

# The Synthesis of Graphene from Coconut Shell Charcoal

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**Keywords:** charcoal,  $sp^2$  orbital, graphene, activated carbon, pyrolysis, coconut shell

**Abstract:** The hybrid coconut shell charcoal is  $sp^3$ , after being mixed with activated carbon and heated to 600°C for 1 hour it produces  $sp^2$  which shows the characterization of graphene. The process of making graphene in this study, namely coconut shell dried under sunlight then hydrolyzed into charcoal then mixed with activated carbon as a reducing agent at 600°C for 1 hour to produce graphene. The resulting graphene is characterized by XRD, SEM-EDX, XRF and BET. The results of the XRD analysis showed that the resulting peaks were not sharp and slightly broadened the diffraction peak at 24° and 44°. The results of SEM-EDX analysis at 4000x magnification showed smaller, thinner surface sizes and structural shapes and reduced buildup in the graphene structure. XRF analysis results show that there are still organic impurities. The results of graphene analysis with BET show the surface area of graphene 82.873  $m^2 / g$  with a pore volume of 0.116  $cc / g$ .

## 1 INTRODUCTION

Coconut shell is a hard part on coconut which has a thickness between 3-8 mm which consists mostly of lignin, cellulose, and hemicellulose. Coconut shells can be converted into charcoal or activated carbon through the carbonization process. Coconut shell can be used as a carbon source in graphene synthesis (Liyanage and Pieris, 2015).

Now, the most popular method for producing single-layered and multi-layered graphene is by solving methods or known as mechanical and chemical methods. For the mechanical method, the graphene produced is single layered. However, the cost needed in making graphene is very expensive, and graphene is produced in small quantities. Meanwhile, with the chemical method, graphene produced in large quantities and the preparation of graphene is very simple, but the graphene produced is still not single-layered (Siburian, 2012).

In general, graphene is made through the Hummer method and got from graphite mining which comes from nature and is a non-renewable resource. Previous researchers were done by Supeno and Siburian (2018) using coconut shells which were converted into graphite and graphene nano switching. The conversion of coconut shell into graphene can be seen from the comparison of Aluminum-vessel effect and Pyrex-Glass effect on the cracking process. On Pyrex-Glass vessels the process of pyrolysis at high temperatures will less contribute in donating electrons to the coconut shell compared to the Aluminum-vessel vessel and then continued pyrolysis at a temperature of 600°C. Previous researchers assumed that coconut plants were an abundant natural resource and the constituent carbon was C-amorphous. Therefore, through this research coconut shell was used as a carbon source in graphene synthesis. Graphene synthesis starts from carbonize coconut shell into charcoal. After the coconut shell structure changed from physical structure to charcoal, then the reducing agent is activated carbon is added which functions to absorb the oxide present in the charcoal with a temperature of 600°C which is expected to synthesize graphene from coconut shell charcoal. Based on the background described, it is necessary to do some research with the title "The Synthesis of Graphene from Coconut Shell Charcoal".

## 2 MATERIALS AND METHODS

### 2.1 Tools

Tools used in this experiment: Glassware, Analytical Balance, Tube Clamp, Watch Glass, Porcelain Saucer, Filter Paper, Mortar, Funnel, Magnetic Bar, Furnace, 100 mesh sieve, 150 mesh sieve, Oven, Spatula, Electron Scanning Microscope, X-Ray Diffraction, Brunauer–Emmet–Teller, Fluorescence Spectroscopy X-ray.

### 2.2 Materials

Materials used in this experiment: coconut shell, activated carbon, aquadest and KOH 1 N.

### 2.3 Procedure

#### 2.3.1 Making of Charcoal

Coconut shells are dried in the sun until dry. Then, 1 kg of coconut shell is taken, then hydrolyzed in the furnace in an oxygen free condition for 5 hours at 600°C until became charcoal. Smoothed using mortar. Furthermore, it is sifted using a 100-mesh size sieve. Then, it was characterized using XRD and SEM.

#### 2.3.2 Synthesis of Graphene

Charcoal from the coconut shell is in chip form, then weighed as much as 15 g and mixed with activated carbon powder. Then it was heated at 600°C for 1 hour. Then sifted using 150 mesh sieves. Coconut shells are washed with distilled water until clean, and dried in an oven at 70°C. Furthermore, it was characterized using XRD, XRF, SEM-EDX, and BET.

#### 2.3.3 Effect of Addition of KOH 1 N Solution on Surface Area and Graphene Pore Size

Weighed 15 g of graphene and then soaked with KOH 1 N for 2 hours. Then precipitated in the oven for 1 h until dry. Furthermore, the graphene is heated in a furnace at 600°C for 1 hour. Furthermore, it was characterized by using XRD and BET.

## 3 RESULTS AND DISCUSSIONS

### 3.1 Making of Charcoal

Coconut shells are cleaned then the coconut shell is burned at 600°C for 5 hours until it turns black and turns into charcoal. According to Liyanage and Pieris (2015) the heating process in the coconut shell will produce gradual changes. Coconut shell charcoal produced, mashed with a 100-mesh sieve. The coconut shell charcoal powder produced was characterized using XRD and SEM-EDX.

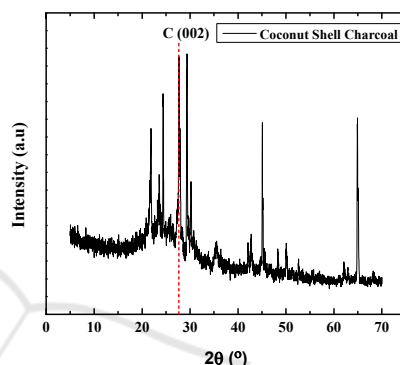


Figure 1: XRD diffractogram coconut shell charcoal powder

The diffractogram showed by Figure.1, which is XRD analysis shows that there are sharp peaks and densities in several regions  $2\theta$  starting from 28° diffraction angle which indicating that the phase formed is the crystalline phase. The data obtained is characteristic of the crystal structure of graphite (Chen and Yan, 2009).

The results of the surface morphology scale analysis by Scanning Electron Microscope (SEM) on coconut shell charcoal powder with 4000x magnification can be seen in Figure 2.

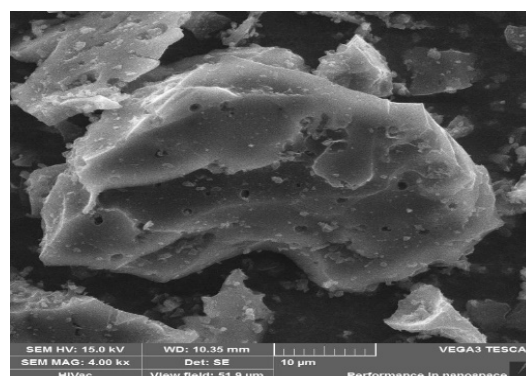


Figure 2: Surface morphology scale by scanning electron microscope (SEM) on coconut shell charcoal powder with 4000x magnification

SEM testing aimed to observe the surface morphology and particle shape of the sample. At 4000x magnification, the coconut shell charcoal powder in the form of piles showed that the coconut shell charcoal powder had a layer structure.

### 3.2 Analysis of Graphene Powder Structure from Coconut Shell Charcoal

Graphene powder from coconut shell charcoal in this experiment was produced from the pyrolysis of old coconut shell by mixing activated carbon as a reducing agent with a temperature of 600°C in the furnace for 1 hour. The structure and phase analysis of graphene from coconut shell charcoal used X-Ray Diffraction (XRD), SEM-EDX, XRF and BET.

#### 3.2.1 XRD Data Graphene Powder from Coconut Shell Charcoal

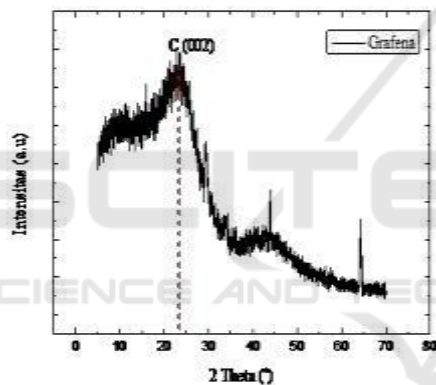


Figure 3: XRD diffractogram graphene powder from coconut shell charcoal

Diffraction peaks resulted are weaker and wider indicating a reduction in some functional groups.

#### 3.2.2 SEM-EDX Data Graphene Powder from Coconut Shell Charcoal

The results of the surface morphology scale analysis by Scanning Electron Microscope (SEM) analysis of graphene powder from coconut shell charcoal with 4000x magnification can be seen in Figure 4.

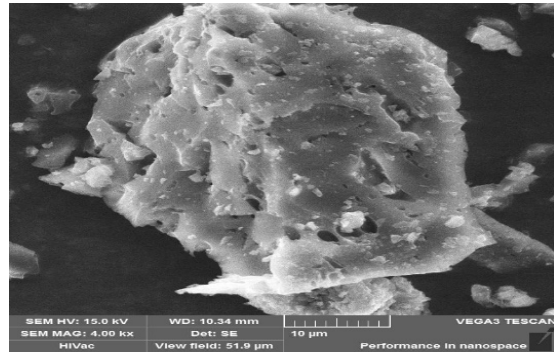


Figure 4: Surface morphology scale by sem of graphene powder from coconut shell charcoal with 4000x magnification

Based on the results of SEM analysis, it is seen that the formation of graphene layers piled on the 4000x scale (Supeno and Siburian, 2018) was formed due to the reduction of carbon on the surface of coconut shell charcoal.

#### 3.2.3 Analysis of Composite Graphene Powder from Coconut Shell Charcoal

To analyze impurity phase and to determine the composition of the elements contained in the material used X-Ray Fluorescence (XRF) which can analyze elements of Sodium to Uranium. However, testing using XRF has not been able to measure the percentage of the main content of graphene in the form of C, H, O, N, and S because, it has a lower atomic number than Sodium.

Coconut shell has a lot of lignin, so it is common to find a lot of potassium content from the resulting graphene powder. Other impurities such as Sulfur and Phosphorus are natural materials which are also found in natural materials such as coconut shells (Campbell, 2006).

#### 3.2.4 Adsorption-Desorption Nitrogen Isotherm Analysis of Graphene Powder from Coconut Shell Charcoal

To measure porosity of mesopore graphene material and pore size distribution, isotherm adsorption-desorption of nitrogen is carried out. Graphic curve adsorption-desorption of graphene powder with the BJH method can be seen in Figure 5.

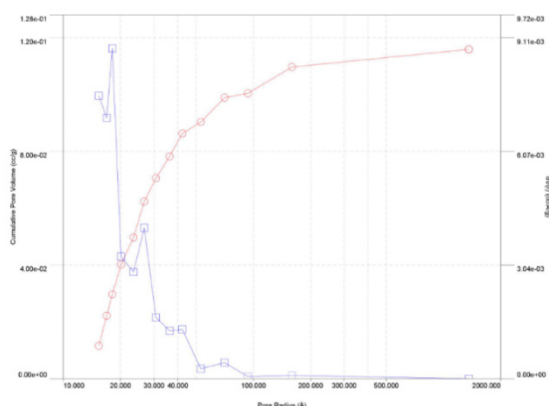


Figure 5: Adsorption-desorption curve of graphene powder by BJH method

Figure 5 showed type VI adsorption isotherm according to the IUPAC classification. The non-uniform surface of graphene to produce Type VI is a very homogeneous and non-porous characteristic of two-dimensional solids. To find out the graph of the distribution of pore size and surface area adsorption, the Barrett-Joyner-Halenda (BJH) method can be seen in Figure 6.

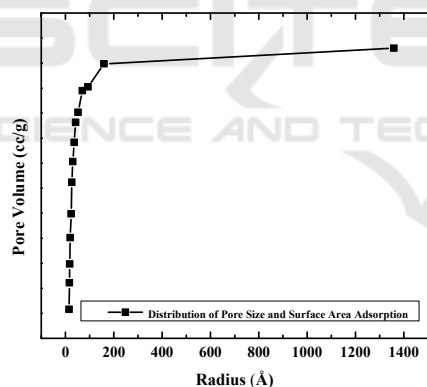


Figure 6: Distribution of pore size and surface area adsorption by Barret-Joyner-Halenda method

Based on Figure 6 graphene powder from coconut shell charcoal absorbs nitrogen gas (adsorption isotherm) on the surface of the sample at low pressure. The results of adsorption- desorption analysis of nitrogen powder gas graphene BJH method produce the surface area and pore size shown in Table 4.1

Table 1: The results of adsorption- desorption analysis of nitrogen powder gas graphene BJH method

Adsorption-desorption data	Graphene
Surface area	82.873 m <sup>2</sup> /g
Pore volume	0.116 cc/g
Pore radius	1.8098 nm

### 3.3 Structure Analysis of Graphene Powder from Coconut Shell Charcoal by Adding KOH 1 N Solution

Graphene powder from coconut shell charcoal with the addition of 1 N. KOH solution. Analysis of the structure and phase of graphene from coconut shell charcoal using X-Ray Diffraction (XRD) and BET.

#### 3.3.1 Analysis of XRD Graphene Powder from Coconut Shell Charcoal by Adding KOH 1 N Solution

The results of XRD diffraction analysis of graphene powder from coconut shell charcoal can be seen in Figure 7.

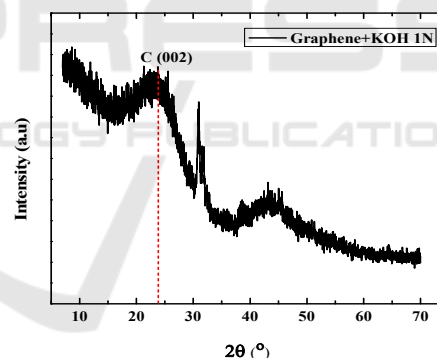


Figure 7: XRD from graphene powder with adding KOH 1 N from coconut shell charcoal

The resulting diffraction peak is at 24°. The resulting diffraction peak is weak and wide which indicates formation of graphene. The effect of adding KOH to activate graphene produces a large surface area and pore size.

#### 3.3.2 Adsorption-desorption Nitrogen Isotherm Analysis of Graphene Powder from Coconut Shell Charcoal byA KOH 1 N

To measure the porosity of mesopore graphene material by adding KOH 1 N solution and pore size

distribution, adsorption-desorption nitrogen isotherm analysis carried out. Potassium hydroxide as an activator solution played an important role in yield results. The presence of KOH during activation results in degradation of the material that will form the pore. Graphic curve adsorption-desorption of graphene powder with the addition of KOH 1 N with the BJH method can be seen in Figure 8.

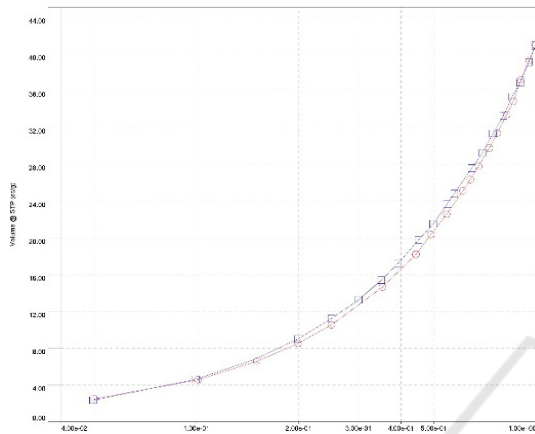


Figure 8: Graphic curve adsorption-desorption of graphene powder with the addition of KOH 1 N with the BJH method

From Figure 8 showed type IV adsorption isotherm according to IUPAC classification. From the graph of adsorption-desorption of nitrogen isotherm from graphene with the addition of KOH 1 N. The results showed that the porous material subjected to nitrogen gas was included in the mesoporous category. The results of gas adsorption-desorption analysis nitrogen graphene powder with the addition of KOH 1 N with the BJH method produced the surface area and pore size shown in Table 2

Table 2: The results of gas adsorption-desorption analysis nitrogen graphene powder with the addition of KOH 1 N with BJH method produced surface area and pore size

Adsorption-desorption	Graphene
Surface area	40.494 m <sup>2</sup> /g
Pore volume	0.060 cc/g

From Table 2, it can be concluded, in this activation process carbon will react with KOH so that carbon will be eroded (forming holes) resulting in the formation of pores. In this experiment the pore size and surface area were smaller than before the addition of KOH 1 N, graphene surface area 82.873 m<sup>2</sup> / g and the graphene pore size 1.8098 nm. The

researcher thinks this is because the concentration of the activating solution is low and between the substances reacting between the mixed substances do not touch each other so that it produces a small surface area and pore size (Erliana, 2015).

## 4 CONCLUSIONS

Based on the results of the experiment conducted, it can be concluded as follows: Graphene can be synthesized from coconut shell charcoal using activated carbon as a reducing agent. The results of characterization by XRD analysis showed diffraction peaks at 24°. The results of SEM-EDX analysis at 4000x magnification showed smaller, thinner surface sizes and structural shapes and reduced build-up in the graphene structure. XRF analysis results showed that there are still organic impurities. The results of graphene analysis with BET showed the surface area of graphene 82.873 m<sup>2</sup>/g with a pore volume 0.116 cc/ g. Activated carbon can reduce graphene oxide to graphene.

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