

Short communication

An optimized pretreatment method in the analysis of negative plate of lead acid battery

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Abstract

The composition of the plate in lead acid battery plays a vital role on battery performance. Therefore, controlling and monitoring of plate composition is utmost important in order to attain required battery performance. While monitoring negative plate, it has to be ensured that they are not exposed to air, which would otherwise result in Pb oxidation; thereby affecting the accuracy of the test data. Nevertheless, published information is scarce on the “pretreatment and preservation of the negative plates”. The objective of this study is to develop a suitable procedure to “preserve and pretreat the negative plate” prior to chemical composition analysis. A host of experimental parameters like immersion time in water, effect of drying reagent and air drying time were studied. Two drying solvents; namely, methanol and acetone were studied to evaluate their suitability.

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1. Introduction

Lead acid batteries have been used for diverse and demanding applications where the energy needs vary significantly. The hostile conditions that these batteries would experience also differ remarkably depending upon the application.

The best way of achieving the required performance for different applications is by optimizing the “quantity and quality” of the materials that are used in the plates. While the quantity of the material can be controlled precisely by adopting advanced manufacturing techniques, the quality of plate is achieved by optimizing the plate composition and hence monitoring of plate composition assumes significance.

The plate composition is determined by different methods. Standard test procedures describing the analysis of lead and its compounds in the plate are available in Greninger [1] and Vogel [2] using EDTA and electro-gravimetric methods, respectively. There have also been other methods adopted by the battery industry [3,4] including a two-step gravimetric method [5].

In case of negative plate composition analysis, plates have to be preserved properly to prevent the oxidation of lead. The

problem of lead oxidation is overcome by immersing the plates in water or under vacuum till the analysis is taken up [3,4]. Of the two methods mentioned, the former method is the most convenient and cost effective. In this method; the plates are dried by treating with a suitable organic solvent followed by air drying and then subjected to chemical analysis [3]. It means that a host of parameters such as immersion time in water, effect of drying reagent, air drying time etc., may influence the lead oxidation phenomena. However, published information on the role of each of these parameters is scarce. This paper primarily deals with the optimizing “preservation and pretreatment techniques” for negative plate analysis.

The negative plate composition is determined by gravimetric method [5] wherein, a particular compound is selectively dissolved and the weight of the rest of the compounds is estimated.

2. Experimentation

For the purpose of present study, automotive batteries of 38B20R type conforming to JIS specification were selected. The test samples were assembled with positive and negative plates having a weight tolerance of 2%. Subsequently, these batteries were formed using sulphuric acid of 1.225 sp. gr. in an environmentally controlled atmosphere. At the end of formation,

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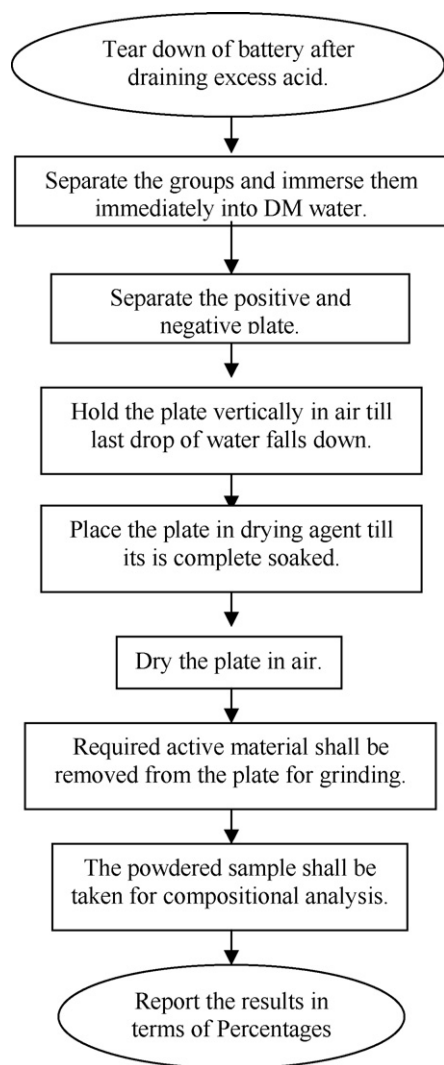


Fig. 1. The sequence followed in preservation and pretreatment of negative plates.

the integrity of the test samples was evaluated by subjecting to “high rate discharge” test.

To collect the plate for analysis, the acid in the battery was initially drained up to the top of plate. The cover and container were then separated. A particular group(s) was arbitrarily removed

from the pocket. On removal, it was immersed in distilled water immediately to avoid oxidation of lead. The positive and negative plates were separated from each other by keeping the group in water. The negative plates were subjected to the chemical analysis and the effect of various test parameters was studied. The sequence of “pretreatment and preservation” is as described in Fig. 1.

All the chemicals used for analysis were of AR grade. Distilled water obtained from MilliQ distillation unit of MILLIPORE and having conductivity of $<18 \text{ m}\Omega \text{ cm}$ was used for the experiments. Weighing balance (PRECISA, Model Number-40SM-200A) with an accuracy of 0.5% was used for weighing the plates.

3. Results and discussion

3.1. Selection of test samples for the experiment

Usually, all the plates in a battery are not homogeneous even though the test samples are manufactured under identical conditions. The degree of variation within and among the plates is therefore analyzed to decide on the sampling procedure to be followed for studying the effect of different variables.

Firstly, three negative plates collected from a cell were cut into top, middle and bottom pieces of uniform width. The cut pieces were dried in methanol followed by air drying. The test samples were analyzed for their composition using a two step analysis technique. The results of the analysis are given in Table 1. It is evident that the variation within the plate is less than that of the variation observed among the plates. Therefore, the samples prepared from a single plate were used to study the effect of each of the test parameters.

3.2. Effect of immersion time in water

The negative plate was cut into four pieces, which were then immersed in distilled water for different time periods. The selection of time was based on operational convenience. Each sample piece at the end of a particular immersion time was analyzed for its composition, to establish the detrimental effect of prolonged immersion of plate in water before subjecting to composition analysis. The test results are presented in Table 2. It is

Table 1

Composition	Plate-1			Plate-2			Plate-3		
	Top	Middle	Bottom	Top	Middle	Bottom	Top	Middle	Bottom
(a) Estimated composition within a plate									
Percentage of Pb	70.11	69.83	70.7	63.05	63.15	62.8	68	67.13	69.11
Percentage of PbO	14.8	14.22	13.46	19.99	20.8	20.02	16.1	15.44	16.11
Percentage of PbSO ₄	15.09	15.95	15.84	16.96	16.05	17.18	15.9	17.43	14.78
(b) Estimated composition among the plates									
Percentage of Pb	70.21			63.67			68.08		
Percentage of PbO	14.16			20.27			15.88		
Percentage of PbSO ₄	15.63			16.06			16.04		

Table 2
Estimation of composition on immersion of plates in water for different time intervals

S. no.	Composition	0 h	6 h	24 h	48 h
1	Percentage of Pb	73.40	74.99	67.72	62.59
2	Percentage of PbO	14.81	15.31	19.9	28.19
3	Percentage of PbSO ₄	11.79	9.70	12.38	9.22

evident that, lead oxidation was not predominant in first 6 h, whereas the prolonged immersion caused oxidation of lead. In order to prevent lead oxidation with prolonged immersion, the usage of deoxygenated water may help instead of static distilled water.

3.3. Determination of soak time in drying reagent

In the process of estimating the composition of the plate as described earlier, the plates were immersed in distilled water till the analysis is started. The plates upon removal from the water were subsequently dried using a drying reagent. The objective of drying reagent is to remove the water from plate, which means that the drying reagent shall have complete miscibility with water.

In the present study, the drying efficiency of “acetone and methanol” was studied. It shall be noted that while acetone and methanol are completely miscible with water, on the other hand, acetone has the following advantages over methanol:

- low boiling point [6,7],
- higher auto-ignition temperature (465 °C) [6] than that of methanol (385 °C) [6,7],
- lesser cost and
- lower toxic ratings.

The effect of “soak time” with respect to each drying reagent is presented in Table 3a. The test samples were immersed in acetone and methanol for 5 and 15 min, respectively, followed by air drying of 20 min and then subjected to the chemical analysis. To validate the sufficiency of 15 min soak time, another set of test samples were soaked in acetone for 15, 30 and 60 min, respectively. The results as listed in Tables 3a and 3b infer that further soaking beyond 15 min does not show any improvement. The results also infer that the amount of lead reported with acetone was higher in comparison to that obtained with methanol by about 6% to 8%.

Table 3a
Estimation of plate composition on soaking them in methanol and acetone

Drying agent	Time	Pb (%)	PbO (%)	PbSO ₄ (%)
Acetone	5 min	72.114	17.228	10.658
Methanol		66.486	21.582	11.932
Acetone	15 min	75.842	11.750	12.408
Methanol		67.928	18.394	13.678

Table 3b
Determination of extended soaking time with acetone

Time	Pb (%)	PbO (%)	PbSO ₄ (%)
15 min	88.764	4.072	7.164
30 min	87.600	5.344	7.056
60 min	88.026	5.034	6.940

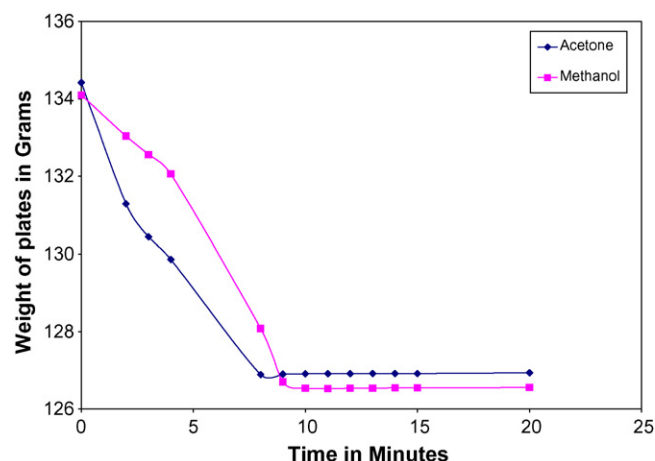


Fig. 2. Weight of plates on air drying at different lengths of time.

3.4. Effect of air drying time

The test sample was parted into two pieces. They were soaked in acetone and methanol for 15 min. Subsequently they were dried under ambient conditions in an open atmosphere, wherein the temperature was observed to be between 33 and 37 °C. The weights of the samples were measured up to 20th minute with a time interval of 1 min. The results are presented in Fig. 2. The onset of stable weight indicates the completion of drying.

As evident, the plate loses its weight continuously indicating the process of drying. The weights started stabilizing at 9th minute in case of acetone and 11th minute in case of methanol. The faster drying in case of acetone can be attributed to its lower boiling point and vapor pressure.

The results obtained with optimized test conditions are listed in Table 4. To ensure the sufficiency of 10 min drying time with acetone as evident from “weight stabilization study”, another set of experiment was carried out by soaking the test sample in acetone for 15 min and drying in air for 10 and 20 min, respectively. The results infer that 10 min of air drying is sufficient. It is noteworthy, that the ambient temperature and air stream are the two vital factors, which can have significant influence on

Table 4
Analyzing the plate composition after different air drying time

S. no.	Material/test	Air drying time	
		10 min	20 min
1	Percentage of Pb	84.412	85.067
2	Percentage of PbO	5.605	5.375
3	Percentage of PbSO ₄	9.983	9.558

the drying time and these aspects deserve further investigation. Further, drying in oxygen free atmosphere is another interesting area to explore.

4. Conclusion

In the above said work, the following are the major conclusions:

- The amount of lead was found to be within close tolerance when immersed in water till the end of 6 h, but prolonged immersion causes more Pb oxidation. However, given a possibility, the analysis should be taken up as quickly as possible.
- Usage of acetone was found to provide better results than methanol.
- A minimum soak time of 15 min in drying reagent is suggested to ensure the complete removal of water from the plate.
- Air drying for 10 min (with respect to soaking in acetone) was found to be sufficient for the complete removal of drying reagent from the plate.

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