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5 PREPARATION OF KEROSENE SOOT CARBON ELECTRODE AND ITS APPLICATION IN LITHIUM ION BATTERY

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Abstract—The research of the development kerosene soot carbon particles and its application as electrode for lithium ion battery has been done. The carbon was prepared from kerosene by using thermal plasma pyrolysis and characterized by Boehm titration in order to determine the carbon functional group, point zero charge for surface charge and X-ray diffraction for crystallography. Carbon was mixed with polianilin to form anode and mixed with lithium, iron salts and phosphate to form LiFePO_4 as cathode. Both electrodes were fabricated into lithium ion battery with Li_2SO_4 as electrolyte. The performance of battery was measured with the variation of electrolyte concentration i.e., 0.1 M, 0.5 M, and 1 M. Boehm titration result showed that the carbon has acid functional groups greater than base functional group as 6.90 mgeq. 4.80 of point zero charge pH was in acid condition. The crystallography of carbon as anode presented in diffractogram showed $2\theta = 23.68$ and 42.33 of diffraction peaks and indicated graphite crystal in the carbon. CV measurement reveal that the oxidation occurred at the peak of anode current, i.e., 0.015 A for 0.3 V whereas the reduction occurred at the -0.3 V and 0.007 A. A Galvanostatic charge discharge measurement showed that The highest capacity is obtained in 0.52 mAh/g with a voltage of 0.71 Volts, which is the 500th cycle. The lowest capacity is obtained in only 0.15 mAh/g of 0.2 Volts, that is in cycle 1st.

Keywords—electrode, kerosene, boehm titration, pH, pzc, voltammogram, diffractogram, LIB, galvanostatic

I. INTRODUCTION

Carbons with large surface area become very important as their application in LIB electrode material because they significantly contributed high energy density storage [1]. Such materials can be obtained from methane plasma jet pyrolysis [2], thermal plasma pyrolysis of four polymer waste [3], oily sludge [4] and the plasma pyrolysis from fatty oil [5]. Based on the research conducted by Hossain (2013), carbon nanoparticles were produced from the pyrolysis of petroleum diesel fuel [6].

Carbon particles can also be obtained from petroleum kerosene using thermal plasma pyrolysis as stated by Chen et al [7]. Kerosene produced directly from traditional well

become fascinating because it contains chemicals that provide a manageable oil for producing heat, combustion and pyrolytic capability [8]. Soot as pyrolytic product has higher price compare to the others product because it has a specific feature that valuable for a specific application, such as nano feature of carbon in the battery electrodes.

The lithium ion batteries (LIB) electrodes generally use graphite and its composites[9]. Graphite has intercalation space that serves as the host for ions of electrolyte to accommodate charging – discharging process[10]. It has disadvantage of having a low surface area so that the intercalation value becomes low [11]. Various attempts have been made to increase the energy capacity of LIB by replacing its crystallography and the surface of the materials [12]. Carbon with large surface area is required for large amounts of lithium ions contributing high energy density storage. In this research, the carbon electrodes prepared from the carbon of kerosene soot. The soot was prepared by thermal plasma pyrolysis.

Intercalation of lithium ion to the graphite matrices is one factor for the mass and charge transfer during charging-discharging processes. It is expected that the carbon in lithium ion battery[3] has a large intercalation value. The intercalation value can be represented by the results of the measurement of pH_{pzc} values. FTIR measurement was applied for carbon functional groups analysis. The actual electrochemical involved in the electrode were revealed by using cyclic voltammetry technique. The performance of LIBs were evaluated by using galvanostatic technique.

II. METHODOLOGY

A. Carbon Preparation

Carbon preparation was conducted by using flame pyrolysis. The method was previously used by other authors [6]. Kerosene was put into bunsen and then burned through the cotton wick to produce a flame. The flame was managed to have good flame with dark smoke. The dark smoke was captured using a stainless steel plate. Soot is particles carried by smoke and attached to metal surfaces The soot was harvested using a brush or razor blade stuck after which the soot was taken.

B. Carbon Characterization

Crystallography [16] the carbon was characterized using XRD technique. X-ray diffraction (XRD) patterns of the carbons were obtained on a Shimadzu X-ray diffractometer XRD 7000 operating at 40 kV and 30 mA, using Cu-K α radiation.

Qualitatively, the existence of functional groups on activated carbon is known from the IR spectrum resulting from FTIR measurements. Whereas quantitatively, the functional groups are calculated by the Boehm titration method [13]. All acidic sites in carbon were neutralized with 0.1N NaOH solution while the base sites were neutralized with 0.1N HCl solution. Specifically at acidic sites, the reaction between reagents and oxygenated functional groups is based on the difference between the strength of acids and bases. Acid sites in activated carbon can be divided into carboxylates, lactonates and phenolics. The strength of the acid is arranged according to carboxylate series> lactonate> phenolics. The carboxylate site is the result of titration with 0.1N NaHCO₃ solution. While the phenolate site is the difference between the calculated Na₂CO₃ and NaOH results and the lactonate site is the difference in the results of the titration results with NaHCO₃ and Na₂CO₃.

0.5 gram of carbon was put into the beaker, that contained 0.1 N solution of NaOH, Na₂CO₃, NaHCO₃ and HCl. The mixture was stirred for 24 hours on a vortex mixer. After 1 day the mixture was allowed to stand for 30 minutes. The mixture was filtered with filter paper and as much as 10 mL of the filtrate was calibrated and titrated with the appropriate titrant. For base analysis, NaOH titrant for filtrate containing HCl. HCl titrants applied for the filtrates containing Na₂CO₃, NaHCO₃ and NaOH for acid analysis

Procedure for pH of point zero charge can be found in elsewhere [14]. A total of 50 mL of 0.01 M NaNO₃ solutions were put into 10 erlenmeyers. The solution was adjusted to the initial pH from 2, 4, 5, 6, 7, 8, 9, 10, 11, 12 by adding 0.1 M HCl or 0.1 M NaOH. 0.2 gram of carbon of kerosene soot were added into the solutions for each erlenmeyers. The mixture was shake for 2 hours using a shaker and were allowed to run the process for 2 days. The initial and final pHs were measured using pH meter. The pH value for pzc was determined based on the point of intersection between the curves.

C. Preparation of Lithium Ion Battery

The LiFePO₄ cathode was synthesized using the hydrothermal method. The synthesis process with this method used a mixture of LiCl, FeSO₄·7H₂O, and H₃PO₄ solution as a base material, with a mole ratio of Li: Fe: P is 1: 1: 1. The basic mixture was added ascorbic acid, to keep Fe from being oxidized to iron (III). The synthesis process begins with stirring or mixing basic ingredients, LiCl, FeSO₄·7H₂O, and H₃PO₄ solution. The mixture was placed in crucible and heated in an oven at 200 °C for 12 hours to form LiFePO₄ powder. The powder was washed by using distilled water and dried in an oven at 110 °C for 12 hours. LiFePO₄ is the active material for cathode. Mixture of LiFePO₄, carbon and PANi with mass ratio 70: 25: 5 was prepared and dissolved in DMSO to make slurry. Preparation polyaniline from aniline is explained by other author [15]. The slurry was coated on the surface of copper plate 2 x 3 cm² by using roller coating technique and heated in 60 °C to form cathode sheet.

The anode was prepared as follows. The carbon, PANi and DMSO were mixed to form slurry and coated on top of Aluminum plate and to form anode sheet. Both sheets were stacked on top separator cm². Cathode and anode were arranged in opposite direction and faced each other with separator in the middle and a few dropped of Li₂SO₄ 0.1, 0.5 and 1M solutions as electrolyte. All works were done in Ar filled glove box to develop LIB.

Cyclic voltammetry (CV) test was performed at a voltage range of 2.7 – 4.5V with a scan rate of 160 mV/s while the charge-discharge (CD) was performed at a voltage of 3.0 – 4.6V. Both tests were conducted in Autolab potentiostat.

III. RESULTS AND DISCUSSIONS

There is a pyrolysis process which occurs the decomposition of petroleum components, namely hydrocarbons and sulfur which are the main components of kerosene. This decomposition process takes place at temperatures around 800°C, which is marked by the release of black-colored smoke [6]. In this process carbon is produced in the form of soot.

A. Preparation of Carbon and Characterization

Boehm titration consists of two analyzes, namely acid group analysis and base group analysis. Based on Boehm titration calculations, the data is as shown in table 1.

TABLE 1. FUNCTIONAL GROUPS OF THE CARBON OF KEROSENE SOOT

Functional Groups	Concentration (mgeq)
Carboxylates	3.87
Lactonates	0.83
Phenolics	2.20
Bases	6.76

Table 1 shows the functional groups of the carbon, i.e., carboxylates, lactonates, phenolics which are acidic functional groups and bases functional groups. The acid functional groups are larger than bases functional groups. The functional group of the acids and bases in the carbon are 6.90 mgeq and 6.76 mgeq.

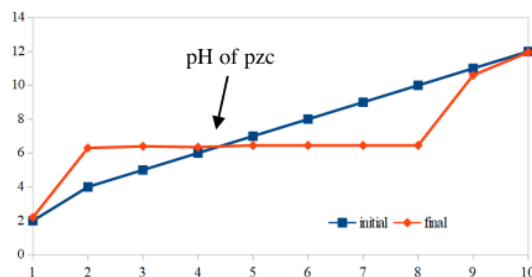


Fig. 1. Initial and final pH curves and the pzc of the carbon

pH of point zero charge (pH_{pzc}) is interpreted as a condition when the surface is neutral. This data can be used to estimate the affinity of carbon adsorption for a substance. The determination of pH_{pzc} is done by flowing the graph between the initial pH and the final pH of the solution. Based on Figure 1, the pH_{pzc} value of the carbon is 4.80.

In the alkaline surface the carbon surface will form R^-OH^{2-} whereas in the acidic state the carbon surface has an R^+O^- affinity. In basic conditions, carbon surface more easily absorbs anions. Inversely, in acidic surfaces, carbon surface more easily absorb cations [16]. Carbon particles have pH_{pzc} in acidic conditions so that the surface charge of carbon nanoparticles is easier to absorb cations. This shows that the carbon are best used as lithium ion battery electrodes because they function as a host or matrix for the entry and exit of Li^+ ions.

XRD is a technique commonly used to determine the crystallographic characterization of a material through peaks of intensity that arise. In this characterization, $2\theta = 0$ to 80° were used to identified the crystal on both electrodes, i e., anode and cathode.

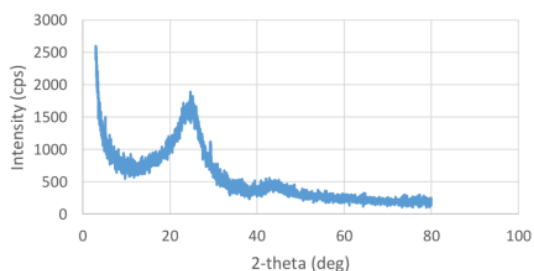


Fig. 2. Diffractogram of kerosene soot carbon as anode

The results (Fig. 2) shows that the carbon on anode has small crystallite sizes indicated by two broad peaks at $2\theta = 23.68$ and 42.33° . This result is confirmed by standard measurement of crystalline graphite in JCPDS database entry 00-026-1077 [7].

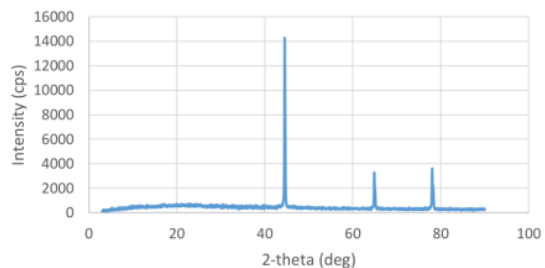


Fig. 3. Diffractogram of kerosene soot carbon

Diffractogram of cathode (Figure 3) shows the peaks at $2\theta = 44.51^\circ$, 64.90° , and 78.06° . Standar diffraction $LiFePO_4$ is provided by JCPDS, entry number 00-040-1499. The peaks can be interpreted that the crystal has orthorhombic unit cell.

B. Electrochemical Test

The electrochemical character of the electrodes were determined by using cyclic voltammetry (CV) method on a potentiostat. CV measurements were carried out with a scan rate of 5 mVs^{-1} with electrical potential intervals of -2.5 to 2.5 Volts using variations of Li_2SO_4 electrolytes concentrations, i e., 0.1 M, 0.5 M, and 1 M (Fig. 4).

This test was carried out to determine the existence of reduction oxidation (Faradaic processes) on LIB. It can be shown that the expected reaction is observed (Fig. 5(a)). The reactions was found in low rate of voltage scan, i e., 5 mVs^{-1} [17] with 0.1 M of electrolyte concentration. It can understand that the reactions (reduction and oxidation) take place when the anode and cathode on a LIB has a potential difference. Acurate detection can be provided by apping low scan rate.

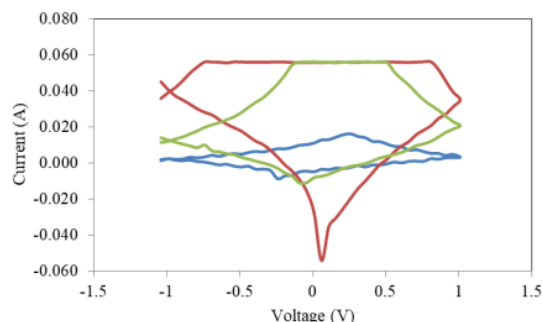


Fig. 4. Voltammograms of LIB in various concentration of electrolyte Li_2SO_4 i e., (a) 0.1 M (b) 0.5 M and (c) 1 M with scan rate 0.05 V/s

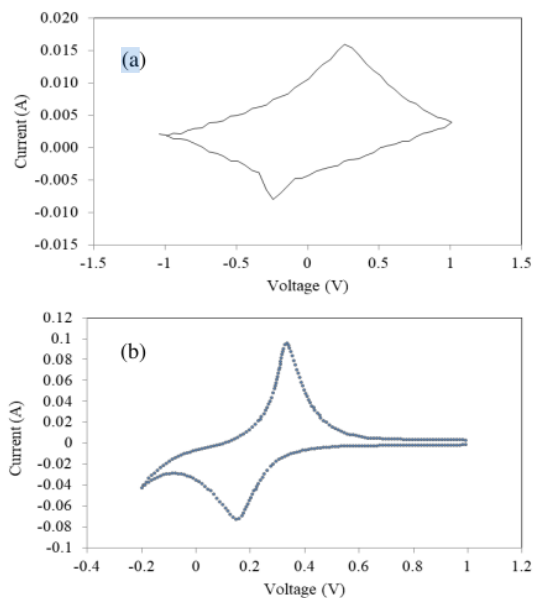


Fig. 5. Comparison of $LiFePO_4$ of LIB voltammograms from (a) this study and (b) other author[18].

The cyclic voltammetry measurements of LIB performance with the cathode of $LiFePO_4$ has been done by other author [18] as in Fig. 5(b). It can be seen that the oxidation reaction occurred at the peak of anode current, i e., 0.015 A for 0.3 V of applied potential whereas the reduction reaction occurred at the -0.3 V of potential applied when current reached peak at 0.007 A .

C. Stability of Charging – Discharging

The charge - discharge cycle is an important parameter in determining practical applications of battery. It reveals its lifetime and durability. The cycles measurements are performed at the beginning and several subsequent cycles. Galvanograms (Fig. 6) show the transition times of the cycles work maximum at the beginning and have tendency to decay after be used for a certain time.

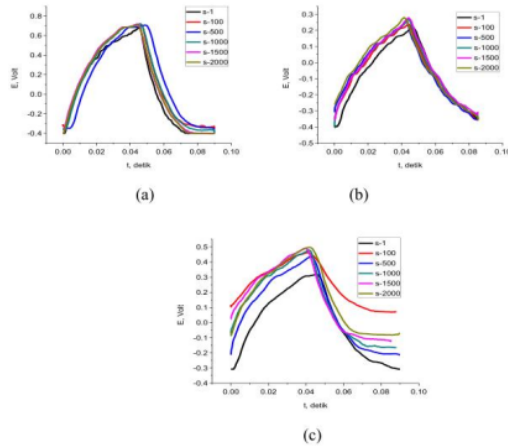


Fig. 6. Galvanogram of charging discharging in LIB with electrolyte Li₂SO₄ vary (a) 0.1, (b) 0.5 and (c) 1 M.

Fig. 6(a) shows that the batteries experienced a change in which the minimal voltage rise reached the maximum voltage occurred in the 500th cycle and then dropped again in the 1500th cycle. Figure 6 (b) shows that the batteries experienced a change in which the minimum voltage rose reached the maximum voltage occurred from the 1st cycle to the 2000th cycle, whereas Figure 6(c) shows that the batteries the change in which the minimum voltage rises in the 100th cycle then drops in the 500th cycle, then the voltage rises again in the 1000th cycle until it reaches the maximum voltage in the 1st cycle. Figure 6 (a) show that the LIB with 0.1 M of electrolyte concentration has the highest voltage, i.e., 0.71 volts occurred in the 500th cycle, while the lowest voltage is shown in Figure (b) with 0.5 M of electrolyte concentration, i.e., 0.2 volts occurred in the 2000th cycle.

The transitions at the beginning of the cycle 6 days occur when LIB at the first used. The differences between the initial cycle and the subsequent cycle were due to the distribution and dislocation arrangements via electrolytes at the molecular scale [19]. This arrangement can be detected as a macro phenomenon and expand - shrinkage of the electrode matrix performed with dilatometer equipment [20]. The electrode expanded when charged and shrank when discharged.

D. Rate of Charging Discharging

Charging and discharging plots are employed to see the rate of the two process involved when LIB is under application. The measurement of charging and discharging rates was done in the 1000th cycle, where the process is stable.

The slope values in Tables 1 to 3 show the results of the galvanostatic test conducted on LIB. Negative value indicates of the decline direction which means that the battery discharges.

TABLE I. SLOPE VALUES OF THE CHARGING AND DISCHARGING CURVE OF THE BATTERY WITH 0.1M OF Li₂SO₄.

Cycle	Slope of Charging	Slope of Discharging
1 st	20.981	-34.930
100 th	22.183	-39.363
500 th	23.626	-41.159
1000 th	22.964	-35.963
1500 th	22.572	-34.495
2000 th	23.003	-36.711

TABLE II. SLOPE VALUES OF THE CHARGING AND DISCHARGING CURVE OF THE BATTERY WITH 0.5M OF Li₂SO₄.

Cycle	Slope of Charging	Slope of Discharging
1 st	13.086	-14.117
100 th	12.204	-13.572
500 th	11.966	-14.768
1000 th	12.453	-14.217
1500 th	12.598	-14.184
2000 th	11.95	-13.729

TABLE III. SLOPE VALUES OF THE CHARGING AND DISCHARGING CURVE OF THE BATTERY WITH 1M OF Li₂SO₄.

Cycle	Slope of Charging	Slope of Discharging
1st	13.269	-12.763
100th	7.9898	-7.6283
500th	12.037	-11.831
1000th	11.163	-13.898
1500th	10.31	-12.281
2000th	11.665	-17.72

It can be shown that charging slope (12,453) is smaller than the value of the discharging slope (-14,217).

These results show that there is a complex (non-linear) relationship to the electrolyte-electrode interactions that contribute to the capacity and working voltage of LIB. The best discharge rate is obtained in electrolytes with a concentration of 0.5 M, this is because at a concentration of 0.5 M shows the characteristics of LIB where the charging rate is smaller than the rate of discharge and the distance between the charging and discharging slope values is not too large and stable.

E. The Capacity of LIB

Battery capacity is a measure of the charge stored by a battery and determined by the mass of the active ingredient contained in the battery. The unit for battery capacity is

mAh/g. Unit mAh/g (milliampere hour per gram) is combination of mA (milliampere) and hour (hour) divided by the mass of the electroactive active material in the battery. Fig. 7 (a), (b), and (c) represent the capacity LIB with varying electrolyte concentrations, i.e., 0.1 M, 0.5 M, and 1 M.

During the charging process, an oxidation reaction occurs where the cathode releases Li^+ ions and electrons to the anode, while in the emptying process, a reduction reaction occurs, where Li^+ ions and electrons reenter the cathode. The highest capacity is obtained in Fig. 7 (a) of 0.52 mAh/g with a voltage of 0.71 Volts, which is the 500th cycle. The greater the voltage applied, the greater the capacity of the battery. While the lowest capacity is obtained in Fig. 7 (b) which is only 0.15 mAh/g with a given voltage of 0.2 Volts, that is in cycle 1.

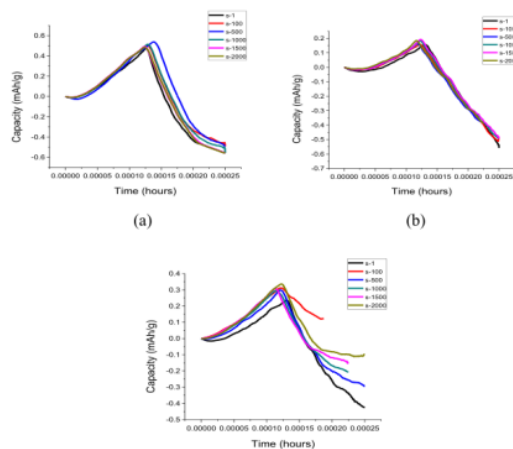


Fig. 7. Battery capacity with (a) 0.1 M (b) 0.5 and (c) 1 M of Li_2SO_4 of electrolyte concentrations.

The effect of electrolyte concentration on the capacity of the batteries can be seen in Fig. 7. In Fig. 7 (a) and (b) show that the capacity of the battery is more stable than in Fig. 7 (c) which shows the capacity of the battery is unstable. This is due to the high concentration of ionic mobility being disrupted due to the limited space of Li^+ ions to exit and enter the matrix whereas at low electrolyte concentrations the Li^+ ions have enough space for ions to move more easily in and out of the matrix. In addition, the surface area area greatly affects the capacity of the lithium ion battery. A larger surface area increase contact electrode – electrolytes contact so that more lithium ions enter and exit the cathode, thereby increasing the capacity of lithium ion batteries [21].

The results showed that the capacity of the battery using kerosene soot carbon in its electrode is lower than the capacity of the battery using diesel soot carbon electrodes[22]. Carbon from diesel oil soot has a longer carbon chain compared to carbon derived from kerosene soot. The surface area formed by carbon chain structure of precursors affect the energy storage capacity of the battery. The longer carbon chain is the larger in capacity.

IV. CONCLUSIONS

Carbon of kerosene soot is qualify to be used as electrodes on lithium ion batteries. The results of boehm titration and pHpzc show that carbon nanoparticles are acidic, so that the surface charge of carbon nanoparticles is easier to absorb cations. The carbon act as the matrices for Li^+ ions transfer. The XRD diffractogram shows that there are two peaks at $2\theta = 23.68$ and 42.33 which indicate the presence of graphite crystal in carbon particles. When testing a LIB using the CV method, the expected reaction is not formed. This is because the device used is not able to read the voltage applied to the lithium ion battery electrodes because the reaction is slow, so a smaller scan rate is needed. The largest value of LIB capacity with all variations is 0.52 mAh / g with a voltage of 0.71 volts. For the largest energy density value of LIB is 3.69×10^{-4} Wh/g LIB performance testing analyzed by the galvanostatic method shows that concentration and cycle are the most influential factors on the value of the capacity and energy density of lithium ion batteries.

It is suggesting to select other cathodes in order that needs to increase the capacity of energy storage of lithium ion batteries. It is also necessary to utilize of carbon soot electrodes other energy storage devices, such as supercapacitor and fuelcell.

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