

# Model of Formation Graphene from Graphite with Ammonia

H. Sitohang<sup>1</sup>, N. Pasaribu<sup>1</sup>, R. Siburian<sup>1\*</sup> C. Simanjuntak<sup>1</sup>

<sup>1</sup>Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Sumatera Utara, Medan

Keywords: Graphene, Graphite, FTIR, Hummer's Method.

Abstract: The reaction mechanism of commercial graphite using ammonia reducing agents has been carried out based on Hummer's modification method. The purpose of this study was to determine the reaction model of graphene formation and characterization using an infrared spectrophotometer (FTIR). The results showed that the characterization using infrared spectrophotometer (FTIR) analysis on graphene oxide showed a ring consisting of three one oxygen atom or called an epoxy group (C-O-C) at a number wave  $1396\text{ cm}^{-1}$  which reacted with ammonia thus replaced by the availability of CN groups in wave numbers  $1033\text{ cm}^{-1}$  on graphene. This happens due to the deoxygenation process. These data proved that graphene can be synthesized from graphite using ammonia reducing agents.

## 1 INTRODUCTION

Graphene is believed to be a promising material in the future (1). Graphene is a two-dimensional carbon from the structure of graphite. Graphene has various extraordinary properties, such as mechanical resistance, strength and elasticity, thermal and electrical conductivity and high surface area (2,3). That is, graphene can replace other materials and applications. By laboratory, graphene can be produced from a simple, high-quality and inexpensive method. Graphene characterization produced approaches the theoretical parameters namely electron mobility ( $2.5 \times 10^5\text{ cm}^2\text{ V}^{-1}\text{s}^{-1}$ ) (4), Young's modulus 1 TPa and 130 GPa intrinsic strength (5,6), high thermal conductivity ( $> 3000\text{ W mK}^{-1}$ ) (7), optical absorption  $\pi\alpha = 2.3\%$  ( $\alpha =$  structural stability) (8), can react with various gases (9), ability high density of electric current (1000 times that of Cu) (10) and has a functional chemistry (11). Graphene is also called a magical material (11) and can be deposited on special substrates such as hexagonal boron nitride (4, 13). Thus, graphene will be of particular concern to be applied to the industry. Thus, the production of graphene on a large scale becomes the target of scientists.

## 2 MATERIALS AND METHODS

### 2.1 Materials

The materials used are: Graphite; strong acids, oxidizing agents and ammonia reducing agents.

### 2.2 Synthesis of Graphene Oxide

A total of 0.2 g of graphite powder were fed into a 250 mL erlenmeyer, then 0.2 g of NaNO<sub>3</sub> and 15 mL of 96% H<sub>2</sub>SO<sub>4</sub> were added. The solution is stirred for 2 hours. Furthermore, the Erlenmeyer containing the mixture was placed in an ice container and added gradually 1 gram of KMnO<sub>4</sub> then stirred for 24 hours. After stirring for 24 hours, 20 mL 5% H<sub>2</sub>SO<sub>4</sub> and 1 mL H<sub>2</sub>O<sub>2</sub> 30% were added to the solution and stirred for 1 hour. The solution is confused with a speed centrifuge of 6500 RPM for 20 minutes to separate the filtrate and supernatant. Then into the solution added 25 ml distilled water and messed up using a centrifuge with a speed of 6500 RPM (Rotor PerMinute) for 20 minutes. The solution was transferred to a beaker glass and added with 100 mL distilled water and then ultrasonicated for 5 hours, then allowed to cool and produced graphene oxide.

### 2.3 Synthesis of Graphene

The second graphene oxide solution was added with 10 ml of ammonia then stirred for 72 hours. The solution was filtered and dried at 80°C for 24 hours and characterized by using XRD, FTIR, and SEM-EDX.

## 3 RESULTS

Characterization using FTIR was carried out to determine changes in functional groups before and after the oxidation process in graphite, the synthesis of graphite oxide, graphene oxide and graphene and to determine the interaction between graphene and ammonia reducing agents. FT-IR analysis using Shimadzu IR Prestige-21 tool was carried out in the wave number range of 500-4500  $\text{cm}^{-1}$ . All samples were analyzed in the form of smooth curls. In Figure 1 shows that the peak wavelength FT-IR has occurred in graphite, graphene oxide and graphene.

In the Graphite FT-IR spectrum, the wave number 1581  $\text{cm}^{-1}$  shows the bond between the aromatic group C = C, and the presence of a support group in another wave number that appears at peak 1026  $\text{cm}^{-1}$  which is the bond between C-C. Based on the data in Figure 1 it can be seen that graphite only has the structure C = C ( $\pi$  bond) and C-C.

In the FT-IR spectrum of Graphene Oxide has formed bonds between oxygen and hydrogen (OH) groups occurring at wave numbers 3402  $\text{cm}^{-1}$ , a weak absorption spectrum occurring at wave numbers 1705.07  $\text{cm}^{-1}$  indicates the bond between C = O of the carboxylic acid group (COOH), the spectrum of the wave number is formed by the aromatic group C = C in the wave number 1581  $\text{cm}^{-1}$  and there is also a weak spectrum at wave number 1396  $\text{cm}^{-1}$  indicating the bond between C-OH or CO (epoxy) bond at 900-1300  $\text{cm}^{-1}$ .

In the Graphene FT-IT spectrum shows the appearance of absorption peaks in the wave number 1033  $\text{cm}^{-1}$  which indicates the bond between C-N. The wavelength range ~ 900  $\text{cm}^{-1}$  is the range of wave numbers for epoxy groups (C-O) (Li, et al, 2008). Based on these data it can be seen that there has been a de-epoxidation process. However, the stretch vibration of O-H at 3410  $\text{cm}^{-1}$  and the stretching vibration of C-O at 1404  $\text{cm}^{-1}$  are still observed due to the presence of hydroxyl groups and carboxylic groups even after being reduced by ammonia. Synthesis of graphene oxide from graphite has occurred the process of peeling graphite oxide into graphene oxide by ultrasonication of graphite oxide.

The mechanism of the oxidation reaction can be stated in equations 1.1 (a) and 1.1 (b).

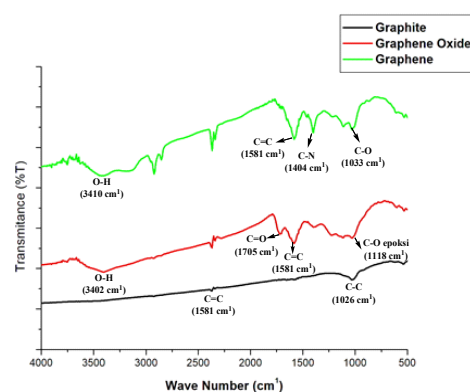
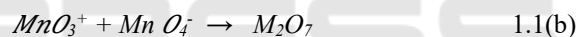
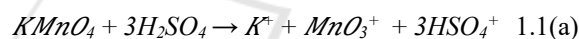


Figure 1: FTIR data of Graphite, Graphene Oxide and Graphene.

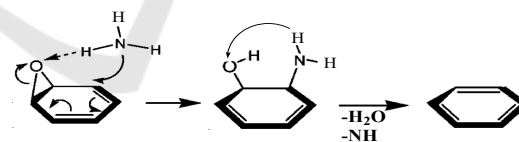
This transformation process can only occur under strong acid conditions, so the presence of sulfuric acid as well as solvent from graphite also plays a role in further oxidation processes.



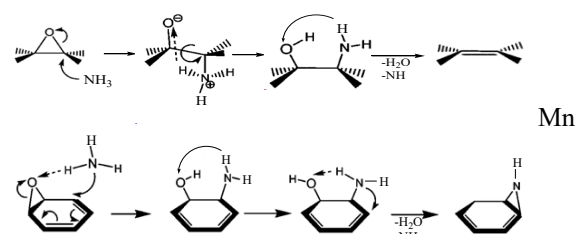
(Rizkietal, 2014).

Mechanism of reaction between epoxy and ammonia:

#### Phase 1:

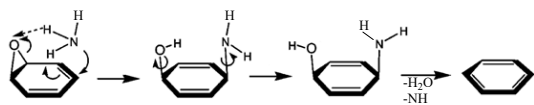


#### Phase 2:



Mn

**Phase 3 :**



**4 CONCLUSION**

Synthesis of graphene oxide with the addition of ammonia peroxide to produce graphene from commercial graphite has been successfully carried out with the modified Hummer's method. The success of this synthesis is evidenced by the presence of FTIR data.

Graphene formation reaction model of graphite and graphene oxide reduction has been obtained according to FTIR data.

**ACKNOWLEDGEMENT**

We would like to thank for “TALENTA”, Universitas Sumatera Utara, No. 2590/UN5.1 R/PPM/2017, 16 March 2018 who funding supported on our research.

**REFERENCES**

Novoselov, K.S., Fal'ko, V.I., Colombo, L., Gellert, P.R., Schwab, M.G. and Kim, K. 2012. A road map for graphene. *Nature*. 490: 192– 200.

Geim, A. K., and Novoselov, K. S. 2007. The rise of graphene. *Nature Mater*. 6: 183–191.

Geim, A. K. 2009. Graphene: status and prospects. *Science*. 324: 1530–1534.

Mayorov, A.S.et al. 2011.Micrometer-scale ball is tic transport in encapsulated graphene at room temperature. *Nano Lett*. 11: 2396–2399.

Lee, C., Wei, X.D., Kysar, J.W.and Hone, J.2008. Measurement of the elastic properties and intrinsic strength of monolayer graphene. *Science*. 321: 385–388.

Liu, F., Ming, P. M. and Li, J. 2007. Ab initio calculation of ideal strength and phonon in stability of grapheme under tension. *Phys. Rev. B*. 76: 064120.

Balandin, A.A. 2011. Thermal properties of grapheme and nanostructured carbon materials. *Nature Mater*. 10: 569–581.

Nair, R.R.et al. 2010. Fluoro graphene: a two-dimensional counter part of Teflon. *Small*. 6:2877–2884.

Bunch, J.S.et al. 2008. Impermeable atomic membranes from grapheme sheets. *Nano Lett*. 8:2458–2462.

Moser, J., Barreiro, A. and Bachtold, A. 2007. Current-induced cleaning of graphene. *Appl. Phys. Lett*. 91: 163513.

Elias, D.C.et al. 2009. Control of graphene's properties by reversible hydrogenation: evidence for graphene. *Science*.323: 610–613.

Novoselov, K.S. et al. 2004. Electric field effect in atomically thin carbon films. *Science*.306:666–669.

Dean, C.R. et al. 2010. Boron nitride substrates for high-quality grapheme electronics. *Nature Nanotechnology*. 5, 722–726.