

Effect of Para-Dimethyl Aminobenzaldehyde additive on Electrochemical performance of Lead–Acid Battery

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Abstract

Effects of para-dimethyl aminobenzaldehyde (p-DMAB) additive on electrochemical performance of lead-acid battery have been studied by electrochemical impedance spectroscopy (EIS), linear sweep voltammetry (LSV), cyclic voltammetry (CV) and scanning electron microscopy (SEM). Results show that p-DMAB improves the reduction and oxidation process of electrode materials, increases the hydrogen overpotential, facilitates the diffusion of substances into and out of the electrode. The p-DMAB additive is stable under the operating conditions of lead-acid batteries. The results showed that p-DMAB is promising for use as a suitable additive for lead-acid batteries.

Keywords: Lead-acid battery, electrochemical performance, para-dimethyl aminobenzaldehyde additive.

Introduction

Lead acid batteries (LABs) play an important role in the industry and social life. Although used in many different fields and produced with stable processes, LABs still have some disadvantages such as: low material efficiency, short cycle life, gas evolution when battery is overcharged [1,2].

Studies to improve the performance of LABs are carried out in different directions such as: changing composition of electrode active materials, changing components of grid, improving separator and structure of the battery and using additives for the electrolyte solution [3-8]. The latter is of interest because there are many advantages such as: comprehensive impact on electrode processes, little change in manufacturing processes, ease of application for both production and use [2,9]. Additives for electrolyte solution in lead-acid batteries include: inorganic additives, suspension carbon and organic additives [9]. In particular, organic additives have the advantage of being designable and strongly inhibiting hydrogen evolution. Benzaldehyde and acetophenone derivatives have been reported to have effects such as: reducing hydrogen evolution [10-13], preventing precipitation of Sb to Pb [12]; reduced lead sulfate crystal size [14], increased porosity of electrode active material [11] resulting in an increase in capacity;

assisting the elimination of oxygen [10]. Hydrogen inhibitors can be used in flooded batteries to improve maintenance-free effects and in airtight storage batteries to reduce hydrogen pressure [9]. The above beneficial effects are the result of the interaction between carbonyl group in benzaldehydes/ acetophenones and surface of the electrode materials following the model of W. Böhnstedt *et al.* [13]. Recently, several ionic liquids (ILs) have been studied for use as an additive for electrolyte solutions in lead-acid batteries. These additives exhibit the following effects: increasing the hydrogen drainage potential [15-18], reducing the rate of corrosion [19-21], making porous particles of active material [18,20], enhancing the conversion of PbO into Pb and PbO₂ [20]. In the electrolyte solution, cations of ionic liquids are transported from the solution to the electrode surface and adsorbed onto the surface by physical and electrostatic interaction. The different behaviors of ionic liquids result from differences in the size and charge density of ILs, depending on the structure and number of chains of cations in those ILs [15]. Para – Dimethyl aminobenzaldehyde (p-DMAB) is an organic compound containing an amino group and a benzaldehyde base with the formula (CH₃)₂NC₆H₄CHO. It is used in Ehrlich reagent and Kovac reagent to test indoles. The structural formula of p-DMAB is shown in **Figure 1**.

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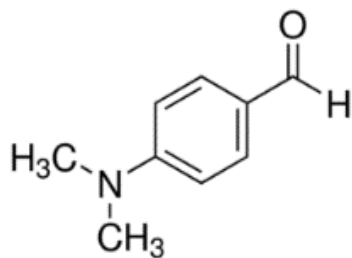


Figure 1. Structure of p-DMAB

There have been no reports of the use of p-DMAB for electrolyte solutions in lead-acid batteries. However, based on structural characteristics, p-DMAB is expected to have a positive effect on the performance of lead-acid batteries - as a hybrid additive between ionic liquids and benzaldehyde.

This paper investigates the effect of p-DMAB additive on the electrochemical performance of lead-acid batteries on the following aspects: the redox reaction of electrode active materials, the release of hydrogen gas and the change in morphological structure of material.

2. Experiment

2.1. Preparation of working electrode.

The working electrodes are the flat plates made of Pb-Sb (3% wt. Sb) alloy or 99.99% pure lead. Except that the working surface area of electrodes were exposed, remaining parts were covered with an epoxy to avoid any contact with electrolyte solution.

2.2. Materials and electrolytes

The 98% concentrated sulfuric acid is pure chemical from China. The H_2SO_4 electrolyte ($d = 1.275 \text{ g/cm}^3$) is prepared from concentrated H_2SO_4 and double distilled water. The p-DMAB additive is pure chemical from Germany. Electrolyte solutions containing 10, 50, 80, 120, 160, 200 and 240 mg/L of p-DMAB additive were prepared by adding an appropriate calculated amount of p-DMAB additive to the electrolyte.

2.3. Electrochemical measurements

Electrochemical measurements were carried out with a potentiostat/galvanostat equipment (AUTOLAB PGSTAT 302N-Netherlands) using the three electrode system. Working electrodes were 99,99% pure lead or Pb-3%Sb alloy plates with surface area of 0.5665 cm^2 and 0.4913 cm^2 , respectively. The counter electrode and reference electrode were a pure lead sheet and an Ag/AgCl electrode, respectively. Before every measurement, the working electrode was mechanically polished with fine paper and cleaned with acetone and double distilled water.

For evaluation of the effect of the p-DMAB on redox reaction of negative active material, cyclic voltammograms were obtained at the scan rate of 50 mV/s, in potential range of -0.9 to -0.2 V (Ag/AgCl). The working electrode used is a pure lead plate. The electrochemical impedance measurements were obtained at the potential of -0.62 V (Ag/AgCl), in the frequency range from 10 kHz to 10 mHz. The voltage amplitude is 10 mV.

For evaluation of the effect of the p-DMAB on redox reaction of positive active material, cyclic voltammograms were obtained at the scan rate of 50 mV/s, in potential range of 1.14 to 2.5 V (Ag/AgCl). The working electrode used is the pure lead plate. Before each measurement, the working electrode is polarized at 1.8V (Ag/AgCl) for 900 seconds. The electrochemical impedance measurements were obtained at the potential of 1.51 V (Ag/AgCl), in the frequency range from 10 kHz to 10 mHz. The voltage amplitude is 10 mV.

For evaluation of the effect of p-DMAB on hydrogen evolution, polarization measurements were carried out with the scan rate of 5 mV/s, in the potential range of -1.4 to -0.9V (Ag/AgCl). The working electrode is the Pb-3%Sb alloy plate.

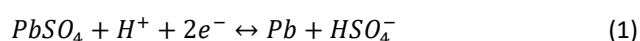
2.4. SEM imaging

The SEM images of the electrode active materials were obtained with JSM 6610-LA scanning electron microscopy (Jeol-Japan). To determine the microstructure of active materials, before taking SEM images, electrodes were polarized by cyclic voltammetry with 10 cycles in H_2SO_4 solution ($d = 1.275 \text{ g/cm}^3$), without and with the different concentrates of p-DMAB. The potential ranges are from 1.14 to 2.5 V (Ag/AgCl) and from -0.9 to 0.2 V (Ag/AgCl) for positive and negative active material, respectively.

3. Results and Discussion

3.1. Redox reaction of negative active material

Figure 2 shows the cyclic voltammetry spectra of negative active material (NAM) in H_2SO_4 solution ($d = 1.275 \text{ g/cm}^3$) without and with different concentrations of p-DMAB. The scan rate is 50 mV/s. The voltage range is from -0.9 V to -0.2 V (Ag/AgCl). There are two peaks on the CVs. The oxidation peak corresponds to the conversion of Pb into $PbSO_4$. The reduction peak corresponds to the conversion of $PbSO_4$ into Pb according to the following equation:



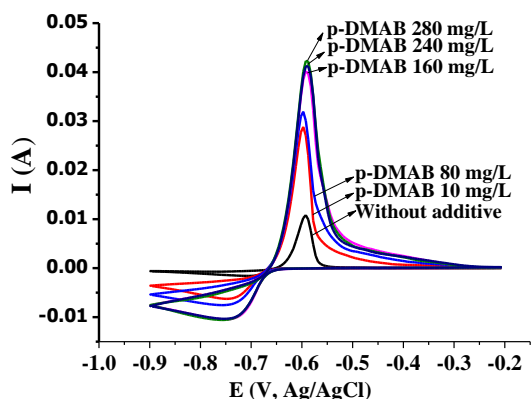


Figure 2. The cyclic voltammety spectra of the negative active material in the H₂SO₄ solution (d = 1.275 g/cm³) without and with different concentrates of p-DMAB. Scan rate: 50 mV/s.

Thus, the presence of the additive in the electrolyte solution does not change the electrode process on the NAM. However, there are changes in height of the oxidation and reduction peaks. To assess the effect of the additive on the redox reaction of the negative active material, the Q_{Add}^{N+}/Q_0^{N+} and Q_{Add}^{N-}/Q_0^{N-} ratios were calculated. The area under the anode peak is the amount of charge for oxidation of Pb to PbSO₄, denoted by Q_{Add}^{N+} and Q_0^{N+} . Here, the “N+” symbol refers to the oxidation process on the NAM, the “0” and “Add” symbols refer to the electrolyte solution without and with the additive. Similarly, the area under the cathode peak is the amount of charge for the reduction of PbSO₄ to Pb, denoted by Q_{Add}^{N-} and Q_0^{N-} . The ratios mentioned above are called conversion ratios, which show the degree of improvement in the reactivity of the negative active material with p-DMAB. The variation of the conversion ratios is shown in **Figure 3**. It is found that the values of the conversion ratios tend to increase with increasing p-DMAB concentration in the electrolyte solution. Thus, the presence of the additive greatly increases the conversion of Pb to PbSO₂ and vice versa. This is the result of the interaction between p-DMAB and the surface of the electrode material. The p-DMAB additive can enhance the ability of conversion reaction at low concentrations.

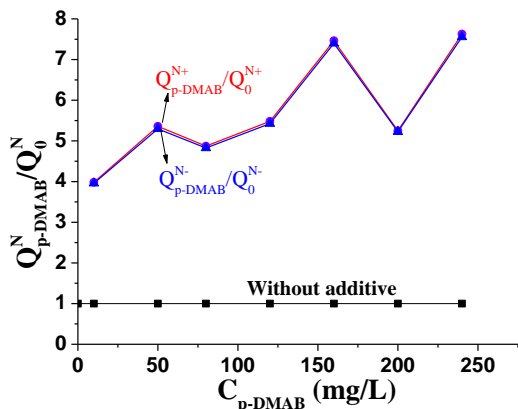


Figure 3. The dependence of the conversion ratios on the negative active material on the concentration of p-DMAB

Figure 4 shows the Nyquist-type electrochemical impedance spectra of pure lead electrode in H₂SO₄ solution (d = 1.275 g/cm³) without and with different concentrations of p-DMAB at -0.62 V (Ag/AgCl). The electrode potential E = -0.62 V corresponds to the equilibrium electrode potential of the negative active material. Therefore, the kinematic parameters derived from impedance measurements are typical parameters for the redox reaction of the NAM according to Equation (1). It is shown from **Figure 4** that the presence of p-DMAB in the electrolyte solution reduces the electrochemical impedance of the redox reaction on the negative active material. This result explains the enhancement of the redox reaction of the NAM with p-DMAB. The descent to the horizontal axis of the impedance spectra at the low frequency region (**Fig. 4b**) demonstrates the adsorption of p-DMAB onto the electrode surface.

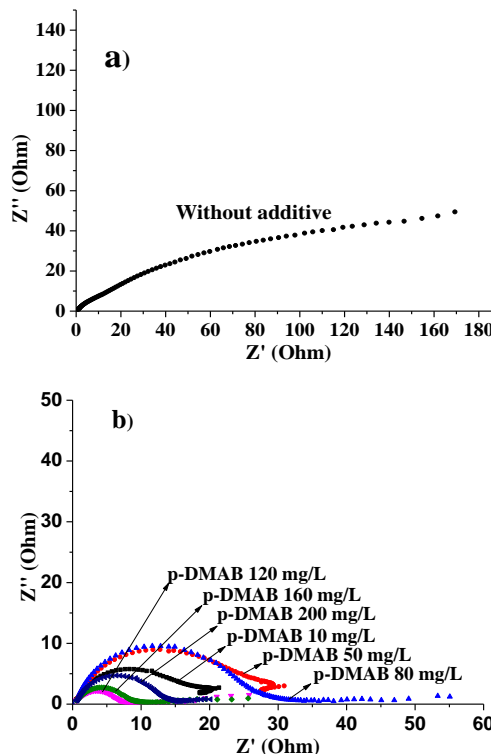


Figure 4. Nyquist-type impedance spectra on the pure lead electrode at -0.62 V (Ag/AgCl) without (a) and with different concentrations of p-DMAB (b).

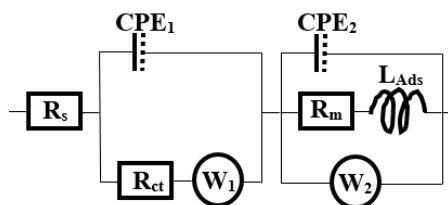


Figure 5. The equivalent circuit describes the reaction process on the negative active material.

The kinematic parameters of the electrode response are drawn through the use of an equivalent circuit in **Figure 5**.

Here, R_s is the solution resistance; CPE_1 is a constant phase element characteristic for double layer capacitance; R_{ct} is the charge transfer resistance; W_1 is the Warburg impedance representing the diffusion in the double layer. CPE_2 is a constant phase element that characterizes the capacitance of the semi-permeable membrane formed by sulfate ions and (or) adsorbent additives on the surface of electrode active material. R_m is the resistance of the semi-permeable membrane. L_{Ads} is specific to the resistance produced by the adsorption process. W_2 is the Warburg impedance representing the diffusion of substances through semi-permeable membranes. The variation of kinematic parameters that derive from fitting the measured impedance datas with the equivalent circuit is shown in **Figure 6**. Here, σ_1 and σ_2 are Warburg constants of diffusion processes of substances through the double layer and the semi-permeable membranes, respectively.

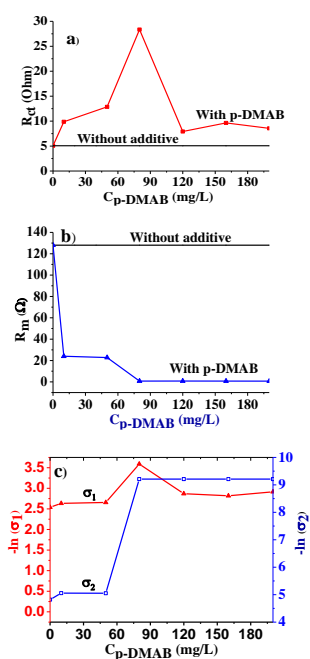


Figure 6. Dependence of kinematic parameters of redox reaction on the NAM on the concentration of p-DMAB additive in H_2SO_4 solution ($d= 1.275 \text{ g/cm}^3$): (a) R_{ct} , (b) R_m , (c) σ_1 and σ_2 Warburg constants.

It is shown in **Figures 6a** and **6b** that p-DMAB increases the charge transfer resistance but reduces the semi-permeable membrane resistance. It is known that the Warburg constant σ is inversely proportional to the square root of the diffusion coefficient of reactants and reaction products [22]. Because the values on the vertical axis of **Figure 6c** are $-\ln(\sigma_1)$, $-\ln(\sigma_2)$, these values are proportional to the diffusion coefficient of the substances involved in the reaction. In the case of the p-DMAB additive, it is noticed that the values of $-\ln(\sigma_1)$ and $-\ln(\sigma_2)$ are greater than without the additive. Thus, the presence of p-DMAB has improved the diffusion ability of

substances in double layer and through semi-permeable membrane. This is important because delays in the diffusion of substances increase the concentration polarization and make the reaction process more difficult. Increasing the concentration of p-DMAB to 80 mg/L increases the diffusion ability. Continuing to increase the concentration of p-DMAB in excess of 80 g/L, the diffusion ability in the double layer decreases and through the membrane holds steady.

3.2. Redox reaction of the positive active material

Figure 7 is the cyclic voltammetry (CV) spectra of positive active material (PAM) in H_2SO_4 solution ($d = 1.275 \text{ g/cm}^3$) without and with different concentrations of p-DMAB. The scan rate is 50 mV/s. The voltage range is from 1.14 V to 2.5 V (Ag/AgCl). There are two peaks on the CV spectra. The oxidation peak corresponds to the conversion of $PbSO_4$ into PbO_2 . The reduction peak corresponds to the conversion of PbO_2 into $PbSO_4$ according to the following equation:

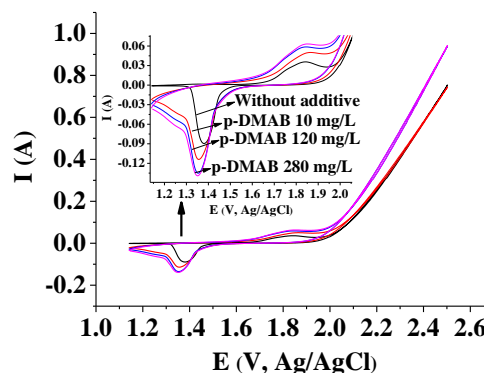
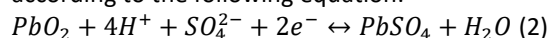


Figure 7. The cyclic voltammetry spectra of the positive active material in the H_2SO_4 solution ($d = 1.275 \text{ g/cm}^3$) without and with different concentrates of p-DMAB. Scan rate: 50 mV/s.

As with negative active materials, the presence of additives does not change the redox process of the positive active material. The values of Q_{Add}^{P+} and Q_0^{P+} are, respectively, the amount of charge used for the oxidation of $PbSO_4$ with and without additive in the electrolyte solution. The values of Q_{Add}^{P-} and Q_0^{P-} are, respectively, the amount of charge used for the reduction of PbO_2 with and without additive in the electrolyte solution. **Figure 8** shows the conversion ratios of the oxidation reaction $PbSO_4$ to PbO_2 (Q_{Add}^{P+}/Q_0^{P+}) and vice versa reduction (Q_{Add}^{P-}/Q_0^{P-}) on the PAM. It was found that the conversion ratios of active materials were greater than 1 with p-DMAB in the solution. The value of conversion ratios gradually increases with increasing concentration of p-DMAB additive up to 240 mg/L. Then this value decreases slightly as the additive concentration continues to rise.

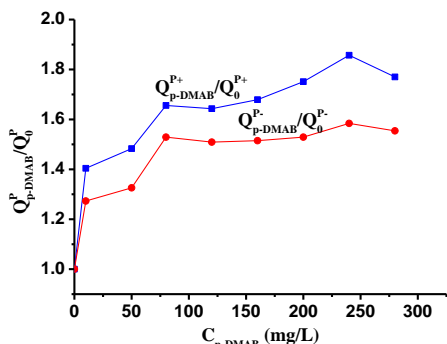


Figure 8. The dependence of the conversion ratios on the positive active material on the concentration of p-DMAB

Figure 9 is the electrochemical impedance spectra of the lead electrode in solution of H₂SO₄ (d = 1.275 g/cm³) at 1.51V (Ag/AgCl) without and with different concentrations of the additive. The frequency range is from 10 mHz to 10 kHz. The amplitude is 10 mV.

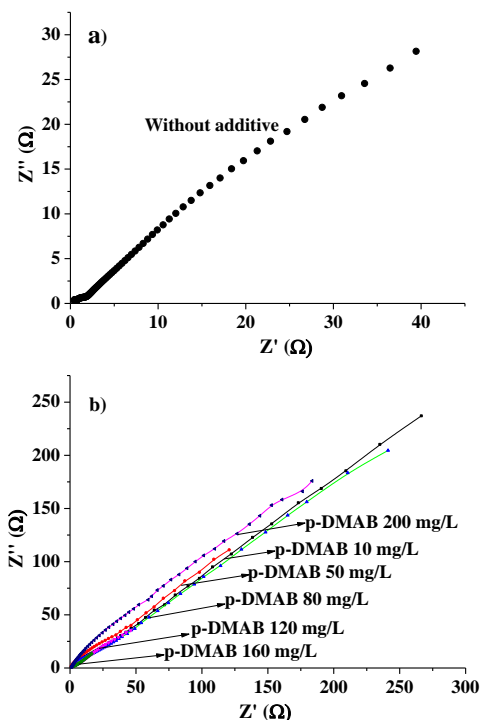


Figure 9. Nyquist-type impedance spectra on the pure lead electrode at 1.51 V (Ag/AgCl) without (a) and with different concentrations of p-DMAB (b).

The electrode potential of 1.51 V (Ag/AgCl) is the equilibrium potential of the positive active material. Therefore, electrochemical parameters derived from impedance measurement are typical parameters for the conversion of PbSO₂ and PbO₂ according to reaction equation (2). The kinematic parameters were drawn by fitting the measured impedance datas with the equivalent circuit in **Figure 10**. In this circuit, R_s is the solution resistance; CPE₁ is a constant phase element characteristic for double layer capacitance; R_{ct} is the charge transfer resistance; W₁ is the Warburg impedance

representing the diffusion in the double layer. C_m is the typical capacity that characterizes the capacitance of the semi-permeable membrane formed by sulfate ions and/or adsorbent additive on the surface of electrode active material. R_m is the resistance of the semi-permeable membrane. W₂ is the Warburg impedance representing the diffusion of substances through semi-permeable membranes.

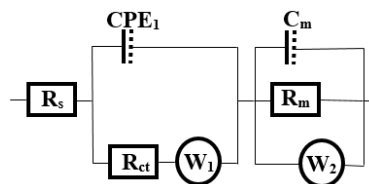
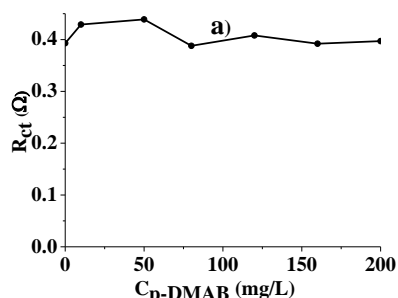


Figure 10. The equivalent circuit describes the reaction process on the positive active material.

Figure 11 shows the variation of kinematic parameters by p-DMAB additive concentration. It is seen from **Figure 11a** that the charge transfer resistance increases when increasing the p-DMAB concentration up to 50 mg/L. However, when the concentration of p-DMAB increases in excess of 50 mg/L, the charge transfer resistance decreases to the value that is achieved without p-DMAB in the solution.

Figure 11b shows the variation of the semi-permeable membrane resistance formed on anode-active material according to the concentration of p-DMAB. This additive reduces more than 50% of semi-permeable membrane resistances. The above effect is due to the presence of p-DMAB that makes the semi-permeable membrane form discontinuously, facilitating ion conduction and particle-particle contact become better.

Figure 11c shows the variation of the Warburg constant σ_1 and σ_2 corresponding to the diffusion in the double layer and diffuse through semi-permeable membranes on the positive active material according to the concentration of p-DMAB. It is recognized that the values of $-\ln(\sigma)$ in the presence of the additive are greater than those without it. This means the additive improves the diffusion of reactants and reaction products in the double layer and semi-permeable membranes. The diffusion in the double layer and through semi-permeable membranes on the positive active material is best improved at a concentration range of 10 to 150 mg/L.



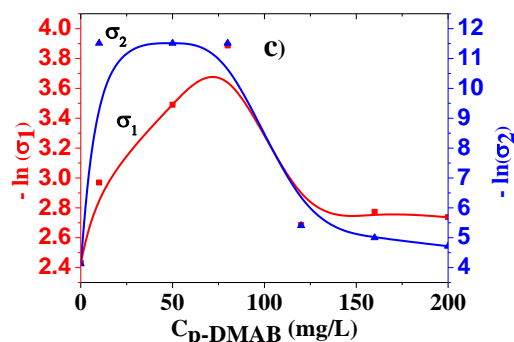
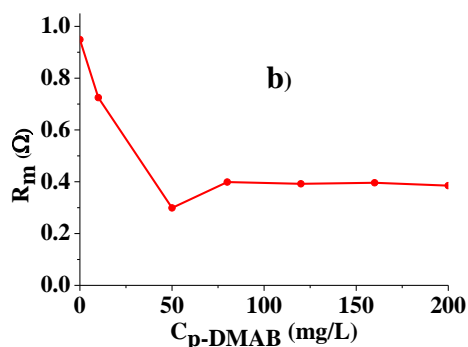


Figure 11. Dependence of kinematic parameters of redox reaction on the PAM on the concentration of p-DMAB additive in H₂SO₄ solution (d= 1.275 g/cm³): (a) R_{ct}, (b) R_m, and (c) σ₁ and σ₂ Warburg constants.

3.3. Hydrogen evolution

Figure 12 shows the linear sweep voltammograms (LSV) of pure Pb and Pb-3%Sb alloy electrodes in H₂SO₄ solution (d = 1.275 g/cm³) without and with different concentrations of p-DMAB in the region of hydrogen evolution voltages. It is seen that, the presence of p-DMAB additive in electrolyte causes the polarization curves of both pure lead electrode and alloy electrode to shift towards negative potentials compared with the absence of the additive. This means that the p-DMAB additive has inhibitory effects on the hydrogen evolution on both pure lead and alloy lead electrodes. The polarization curves of alloy electrode shift to more negative potentials with only a small amount of the additive.

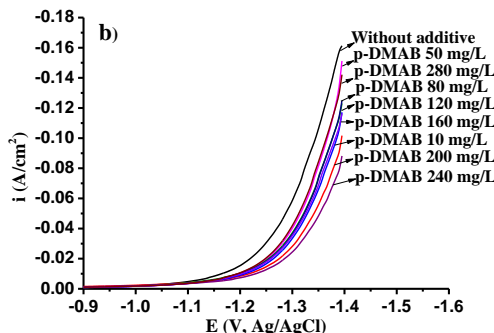
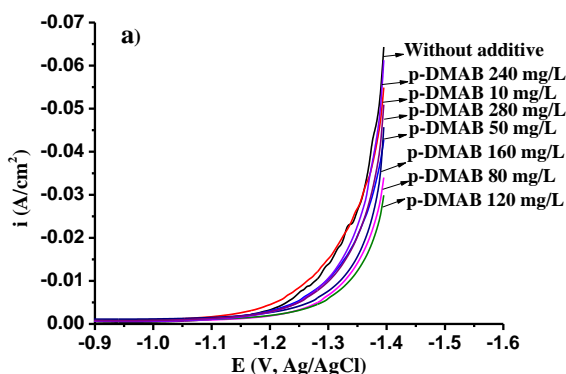


Figure 12. The linear sweep voltammograms (LCV) of Pb and Pb-3%Sb alloy in H₂SO₄ solution (1.275 g/cm³) without and with various concentrations of p-DMAB additive. Scan rate: 5 mV/s.

Figure 13 shows the dependence of the H₂ evolution potentials on the Pb-3%Sb alloy electrode and the pure lead electrode on the concentration of the additive p-DMAB in the H₂SO₄ solution (d = 1.275 g/cm³). The values of the H₂ evolution potentials are recorded from LSVs at a current density of 5 mA/cm². According to the results from **Figure 13**, the addition of the p-DMAB additive makes the hydrogen evolution potential on both the pure lead electrode and alloy lead electrode to shift to more negative values. This means that the presence of the p-DMAB additive in the electrolyte increases the hydrogen evolution overvoltage.

To evaluate the inhibitory ability of the additive on the hydrogen evolution, the I_{Pb}(%) and I_{Alloy}(%) inhibition coefficients are calculated according to the formula (3.1) and (3.2), respectively. The results are presented in **Table 1**. Here, Q_{H,0}^{Alloy} and Q_{H,Add}^{Alloy} indicate that the amount of charge used during hydrogen evolution on Pb-Sb alloy electrode without and with p-DMAB additive in H₂SO₄ solution (the area under the LSV curves for hydrogen evolution in Fig. 12). Similarly, Q_{H,0}^{Pb} and Q_{H,Add}^{Pb} are the amount of charge used during hydrogen evolution on pure Pb electrode without and with p-DMAB additive in H₂SO₄ solution.

$$I_{Pb}(\%) = \frac{Q_{H,0}^{Pb} - Q_{H,Add}^{Pb}}{Q_{H,0}^{Pb}} \times 100\% \tag{3.1}$$

$$I_{Alloy}(\%) = \frac{Q_{H,0}^{Alloy} - Q_{H,Add}^{Alloy}}{Q_{H,0}^{Alloy}} \times 100\% \tag{3.2}$$

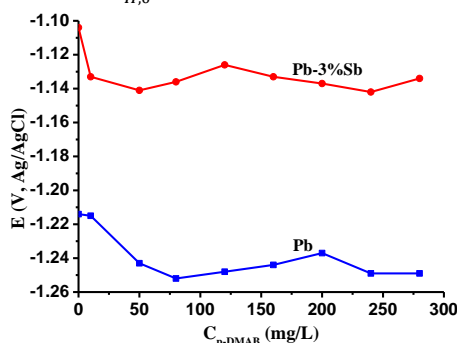


Figure 13. The dependence of the hydrogen evolution potential on the pure lead and the Pb-3% Sb alloy electrodes on the p-DMAB concentration at the current density of 5 mA/cm².

It is seen from Table 1, the p-DMAB additive inhibits hydrogen gas release on both pure lead electrode and Pb-3% Sb alloy lead electrode even at low concentrations of 10 mg/L. For alloy electrode, with increasing p-DMAB concentration upto 240 mg/L, the value of inhibition coefficients increases and reaches a maximum of 52.24 %. As the additive concentration of the additive exceeds 240 g/L, this value decreases. For pure lead electrode, the ability of p-DMAB to inhibit the hydrogen gas release reaches 49.9 % at a p-DMAB concentration of 120 mg/L. At concentrations of p-DMAB greater than 120 mg/L, the inhibitory ability of the additive decreases.

Table 1. The dependence of inhibition coefficients on the alloy lead and pure lead electrodes on the concentrations of the additive

C _{Add} (mg/L)	0	10	50	80	120	160	200	240	280
I _{Pb} (%)	0	0,2	22,2	45,6	49,9	33,1	23,8	13,5	21,4
I _{Alloy} (%)	0	13,4	24,8	31,3	32,3	35,1	44,9	52,2	23,5

3.4. The surface morphology of alloy electrode

Figure 14 and Figure 15 show SEM images of pure Pb surface after 10 cycles of CV (in H₂SO₄ solution (d = 1.275 g/cm³) without and with various concentrations of p-DMAB. It is seen from Fig. 14 and Fig. 15 that formed crystals on the lead electrode surface in the presence of p-DMAB additive are smaller in size. It seems that p-DMAB additives adsorbed on the electrode surface, hence, change the structures of crystals formed on the electrode surface.

3.5. Durability of the additive

Figure 16 shows multiple cycles of the cyclic voltammetry spectra of the pure lead electrode in the H₂SO₄ solution (d = 1,275 g/cm³) without and with p-DMAB additive. The CV spectra in Fig. 16a are obtained in the voltage range from -0.9 V to -0.2 V (Ag/AgCl) corresponding to the conversion region of the positive active material. The CV spectra in Fig. 16b are obtained in the voltage range from 1.14 V to 2.0 V (Ag/AgCl) corresponding to the conversion region of the negative active material. It was found that p-DMAB additive enhances the electrochemical reactivity of positive and negative active materials as described above. This improvement was maintained consistently after 200 cycles of the CV showed that the additive was stable under the operating environment of the lead acid battery.

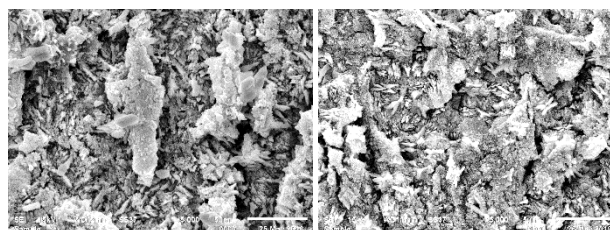
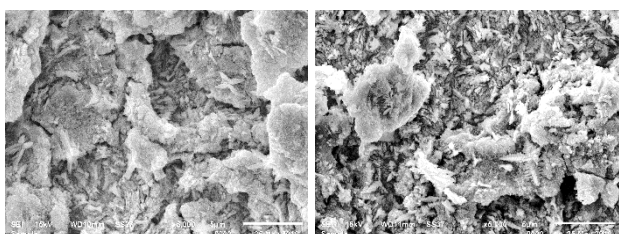


Figure 14. SEM images of Pb electrode after 10 cycles of CV in H₂SO₄ (d = 1.275 g/cm³): (a) without additive, (b) p-DMAB 80 mg/L (c) p-DMAB 120 mg/L and (d) p-DMAB 160 mg/L. The voltage range of CV is from 1.14 V to 2.5 V (Ag/AgCl).

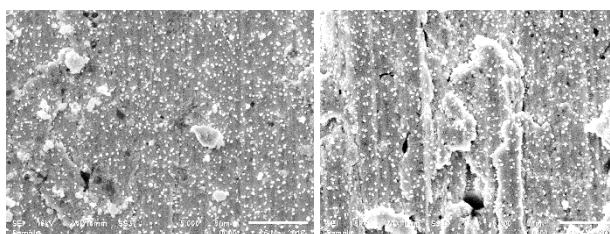
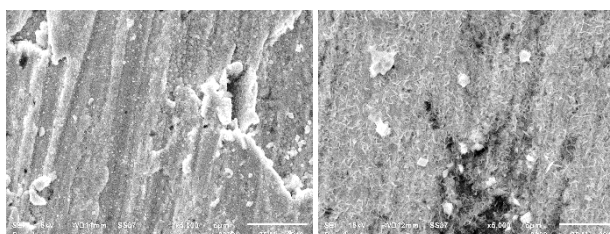


Figure 15. SEM images of Pb electrode after 10 cycles of CV in H₂SO₄ (d = 1.275 g/cm³): (a) without additive, (b) p-DMAB 80 mg/L (c) p-DMAB 120 mg/L and (d) p-DMAB 160 mg/L. The voltage range of CV is from -0.9 V to -0.2 V (Ag/AgCl).

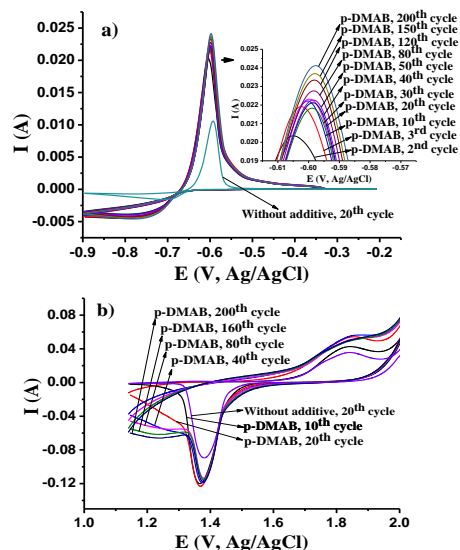


Figure 16 The multiple cycles of the cyclic voltammetry spectra of the pure lead electrode in the H₂SO₄ solution (d = 1,275 g/cm³) without and with p-DMAB additive. Scan rate: 50 mV/s. Voltage range (vs Ag/AgCl): (a) -0.9 V to -0.2 V, (b) 1.14 V to 2.0 V.

Conclusions

The effects of p-DMAB additive on electrochemical behavior of lead-acid battery were drawn through this study as:

- 1) The additive p-DMAB enhances the electrochemical reactivity of the negative and positive active materials through reducing the semi-permeable membrane resistance, increasing the diffusion ability of substances.
- 2) The additive p-DMAB increases the overpotential of hydrogen, thereby inhibiting the release of hydrogen gas on both the lead electrode and the lead alloy electrode. For the pure lead electrode, the ability to inhibit hydrogen release reached the maximum value of 49.9 % at the p-DMAB concentration of 120 mg/L. For the alloy lead electrode, this value and 52.2 % at the additive concentration of 240 mg/L for the alloy electrode.
- 3) The presence of p-DMAB in the electrolyte solution changes surface morphology of activity materials. The formed crystals are smaller in size.
- 4) The p-DMAB additive is stable under the operating environment of the lead acid battery. Therefore, p-DMAB is promising for use as an additive for electrolyte solutions in lead-acid batteries.

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