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## The Influence of Microwave on Reduced Graphene Oxide (rGO) Crystallinity from Inorganic Waste

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**ABSTRAK.** The synthesis of reduced graphene oxide was carried out using the smoke catcher method and inorganic waste. The inorganic waste smoke obtained became the basic material as a carbon source. After burning the inorganic waste, the smoke collected was in the form of a powder and exposed to microwaves. Based on FTIR characterization, the synthesis was successfully carried out by obtaining  $\sim 1600 \text{ cm}^{-1}$  as the peak of the aromatic functional group (C = C) or graphene functional group. After the inorganic waste smoke sample was put in the microwave, the rGO sample was obtained which had a higher crystallinity than before heating in the microwave.

### 1. Introduction

Graphene is one of the two-dimensional (2D)  $sp^2$ -hybridized carbon allotropes with a hexagonal lattice (hexagon like a honeycomb). The two-dimensional structure causing graphene has a zero bandgap and is semi-metallic (Loryuenyong et al., 2013). With superior physical and chemical properties compared to other materials, graphene has recently become a desirable material in the electronics industry and is interesting to research and develop. Graphene has properties that can substantially improve the performance of electronic devices and can also be very potential for applications in various fields of technology such as batteries

(Iskandar et al., 2015), fuel cells (Hsieh et al., 2012), capacitors (Chang et al., 2012), sensors (Raj et al, 2013), electrocatalysis (Wang et al, 2013), solar cells (Wang X et al, 2008), nanocomposites, and others (Stankovich et al, 2006). Until now, many studies have been carried out on the synthesis of graphene to produce graphene, one of the methods often used is the chemical synthesis method in the presence of oxidation, and this method is able to produce products in large-scale, easy, and economical production. Graphene produced from chemical synthesis methods is known as reduced graphene oxide (rGO) through the GO reduction process. Reduced graphene oxide (RGO) is GO processed by chemical, thermal, and other methods to reduce oxygen content, while graphite oxide is a material produced by the oxidation of graphite which leads to the increased distance between cast and functionalization of the graphite base plane.

Carbon, the basic material for making graphene, is very abundant on earth. Carbon is the 15th most abundant element on earth and the 4th most abundant element in the universe. Carbon is present in all types of living things, and in humans, carbon is the second most abundant element (about 18.5%) after oxygen. One of the sources of carbon is the waste (smoke) burning. In this study, combustion smoke from various kinds of inorganic waste was used as a base material for graphene oxide (GO). As is well known, the waste problem in Indonesia is a very serious environmental problem, especially in the problem of inorganic waste. Inorganic waste is very difficult to decompose or recycle naturally in the soil. The difficulty of inorganic waste decompose and the increasing amount of waste produced every year is the source of the waste pile problem in Indonesia.

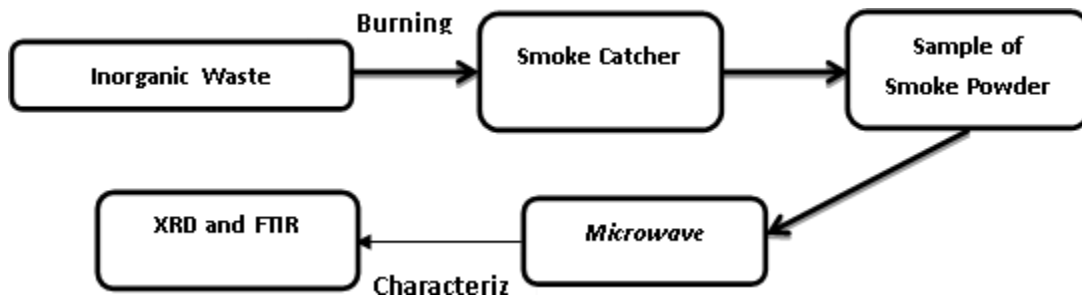
The waste problem has no end. The waste problem has become a serious problem, especially in big cities, not only in Indonesia but throughout the world. Developed countries have made various efforts to overcome this problem, as well as for local governments where solid waste is a serious problem. Waste production continues to increase along with the increase in population, changes in consumption patterns, and lifestyles have increased the number of waste piles, types, and diversity of characteristics of waste.

Buring the waste is an easy waste-handling process. This is one of the reasons for overcoming the problem of solid waste, especially hazardous infectious waste. Combustion is the process of reacting fuel (biomass, oil, etc.) with oxygen, or in other terms, it is called oxidation (Pradipta, 2011). To carry out the combustion process, a combustion chamber furnace is needed which can reduce the volume of solids so as not to cause solids (waste) piles.

Burning inorganic waste can cause problems for the environment and human health and also combustion is one of the causes of the greenhouse effect due to the effect of increasing concentrations of carbon dioxide ( $\text{CO}_2$ ), which goes beyond the ability of plants to absorb it. This is the reason to utilize combustion fumes ( $\text{CO}$  and  $\text{CO}_2$ ) or utilize carbon materials produced from combustion fumes of various kinds of inorganic waste to be used to obtain graphite oxide as a basic material for making graphene oxide (GO) by exfoliating the resulting graphite oxide material so that the smoke from the combustion is not wasted and also want to see the effect of microwaves on the smoke from burning the waste.

## 2. Method

This study was conducted in three stages. The first stage experiment was to find organic waste and manufacture a smoke catcher. The second stage was the burning of inorganic waste using the smoke catcher method with a glass preparation. The method was carried out using two glass preparations arranged at 90 angles and placed on the furnace to catch the smoke inside the furnace. The smoke attached to the glass was dredged using a spatula and dissolved in distilled water to produce a powder that will be used as a base for making Reduced Graphene Oxide (rGO). The third stage was the synthesis of Reduced Graphene Oxide (rGO) by exposing microwaves to break bonds between layers in the atomic arrangement of the sample. In general, several stages in forming reduced graphene oxide can be seen in the following figure 1:

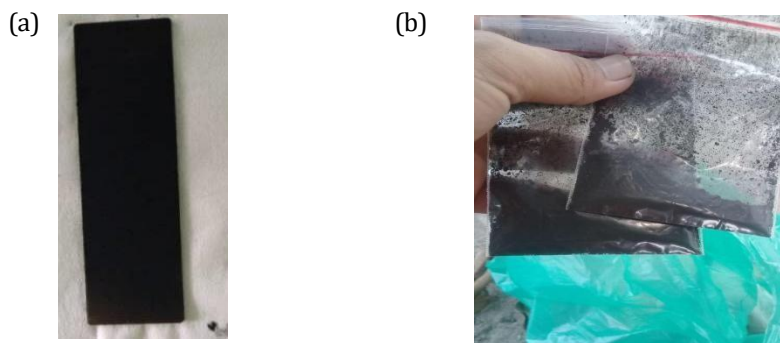


**Figure 1.** Flow chart of reduced graphene oxide (rGO) production from inorganic waste smoke catcher.

This study was started by filling a heating stove (smoke catcher) with various inorganic waste and then burning it for 30 minutes. During combustion, the smoke produced was captured using a glass preparation above the combustion, the glass affixed with smoke was put into distilled water so that the smoke powder escaped from the glass preparation. The solution was then exfoliated using a microwave with a power of 300W for 20 minutes. Then, the solution was added with 150 ml of distilled water and 100 ml of ethanol and continued by sonication using a sonicator and high-speed mixer. Then, the solution was filtered and heated in an oven at 60°C for 2 hours. The resulting samples were characterized using XRD (X-Ray Diffractometer ) 6100 shimazu and FTIR (Fourier Transform Infrared Spectroscopy ) utilize Alpha FTIR Spectrometer Bruker 1 176 396.

### 3. Results and Discussion

The success of reduced graphene oxide (GO) synthesis using inorganic waste smoke as the base material was analyzed and proven through several types of characterization. In this study, there were 2 samples, sample 1 was inorganic waste powder and sample 2 was inorganic waste powder by microwave heating.



**Figure 2.** Inorganic waste smoke (a) attached to the glass preparation and (b) inorganic waste powder.

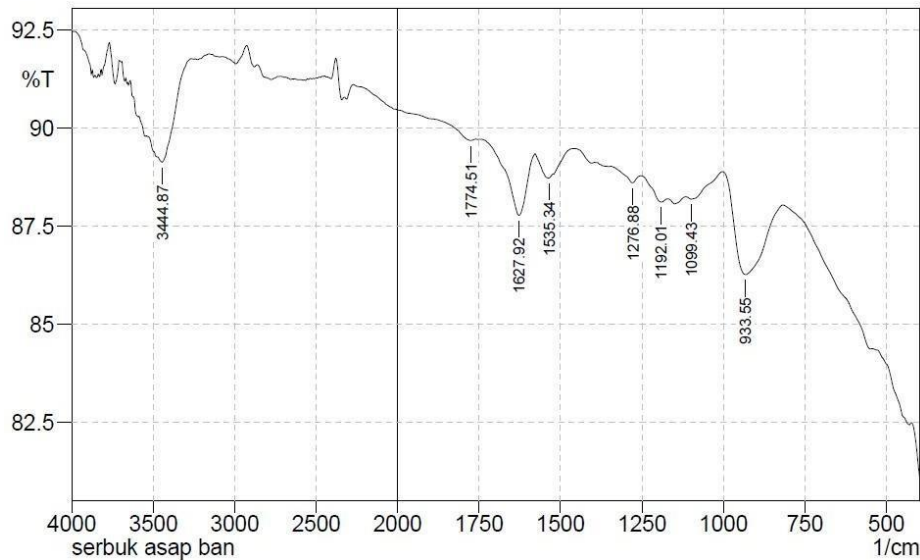
The inorganic waste powder obtained can be seen in Figure 2. The success of the graphene oxide sample from inorganic waste smoke both before and after being exposed to the microwave was analyzed from the results of XRD and FTIR characterization.

#### 3.1 Functional Groups (FTIR Characterization)

Functional group analysis from the results of Fourier Transform Infrared Spectroscopy (FTIR) characterization showed a peak transmittance of the FTIR graphic pattern. Each functional group has a different wavenumber based on the ability of the functional group to vibrate and absorb energy from the

infrared spectrum.

In the FTIR graph, there are basins or waves in the transmittance spectrum that indicate the presence of particles that interact with infrared radiation at that wavelength. The basin shows the elemental bonds in the sample under test. The results of FTIR characterization showed that the sample obtained using a smoke catcher was in the form of Reduced Graphene Oxide (rGO) as shown in Figure 3.



**Figure 3.** FTIR graph of inorganic waste powder.

The results of FTIR spectrum from inorganic waste powder samples showed GO as the absorption peak of FTIR, indicated the OH bond at  $3444.87\text{ cm}^{-1}$ , then it was found at  $1774.51\text{ cm}^{-1}$  as functional group C = O. Furthermore, the results of the FTIR graph show results at wavelengths of  $1627.92\text{ cm}^{-1}$  and  $1535.34\text{ cm}^{-1}$  which are aromatic functional groups (C=C), where functional groups C=C are functional groups in graphene (Husnah, 2017), while the next absorption peak occurred at  $1276.88\text{ cm}^{-1}$  as the functional group of CO, then at  $1192.01\text{ cm}^{-1}$  as the functional group from CN, at  $1099.43\text{ cm}^{-1}$  as the functional group of CO, and at  $933.55\text{ cm}^{-1}$  as the functional group of CH.

### 3.2 Sample Crystallinity (XRD Test)

The crystal phase and structure of the synthesized graphene material were characterized using XRD. X-ray diffraction (XRD) is one of the characterization tools needed to analyze graphene properties. Figure 4 shows the results of XRD characterization in both samples. Sample 1 was powder from inorganic waste smoke before being exposed by microwave and sample 20 was powder from inorganic waste smoke after being exposed by microwave. XRD results show that the samples obtained are not in the shape of graphite, because the results showed diffraction peaks not corresponding to JCPDS card no. 41-1487 (Fakhri, 2017), but the peaks formed based on reference (Yang, Y.-Y., et al., 2012), it can be seen that powder from inorganic waste smoke (Sample 1) was rGO powder (reduced graphene oxide), due to diffraction peaks (002) at  $2\theta \sim 24.04^\circ$  indicating the distance between the layers on rGO (Stobinski, 2014) and the diffraction peaks (101) at  $2 \sim 44.02^\circ$ , whereas at  $2\theta = 9.8^\circ - 11.5^\circ$  (Mei dkk, 2015), as the graphene oxide peaks did not appear in the samples characterized This is very different from what has been reported by previous researchers (Puspitasari, 2017) who concluded that waste smoke from inorganic patchwork was graphene oxide. Based on the Bragg equation, the distance between crystalline planes was  $\sim 0.410\text{ nm}$  in sample 1 and  $0.408\text{ nm}$  in sample 2. The size of crystallinity (H) was calculated by the Scherrer equation as shown in Table 1.

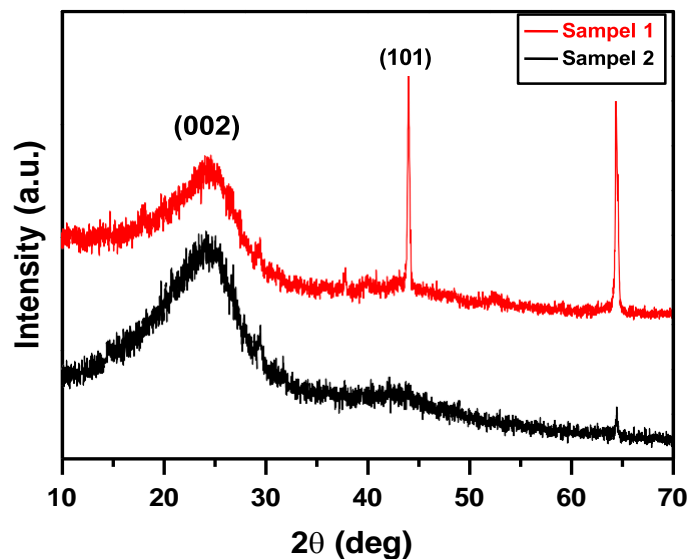
From the results of the XRD test in sample 1, the diffraction peaks were obtained at  $2\theta \sim 64.34^\circ$ , while the reference graphene sample did not have diffraction peaks at  $2\theta$ , the peaks probably came from other elements in sample 1 which were most likely impure. To find out the elements contained in sample 1, further

tests such as SEM (Scanning Electron Microscopy), EDS (Energy Dispersive X-Ray Spectroscopy), or other tests are necessary. The results of XRD characterization in sample 2, namely inorganic waste smoke samples with a microwave, showed a significant decrease in diffraction peaks at  $2\theta = \sim 44.02^\circ$  and at  $2\theta = \sim 64.34^\circ$  contained in the XRD results for sample 1. This shows that the microwave exposure process can remove impurities in the sample.

**Table 1.** Comparison of crystal structure parameters of XRD processed samples

Sample	Peak (002)				
	$2\theta$ (deg)	FWHM ( $\theta$ )	$\beta$ (rad)	H (nm)	d (nm)
Sample 1	24.04	5.197	0.0905	1.559	0.410
Sample 2	24.16	7.930	0.1383	1.020	0.408

Based on the graph of XRD characterization results, it can also be seen that the intensity of the diffraction peak (002) was the highest in rGO of sample 2, so it can be said to have a higher crystallinity than the rGO of sample 1.



**Figure 4.** Results of XRD Characterization

#### 4. Conclusion

Reduced graphene oxide (rGO) was successfully synthesized using the smoke capture method with inorganic waste as the main material. After inorganic waste smoke was exposed to microwave (with microwave heating), rGO samples were obtained which had a higher crystallinity than before being heated by microwave. The characterization results showed that there was a peak at  $\sim 1600\text{ cm}^{-1}$  which is the peak of the aromatic functional group (C = C) are a functional group in graphene.

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