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The effect of microwave irradiation on reduced graphene oxide from coconut shells

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Abstract. Reduced graphene oxide has been synthesized by microwave irradiation assisted. The effect of microwave irradiation on its crystal structure and electrical conductivity was investigated. Graphene oxide was synthesized using a modified hummers method then reduced by LAA (L-Ascorbic Acid) as a reducing agent with microwave irradiation assisted. The irradiation time varied by 10, 20, 30, and 40 minutes. The samples characterized by Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy-EDAX (SEM-EDAX), X-Ray Diffractometer (XRD), and Inductance Capacitance and Resistance Meters (LCR-Meters).

1. Introduction

Graphene is a two-dimensional carbon material with unique properties that have the potential to be used in a variety of applications. Graphene can be applied such as in the fields of batteries, energy conversion, polymer fillers, sensors, and energy storage devices [1]. Because of it, the need for graphene is to be increased. In the world of research, graphene has become the most sought-after raw material, but the availability of graphene is still very limited, to produce this material in large quantities, it has become a topic of interest among researchers. The development of a synthesis method that is simple, efficient and does not cause toxic materials in the procurement of graphene is needed.

The most widely used method for graphene synthesis is graphite oxidation chemically. This method involves the oxidation of graphite into graphene oxide (GO) using a strong oxidizing reagent, then GO can be converted to graphene through a process of reduction using various reductants. One of the advantages of this method is the formation of graphene in large amounts in powder form, which is dispersed in both polar and non-polar solvents. Chemical graphite oxidation is a method that uses concentrated acids such as sulfuric acid, nitric acid, and phosphoric acid and strong oxidizing agents such as potassium permanganate and potassium perchlorate. However, this oxidation method requires several steps and oxidation temperature control to make GO.

The coconut shell charcoal widely used as activated or absorbent materials, here we utilized the carbonized coconut shell as the starting material of reduced graphene oxide. The reduced graphene oxide (rGO) was synthesized by chemical exfoliation, and use LAA (L-Ascorbic Acid) as a reducing agent assisted by microwave irradiation during the reduction process. In this research, we investigate the effect of microwave irradiation time on the electrical conductivity of the samples.



2. Methods

2.1. Synthesis of GO materials

The commercial coconut shell charcoal was crushed to obtain the powder of the coconut shell charcoal. The powder then dispersed in 0.4 M HCl solution for 8 hours. The solution then neutralized with DI water. Coconut shell charcoal powder (5 g) was added to the mixed acid solution of sulfuric acid (H_2SO_4) and 12.75 mL phosphoric acid (H_3PO_4). The solution was stirred and added KMnO_4 (15 g) gradually and the temperature of the mixture was controlled under 20 °C, then added 230 mL of distilled water. The suspension was stirred for 40 minutes at 50 °C with a stirring speed of 730 rpm then adding 230 mL of DI water gradually. The hydrogen peroxide (5 mL) was used to stop the oxidation process. During this stage, the colour of the mixture was change from dark brown to yellow-brown. The mixture was washed with HCl and NaOH solution, then using DI water to achieve a neutral solution. The sample then dried at 60 °C for 12 hours to obtain GO powder.

2.2. Synthesis of rGO materials

The Graphite oxide powder (2 g) was dispersed into 268 ml of ethylene glycol then proceed with the sonication process for 2 hours. The reduction process was used L-ascorbic acid (16 g) as a reduction agent and assisted by microwave irradiation (Panasonic Microwave 2.45 MHz, 800 W). The microwave irradiation time was varied for 10, 20, 30, and 40 minutes using low mode. The reduction results then filtered and washed using DI water and alcohol three times respectively. The sample further dried for 12 hours at 60 °C.

3. Results and discussion

Reduced graphene oxide (rGO) from coconut shells charcoal has been synthesized using the modification chemical exfoliation method reported previously by Husnah. In this research, the raw material of graphite used commercial coconut shell charcoal. The synthesis consist of three steps first is the oxidation process, then the exfoliation process and the last is the reduction process. The oxidation process used the modified Marciano method to obtain graphite oxide [2]. The exfoliation process carried out by sonication for two hours, and the reduction process used L-ascorbic acid (LAA). LAA was used because it is a nontoxic reduction agent [3]. The microwave irradiation was varied for 10, 20, 30 and 40 minutes. The resulted rGO sample appears in black powder depicted in Figure 1.

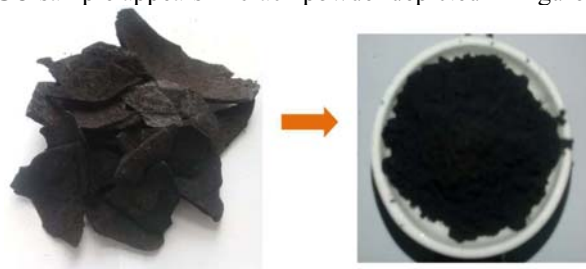


Figure 1. Coconut shell charcoal and resulted in reduced graphene oxide.

To investigate the structure of the obtaining sample, the FTIR and XRD characterization was performed. Fourier Transform - Infrared (FTIR) characterization is carried out to determine the functional groups formed during the synthesis process. This test uses FTIR at the Maliki UIN Chemistry Laboratory Malang. FTIR results of coconut shell charcoal, graphene oxide, and reduced graphene oxide are shown in Figure 2. From the FTIR results, the samples have similar characteristics peaks. There are absorption peaks at wave numbers 3700 and 3400 cm^{-1} describing the vibration of the hydroxyl group. There is also a peak at wavenumber 1620, 1384 and 667 cm^{-1} were caused by aromatic C=C bond, epoxy C-O bond and alkoxy C-O bond respectively [3-5]. After the reduction process, the absorption peak at wave number 3700 cm^{-1} is no longer appear. This indicates that the graphene oxide successfully reduced.

This is in accordance with previous research by Thema, etc., which states that after the reduction process, the oxygen-containing functional group will decrease and even disappear [6]. The oxygen-containing functional group usually provide strong bonds. A strong bond is usually characterized by the depth of the valley produced in the FTIR spectrum. Even so, it is difficult to make a conclusion based on a percentage because it is very dependent on the number of samples when testing, and it is not well controlled [3].

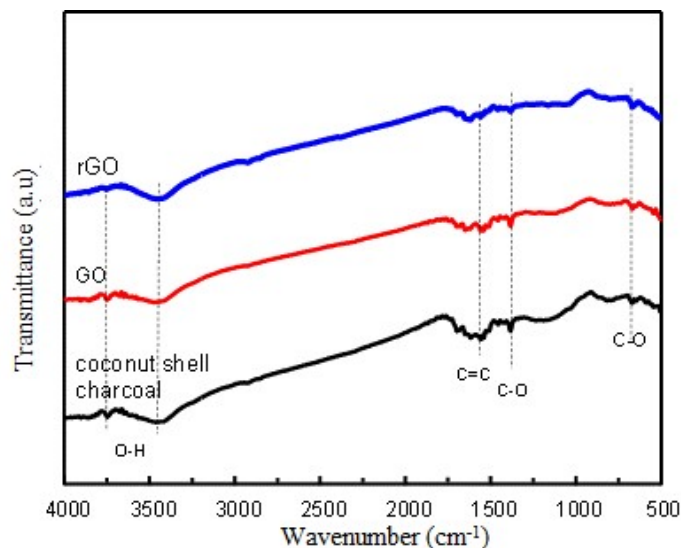


Figure 2. The FTIR results of Coconut shell charcoal, graphene oxide (GO) and reduced graphene oxide (rGO).

The crystal structure of the obtaining sample was investigated by using X-ray diffraction (XRD). X-ray diffraction result is important to determine whether the sample is graphite, graphene oxide or rGO. Graphite and rGO have a characteristic peak at 2θ : $24-28^\circ$, whereas graphene oxide has a 2θ peak at $10^\circ-12^\circ$. The XRD results in Figure 3. Show the XRD pattern of Coconut shell charcoal, graphene oxide (GO) and reduced graphene oxide (rGO). The coconut shells charcoal has a sharp peak at $2\theta=27.8^\circ$, and a lower intensity peak at $2\theta=43^\circ$. After the oxidation process, there is a peak at $2\theta=8.1^\circ$, and high-intensity peak at $2\theta=27.8^\circ$ disappear as the characteristic of GO. This phenomenon due to the appearance of the oxygen-containing functional group during the oxidation process [7]. A broad peak at $2\theta=24^\circ$ appears in the XRD pattern of rGO, this results in accordance with previous XRD result reported by Setiadji [8].

The morphology of the rGO has been investigated by SEM characterizations, as shown in Figure 4. The SEM results showed thin sheets which are the intrinsic properties of graphene [5]. From the EDX result, the rGO sample contains C and O element atom of 86.2% and 13.8% respectively.

High electrical conductivity and stable electrochemical properties are important for energy catalyst and storage applications [9]. Therefore, LCR-meter was conducted to define the electrical conductivity of the rGO samples. LCR-Meter testing was carried out at the Magnet Electric Laboratory of Physics Department, Faculty of Physics, Saintek, UIN Maliki, Malang. This test uses a HICRI 3532-50 LCR HiTESTER LCR-Meter. The electrical conductivity results of the samples are depicted in Figure 5.

Figure 5. Show the electrical conductivity of the rGO samples with varied microwave irradiation time of 10, 20, 30, and 40 minutes. The use of microwave irradiation in this experiment is because it is more efficient (reduce the time consuming during the reduction process), environmentally friendly, scalable and cost-effective to produce graphene-based materials [10]. The electrical conductivity of the rGO increase in line with increasing the irradiation time. But, the highest electrical conductivity is stag after 20 minutes of irradiation time.

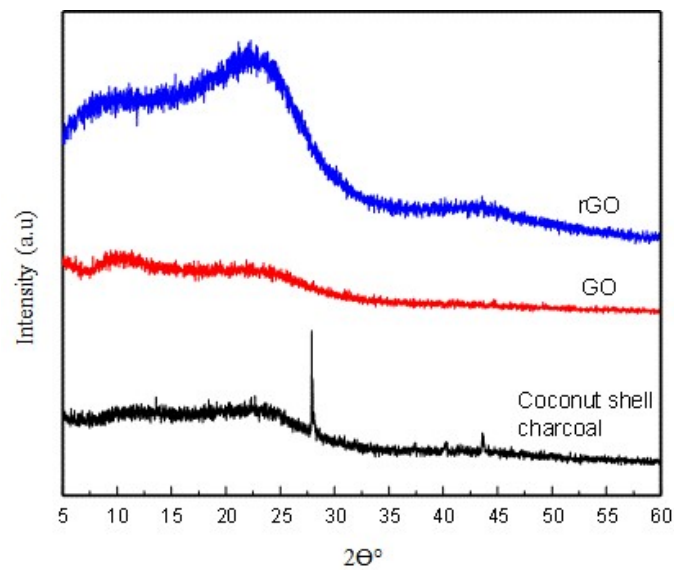


Figure 3. XRD pattern of Coconut shell charcoal, graphene oxide (GO) and reduced graphene oxide (rGO).

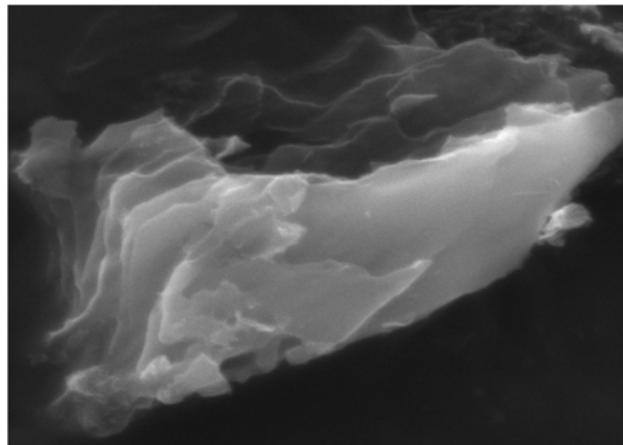


Figure 4. The morphology of the rGO sample.

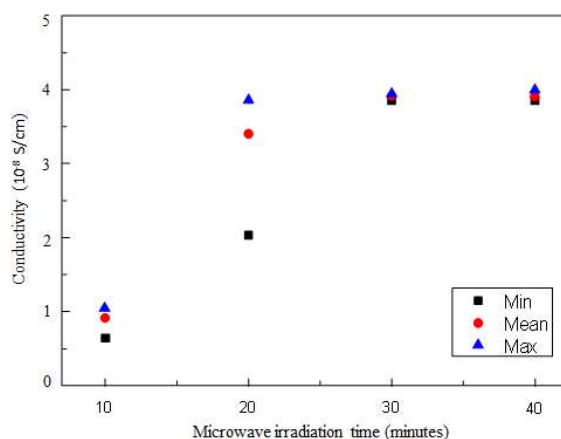


Figure 5. The electrical conductivity of the rGO sample with varied microwave irradiation time 10, 20, 30 and 40 minutes.

4. Conclusion

The reduced graphene oxide (rGO) from coconut shells has been successfully synthesized using the chemical exfoliation method assisted by microwave irradiation. The structure and morphology of rGO were confirmed by FTIR, XRD, SEM, and EDX results. The highest electrical conductivity of the sample rGO was obtained with 40 minutes of microwave irradiation time.

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