Fabrication Of Lithium-Carbon Composite Material From Pepper Peel Waste As Battery Electrodes

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Abstract. The manufacture of battery cathode electrodes made of LiFeO4 and activated carbon from pepper peel has been successfully carried out. The electrodes were made using the solid state reaction method with the addition of adhesives in the form of PVdf and NMP. The resulting homogeneous mixture is then placed in a mold and dried in an oven at 90° to dry to form sheets. The dried electrode sheets were then characterized using FTIR and tested for conductivity values using EIS. The FTIR results showed that there was no change in the functional group with the addition of carbon mass. Meanwhile, the results of the conductivity test showed that the optimum conductivity value occurred in the sample with the addition of 10% carbon mass is 7.42 × 10⁻⁴ S/cm.

1. Introduction

Batteries are divided into two types, namely primary batteries which can only be used once and secondary batteries which are rechargeable so as to reduce the negative impact of primary batteries in the form of saving power and reducing waste pollution [1]. The secondary battery that is currently of interest to researchers is the lithium ion battery [2], [3], [4]. This is because lithium ion batteries have a long lifecycle, are stable and environmentally friendly [5], [6], [7]. In addition, lithium ion batteries also have a higher capacity when compared to other secondary batteries, such as nickel cadmium (Ni-Cd) and nickel metal hydride (Ni-Mh) [8], [9], [10].

One of the most important components in a battery is the electrode, both anode and cathode. The synthesis of battery electrodes based on activated carbon is a special attraction due to its abundance, large surface area and of course low cost. [11] have studied the electrical properties of the lithium battery electrodes based on candlenut shells by varying the concentration of LiOH and obtained a conductivity value of 2.34×10⁻⁶ S/cm. [12] synthesized a carbon-TiO₂ nanocomposite to obtain a conductivity value of 1.11×10⁻⁷ S/cm at a concentration of C-TiO₂ (10% : 90%). According to [13] the conductivity value of battery electrodes is strongly influenced by pH, temperature and time of synthesis. The highest value of LiFePO₄/C electrical conductivity was obtained in samples with a pH of 5.8 with a sintering temperature of 700°C for 6 hours, namely 1.842×10⁻⁵ S/cm. In addition to the parameters of pH, temperature and time, [14] also stated that the biomass-based LiFePO₄ carbon coating showed a high discharge capacity of 147.3 mAh g⁻¹ and tended to be more stable.
Based on the description of the problems that have been raised, researchers are interested in studying the synthesis of lithium pepper-ion peel activated carbon composite material as a candidate battery electrode material. This research is a continuation of previous studies that have synthesized solid battery electrolytes [15] and also synthesized activated carbon from pepper peel waste as supercapacitor electrodes [16].

2. Materials and methods

2.1. Material and equipment

The tools used in this study were SEM-EDS, BET (JWGB Meso 112), Inert Furnace with nitrogen gas (N₂) and Electrochemical Impedance Spectroscopy (EIS) test equipment, mesh, agate mortar and magnetic stirrer. The materials used are waste of pepper peel, distilled water, and KOH.

2.2. Pepper Peel Activated Carbon Production

The manufacture of activated charcoal from pepper peel was carried out according to previous research methods [17]. The initial stage is to wash the pepper peel waste to remove impurities. The washed waste is then dried in the sun to dry. The dried pepper peel waste was then pre-carbonized using an oven at 130 for 2 hours. After that, the grinding and sifting was carried out using a 100 mesh sieve. The next step is chemical activation using KOH with a ratio of 5 : 1 for the volume of the activator compared to the mass of the sample. To homogenize the mixture, the mixture was stirred using a magnetic stirrer and then allowed to stand for 48 hours. After immersion, the samples were washed to neutral pH (pH - 7) using distilled water. The last stage is that the sample is physically activated at a temperature of 700°C using an inert furnace at a rate of 100°C/hour under N₂ gas flow conditions with a holding of 3 hours. After that, the temperature is lowered naturally.

2.3. Battery Cathode Electrode Manufacturing

The cathode electrode was prepared using the solid state method by mixing LiFePO₄ (LPF), polyvinylidene fluoride (PVDF), N-methyl pyrrolidone (NMP) with a total mass of 1 gram of all mixtures, to obtain a slurry mixture with the composition as shown in Table 1. After that, it was dried at temperature of 90°C using the oven to dry to form a sheet and continued testing.

| Sample Code | Carbon (%) | LPF (%) | PVdf (%) | NMP |
|-------------|------------|---------|----------|-----|
| A           | 0%         | 80%     | 20%      |     |
| B           | 5%         | 75%     | 20%      | 5 ml|
| C           | 10%        | 70%     | 20%      |     |

2.4. Cathode Electrode Characterization

The characterization of battery cathode electrodes that have been made using the solid state method was carried out using FTIR to determine the functional group and Electrochemical Impedance Spectroscopy (EIS) to determine the conductivity value produced.

2.5. Data Analysis

The conductivity measurements were carried out using EIS (Electrochemical Impedance Spectroscopy) and then the measurement results were processed using Zview 2 through the Simplified Randles Cell approach to obtain the magnitude of the charge transfer resistance or polarization resistance (Rct/Rp) [18]. This Rct value is used to determine the magnitude of the conductivity value of the synthesized electrode through the relationship:
\[ \sigma = \frac{t}{AR_{ct}} \]  

(1)

where: \( \sigma \) is conductivity (S/cm), \( t \) is sample thickness (m), \( A \) is surface area (m\(^2\)) and \( R_{ct} \) is charge transfer resistance (\( \Omega \)).

3. Result and Discussion

3.1 Results of Preparation of Pepper Peel Waste Activated Carbon

Pepper peel waste that has been activated chemically and physically was characterized using Scanning Electron Microscopy - Energy Dispersive X-ray (SEM-EDX). The results of the SEM test at 5000× magnification in Figure 1 show that the activated carbon sample has a porous morphology. These pores will be the path of charge or ion transfer on activated carbon-based electrodes. In addition to being porous, the sample's dominant element content consists of carbon elements of 82.47 At% and other elements as shown in Table 2.

Figure 1. SEM Image Results at 5000× magnification.

Table 2. Active Carbon Sample Element Content

| Element | C     | O     | Mg    | Al    | Si    | Ca    |
|---------|-------|-------|-------|-------|-------|-------|
| At%     | 82.47 | 13.89 | 00.94 | 00.65 | 00.59 | 01.47 |

3.2 FTIR Test Result

Based on the FTIR analysis, it shows that the functional groups present on the electrodes made include: -OH/NH amide which is indicated by a wave number of 3224 cm\(^{-1}\), C=O at 1600 cm\(^{-1}\), P-O at a wave number of 1027 cm\(^{-1}\), C-F at a wave number of 1027 cm\(^{-1}\), wave number 1204 cm\(^{-1}\), and O-P-O at wave number 627 cm\(^{-1}\). The -OH/NH amide functional group can come from activated carbon or from NMP solvent. Likewise for the C=O group, this comes from the amide in NMP. In this case, the residual NMP solvent was still present in the all electrode samples. While the PVdF polymer on the electrodes made was shown to have a C-F group reinforced with a C-H group at a wave number of ± 2900 cm\(^{-1}\). The electrode composite containing LPF is also supported by the presence of phosphate groups such as P-O and O-P-O.
Figure 2. The FTIR analysis of the electrodes

The difference between the three samples tested is indicated by the addition of the percentage of carbon composition from A to C. This results in an increase in intensity at a wave number of 2155 cm\(^{-1}\). In sample C the higher the intensity at wave number 2155 cm\(^{-1}\). This wave number is a characteristic of the presence of silica (Si-O functional group).

3.3 Conductivity Test Result

The conductivity test result as shown in Figure 3.

Based on Figure 3, the size of the semi-circle curve shows the resistance of the cathode sample [19]. The larger the diameter of the circle, the greater the resistance value and the smaller the conductivity value and vice versa [20].

The magnitude of the charge transfer resistance (Rct) and the conductivity value of the cathode sample based on the results of the Nyquist plot graphic pattern processed using the Z View-2 software are shown in Table 3.
Table 3. Conductivity Data using the Randless Cell Model

| Sampel Code | Thickness (cm) | A (cm²) | Rct (Ω) | Conductivity (S/cm) | χ² |
|-------------|----------------|---------|---------|---------------------|----|
| A           | 0.0370         | 0.7084  | 4268    | 1.22 × 10⁻⁵         | 0.138 |
| B           | 0.0376         | 0.7084  | 705     | 1.05 × 10⁻⁴         | 0.014 |
| C           | 0.0477         | 0.7084  | 90.71   | 7.42 × 10⁻⁴         | 0.002 |

Based on Table 3, it shows that the conductivity value increases as the resistance Rct decreases and is also proportional to the increase in carbon mass. The addition of carbon mass can increase the conductivity value. This is because the addition of activated carbon mass causes electron flow paths formed by the presence of pores in the activated carbon which will result in an increase in the conductivity value.

4. Conclusion
Based on the results of the study, it was found that the optimum conductivity value occurred in samples that experienced an increase in carbon mass of 10%. The resulting conductivity value has met the requirements as a battery cathode electrode material.

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