

Synthesis and characterization of reduced graphene oxidation from corn cob waste

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Keywords

Corn cob waste
Graphene
Graphite
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rGO

Abstract

Graphene is the latest carbon-based nanomaterial with wide applications and has superiority in electrical, thermal, and high conductivity properties. This study aims to synthesize and characterize reduced graphene oxide (rGO) from corn cob waste using the modified Hummer method, which was carried out by removing the NaNO_3 material. This synthesis used corn cob waste that was made into charcoal with a particle size of 100 mesh with an oxidation time of 5 days. Structural analysis of rGO used Fourier Transform Infrared (FTIR) and X-ray diffraction (XRD) testing. The results of FTIR testing showed a wave peak at 1568.82 cm^{-1} with $\text{C}=\text{C}$ bonds which indicated that rGO was formed. The XRD test results showed a diffractogram peak at an angle of $2\theta \sim 24.6635$ which is typical of rGO materials.



Introduction

Graphene is the latest carbon-based nanomaterial with wide applications and has advantages in electrical, thermal, and high conductivity properties. From the very wide application of graphene, it has a lot of potential in the fields of batteries, polymer chargers, sensors, and energy storage (Russo et al. 2013). By considering the wide application of graphene, so the need for graphene is very much needed for raw materials that are very much sought after, but the availability of graphene is still lacking and is still very limited because the manufacturing process is very complicated and expensive. So how to produce graphene in large quantities? which has become an interesting material for research. One of the methods used to fulfill this need is to research a simple and efficient synthesis.

The method that is widely used to synthesize graphene is chemically, this method is through the oxidation of graphite to graphene oxide (GO) using a strong oxidizer, then GO is converted into graphene by a reduction process using various reductants (Habte and Ayele, 2019). Chemical oxidation of graphite is a method that uses concentrated acid (sulfuric acid, nitric acid, and phosphoric acid) and using strong oxidizing agents (potassium permanganate, and potassium perchlorate). requires several stages and temperature control to manufacture GO (Pei and Cheng, 2012).

There are many methods that can be used to produce GO by chemical oxidation, such as the Hummer method, Staudeumaier's method, and Brodie's method (Wang and Dou, 2012). The most widely used method for synthesis of graphene oxide was proposed by Hummers and Offeman (1958). They oxidized graphite with concentrated sulphuric acid, sodium nitrate and potassium permanganate (Selvakumar et al. 2016). This method is very easy and good for generating GO. However, the use of NaNO_3 in this method will produce toxic gases NO_2 and N_2O_4 which are released during the oxidation process. Therefore, a modified Hummer method emerged which does not have to use NaNO_3 so it is no longer dangerous.

Corn cobs in the community are only used as fuel for making tofu and also for animal feed. One of the great benefits of corn cobs is that it can be used as raw material in the manufacture of graphene oxide which



is the raw material for graphene. In this study, reduced graphene oxide (rGO) was synthesized using corncob waste through the modified Hummer method. Then the rGO samples were characterized through XRD analysis and also FTIR.

Method

Materials

This study uses corncob waste obtained from a corn mill in the Jember area, which is used as charcoal to become the main raw material in the manufacture of oxidized graphene. Additional materials used to support this research are H_2SO_4 , $KMnO_2$, HCl , H_2O_2 , and Aquades.

Analytical method

In this study, we used 2 analytical methods, namely XRD and FTIR. In the XRD analysis, it was carried out with an angle range of 2θ between $5-60^\circ$ and a wavelength of 1.54 \AA , while in FTIR, it was carried out with Agilent Technologies carry type 600 series with a wavelength range of $400-4000\text{cm}^{-1}$.

Results and Discussion

Effect of stirring on % yield with various initial masses

In the case of magnetic stirrers, the higher the revolution per minute (rpm), the more homogeneous the mixture and the yield of the product, so that the higher the stirring speed, the more homogeneous graphite material and the more % Yield obtained (Amaia and Beatriz, 2018). The effect on the % Yield results obtained which is shown in Fig. 1.

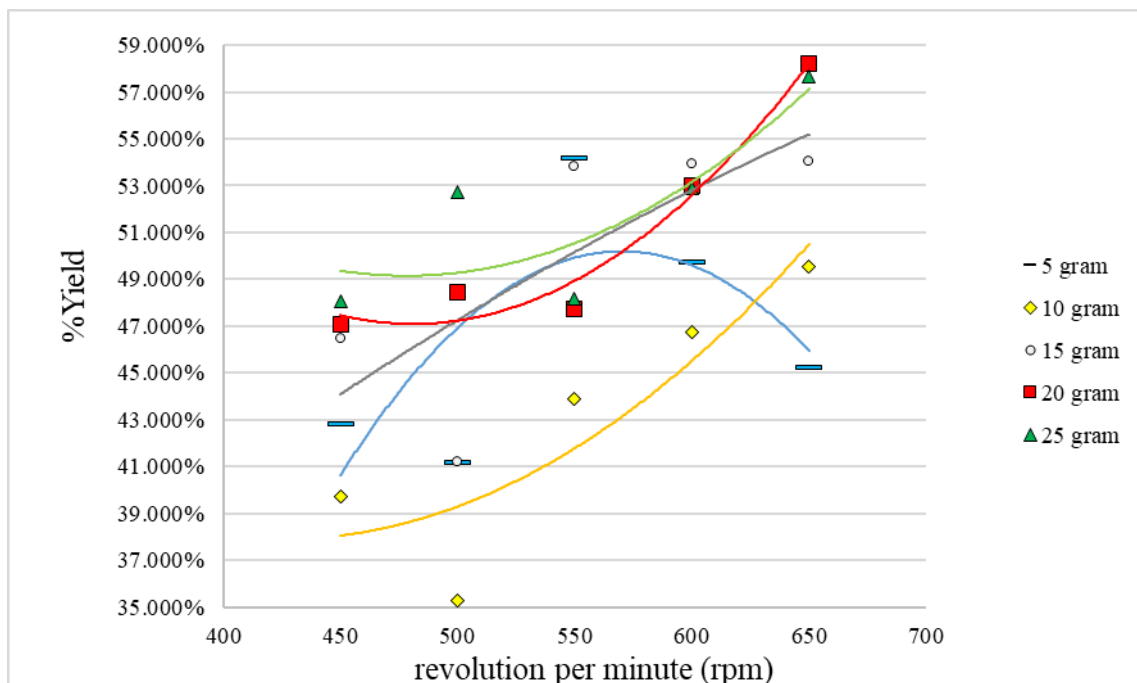


Figure 1. The Effect of stirring on % yield with various initial masses

rGO test results using XRD

The test using X-ray diffraction (XRD) is aimed at determining the crystallinity phase of graphene oxide from reduced graphene oxide (rGO). This diffraction is important because it determines whether the sample shows graphite, graphene oxide, or rGO. Graphite and rGO have a 2θ band at an angle of $25-28^\circ$, while graphene oxide has a 2θ band at an angle of $10-12^\circ$ (Krishnamoorthy et al. 2012; Cooper et al. 2012). The rGO test results using XRD diffractogram is shown in Fig. 2. Based on Fig. 2 with various initial masses and varying stirrer rotations, it is obtained that the rGO results obtained have an amorphous phase, this phase can be seen from the width of the peak obtained from the graph results, and the angle obtained is on average 23.5911° .

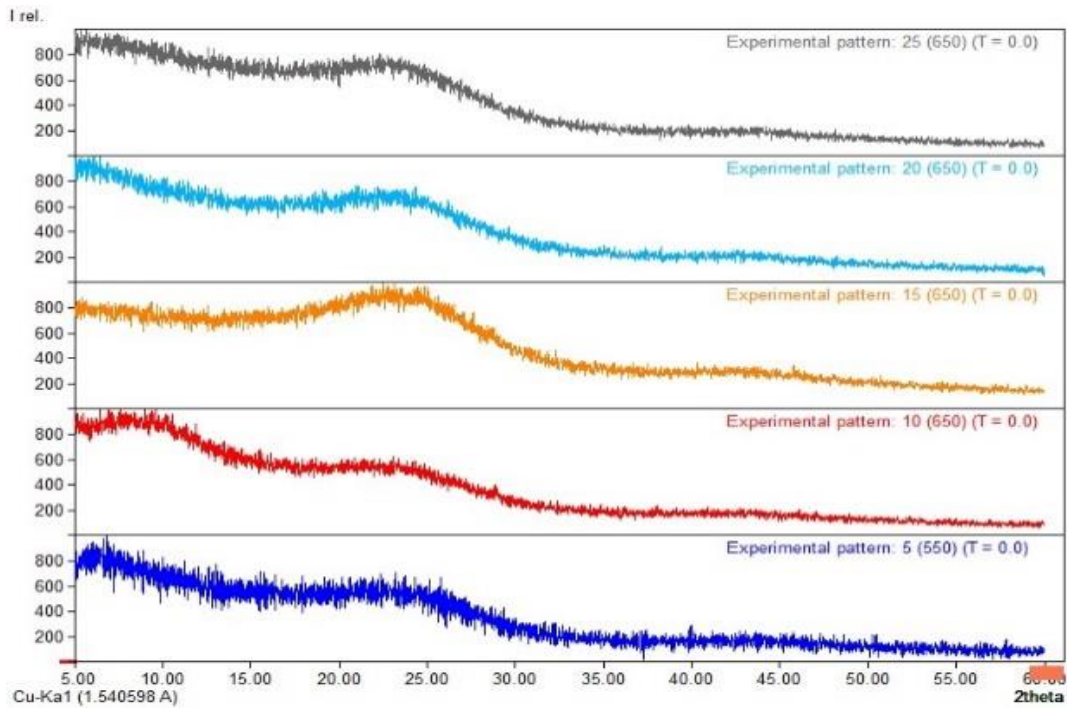


Figure 2. XRD reduced graphene oxide (rGO) diffraction pattern from corn cob waste

rGO test results using FTIR

This test uses FTIR to determine the functional groups contained in the rGO results obtained, this test uses a wavelength range of 400-4000 cm-1. The rGO test results using FTIR is shown in Fig. 3.

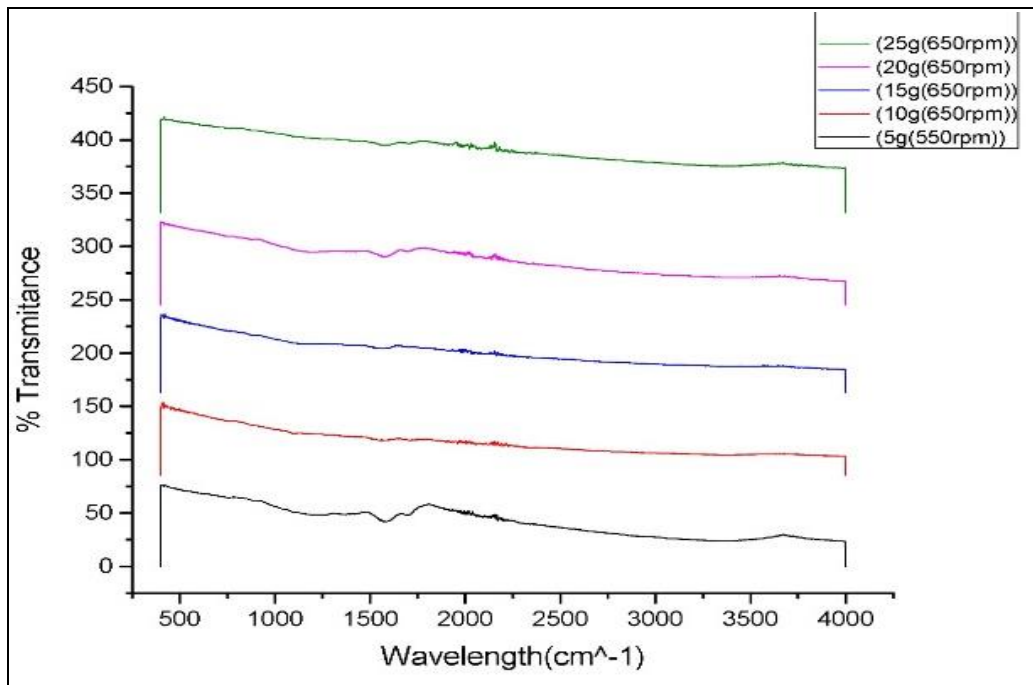


Figure 3. Results of the FTIR reduced graphene oxide (rGO) test from corn cob waste

From the Fig. 3 above, we get a peak at the wavelength of the test results. The results of rGO detected an aromatic C=C functional group at a wavelength of 1500-1600 cm-1, a C=O functional group at a wavelength of 1690-1760 cm-1, and no functional group was detected. O-H at a wavelength of 3590-3650 cm-1. In the process of reducing graphene oxide to rGO, there are things that must be considered, namely the loss of functional groups containing oxygen after the oxidation process. The OH group present in GO will be reduced, While also The aromatic C=C bond indicates the formation of rGO (Setiadji et al. 2018)

Conclusion

It was concluded that for rGO originating from corn cobs waste, namely at 650% the highest yield was obtained, then the effect of rpm on % yield was directly proportional, the two rGO products obtained in this study, which was a mass of 20 grams with stirring at 650 rpm was the best because the test XRD obtained an angle of 23.47170 and the FTIR test showed that there was an aromatic C=C bond, C=O and no O-H group.

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