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Supercapacitors from Reduced Graphene Oxide Material

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Abstract: Reduced graphene oxide (rGO) has been chemically synthesized from graphite powder. Graphite powder is oxidized using a strong oxidizing compound to obtain graphite oxide, this process is called the Hummer Method. Graphite oxide is dispersed using an ultrasonic vibrator to peel off the graphite oxide layer and become graphene oxide. The epoxy group in graphite oxide is reduced using 80 wt% hydrazine compound at temperatures of 70o, 80 and 90oC to obtain reduced graphene oxide. Then rGO is characterized using Uv-Vis spectroscopy, and FTIR. The Uv-Vis spectrum shows that the absorbance of rGO decreases with increasing reduction temperature. Then the absorbance data is calculated for its optical constant using the Kramers-Kronig Method. The refractive index values of the real and imaginary parts of the calculation results show that in the low energy region an increase in the optical constant value is obtained with the addition of rGO. Then rGO was used in supercapacitor fabrication using PVA as a binder, 1 M $H2SO4$ electrolyte, and aluminum foil as a collector and the supercapacitor mass was obtained as much as 0.01 grams. From the cyclic voltammetry measurement, the capacitor capacitance value was obtained as much as 24.5 F/gr.

Keywords: Graphite; Optical Properties; Reduced graphene oxide; Supercapacitor; Synthesis.

Introduction

Supercapacitors, also known as electrochemical double-layer capacitors, have emerged as promising energy storage devices due to their high-power density, long cycle life, and rapid charge/discharge capabilities. These devices store energy through the reversible adsorption of ions at the electrode-electrolyte interface, making them an attractive alternative to traditional battery systems. Supercapacitors have emerged as a promising energy storage technology due to their high power density, long cycle life, and rapid charge/discharge capabilities (Iqbal et al., 2024; Kumar et al., 2024; Şahin et al., 2022).

Among the various materials explored for supercapacitor electrodes, reduced graphene oxide (rGO) has gained significant attention due to its high surface area, excellent electrical conductivity, and mechanical stability. This study investigates the synthesis, characterization, and electrochemical performance of supercapacitors fabricated using rGO as the primary electrode material. However, the relatively low energy density of supercapacitors has limited their widespread adoption, particularly in applications such as electric vehicles where high energy storage is a crucial requirement (Khan et al., 2021; Sindi, 2024; Smith et al., 2019). To address this limitation, significant research efforts have been directed towards the development of high-performance electrode materials.

Among the various materials explored, graphene oxide has garnered considerable attention due to its unique properties, including high surface area, excellent electrical conductivity, and chemical stability. Graphene oxide, a two-dimensional carbon-based material, can be chemically reduced to obtain reduced graphene oxide (rGO), which exhibits enhanced electrical and electrochemical characteristics compared to its oxidized counterpart (Dreyer et al., 2010; Tarcan et al., 2020).

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The reduction of graphene oxide to obtain reduced graphene oxide (rGO) is a crucial step in the development of high-performance supercapacitor electrodes. Graphene oxide, with its abundant oxygen-containing functional groups, can be chemically or thermally reduced to remove these groups and restore the conjugated carbon structure, leading to improved electrical conductivity and electrochemical. Graphene oxide is typically synthesized via the Hummers' method, which involves the oxidation of graphite using strong oxidizers like sulfuric acid (H_2SO_4) and potassium permanganate ($KMnO_4$).

This process introduces oxygen-containing functional groups (like hydroxyl, carboxyl, and epoxy) to the graphene sheets, making them more hydrophilic and easier to disperse in solvents (Kumar et al., 2024). To obtain reduced graphene oxide, the oxygen functional groups of GO are partially removed. The reduction process can be performed via chemical, thermal, or electrochemical methods: Chemical Reduction: Uses reducing agents like hydrazine, ascorbic acid, or sodium borohydride to remove oxygen groups; Thermal Reduction: Involves heating GO at high temperatures to deoxygenate the material; Electrochemical Reduction: Reduces GO via electrochemical reactions under controlled potential, which can be advantageous for supercapacitor applications due to the integration with electrode fabrication. After reduction, rGO is less oxygenated and more conductive than GO, though it typically still retains some functional groups, providing pseudocapacitive behavior (Nam et al., 2018; Rajagopalan & Chung, 2014; Ramabadran et al., 2017).

Reduced graphene oxide (rGO) is chosen for supercapacitors for several key reasons: rGO has a large surface area due to its two-dimensional (2D) structure, which provides ample space for charge storage. The high surface area maximizes the contact between the electrolyte and electrode material, crucial for storing more charge in supercapacitors; rGO possesses good electrical conductivity, though slightly less than pristine graphene. This enhances the rapid transfer of electrons, leading to fast charging and discharging cycles, which is essential for high-performance supercapacitors; The presence of oxygen-containing groups in reduced graphene oxide (compared to pristine graphene) introduces additional sites for charge storage.

This improves the overall specific capacitance of the material, contributing to its effectiveness in supercapacitors; Compared to pristine graphene, rGO is relatively easier and cheaper to produce, especially through chemical or thermal reduction of graphene oxide (GO) (Pei & Cheng, 2012; Rasul et al., 2017; Stoller et al., 2008). This makes it a more feasible option for large-scale applications like supercapacitors; rGO maintains good mechanical strength and flexibility, which are important for the durability and longevity of supercapacitors. These properties allow it to withstand repeated charge/discharge cycles without significant degradation; The reduction process of graphene oxide (GO) allows control over the oxygen content, structural defects, and functional groups.

This tunability lets researchers optimize the material's properties, such as balancing conductivity and capacitance, for specific supercapacitor applications; rGO can be easily integrated with other materials such as metal oxides, conducting polymers, or carbon nanotubes (Sindi, 2024). These composites further improve the electrochemical performance, energy density, and stability of supercapacitors. Graphene oxide (before reduction**)** typically exhibits a prominent absorption peak around 230 nm due to the π → π* transition of C=C bonds. There may also be a shoulder around 300 nm, associated with n → π* transitions of C=O bonds. Reduced graphene oxide **(**rGO**)**: As reduction progresses, the peak at 230 nm typically redshifts (moves to a higher wavelength, around 260-270 nm) as the conjugated sp^2 domains increase in size. The shoulder around 300 nm diminishes, indicating the reduction of oxygen-containing functional groups (Banerjee & Shukla, 2013; El-Kady et al., 2012; Simon & Gogotsi, 2008; Zhang & Zhao, 2009).

Method

Graphite oxide synthesis was carried out using the Hummer method. In this process, graphite powder was used as the basic material and then oxidized using strong oxidizing compounds, namely $H2SO4$, $NaNO3$, and $KMnO4$. 1 gram of graphite powder was mixed into 23 ml of $H2SO4$ and 0.5 grams of $NaNO3$, then stirred for 2 hours. After the stirring process had been running for 1 hour, 3 grams of $KMnO4$ were added gradually (little by little) by keeping the solution temperature below 20 C (in an ice bath). After 2 hours of stirring, the solution is removed from the ice bath and then the stirring process is continued for 20 hours by maintaining the solution at a temperature of $40^{o}C$ (in a heat bath). At this stage the solution will change color to grayish.

Then the solution is removed from the heat bath and the solution is diluted by gradually adding 200 ml of distilled water and maintaining the temperature of the solution not exceeding $90^{o}C$, then the solution will change color to dark brown. At this stage the stirring process continues and is carried out for 1 hour. The color change to dark brown in the solution can be an early indicator that graphite oxide is starting to form in the solution. In order for the oxidation process to run more perfectly, 5 ml of $H2O2$ 30% was added to the solution so that the solution changed color to brownish yellow. To separate the graphite oxide from the solution, a centrifuge was used with a rotation speed of 6000 rpm, so that graphite oxide would be obtained which would settle at the bottom of the solution. The liquid above the sediment from the centrifuge was discarded so that graphite oxide sediment was obtained. To reduce the acidity level, the graphite oxide sediment was dispersed into distilled water and centrifuged again. This process was repeated several times until the pH of the remaining centrifuge liquid was close to neutral.

Then the graphite oxide sediment was dried using an oven at a temperature of 80 ^0C for 4 hours, so that solid graphite oxide was obtained. The graphite oxide solid is then ground using a mortar until the particle size becomes smaller or called graphite oxide powder. Then a graphite oxide solution is made in distilled water with a concentration of 3 mg/ml. Furthermore, the solution is dispersed using an ultrasonic vibrator for 3 hours until a more homogeneous solution is obtained. At this stage, the provision of vibration energy causes the layer to peel off on the graphite oxide structure so that the layer becomes less or called graphene oxide. To reduce the bonds of epoxy and hydroxyl groups in graphene oxide, 1 µg of 80 wt% hydrazine is added to every 3 mg/ml of graphene oxide and stirred for 3 hours by maintaining the temperature of the solution using a heat bath at a certain reduction temperature. In this study, graphene oxide reduction was carried out at different temperatures, namely 70 °C, 80 °C. After stirring for 3 hours, the solution will change color to pitch black so that reduced graphene oxide is obtained. Then the reduced graphene oxide is separated from the solution using a centrifuge and dried using an oven for 4 hours. So that rGO powder is obtained which is reduced at different temperatures, we call it rGO70, rGO80, and rGO90. Reduced graphene oxide is made into electrodes by mixing 20% Polyvinyl Alcohol (PVA) as a binder (adhesive) and 80% rGO.

The mixture is stirred until homogeneous until a slurry-shaped mixture is obtained (like mud). The slurry is then pressed into an electrode with dimensions x, y and z and an electrode weight of 0.03 gr (adjusting the experimental results). The supercapacitor consists of two collectors, two electrodes and a capacitor. The rGO electrode is arranged into a capacitor using Whatman grade 1 filter paper (12 um) as a separator that separates the two electrodes. Aluminum foil is used as a collector. A solution of $H2SO4$ 1M in distilled water is used as an electrolyte. Before being arranged into a capacitor, the separator is first soaked in electrolyte for several minutes and the rGO electrode is the arrangement of capacitor layers can be seen in the Figure (Chen & Yu, 2010; Elias et al., 2009).

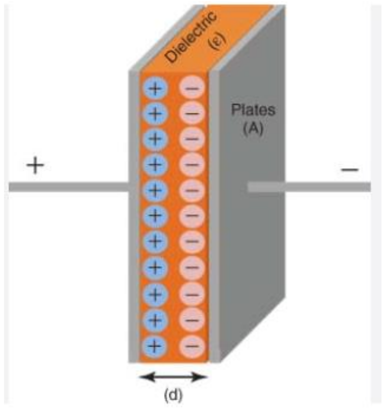

Figure 1. The arrangement of capacitor layers

Result and Discussion

In the figure 2 can be seen that the absorption of graphene oxide has two parts peak at wavelengths around 230 nm and 300 nm. The peak are the characteristics of graphene oxide, each of which occurs as a result electron transition in the $\pi \rightarrow \pi^*$ orbital is transition of electrons in orbitals with sp hybridization and $n \rightarrow \pi^*$. Transition $\pi \rightarrow \pi^*$ is an electron in the sp^2 hybridized orbital and $n \rightarrow \pi*$ represents the existence of bonds between carbon atoms, while sp^3. The existence of the sp^2 orbital represents the presence of carbon oxide bonds. So the degree of oxidation can be observed by observing electron transitions in the material (Dreyer et al., 2010).

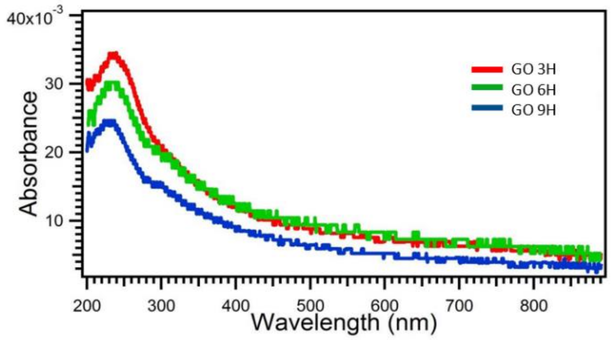

Figure 2. Uv-Vis spectrum of graphene oxide synthesized with different oxidation times

Based on the hypothesis, the longer the oxidation time, the more large degree of oxidation of graphene oxide obtained. This is because of the process long oxidation will form more oxide bonds. The oxidation that occurs in the synthesis process can be seen based on Figure 1 which occurs a reduction in the absorbance value of electron transitions in the orbitals $\pi \rightarrow \pi^*$. As done (Chen & Yu, 2010; Fox, 2010; Geim & Novoselov, 2007), with exposing plasma oxygen to monolayer graphene, it is obtained absorbance of the material by the transition $\pi \rightarrow \pi^*$ will decrease. Subtraction the absorbance value is proportional to the length of plasma oxygen firing time. The longer the firing time, the more oxygen bonds there will be formed on monolayer graphene and causes the absorbance value to be at transition $\pi \rightarrow \pi^*$ becomes smaller. Based on a study by [10] therefore, it can be said that the results of the synthesis of graphene oxide with degrees. The highest oxidation in this study was obtained on graphene oxide oxidized for 9 hours. The degree of oxidation obtained is directly proportional with the length of oxidation time.

To get reduced graphene oxide, reduction is carried out with using the chemical compound hydrazine 80 wt% (N_2H_4OH) in water reduced at 70°, 80° and 90°C. The presence of thermal energy will provide additional energy to break oxide bonds in the material. Hydrazine compound will react with the epoxy group and carboxylic group so that it can breaks oxide bonds in materials. The hydrogen atom in hydrazine will bond to the group oxide to form a H_2O molecule, while the N atom in hydrazine will bond to the surface of the reduced graphene oxide plane. So, reduction graphene using this method will remove a number of oxide atoms on the graphene material, but there is the addition of fewer nitrogen bonds rather than breaking off the O atom. The presence of nitrogen atoms in the material will contributes its own defects to the structure of reduced graphene oxide (Huang et al., 2011; Xiao et al., 2011). The more hydrazine compounds that are added, the more many oxide bonds are broken and more nitrogen bonds are formed on the material. Defects formed in reduced graphene oxide the contribution of oxide atoms will be smaller, but the contribution of defects by the nitrogen atom will get bigger (Nair et al., 2008; Pei & Cheng, 2012).

Figure 3 it can be seen that the reduced graphene oxide only has one absorption peak at a wavelength of 270 nm which is a transition electrons in the $\pi \rightarrow \pi^*$ orbitals. Absorption peaks due to electron transitions in orbitals $n \rightarrow \pi^*$ not visible. This indicates that a reduction has occurred the number of oxide atoms in the material. As the reduction temperature increases, it will further reducing the number of oxide atoms in the material (Prekodravac et al., 2016; Raza, 2012). Reducing oxide bonds in the material will increase the zeta potential value (interaction between surface areas) on the material, causing reduced graphene oxide in solution easily experiences agglomeration and structure experienced layer buildup again. Reduced graphene structure Oxide that undergoes agglomeration does not exactly return to a structure-like form on graphite because the graphene oxide reduction process is not complete removes oxide bonds, and hydrazine compounds will add bonds nitrogen in the material (Ren & Ji, 2011; Santoso et al., 2014; Shulga et al., 2015).

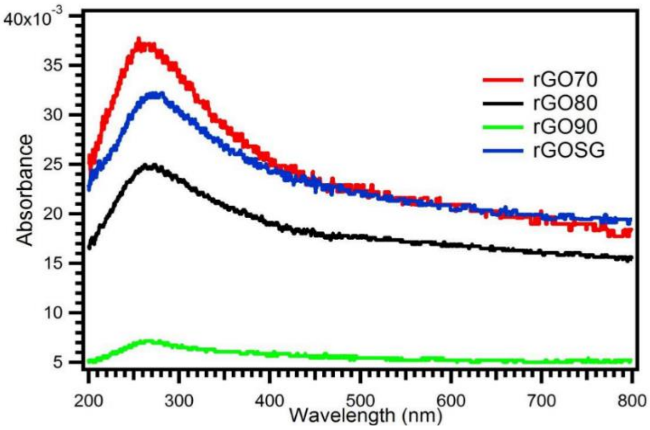

Figure 3. Uv-Vis spectrum of reduced graphene oxide at different reduction temperatures

The presence of oxygen and nitrogen atoms creates distance between them the reduced graphene oxide layer remains larger than on graphite so the electron is in the sp^2 orbital will have more mobility larger than graphite. The higher the reduction temperature, the value the absorbance of the material will be smaller. This happens because, there are fewer transitions electrons in the $\pi \rightarrow \pi^*$ orbitals as a result of the destruction of the hexagonal structure ($C = C$) on reduced graphene oxide. The higher the reduction temperature, the more reduces the $C = C$ bonds in the material thereby contributing to defects. At low energy, the absorption of the material reaches a constant value, as is the case with monolayer graphene which has a constant absorbance value in the visible to infrared region where the characteristic absorbance value is 2.293% (Stoller & Ruoff, 2010; C. Wang et al., 2014; Wu et al., 2010). Absorption spectrum of materials that are similar to reduced graphene oxide purchased from sigma aldrich is a material that synthesized at a reduction temperature of 70^oC. However, there are differences in the form of absorption in the low energy region where the rGO sigma Aldrich absorbance value hasa more constant value compared to synthetic rGO at a temperature of 70^oC. This difference will certainly give the two different energy band shapes materials that can provide different material conductivity values. Based on Figure 2, it can be said that rGO sigma Aldrich is still has better quality compared to synthetic rGO at temperature 70^oC.

Figure 4 shows that GO has a large absorption peak in the wave constant region 3433 and 1620 respectively are stretching vibration of OH molecules and skeletal vibration of C=C molecules. Existence OH molecules show the nature of the material's response to water molecules. On GO has greater OH absorption than rGO sigma Aldrich and synthesis. This shows that GO is hydrophilic while rGO is hydrophobic. In addition to removing the C-O bond, reduction with using hydrazine can also destroy the C=C bond of the sp^2 orbital on the material. This damage will contribute to defects in the material. The more the decrease in the absorbance value of the material at a higher reduction temperature can be This is possible because more and more C=C bonds are broken so that electron transition in the sp^2 orbital decreasing (Berger et al., 2004).

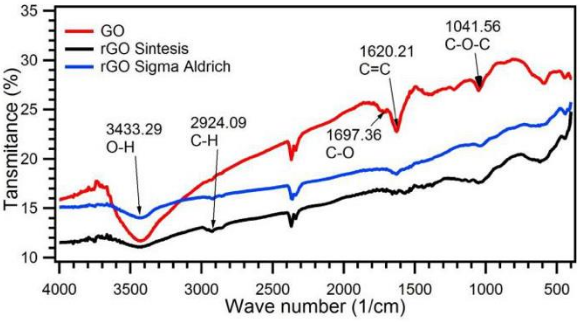

Figure 4. FTIR spectrum of GO, synthetic rGO and sigma Aldrich rGO

Reduced Graphene Oxide (rGO) has a large surface area due to its structure when dispersed in solution in the form of a monolayer. When rGO is dispersed in solution it is brought into the solid phase so it will still have an area larger surface compared to other materials such as graphite, carbon nanotubes, activated carbon, etc. The surface area of rGO can be used as an application in supercapacitor electronic devices. It's known that the capacity of a capacitor will be greater with the greater its area the surface of the electrode used, so that rGO has great potential used as electrode material. Supercapacitors are formed into several layers consisting of two collector (metal), two electrodes (carbon), liquid electrolyte and a separator. In this research, aluminum foil was used as a collector, rGO as electrode, H_2SO_4 1 M in distilled water as electrolyte, and filter paper Whatman grade 1 as separator. Then each layer is arranged. To arrange rGO on the surface of aluminum foil as collector, then the rGO powder must first be glued into a plate shape. In this research, PVA was used as an adhesive (binder). Supercapacitor which has been fabricated, its performance is then measured based on the cyclic test voltammetry. The results obtained are as in Figure 4. Cyclic voltammetry measurements in this study used instruments kithley which can provide electrical voltage values that change linearly. In principle, cyclic voltammetry measurements are carried out by providing positive linear change in voltage (voltage sweep rate) on the sample, then when it reaches a certain desired voltage it changes the voltage will be linear negative (Pham & Dickerson, 2016; Strimaitis et al., 2022).

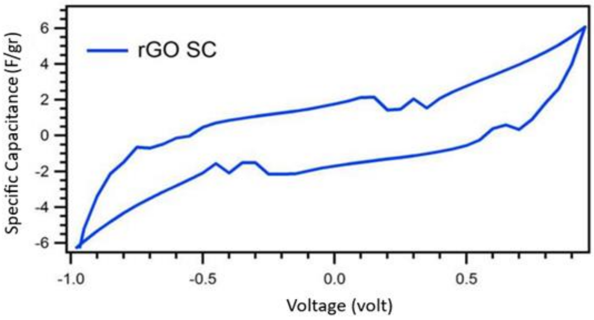

Figure 5. Capacitance value of rGO supercapacitor at different voltages different test results based on the principle of cyclic voltammetry with voltage sweep rate 10 mV/s.

The magnitude of the speed of change in voltage can be adjusted according to needs. In this research, measurements were carried out with a voltage change rate of 70 mV/s. The current flowing in supercapacitor as a response to changes in voltage can be described supercapacitor performance and its capacitance value. Capacitance value rGO supercapacitor obtained is 24.5 F/g. Based on the capacitance value a capacitor is said to be of super value if it has a capacitance of up to 1 Farad. Test results cyclic voltammetry of the rGO supercapacitor shows that the supercapacitor has capable of storing a charge can be seen from the difference between the curve trajectories supercapacitor charging and discharging (Strimaitis et al., 2022). Charge storage mechanism In supercapacitors this occurs by two effects, namely electrical double layer (EDLC) and pseudocapacitance. In this research, the process of storing the cargo desired occurs only because of the EDLC effect. However, from figure 4 it can be seen that the process of storing electric charge also occurs due to effects pseudocapacitance (Strimaitis et al., 2022). This is indicated by the presence of peaks formed at a certain voltage. This pseudocapacitance effect is caused by there is a redox reaction that occurs at the electrode and capacitor collector with electrolyte material. One of the causes of the pseudocapacitance effect in research this is provided by the reaction between PVA as a binder and the electrolyte material. Choosing a good binder is a material that has stable or stable properties does not easily react with water molecules and electrolytes (H. Wang et al., 2011).

Conclusion

From the results obtained that the longer the oxidation process of graphene oxide, the greater the degree of oxidation of graphene oxide or the more C- O bonds formed. The higher the reduction temperature of graphene oxide using hydrazine will cause fewer C- O bonds in the material and the reduced graphene oxide produced will experience damage to the C = C bonds, thereby reducing the number of sp orbitals in the material. Reduced graphene oxide reduced at a temperature of 70°C has the most similar material absorption to reduced graphene oxide purchased from Sigma Aldrich. The addition of reduced graphene oxide to the material provides an increase in absorbance value, real and imaginary refractive index values in the material. An increase in the imaginary refractive index value at low energy indicates an increase in conductivity in the material. The rGO supercapacitor with PVA binder, 1 M $H2SO4$ electrolyte, and aluminum foil as a collector has a capacitance value of 24.5 F/g

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Author Contributions
The contributions of authors: Conceptualization, A and B.; methodology, D.; software, C.; validation, A., and B.; formal analysis, D.; investigation, A.; resources, D.; data curation, C.; writing—original draft preparation, A.; writing—review and editing, B.; visualization, C; supervision, B.; project administration, D.; funding acquisition, A.

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Conflicts of Interest
No conflicts of interest.

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