RSC Advances

1   **Hydrogen evolution inhibition by L-Serine at the negative electrode**

2   **of a lead-acid battery**

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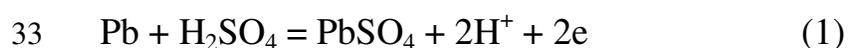
7   **Abstract**

8   The inhibition effect of L-Serine on the hydrogen evolution at the

9   negative electrode of a lead-acid battery (Pb) in 5.0 M H_2SO_4 has been

10   studied by hydrogen evolution and electrochemical methods. The surface

11   of Pb is analyzed with scanning electron microscope (SEM) and energy-

12   dispersive X-ray (EDX). The results demonstrate that L-Serine is an

13   adequate inhibitor, retarding the hydrogen evolution reaction.

14   Polarization curves denote that L-Serine performs as the cathodic

15   inhibitor. The inhibition efficiency boosts with increase in L-Serine

16   concentration but decreases with upturn in temperature. The most

17   efficient inhibitor concentration is 10 mM. Adsorption of L-Serine on the

18   Pb surface is unprompted and complies with Langmuir's isotherm. L-

19   Serine induces an energy barrier for the hydrogen evolution reaction and

20   this barrier increases with increasing L-Serine concentration.

21   **Keywords:** hydrogen evolution; lead-acid battery; inhibition; amino acid
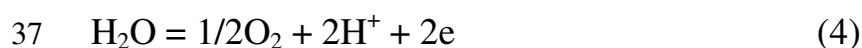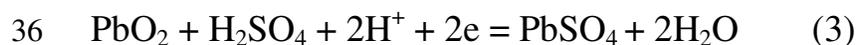
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1. Introduction

The lead–acid battery is the oldest type of rechargeable battery [1-2]. It consists of PbO_2 as a positive electrode, Pb as a negative electrode and an electrolyte of aqueous H_2SO_4. The concentration of H_2SO_4 in a fully charged auto battery measures a specific gravity of $1.265 - 1.285$ [2]. This is equivalent to a molar concentration of $4.5 - 6.0$ M. As the battery discharges, PbO_2 and Pb react with aqueous solution of H_2SO_4 to form $PbSO_4$ and water. On recharge, $PbSO_4$ converts back to PbO_2 and Pb. In the same time, SO_4^{2-} ions are driven back into the electrolyte solution to form H_2SO_4. The reactions can be presented as the following:

At the negative electrode [3]:

$$Pb + H_2SO_4 = PbSO_4 + 2H^+ + 2e \qquad (1)$$

$$2H^+ + 2e = H_2 \qquad (2)$$

At the positive electrode:

$$PbO_2 + H_2SO_4 + 2H^+ + 2e = PbSO_4 + 2H_2O \qquad (3)$$

$$H_2O = 1/2O_2 + 2H^+ + 2e \qquad (4)$$

Under normal conditions, gassing vented from a lead-acid battery is mainly oxygen and hydrogen gas [4-5].

Hydrogen gas can collect at the top of a battery. If this gas is exposed to a flame or spark, it can explode. Hydrogen gas evolution harm the valve regulated lead acid battery during charging and discharging

2

43 processes [6]. In addition to the primary focus on human and system

44 safety, hydrogen evolution also affects on the battery life and

45 maintenance economics. Therefore the inhibition of hydrogen evolution

46 during the charging of a lead-acid battery is an important part of the

47 engineering for any battery system [7-10].

48   The aim of this work is to explore the use of L-Serine to inhibit the

49 hydrogen evolution during the reaction of Pb with 5.0 M H_2SO_4.

50   L-serine is a non essential amino acid. It is manufactured by

51 fermentation from carbohydrate sources [11]. In contrast to most

52 commercial acid corrosion inhibitors which are highly toxic, L-serine is

53 an environmentally friendly corrosion inhibitor. However, there is no

54 literature to date about the corrosion inhibitory effect of L-serine in acid

55 solution.

56 **2. Experimental**

57 **2.1. Materials**

58   The working electrodes were made from pure lead (Pb) 99.99%.

59 Specimens used for hydrogen evolution measurements were mechanically

60 cut into $2.0 \times 1.0 \times 0.2$ cm dimensions. For electrochemical

61 measurements, the cylindrical rod of Pb with exposed surface area of

62 0.452 cm^2 was allowed to contact the test solutions. The working

63 electrodes were abraded with different grads of emery papers up to 1200

3

64   grad. Then they were cleaned by using acetone and distilled water before

65   the tests.

66   Experiments were done in 5.0 M H_2SO_4 in the absence and presence

67   of L-Serine. L-Serine was obtained commercially from Sigma-Aldrich

68   Co.

69   All solutions were prepared from analytical grade chemicals and

70   distilled water. The temperature of the solutions was controlled by a

71   thermostat.

72   **2.2. Hydrogen evolution rate measurements**

73   The schematic diagram of the hydrogen evolution measurements was

74   reported in previous paper [12]. The volume of H_2 gas evolved (ΔV)

75   during the reaction of Pb specimens in 100 ml of 5.0 M H_2SO_4 was

76   measured at specified time intervals (t). The hydrogen evolution rate (H_R)

77   was expressed using the following correlation [13]:

78   $$H_R = (\Delta V) / (t) \qquad (5)$$

79   **2.3. Electrochemical measurements**

80   Electrochemical experiments (Potentiodynamic polarization) were

81   performed in a regular three-electrode cell. A platinum coil was used as

82   the counter electrode and potentials were measured relative to Hg/Hg_2SO_4

83   reference electrode. Electrochemical experiments were conducted using

84   Potentiostat/Galvanostat (Gill AC model no. 947, ACM instruments).

4

85   Before electrochemical measurements, the Pb electrode was immersed in

86   freshly prepared solutions at open circuit potential (OCP) for 2 h, to attain

87   a steady case. The potential of polarization curves was done from - 200

88   mV to + 200 mV versus OCP with 1.0 mV s^{-1} sweep rate.

89   **2.4. SEM-EDX analysis**

90   The surface morphology of the metal specimen was evaluated by SEM-

91   EDX analysis using JOEL-JEM-1200 EX II ELETRON MICROSCOPE

92   and a Traktor TN-2000 energy dispersive spectrometer.

93   **3. Results and discussion**

94   **3.1. Effect of L-Serine concentration on the hydrogen evolution**

95   The effect of the addition of various concentrations of L-Serine (0.1-20

96   mM) on the H_R during the reaction of Pb in 5.0 M H_2SO_4 at 303 K is

97   shown in Fig. 1. It should be noted that the presence of L-Serine in 5.0 M

98   H_2SO_4 has a considerable influence on the inhibition of the hydrogen

99   evolution rates H_R. Furthermore, increasing the concentration of L-Serine

100   from 0.1 to 20 mM causes a noticeable decrease in hydrogen evolution

101   rates H_R.

102   The efficiency of L-Serine (I_H %) to inhibit the hydrogen evolution is

103   calculated according to [13] :

104   $$I_H\% = \left(1 - \frac{H_R}{H_{Ro}}\right) \times 100 \qquad (6)$$

5

105   where H_{R0} and H_R are the hydrogen evolution rates without and with L-

106   Serine, respectively.

107   Fig. 2 shows variations of the efficiency of L-Serine I_H % as a function

108   of the logarithmic of L-Serine concentration. The plot of Fig. 2 exhibits

109   S-shaped adsorption isotherm. This suggests, but does not prove, that L-

110   Serine inhibits the hydrogen evolution during the reaction of Pb in 5.0 M

111   H_2SO_4 by adsorption at the Pb/ H_2SO_4 solution interface [14]. It is evident

112   from Fig. 2 that upon increasing L-Serine concentration to 10 mM, I_H %

113   values significantly increases reaching a maximum value (I_H % = 88).

114   However, a further increase in L-Serine concentration to 20 mM does not

115   lead to a change in I_H % values. This may be due to the adsorption of L-

116   Serine on the surface of Pb have reached the state of equilibrium at 10

117   mM and consequently, any further addition will not yield any increase in

118   the inhibition efficiency [15]

119   **3.2. Effect of temperature and activation energy calculation**

120   The rate of the chemical reactions inside the lead-acid battery depends

121   on the temperature. In general, the increase of the temperature causes the

122   acceleration in the reaction rate of the internal processes, e.g. corrosion

123   (aging), self discharge and the corresponding hydrogen and oxygen gas

124   evolution rates [16-17].

6

125   To assess the influence of temperature on the hydrogen evolution rate

126   and the hydrogen evolution inhibition process, the hydrogen evolution

127   rates H_R are performed at different temperatures (303 and 323 K) without

128   and with different concentrations of L-Serine. The results are recorded in

129   Table 1. The results obtained indicate that the rates of the hydrogen

130   evolution rate in all cases increased with temperature while I_H %

131   decreased. The increase in the H_R in the absence of L-Serine with the rise

132   of temperature may be arises from an increase in $PbSO_4$ solubility with

133   temperature [18]. In the same time, the raise in temperature accelerates

134   both of the diffusion and migration rates for the reactant and product

135   species. This drives to an increase in the rate of hydrogen evolution

136   reaction. On the other hand, the increase in the H_R and the decrease in the

137   I_H % in the presence of L-Serine with the rise of temperature may be

138   assign to the partial desorption of L-Serine molecules from Pb surface

139   with temperature [19].

140   The activation energy (E_a) for the process responsible for hydrogen

141   generation (diffusion of both protons and uncharged hydrogen in

142   solution) was computed using the Arrhenius relation [13]:

143
$$Ea = 2.303R\frac{\log\left(\dfrac{H_{R2}}{H_{R1}}\right)}{\left(\dfrac{1}{T_1} - \dfrac{1}{T_2}\right)} \qquad (7)$$

7

144   where R is the ideal gas constant, H_{R1} and H_{R2} are the hydrogen evolution

145   rates at the absolute temperatures T_1 and T_2, respectively.

146   The calculated values of E_a are recorded in Table 1.

147   The data examination shows that the activation energy is higher in the

148   presence of L-Serine than in its absence. The higher E_a value in the

149   presence of L-Serine, implies that physical adsorption exists between L-

150   Serine molecules and the charged surface. Moreover, the values of E_a

151   increase with increasing L-Serine concentration. This means that, the

152   presence of L-Serine induces an energy barrier for the hydrogen evolution

153   reaction and this barrier increases with increasing concentration of L-

154   Serine [20].

155   **3.3. Electrochemical measurements**

156   Fig. 3 shows anodic and cathodic polarization plots (Tafel plots)

157   recorded on Pb electrode in 5.0 M H_2SO_4 containing different

158   concentrations of L-Serine at 303 K.

159   It can be noticed that the addition of L-Serine causes a remarkable

160   decrease in the corrosion rate.

161   It is clear that, the presence of L-Serine shifts cathodic curves to lower

162   values of current densities. Whereas the anodic curves are slightly

163   retarded by L-Serine. This result suggests that the addition of L-Serine

164   retards the hydrogen evolution reaction [21].

165   Table 2 displays the kinetic parameters for corrosion process, i.e.,

166   corrosion potential (E_{corr}) and corrosion current density (j_{corr}) [22].

167   Comparing with blank solution, E_{corr} shifts to negative side more than

168   85 mV in the presence of L-Serine; this elucidates that L-Serine works as

169   cathodic-type inhibitor [23]. Evidently, j_{corr} decreases frequently in the

170   presence of L-Serine and decreases with L-Serine concentration. The

171   corrosion current density j_{corr} values can be used to calculate the inhibition

172   performances of L-Serine ($\eta_j \%$) from the following equation [24] :

173    $$\eta_j \% = \frac{j_{corr(0)} - j_{corr}}{j_{corr(0)}} \times 100 \quad (8)$$

174   where $j_{corr(0)}$ and j_{corr} are uninhibited and inhibited current densities,

175   respectively.

176   From results given in Table 2, an increase of $\eta_j \%$ with L-Serine

177   concentration, reaching a maximum value (91%) at 10 mM, was

178   observed. A slight a change in $\eta_j \%$ values was found above the 10 mM.

179   This phenomenon is consistent with results obtained form hydrogen

180   evolution measurements.

181   **3.4. Study of the adsorption phenomenon**

182   The adsorption isotherms can supply helpful information about the

183   mechanism of corrosion inhibition [25-26].

9

184   The degree of surface coverage (θ) of L-Serine from hydrogen

185   evolution and electrochemical measurements can be calculated using the

186   following equations [27]:

187    $$\theta = \left(1 - \frac{H_R}{H_{Ro}}\right) \qquad (9)$$

188    $$\theta = \frac{j_{corr(0)} - j_{corr}}{j_{corr(0)}} \qquad (10)$$

189   The surface coverage values θ were fitted to different adsorption

190   isotherm models and best results judged by the correlation coefficient

191   (R^2) were obtained with Langmuir adsorption isotherm as follows [28]:

192    $$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \qquad (11)$$

193   where C_{inh} is L-Serine concentration and K_{ads} is Langmuir isotherm

194   constant.

195   The plot of C_{inh} / θ versus C_{inh} (Fig. 4) yields a straight lines with a

196   correlation coefficients (R^2) are close to one (Table 3). This supports the

197   assumption that the adsorption of L-Serine from 5.0 M H_2SO_4 solution on

198   the Pb surface at the studied temperatures comply with the Langmuir

199   adsorption isotherm [29]. The high values of K_{ads} (Table 3) reflect the

200   high adsorption efficiency of L-Serine on Pb surface [30]. K_{ads} can be

201   used to determine the free Gibbs energy of adsorption (ΔG^0_{ads}) using the

202   following equation [31]:

10

203                            $\Delta G^0_{ads} = -RT \ln (55.5\ K_{ads})$ (12)

204   The calculated ΔG^0_{ads} values obtained from hydrogen evolution and

205   electrochemical data were given in Table 3. The data clearly show that

206   ΔG^0_{ads} values were negative. In essence, this means that the adsorption of

207   L-Serine molecules on Pb surface will be favored and will release energy.

208   Furthermore, the values ΔG^0_{ads} of less than -40 kJ mol^{-1} indicate physical

209   adsorption [32].

210   The heat of adsorption (Q_{ads}) of L-Serine on Pb surface has been

211   determined as a function of the surface coverage (θ) and temperature as

212   follows [33]:

213            $$Q_{ads} = 2.303R\left(\log\frac{\theta_2}{1-\theta_2} - \log\frac{\theta_1}{1-\theta_1} \right) \times \left(\frac{T_1 T_2}{T_2 - T_1} \right)$$ (13)

214   where θ_1 and θ_2 are the degrees of surface coverage (θ_1 and θ_2 values were

215   calculated using equation 9) at T_1 and T_2, respectively.

216   The calculated values for Q_{ads} are presented in Table 4. The negative

217   value of Q_{ads} assumes that the adsorption process is exothermic and

218   physisorption [33].

219   **3.5. SEM-EDX examinations**

220   The aim of SEM-EDX examinations was to confirm the results

221   obtained from the hydrogen evolution and electrochemical measurements

222   that a protective surface film of L-Serine molecules is formed on the Pb

11

223   surface. To attain this aim, SEM-EDX examinations of the Pb surface

224   were performed in 5.0 M H_2SO_4 in the absence and presence of 20 mM of

225   L-Serine.

226   As it is shown in Fig. 5a, the Pb surface was strongly damaged in the

227   absence of L-Serine due to Pb corrosion in 5.0 M H_2SO_4. However, less

228   corrosion attack was found for the sample exposed to H_2SO_4 solution

229   containing L-Serine (Fig.5b). A smoother surface is seen in the presence

230   of 20 mM L-Serine in comparison to that observed in Fig.5a.

231   Fig. 6 presents EDX survey spectra recorded for Pb surface exposed

232   for 5.0 M H_2SO_4 in the absence and presence of 20 mM L-Serine. In

233   H_2SO_4 solution free solution, the EDX spectra (Fig. 6a) show the

234   characteristics peaks of Pb element. In addition, $PbSO_4$ is present, as

235   indicated by the Pb, S and O signals. In solution contain 20 mM L-Serine

236   (Fig. 6b), the EDX spectra showed an additional line characteristic for the

237   existence of N and C. In addition, the intensity of S signal decreased. The

238   appearance of the N and C signals is due to the N and C atoms of the

239   adsorbed L-Serine molecules.

240   These data confirm the adsorption of L-Serine molecules on the Pb

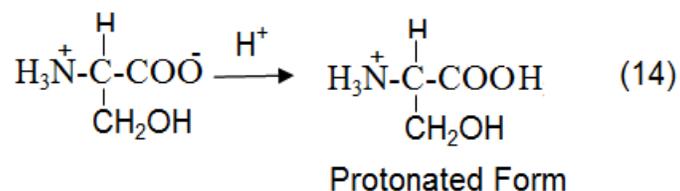241   surface and formulation a protective surface film.

242   **3.6. Explanation for inhibition**

12

243   The effectiveness of organic inhibitor may be due to the adsorption of

244   inhibitor molecules on the metal surface [34-35]. Adsorption process

245   occurs through the electrostatic attractive forces between dipoles and/or

246   ionic charges on the adsorbed molecules and the electric charge on the

247   metal surface. The presence of heteroatoms with loosely bound electrons

248   or π-electron systems or aromatic rings in molecular structure of the

249   inhibitor enhance the adsorption efficiency process [36-37].

250   In acidic solutions the anodic reaction produces metal ions, and the

251   principal cathodic reaction produces hydrogen gas. An inhibitor may

252   decrease the metal dissolution, hydrogen gas evolution, or both processes.

253   The shift of the corrosion potential to the positive direction indicates

254   mainly inhibition of the metal dissolution process (anodic inhibitor),

255   whereas the shift to the negative direction indicates mainly inhibition of

256   the hydrogen gas evolution (cathodic inhibitor) [38].

257   In the present work, the essential step in the inhibition of hydrogen

258   evolution during the reaction of Pb in 5.0 M H_2SO_4 is the adsorption of L-

259   Serine on cathodic sites on the Pb surface. L-Serine has three polar

260   groups, namely, NH_2, COOH, and OH groups. It can coordinate with Pb

261   surface through the nitrogen atom and oxygen atom of the polar groups

262   [39]. In neutral solutions, L-Serine molecules are presented usually as

13

263   Zwitter ions. Whereas in acidic solutions, L-Serine molecules are

264   presented in protonated form as the following:

$$H_3\overset{+}{N}\text{-}\underset{CH_2OH}{\overset{\overset{H}{|}}{C}}\text{-}CO\bar{O} \xrightarrow{\text{ }H^+\text{ }} H_3\overset{+}{N}\text{-}\underset{CH_2OH}{\overset{\overset{H}{|}}{C}}\text{-}COOH \qquad (14)$$

Protonated Form

265

266   This means that in acidic medium, L-Serine molecules are adsorbed

267   through the $^+NH_3$ on the electrode surface (cathodic sites) and decrease

268   the rate of the cathodic reaction, thus the rate of hydrogen evolution will

269   be decreased.

270   **4. Conclusion**

271   The inhibition performance of L-Serine on the hydrogen evolution at the

272   negative electrode of a lead-acid battery (Pb) in 5.0 M H_2SO_4 solution

273   was evaluated using hydrogen evolution and electrochemical methods.

274   The results show that L-Serine works as an adequate inhibitor for the

275   hydrogen evolution at Pb electrode in 5.0 M H_2SO_4. The inhibition

276   efficiency of L-Serine depends on its concentration and solution

277   temperature. The results of electrochemical measurements demonstrate

278   that L-Serine behaves as a cathodic-type inhibitor. The adsorption of L-

279   Serine on Pb surface complies with Langmuir's isotherm. SEM and EDX

280   analysis clearly show that the inhibitor molecules form a good protective

281   film on the Pb surface.

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Table 1: The values of hydrogen evolution rates (H_R) at 303 and 323K, and activation energy (E_a) for Pb in 5.0 M H_2SO_4 in the absence and presence of various concentrations of L-Serine.

L-Serine (mM)	H_{R1} (ml/h) 303 K	H_{R2} (ml/h) 323 K	E_a (kJ mol^{-1})
Blank	25	38	16.92
0.1	23	36	18.09
0.5	17	27	18.68
1	7	14	27.99
5	4	9	32.75
10	3	8	39.61
15	3	7	34.22
20	3	7	34.22

Table 2 Polarization parameters for Pb in 5.0 M H_2SO_4 without and with various concentrations of of L-Serine.

L-Serine (mM)	E_{corr} (mV vs. Hg/Hg$_2$SO$_4$)	j_{corr} (mA cm^{-2})	η_j %
Blank	-940	5.12	-
0.1	-1030	4.60	10.15
0.5	-1045	3.35	34.57
1	-1071	1.25	75.58
5	-1087	0.696	86.40
10	-1112	0.460	91.01
15	-1123	0.452	91.17
20	-1130	0.450	91.21

Table 3 Adsorption parameters for L-Serine adsorption on Pb surface in

5.0 H_2SO_4 at 303 K.

Method	R^2	K_{ads} (M^{-1})	ΔG^o_{ads} (kJ mol^{-1})
Hydrogen evolution	0.9985	1428	-28.36
Electrochemical	0.9991	1666	-28.75

Table 4: The heat of adsorption (Q_{ads}) of L-Serine on Pb surface in 5.0 M H_2SO_4 at various concentrations of L-Serine

L-Serine (mM)	θ_1	θ_2	Q_{ads} (kJ mol^{-1})
	303 K	323 K	
0.1	0.080	0.052	-18.64
0.5	0.320	0.289	-6.51
1	0.720	0.631	-16.57
5	0.840	0.763	-19.93
10	0.880	0.789	-27.43
15	0.880	0.815	-20.67
20	0.880	0.815	-20.67
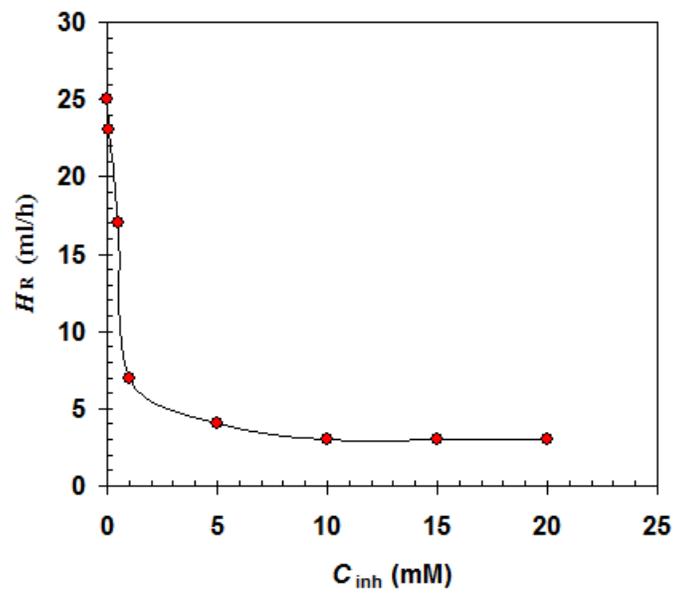
Fig. 1

Fig.2

Fig. 3

Fig .4

Fig.5

Fig.6