

Comparison Mass Composition of Coconut Shell in TiO₂ towards its Characterization for Supercapacitor Applications

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Abstract

This study aims to evaluate the effect of the mass ratio on the capacitance of electrodes made from TiO₂-based composites. The composites were synthesized using a wet chemical method and applied using the doctor blade technique, incorporating TiO₂ and rGO derived from coconut shell activated carbon. Our findings reveal that at a 1:1 mass ratio, the atomic composition of Ti and O was non-uniform, although there was evident adherence of TiO₂ elements to the sample surface. In contrast, at mass ratios of 1:3 and 1:5, a decrease in the concentration of Ti atoms and an increase in O atoms were observed, indicating a reduction in Ti oxidation. SEM analysis further revealed that particle size significantly impacts capacitance: smaller particle sizes yielded higher capacitance. In the 1:1 mass variation, the discharge process was protracted, taking up to 29.3 minutes and generating an electrical energy of 0.000413 joules with a capacitance of 489 μF. These insights are pivotal for optimizing the composition and mass ratio, fostering the development of electrode materials characterized by enhanced capacitance and energy efficiency. Such advancements hold promising potential for a range of applications in the energy storage sector.

Keywords: Activated carbon, Capacitance, Supercapacitor

1 Introduction

Supercapacitors are currently gaining attention as a future energy storage system due to their superior performance in terms of capacitance compared to conventional capacitors. However, supercapacitors have drawbacks, including lower energy density compared to batteries and unstable voltage output. Consequently, many researchers are investigating solutions to these issues for a wide range of applications, from small to large



scale [1]. The supercapacitor fabrication process is efficient, easy and environmentally friendly because it does not produce any pollution. Therefore, the use of supercapacitors is a step that can overcome global issues regarding warming and environmental damage [2]. In principle, capacitors have two types of capacitors based on their storage media ELDC capacitors are capacitors with a type of physical storage by accumulating charge between two electrodes which are separated by a thin electrical double layer and there is an interaction between positive and negative charges which causes the energy storage process to occur. Pseudocapacitors use transition metal oxides as electrode materials. Some transition metal oxides commonly used in pseudocapacitors include ZnO [3], TiO₂ [4], CuO [5], Mn₂O₃ [6], and metal oxides with three metal components [7]. Meanwhile, another type of capacitor is a pseudocapacitor which carries out energy storage by chemical means involving Faradaic redox where electrons are transferred to ions at an electrode. Basically, this type stores more energy than ELDC, but the rate of charging and discharging is too fast due to the influence of chemical properties [8]. Although pseudocapacitors have high capacitance, several material limitations make them not optimal for large-scale applications. Meanwhile, EDLC, which uses carbon materials, offers stability and abundance that makes it a more practical and economical choice for various applications [9]. Basically, TiO₂ material has stable chemical properties at low cost, is abundant in nature and can improve the electrochemical performance of the electrode material. This shows that TiO₂ is considered a potential candidate to increase the efficiency and effectiveness of supercapacitors [10]. Therefore, it is the focus of research in developing electrode materials for supercapacitors. Currently, a new design has been carried out on a composite of activated carbon, conductive carbon, and TiO₂. The material composition has been optimized to obtain electrode performance with high capacitance and longer service life [11]. Carbon material is generally the choice that is often used as an electrode in making supercapacitors because it is easy to find, can be renewed and has high performance in energy storage so that it can be a sustainable energy solution [12]. When compared with commercial carbon-based materials, activated carbon derived from biomass is cheaper, environmentally friendly, and rich in surface functional groups. Therefore, many biomasses have been sought for use as raw materials or precursors in the manufacture of activated carbon, such as durian peel [13], Cassava peel

[14], mangosten peel [15] and cocnut shell [12]. In the research carried out the analysis made was EDX [16]. EDX or Energy Dispersive X-ray Spectroscopy is a method used to evaluate the composition of chemical elements in a sample. This technique can be used to analyze the materials used in supercapacitors, which generally consist of materials such as activated carbon, electrolytes and electrodes.

2 Material and Methods

This research activity was carried out by mixing coconut shell activated carbon that had been synthesized initially with TiO_2 material. TiO_2 : Coconut Shell then put into a glass beaker and mix with 1-Butanol. Next, homogenize the ingredients using a magnetic stirrer for 3 hours and dry in the oven. The dried material is called the TiO_2/rGO composite [17]. The variations used by researchers in the mixing process are 1:1, 1:3 and 1:5. Next, add 50 ml of H_2SO_4 to the TiO_2/rGO composite while stirring for 1 hour, then slowly add 2 g of KmnO_4 and stir again for 1 hour. Then add 100 ml of distilled water. then add 5 ml of H_2O_2 to remove the potassium permanganate level and dry using an oven [18]. Before coating the aluminum plate, there are several things that must be done, namely mixing poly urethane in the TiO_2/rGO composite and then stirring until evenly distributed. Next, apply the material to the aluminum plate and leave it for 2 hours so that the material sticks well to the aluminum. The next step is to drip phosphoric acid on tissue paper which functions as a separator between the anode and cathode as a polarity bridge. Stick the tissue paper on the aluminum filled with graphene. The next stage is to insert the supercapacitor plate into vacuum plastic so that it does not become contaminated with air because this will interfere with the capacitance value [19]. This research is designed to make a supercapacitor material by applying TiO_2/rGO composite material to an aluminum plate as shown in the following figure. Fig. 1 is a supercapacitor design that has a cable attached to one end and is then ready to be connected to a power supply for charging and discharging measurements as shown in Fig. 2.



Figure 1. Composite of TiO₂: Coconut Shell that has been bonded to an Aluminum Plate

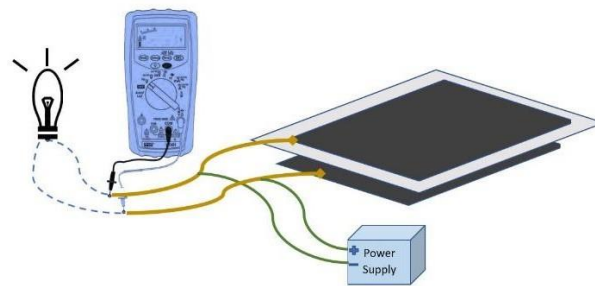


Figure 2. Approximate scheme of charging and discharging

3 Results and Discussions

1. Energy Dispersive X-Ray Analysis

The analysis carried out was TiO₂/Carbon with mass ratios of 1:1, 1:3 and 1:5 respectively. The morphology at a mass ratio of 1:1 can be seen in Fig. 3. Based on Fig. 3, it can be explained that the morphology of the 1:1 mass ratio is known to be not uniform, but the Ti element has adhered to the surface of the sample as indicated by the percentage of Ti atoms of 45.17% and the O atom content of 37.7%, while the P atoms are 1.12% and the K atoms are 2.03%. This compound was formed because during the sample making process using Phosphoric Acid and Potassium Permanganate. However, the content is very small, namely approx.

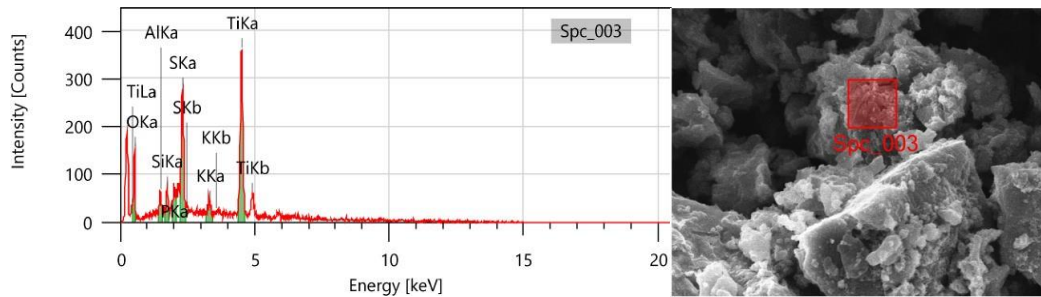


Figure 3. SEM-EDX Comparison 1:1 with magnification 2500x

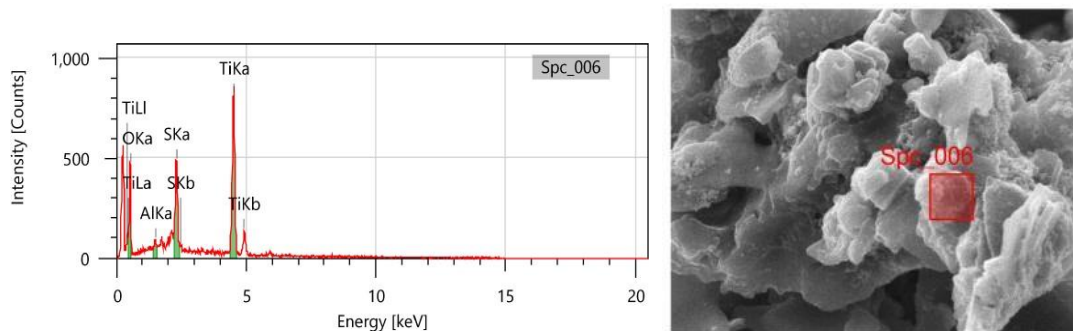


Figure 4. SEM-EDX Comparison 1:3 with magnification 2500x

In Fig. 4 it is known that the morphology looks uniform and the TiO_2 semiconductor is also attached, it is known that Ti atoms are 20% and O atoms are 67%. It can be seen that there is a decrease in the mass of the Ti atom and an increase in the mass of the O atom. In the graph the ratio 1:3 is found to be smoothly coated on the aluminum surface. This happens because contamination from equipment can occur when equipment used in experiments, such as containers, grinders, or other laboratory equipment, accidentally contaminates samples during the preparation or analysis process. In some cases, particles or residues of other metals thrown off or remaining on the equipment can enter the sample and then be detected in the EDX analysis. In this research, the author produced sample preparation results when heating aluminum foil. This can make it a contaminant at a mass ratio of 1:3. The 1:5 comparison of morphological analysis can be seen in Fig. 5.

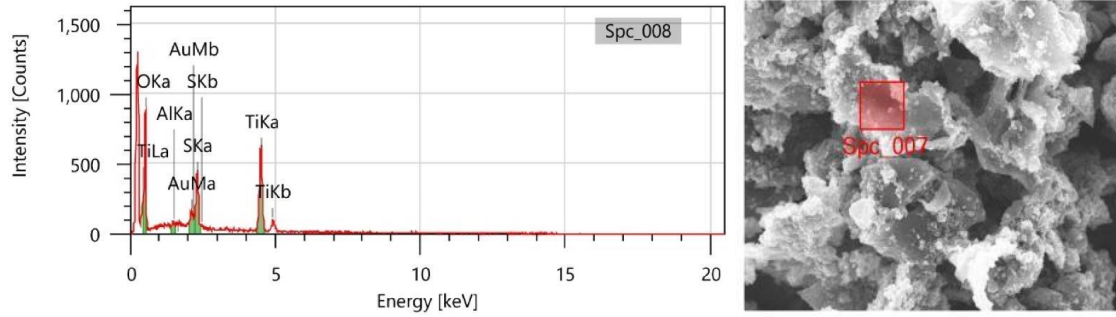


Figure 5. SEM-EDX Comparison 1:5 with magnification 2500x

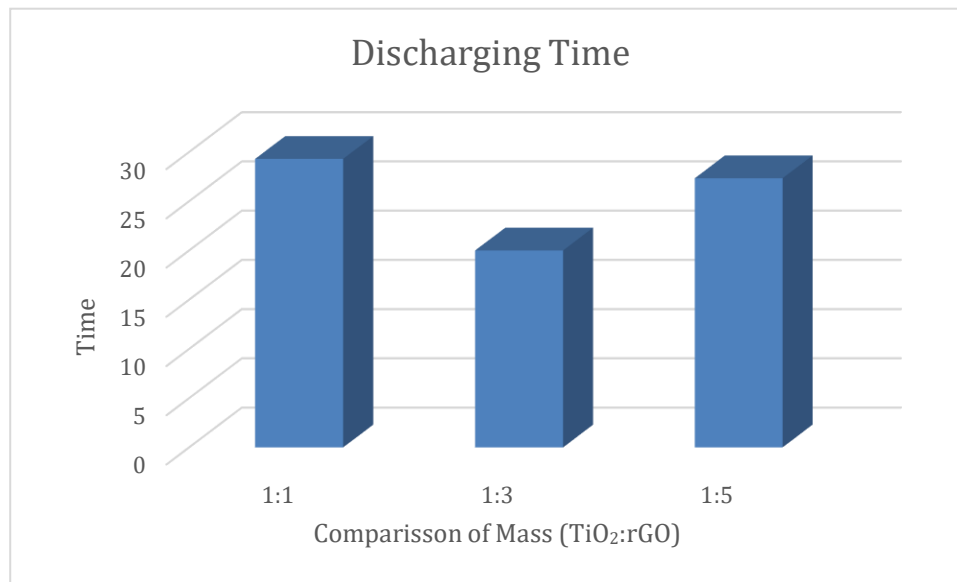


Figure 6. Diagram of Discharging Capacitors

In Fig. 5, the morphology formed based on the measurement results is uniform and semiconductive TiO₂ formed on the attached surface with the respective compositions of Ti and O being 27.01% and 48.28%. This percentage indicates that the Ti element also experiences a decrease in oxidation. This happens because the oxide value is higher compared to the 1:3 composition. The particle size obtained in this study was that at a mass ratio of 1:1 the size was 6.5 μm, while at a mass ratio of 1:3 it was 8.9 μm and the third ratio was 7.7 μm. From the results found, it is known that the smallest particle size is found in a ratio of 1:1. The difference in particle size in the material has an influence on capacitance, this happens because when particles come into contact with electrolyte ions, they can facilitate charge which of course has a role in charging and discharging supercapacitors [20]. An increase in particle size can also occur due to agglomeration,

causing an increase in intraparticle porosity. When smaller particles collect around larger particles, they create gaps or pores in the larger particles [21].

2. Charging and discharging analysis of supercapacitors

Based on Table 1, it can be seen that with a constant variable charging time of around 8 minutes, the emptying time varies. At a mass ratio of 1:1 the time required to empty is 29.3 minutes, while at a mass ratio of 1:3 the emptying is 20 minutes. There was a decrease in emptying time by around 9 minutes. This is because the mass of carbon produced is less than the mass of 1:1, apart from that, based on FTIR analysis, O-H bonds or carboxyl acids are formed which have an impact on supercapacitor applications. At a mass ratio of 1:5, the emptying time is 27.33 higher than 1:3. This happens because the composition of oxide and carbon is higher than 1:3 but lower than 1:1. Based on the Table 1, the mass ratio of 1:1 takes longer to empty compared to the others. This happens because more carbon elements are formed in this composition. The longest emptying time is at a mass ratio of 1:1, which is in line with the smallest particle size in that ratio. From the results of this research, it was found that particle size influences the discharge of supercapacitors.

3. Electrical Energy Analysis

In this research, electrical energy values were also calculated which can be seen in Table 2. The varying capacitance values are due to a decrease in specific capacitance and rapid mechanical degradation due to swelling, shrinkage and cracking during the doping/dedoping process. In this context, the doping/dedoping process refers to the entry and exit of ions or molecules into/from the electrode structure, which is part of the operational mechanism of energy storage and release [22]. Apart from that, the decrease

Table 1. Charging and discharging times

TiO ₂ : rGO	Charging (Minute)	Discharging (Minute)
1:1	8	29.3
1:3	8	20
1:5	8	27.33

Table 2. Electrical Energy

Mass Comparison	Kapasitance (μf)	V Saving (Volt)	Electrical Energy (Joule)
1:1	489	1.3	0.000413
1:3	58	1.1	0.000035
1:4	211	0.9	0.000085

also occurs because the morphological structure is not uniform, resulting in differences in surface area as a load storage. Based on Table 2, it can be seen that the greatest electrical energy is found at a mass ratio of 1:1, namely 0.000413 joules, and the lowest energy is at a mass ratio of 1:3, namely 0.000035 joules. The greater the capacitance value, the greater the electrical energy and vice versa, if the capacitance value is low, the electrical energy is also low. It was also reported that the activation method, type of activator, pyrolysis conditions provide different surface areas for the activated carbon produced, and this will have an influence on the capacitance values obtained [23]. Apart from that, differences in capacitance values can also occur due to inhomogeneous grains on a sample surface [24]. This is in accordance and store with $E = \frac{1}{2} CV^2$ where the E value is directly proportional to the capacitance and stored voltage obtained in each sample that has been made.

4 Conclusions

In research related to the addition of activated carbon mass in supercapacitor applications, it can be concluded that:

1. The more coconut shell mass mixed with activated carbon, the Ti value in the EDX results decreases. The highest Ti mass value is at a mass ratio of 1:1.
2. The fastest emptying time is in the ratio 1:3 and the longest emptying time is in the ratio 1:1 up to 29.3 minutes.
3. The largest capacitance value is at a mass ratio of 1:1 and this also results in an increase in the value of the electrical energy produced.

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