Porosity Characteristics and Electrochemical Performance of rGO Coconut Shell as Supercapacitor Electrodes

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Abstract. Supercapacitor is a device which is currently being researched. Research on supercapacitors can be done from various aspects, including the supercapacitor electrodes, electronic materials or dielectric materials as well as the electrochemical performance of supercapacitors. In the research that has been done, has been obtained rGO material that has a specific capacitance value of 485.97 F / gram with calcination temperature of 1000°C and an ultrasonication time of 2.5 hours. The reduction of Graphene Oxide has fulfilled the standards as a supercapacitor. Another characteristic that must be fulfilled as a supercapacitor is porosity, to determine the ability of adsorption and desorption, as well as electrochemical performance. The results of BET characterization for rGO produced from coconut shells that the largest distribution in the adsorption process was 0.00192 cc/nm/g at a pore diameter of 35.68 nm, while the largest desorption distribution was at 0.00211 cc/nm/g at pore diameter 35.63 nm. So that it can be categorized that rGO is a mesoporous category with a pore diameter of 35.63 - 35.68 nm. Cyclic Voltammetry characterization results obtained that rGO has a reversible curve and is very wide in rGO with 2.5 hours ultrasonication.

1. Introduction

The development of material developed very rapidly in this era. Advanced materials can be fabricated from natural materials that are available or from materials that are waste, or from basic materials of industrial production that are reprocessed to have better applications. One of the abundant natural resources in Indonesia is coconut plants. Apart from being able to use coconut plants in the fruit, stem, roots, leaves, other parts that have been considered as waste, namely the coconut shell can also be used. From the test of carbon content, the coconut shell fixed carbon value was 74.62% [1]. The high carbon content can be used as a basic material for making graphene with a fairly good level of application, as an absorber, water purifier and supercapacitor. Graphene characteristics that support potential applications include large surface area so that it can absorb more electrons, and result in high capacitance values [2]. But graphene is a material whose synthesis process is very difficult, but can be made into another carbon allotrope with a simpler synthesis process, namely reduced graphene oxide (rGO). The characteristics of rGO are not much different when compared to graphene. Based on research conducted by Nurdiansah and Susanti (2013) successfully synthesizing coconut shell carbon was applied as a supercapacitor using the carbonation method at a temperature of 800°C and holding time of 2 hours obtained the specific capacitance value of the coconut shell was 844 mF / gr and the
The surface area of the coconut shell was 548.542 m$^2$/gr. Whereas from previous studies [3], rGO has been synthesized from coconut shell material, with variations in calcination temperature. The research of Nugraheni, et al. (2015), also succeeded in synthesizing rGO from the exemplary shell in ultrasonication with a time variation of 1-2.5 hours and obtained the largest capacitation value of 485.97 farad/gram, at a time variation of 2.5 hours.

2. Materials and Method
Coconut shell as the basic ingredient of making RGO is first cleaned from impurities and fibers attached to the old coconut shell. The synthesis of rGO uses the calcination method, by adopting the calcination process from the research of Prasetya (2014) [4]. Coconut shell that has been cleaned is cut into sizes 1-2 cm$^2$ using mortar and pestle. Coconut shell that has been cut to a smaller size, is dried at a temperature of 100°C with the aim of removing the moisture content of the coconut shell. The process is continued by pounding the dried coconut shell, until it becomes powder and passes the 100 mesh filter. The carbon powder is heated at a temperature of 1000°C for two hours. The powder which has been heated is filtered again until it passes the 200 mesh filter. The results of the synthesis of the powder were ready to be characterized and cultivated for 2.5 hours [5].

Characterization carried out in this research is BET to determine porosity and surface area of rGO that influence the identification of the ability of rGO in binding and storing loads and CV to determine the performance and electrochemical stability of rGO which has been 2.5 hours cultivated with capacitance 485.97 F/gram.

3. Results and Discussion
The synthesis of rGO from a coconut shell begins with the process of washing the coconut shell from impurities, cut into smaller sizes, followed by the drying process using a furnace with a temperature of 110°C for 1 hour, to remove the water content from the coconut shell. Coconut shell that has been dried, crushed and filtered to pass the filter. The powder formed was calcined at a temperature of 1000°C in 2.5 hours. The calcined powder was crushed and filtered so that it passed the filter with a size of 200 mesh, then the resulting powder was characterized. Preliminary test results in previous studies through Raman spectroscopy obtained that the resulting powder was rGO with an $I_D/I_G$ ratio of 2.21 [6].

BET characterization on rGO powder was carried out to determine the surface area and pore size of the material. This is because one of the criteria for supercapacitor electrode material has a pore size between 2-50 nm, which is categorized as mesoporous [2]. Mesoporous nature is needed in the process of charge intercalation in the electrode so that the charge can be well absorbed in the supercapacitor electrode [7]. The results of BET characterization are obtained as a curve as shown figure 1.

BET characterization consists of two parts, desorption (red curve) and adsorption (blue curve). Figure 1.a shows the pore size with respect to the change in potential, where by the BJH (Barrett Joyner Halenda) method, the largest pore size distribution is obtained in desorption and absorption conditions. The results of the analysis by the BJH method showed that the desorption process obtained the largest distribution at 0.0021 cc/nm/g with a pore diameter of 35.63 nm, while the largest distribution at the time of adsorption was 0.0019 cc/nm/g at the pore diameter 35.68 nm. So that the pore diameter of rGO is around 35.63 - 35.68 nm, which is in the pore size range for mesoporous material (2 nm-50 nm), so that rGO material meets one of the requirements as a supercapacitor material. Besides pore diameter, BET characterization can also see the surface area of the material. In Figure 1.b, it is known that the surface area of rGO is 263.2 m$^2$/g. The resulting curve in the BET characterization is in the form of a slit-shaped pore [8], meaning that the rGO formed has a gap that functions to bind cations and anions, so that the capacitance is large.
Cyclic Voltammetry (CV) characterization was performed to determine the electrochemical performance of rGO to be used as a supercapacitor electrode. Preparation for CV characterization is to form material into electrodes by the compacting process using PEG adhesive. The electrodes are formed into solids with a diameter of approximately 1 cm. The electrodes were characterized by cyclic voltammetry at voltages of -0.7 to 0.8 volts, with a scan rate of 5 mV/s [9][10]. The results obtained are shown in Figure 2. In Figure 2 it can be seen that the CV curve is a reversible hysteresis curve. The area under the curve is wide enough, with a capacitance value of 485.97 F/gr.

**Figure 1.** Pore size analysis using the BJH method (a) and rGO electrode isotherm curve (b)
The capacitance of the rGO meets the supercapacitor material standards, but it is necessary to test the stability of the electrochemical performance of the rGO. So that the electrochemical performance testing is continued until the next 10 cycles, and the stability of the performance is observed by observing the hysteresis curve of the CV characterization and the capacitance calculation. CV test results for 10 cycles can be seen in Figure 2.b. In Figure 2.b it can be seen that the hysteresis curve from rGO up to the 10-th cycle is still reversible, so it can be concluded that the rGO is still stable until the 10-th cycle. Identification of the stability of electrochemical performance can be observed from the capacitance value which does not change significantly. The capacitance value for 10 cycles can be seen in Figure 3. Where the capacitance value is in the range of 485.97 - 482.72 F/gr.
Capacitance value does not change significantly but still decreases, this is because if it has been used several times the stored charges will decrease if carried out continuously in several cycles. Capacitance is still good, characterized by a reduction in capacitance of no more than 4% after 10 cycles of electrochemical performance testing.

The charging-discharging characteristics of the rGO electrode also show good performance, can be seen in Figure 4. The charging process on the "a" line which is the process of absorbing ions on the electrode surface, which takes 173 seconds. The "b" line shows the discharging process, in which the process of releasing ions on the electrodes takes the same time as the charging process. So it can be concluded that the electrochemical performance of rGO as a supercapacitor electrode is very good in terms of its charge-discharge process [11].

4. Conclusion
RGO material as a supercapacitor meets several criteria including pore size, surface area and electrochemical performance. RGO pore size with 2.5 hours ultrasonication is included in the mesoporous that meets the supercapacitor criteria, while its chemical performance can be seen from the reversible capacitance and hysteresis curve stability up to 10 cycles, as well as a stable charge-discharge process.

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