsorption isotherm. The corrosion inhibition of three cationic surfactants of 4-hydroxy-3methoxy-benzaldehyde on mild steel in HCl media was evaluated by Shaban et al. through weight loss, polarization resistance and impedance spectroscopy. The inhibitors displayed high inhibition efficiency with the surface coverage obeying the vilamil isotherm (Shabana et al., 2015). The aim of this research is to assess the synergistic effect of 4-hydroxy-3methoxybenzaldehyde and hexadecyltrimethylammoniumbro mide as suitable inhibitors for mild steel in dilute H<sub>2</sub>SO<sub>4</sub> and HCl media and to investigate the mechanism of inhibitor metal interaction.

## 2. Materials and methods

# 2.1. Material

Mild steel commercially obtained and analysed at the Materials Characterization Laboratory, Department of Mechanical Engineering, Covenant University gave an average nominal per cent (%) composition shown in Table 1. 14 cylindrical mild steel samples were cut with an average length of 10 mm. Their exposed surface ends were metallographically prepared in accordance with ASTM G1-03 (2011).

## 2.2. Inhibiting Compound and Acid Test Media

4-Hydroxy-3-methoxybenzaldehyde and hexadecyltrimethy lammoniumbromide (CBH) were obtained in synthesized form from Sigma Aldrich, USA and SMM Instrument, South Africa in synthesized form. Their structural formulas are shown in Fig. 1, and the properties in Table 2.

CBH was prepared in molar concentrations of  $4.8 \times 10^6$ ,  $9.7 \times 10^6$ ,  $1.45 \times 10^5$ ,  $1.94 \times 10^5$ ,  $2.42 \times 10^5$  and  $2.9 \times 10^5$  respectively per 200 mL of the acid solution. 1 M H<sub>2</sub>SO<sub>4</sub> and HCl acid solution were prepared with grade of H<sub>2</sub>SO<sub>4</sub> acid (98%) and HCl acid (37%) with deionized water.

# 2.3. Weight-loss measurement and potentiodynamic polarization test

Weighed steel samples individually immersed into 200 mL acid solution for 432 h (Mathur and Vasudevam, 1982) were weighed every 24 h interval according to ASTM G31-72 (2004) (ASTM NACE/ASTM G31-12a). The corrosion rate  $(\gamma)$  calculation is defined in Eq. (1) (Venkatesan et al, 2009).

$$\gamma = \left[ \frac{87.6\tilde{\omega}}{DAT} \right] \tag{1}$$

where  $\tilde{\omega}$  is the weight loss in mg, *D* is the density in g/cm<sup>3</sup>, *A* is the total area in cm<sup>2</sup> and 87.6 is a constant. Inhibition efficiency ( $\eta$ ) was calculated from Eq. (2) (Gülşen, 2008).

$$\eta = \left[\frac{\tilde{\omega}_1 - \tilde{\omega}_2}{\tilde{\omega}_1}\right] \times 100 \tag{2}$$

where  $\tilde{\omega}_1$  and  $\tilde{\omega}_2$  are the weight loss with and without specific CBH concentrations. Surface coverage was determined from the mathematical relationship (Eq. (3)) (Moussa et al., 2007).

$$\theta = \left[1 - \frac{\tilde{\omega}_2}{\tilde{\omega}_1}\right] \tag{3}$$

where  $\theta$  is the amount of CBH compound, adsorbed per gram of the mild steel. Electrochemical test was performed with cylindrical mild steel electrodes embedded in acrylic resin with an exposed surface area of 154 mm² according to ASTM G59-97(2014). Tests were performed with Digi-Ivy 2300 potentiostat with platinum counter electrode and Ag/AgCl reference electrode from -1.5 V to 1.5 V at a scan rate of 0.0016 V/s according to ASTM G102-89(2015). The corrosion rate ( $\gamma$ ) was calculated from Eq. (4).

$$\gamma = \frac{0.00327 \times J_{\text{corr}} \times E_{\text{q}}}{D} \tag{4}$$

where  $j_{\text{corr}}$  is the corrosion current density, D is the density in g/cm<sup>3</sup>;  $E_q$  is the specimen equivalent weight in grams. 0.00327 is a constant for corrosion rate calculation in mm/y (Venkatesan et al., 2009).

## 3. Results and discussion

# 3.1. Weight-loss Measurement

Results from weight loss test for mild steel in both acid solutions are shown in Tables 3. Fig. 2 shows the plot of corrosion rate versus exposure time in the acid media. The control sample (sample A) corroded significantly in the acid media in com-

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Table 1 Percentage	compositio	n of mild stee	el.						
Element Symbol	С	Si	Mn	P	S	Cu	Ni	Al	Fe
% Composition	0.401	0.169	0.440	0.005	0.012	0.080	0.008	0.025	98.86

Figure 1 (a) 4-Hydroxy-3-methoxybenzaldehyde, (b) hexadecyltrimethylammoniumbromide.

Table 2	Properties of the CBH inhibiting compound.		
S/N	Compound	Molecular Formula	Molar Mass (gmol <sup>-1</sup> )
1	4-Hydroxy-3-methoxybenzaldehyde	$C_8H_8O_3$	152.15
2	Hexadecyltrimethylammoniumbromide	$C_{19}H_{42}BrN$	364.45

0.5 M H <sub>2</sub>	SO <sub>4</sub>				0.5 M H	Cl			
Samples	CBH Conc. $(M \times 10^3)$	Weight Loss (g)	Corrosion Rate (mm/y)	CBH Inh. Efficiency (%)	Samples	CBH Conc. $(M \times 10^3)$	Weight Loss (g)	Corrosion Rate (mm/y)	CBH Inh. Efficiency (%)
A	0	8.9779	1.40	0	A	0	2.829	0.012	0
В	4.84E - 06	0.4758	0.07	94.7	В	4.84E - 06	1.992	0.009	29.6
C	9.68E-06	0.3927	0.06	95.6	C	9.68E-06	1.078	0.005	61.9
D	1.45E-05	0.3927	0.06	95.6	D	1.45E-05	0.706	0.003	75.0
E	1.94E-05	0.3000	0.05	96.7	E	1.94E-05	0.661	0.003	76.6
F	2.42E-05	0.3817	0.06	95.7	F	2.42E-05	0.465	0.002	83.6
G	2.90E-05	0.2456	0.04	97.3	G	2.90E-05	0.415	0.002	85.3

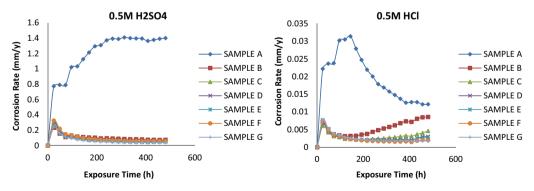


Figure 2 Corrosion rate vs exposure time for  $0.5 \text{ M H}_2\text{SO}_4$  and HCl solution at  $0-2.9 \times 10^5 \text{ M CBH}$ .

parison to samples with varying degrees of CBH concentration, however the corrosion rate in  $H_2SO_4$  acid is much higher than in HCl even though the solubility product constant of  $FeCl_2$  (pKs = 39.4) is greater than that of  $FeSO_4$ 

(pKs = 22.8) at 25 °C due to two reasons; (a) the relative concentration of the  $H_2SO_4$  is much higher than HCl, (b) at higher concentration  $H_2SO_4$  completely ionizes in the solution releasing two protons that strongly reacts with the steel surface com-

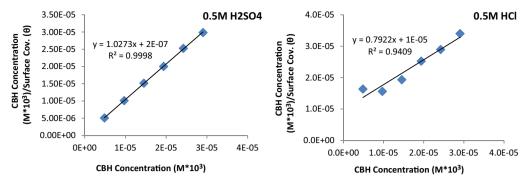
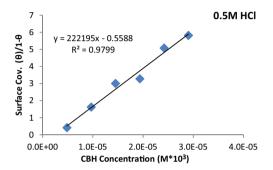


Figure 3 Langmuir Isotherm plot for CBH in 1 M H<sub>2</sub>SO<sub>4</sub> and HCl acid solution.



**Figure 4** Frumkin Isotherm plot for CBH in 1 M HCl acid solution.

pared to HCl which releases one proton at a weaker concentration. The steel being a ferrous alloy spontaneously reacts with acid resulting in hydrogen evolution and oxidation reactions causing the release of  $Fe^{2+}$  ions into the solution through the action of sulphate and chloride anions. The presence of CBH compound in the solution (sample B to G) suppressed the reduction–oxidation reaction responsible for corrosion degradation. CBH protected the steel from corrosion through adsorption of its cations onto the mild steel surface. This explanation is confirmed from the significant difference in corrosion rate between the CBH inhibited and uninhibited samples (Tables 3). CBH inhibitor has higher inhibition efficiency in  $H_2SO_4$  acid despite the high dissociation constant of the acid. This can be explained on the basis that in  $H_2SO_4$ 

Table 4	Data for Gib	bs free ene	rgy, surface cover	age and equili	brium cons	tant of adsorp	tion for 0-	7.5% CBH in 1 M	$H_2SO_4$ .
0.5 M H2	SO4				0.5 M HC	71			
Samples	CBH Conc. $(M \times 10^3)$	Surface Cov. (θ)	Equi. Constant of Ads. (K)	Gibbs Free Energy, ΔG (kJ/Kmol)	Samples	CBH Conc. $(M \times 10^3)$	Surface Cov. (θ)	Equi. Constant of Ads (K)	Gibbs Free Energy, ΔG (kJ/Kmol)
A	0	0	0	0	A	0	0	0	0
В	5E-06	0.947	4E + 06	-47.42	В	4.8E - 06	0.296	86744.821	-38.13
C	1E-05	0.9563	2E + 06	-46.21	C	9.7E - 06	0.619	167875.82	-39.77
D	1E-05	0.9563	2E + 06	-45.20	D	1.5E-05	0.750	207039.95	-40.29
E	2E-05	0.9668	2E + 06	-45.20	E	1.9E-05	0.766	169366.03	-39.79
F	2E-05	0.9575	9E + 05	-44.00	F	2.4E - 05	0.836	209953.7	-40.32
G	3E-05	0.9726	1E+06	-44.69	G	2.9E-05	0.853	200404.6	-40.20

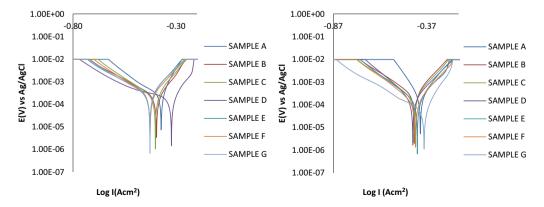


Figure 5 Polarization plot for mild steel in 0.5 M  $H_2SO_4$  and HCl acid at  $0-2.9 \times 10^5$  M CBH.

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CBH protonates strongly allowing more molecules to release electrons, the phenomenon increases the reactivity of the compound enabling strong interaction with the steel surface. Due to the large molecular weight of the Hexadecyltrimethylammo niumbromide component of CBH [C<sub>16</sub>H<sub>33</sub>N<sup>+</sup>(CH<sub>3</sub>)<sup>3</sup>Br<sup>-</sup>], the compound easily attached itself to the mild steel through intermolecular attraction. It consists of hydrophilic [<sup>+</sup>N(CH3)<sub>3</sub>] which enhances its solubility in the solution and facilitates adsorption to the steel while the main hydrophobic part (C<sub>6</sub>H<sub>33</sub>) enables substitutional adsorption mechanism. These attributes allow for chemical interaction with the steel at steel/solution interface through covalent bonding resulting from electrostatic attraction between the nitrogen cations and negatively steel surface (El-Maghraby and Soror, 2010; Luo et al., 1998).

The physicochemical property of 4-hydroxy-3methoxybenzaldehyde is related to its functional groups (aldehyde, hydroxyl, and ether) and their chemical reactivity. Ethers are slightly polar, the presence of two lone pairs of electrons on the oxygen atoms enables hydrogen attachment with water molecules possible (James). The hydroxyl functional group has two active covalent bonds, the C-O bond and the O-H bond (Alcohol Nomenclature). The aldehyde functional group has the carbon atom which shares bonds with oxygen, hydrogen atom and other group of atoms. The reactivity of these compounds arises largely through the polarity of the group and the acidity of any α-hydrogen that are present in their structure which reacts with the nucleophide atoms of the corrosive species (Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup>) to form a new covalent bond (Aldehyde Chemical compound).

# 3.2. Adsorption isotherm

Adsorption characteristics of CBH were studied to understand the mechanism of interaction between the compound and mild steel (Limousin et al., 2007). Langmuir isotherm had the best fit for CBH in H<sub>2</sub>SO<sub>4</sub> and HCl acid. Frumkin isotherm had a good fit for CBH in HCl solution only. The Langmuir isotherm for HCl (Fig. 3) did not show a slope around unity. According to Langmuir CBH cations occupy specific adsorption sites at the metal/solution interface. Interaction between the adsorbed CBH species and changes in adsorption heat with increasing surface coverage would affect the slope resulting in the slight deviation of the slope from unity (Abiola, 2006). Frumkin isotherm (Fig. 4) assumes unit coverage at high inhibitor concentrations over heterogeneous electrode surface.

## 3.3. Thermodynamics of the corrosion process

Gibbs free energy ( $\Delta G^o_{\rm ads}$ ) values for the adsorption process are shown in Table 4. The heterogeneous nature of the steel surface is responsible for the changes in  $\Delta G^o_{\rm ads}$  with respect to surface coverage (Damaskin and Frumkin, 1971). The average value of  $\Delta G^o_{\rm ads}$  for CBH adsorption in H<sub>2</sub>SO<sub>4</sub> and HCl acid solution is above 40 KJmol<sup>-1</sup> which depicts chemisorption adsorption mechanism (Benali et al., 2013).

## 3.4. Potentiodynamic polarization studies

The potentiodynamic polarization plots of the electrochemical behaviour of CBH compound on the corrosion inhibition of

Table 5	Potentiody	ynamic pola	arization res	Table 5 Potentiodynamic polarization results for mild steel	el in 0.5 M i	H <sub>2</sub> SO <sub>4</sub> and HC	in 0.5 M $\rm H_2SO_4$ and HCl solution at 0–2.90 $\times10^5$ M CBH.	$2.90 \times 10^5  \mathrm{l}$	M CBH.				
		$0.5 \text{ M H}_2\text{SO}_4$	$SO_4$					0.5 M HCI	1				
Sample CBH Conc (M ×	CBH Conc. $(M \times 10^3)$	Corr. Rate (mm/y)	CBH Inh. Eff. (%)	Corr. Current Density (A/ cm <sup>2</sup> )	Corr. Potential (V)	Cathodic Tafel Slope (V/Dec)	Anodic Tafel Slope (V/ Dec)	Corr. Rate (mm/y)	CBH Inh. Eff. (%)	Corr. Current Density (A/ cm <sup>2</sup> )	Corr. Potential (V)	Cathodic Tafel Slope (V/Dec)	Anodic Tafel Slope (V/ Dec)
A	0	1.003	0	8.64E-05	-0.373	-4.86	14.63	0.84	0.00	7.24E-05	-0.404	-12.110	7.601
В	4.84E - 06	0.061	94.0	5.22E-06	-0.395	-4.91	10.89	0.83	0.74	7.19E-05	-0.435	-6.251	9.3
C	9.68E - 06	0.064	93.6	5.54E-06	-0.401	-5.97	10.18	0.46	44.73	4.00E-05	-0.438	-6.537	6.8
О	1.45E - 05	0.048	95.3	4.10E - 06	-0.323	-2.57	10.51	0.20	76.57	1.70E-05	-0.445	-8.126	9.8
田	1.94E - 05	0.078	92.2	6.76E-06	-0.427	-4.74	9.47	0.16	81.10	1.37E-05	-0.421	-6.238	-6.2
Ţ	2.42E - 05	0.065	93.5	5.63E-06	-0.427	-5.85	9.48	0.13	84.78	1.10E-05	-0.430	-7.109	9.4
ŭ	2.90E-05	0.070	93.0	6.03E-06	-0.427	-5.39	9.21	0.10	87.60	8.98E-06	-0.384	-4.083	12.6

the mild steel sample in 0.5 M H<sub>2</sub>SO<sub>4</sub> and HCl solution are presented in Fig. 5. Table 5 shows the data obtained from the polarization scans. Significant difference in corrosion rate values between the CBH inhibited and uninhibited steel specimens (0 M &  $4.8 \times 10^6 - 2.9 \times 10^5$  M CBH) is observed, however the corrosion rate in H<sub>2</sub>SO<sub>4</sub> is much higher than in HCl. At 0 M CBH the steel oxidizes in the acid solution causing the formation of porous oxides which accelerates the rate of corrosion. The corrosive species (Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup>) within the solution aggravates the corrosion mainly due to depassivation effect in iron dissolution as a result of the presence of numerous anodic and cathodic reaction cells. The corrosion rate reduced dramatically after 0 M CBH (sample A) in H<sub>2</sub>SO<sub>4</sub> acid solution and remained generally the same till  $9.68 \times 10^6$  M CBH at sample G with inhibition efficiency above 90% throughout, however there was a gradual decrease in corrosion rate in the HCl solution before sample D  $(1.45 \times 10^5 \,\mathrm{M}\,\mathrm{CBH})$ , after which increase in corrosion rate was appreciable till sample G (9.68  $\times$  10<sup>6</sup> M CBH) with a maximum inhibition efficiency above 85%. At low concentrations CBH molecules is unable to effectively withstand the electrochemical action of Cl<sup>-</sup> ions in HCl in contrast to SO<sub>4</sub><sup>2-</sup> ions in H<sub>2</sub>SO<sub>4</sub>. The electrochemical reactions of CBH within both acids cause the formation of an adherent film that generally inhibits mild steel corrosion. The corrosion inhibiting molecules of CBH tend to interact with the electrochemical cells, through retardation of the redox electrochemical process and/or inhibition of the diffusion of active corrosive anions from the acid solution to the steel and diffusion of Fe<sup>2+</sup> ions into the acid solution. Increase in CBH concentration did not affect the corrosion rate values in H<sub>2</sub>SO<sub>4</sub>. Its inhibition efficiency is independent of CBH concentration and acts instantaneously from the lowest CBH concentration.

The polarization plots (Fig. 5) show that CBH compound significantly influenced the corrosion polarization behaviour of the mild steel specimen under the inhibiting action of CBH compound and that the inhibition mechanism is through surface coverage of the active regions preventing the diffusion of iron cations into the acid solution and suppression of hydrogen evolution and oxygen reduction reactions through selective precipitation on the cathodic sites. This increases the surface impedance and limits the diffusion of reducible species (Hosseini and Azimi, 2009). The maximum change in corrosion potential in H<sub>2</sub>SO<sub>4</sub> is 54 mV while in HCl it is 41 mV, thus CBH is a mixed type inhibitor in both acids inhibiting the total redox process (Susai et al., 2002; Sahin et al., 2002).

# 4. Conclusion

The combined admixture of 4-hydroxy-3-methoxybenzaldehyde and hexadecyltrimethylammoniumbromide showed excellent corrosion inhibition at all concentrations  $\rm H_2SO_4$  while in HCl there was a gradual but appreciable increase in corrosion rate with increase in inhibitor concentration due to the effective inhibiting action of the compound. The compound showed mixed inhibition properties suppressing the redox electrochemical process. Molecular adsorption of the compound onto the steel surface was observed to obey the Langmuir and Frumkin adsorption isotherm models through chemisorption adsorption mechanism from thermodynamic calculations.

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