©2019 Published in 7th International Symposium on Innovative Technologies in Engineering and Science 22-24 November 2019 (ISITES2019 SanliUrfa - Turkey)
https://doi.org/10.33793/acperpro.02.03.59

Obtaining of Reduced Graphene Oxide from Graphite by using Hummer's and Chemical Reduction Method

[1]Kübra YILDIZ And *[2]Muhammet UZUN
[1]Harran University, Vocational School Of Technical Sciences, Department Of Textile Technology, Şanlıurfa Turkey
*[2] Marmara University, Faculty Of Technology, Department Of Textile Engineering, Istanbul Turkey

Abstract

In this study, graphene oxide (GO) was synthesized from graphite using modified Hummers method. According to other methods known in the literature, modified Hummers method; it is simpler and less costly in terms of process steps.
In addition, it is safer and environmentally friendly than the Hummers method. Reduced Graphene Oxide (RGO) was obtained by reduction of graphene oxides (GO) synthesized by modified Hummers method.
It is understood from the obtained results that GO is synthesized successfully from graphite powder by modified Hummers method and RGO is obtained successfully by reduction of graphene oxides (GO).

Key words: Graphene, Graphene Oxide, Hummers Method, Reduced Graphene Oxide

1. Introduction

Its unique properties such as high flexibility, mechanical strength, thermal conductivity, high electrical conductivity and transparency make graphene an interesting material [1-3]. Graphene is the basic building block of all other graphitic materials of different sizes, packaged in a two-dimensional honeycomb cage consisting of a uniform, monolayer sp2 hybrid honeycomb appearance of covalent bonded carbon atoms [4, 5]. The distance between two carbon atoms in the graphene layer is 1.42 Å (0.42 nm), which makes it transparent in a single layer and provides excellent conductivity [6].

The carbon atom has four different allotropes in nature: fullerene, carbon nanotube, graphite and graphene. The properties of carbon allotropes are given in Table 1 comparatively [7, 8].

Table 1. Comparison of properties of carbon allotropes [7, 8]

Feature	Graphene	CarbonNanotube	Graphite F	Fullerene
Surface area (m^2 / g)	2630	1315	10	5
Thermal Conductivity (W/mK)	5000	>3000	3000	0,4
Mobility	15,000 SiO over	100000	13000	0,56

*Corresponding author: Address: Harran University, Vocational School Of Technical Sciences, Department Of Textile Technology, Şanlıurfa TURKEY. E-mail address: kubra.yildiz@harran.edu.tr

(Cm^2 / Vs)	200,000 free state			
Young module (TPa)	1	0,64	1,06	0,01
Optical Permeability (%)	97,7	-	-	-

2. Materials and Method

2.1. Material

The all materials required for the GO synthesis using the modified Hummers method were provided. Graphite powder (<20 micron), hydrogen peroxide (H2O2), sulfuric acid (H2SO4) were obtained from Sigma Aldrich company and sodium nitrate (NaNO3), potassium permanganate (KMnO4) were obtained from Merck company. The materials required for the reduction from GO (NH2 NH2.H2O) to graphene, Hydrazine hydrate and ethanol (CH3CH2OH) were obtained from Sigma Aldrich company.

GRAFEN®-İGP 2 / Industrial Graphene Nano platelets for General Purposes was obtained from the Graphene Chemical Industries Company (Grafen Co.). The density of this material was 0.05 g / cm^3, the thickness of it was 5 - 10 nm. and the diameter of it was 5 - 10 μm.

2.2. Method

2.2.1. Graphene Oxide Production

Graphite powder was converted to graphene oxide using modernized Hummers method [5]. 5 g of graphite, 2.5 g of sodium nitrate ($NaNO_3$) and 115 ml of sulfuric acid (H_2SO_4) was added into the ice bath and stirred for 1 hour. 15 g of potassium permanganate ($KMnO_4$) was added to the mixture in the ice bath slowly. Meanwhile, during the addition attention should be paid that the temperature was below 5 °C. the mixture removed from the ice bath and it was stirred for 2 hours. The solution temperature had to be range between 35 - 40 °C.

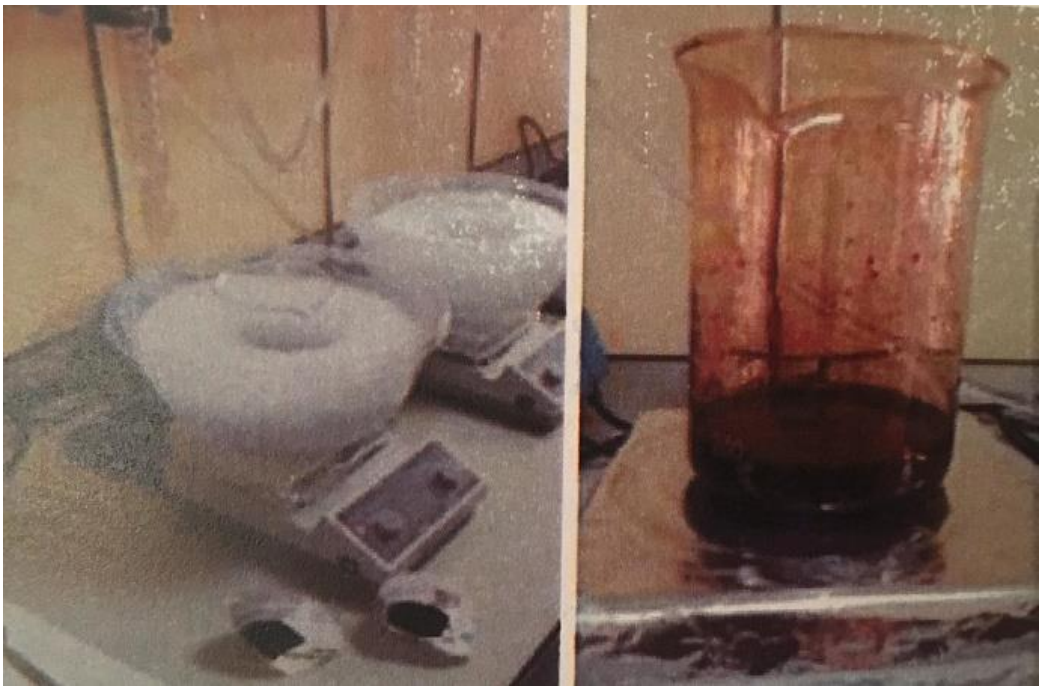

Figure 1. GO Synthesis Stages1

500 ml of deionized water was slowly added to the solution and stirring was continued for 1 hour 8.4 ml of hydrogen peroxide (35.7'lik% H_2O_2) was added to the solution and solution was stirred for 2 hours at a temperature that was not exceeding 40 °C. The colour of the solution turned to brown to black at this stage. At the end of this process, solution was washed with deionized water and filtered. After repeating filtration for several times, remaining material was dried in an oven at 50 ° C for 24 hours and then it was obtained as a powder GO.

Figure 2. GO Synthesis Stages 2

2.2.2. Reduced Graphene Oxide (Graphene) Production

The graphite is first subjected to oxidation in aqueous medium, then the layers are separated and then separated by oxygen, chemical or thermal combined reduction processes to obtain products close to the original structure of graphene.

1 gr GO was introduced into 300 ml of deionized water and dispersed in an ultrasonic bath for 2 hours. Subsequently, 30 ml of hydrazine hydrate was slowly added to the solution. This mixture was subjected to the mixing in a magnetic stirrer at 95 ^{0}C for 24 hours depending on the reflux system. Then the solution was filtered. After filtering once with acetone or ethanol, it was filtered with deionized water until pH was about 6-7. Filtering finished material was left to stand in an oven at 50 ^{0}C for 24 hours. At the end, reduced graphene oxide was obtained.

Table 1. RGO Synthesis Stages

1. stage	2. stage	3. stage	4. stage
Ultrasonic bath at 30 ^{0}C 300 ml Pure Water 1 g GO 2 hours	Magnetic stirrer 95 ^{0}C Hydrazine hydrate 25 Rpm 24 hours	Filtration 1 time Acetone or Ethanol, Deionized Water pH 6-7	Drying Oven at 50 ^{0}C 24 hours

3. Conclusions

As a result of all the measurement results, In SEM analysis, images of layered GO structures were obtained and layered structures were determined. EDX Elemental analysis showed that functional groups were formed in GO structure. The loss of mass in the TGA analysis shows that hydroxyl and oxygen containing groups decay from the structure. Raman spectroscopy showed characteristic peaks of GO. The characteristic peaks of GO were observed on FT-IR spectroscopy. The XRD analysis showed that GO was successfully synthesized from graphite powder by modified Hummers method. Subsequently reduced graphene oxide (RGO) was obtained by reduction method from graphene oxide (GO) using hydrazine hydrate. SEM images of RGO showed randomly distributed thin layers with wrinkled, curved surfaces and similar images. At the same time, according to EDX elemental analysis results, it is seen that the percentage of carbon increases because the functional groups are removed by the conversion.

Reference

[1] J. E. An, G. W. Jeon ve Y. G. Jeong, "Preparation and properties of polypropylene nanocomposites reinforced with exfoliated graphene", Fibers And Polymers, cilt 13, no. 4, pp. 507-514, 2012.

[2] Y. J. Yun, W. G. Hong, W. J. Kim, Y. Yun ve B. H. Kim, "A novel method for applying reduced graphene oxide directly to electronic textiles from yarns to fabrics", Advanced Materials, no. 25, pp. 5701-5705, 2013.

[3] E. Skrzetuska, M. Puchalski ve I. Krucińska,, "Chemically driven printed textile sensors based on graphene and carbon nanotubes", Sensors, no. 14, pp. 16816-16828, 2014.

[4] M. Arseven, "Polikristalin bakır folyo üzerinde grafen sentezi", Ankara: Yüksek Lisans Tezi, Fen Bilimleri Enstitüsü, Hacettepe Üniversitesi, 2011.

[5] C. Baykasoğlu ve A. Muğan, "Tek katmanlı bir grafen tabakasının kırılma davranışının incelenmesi," 17-18 Haziran 2011, 1. Ulusal Nanoteknoloji Kongresi, İstanbul, 2011.

[6] B. Kozal, "Karbon tabanlı petek örgülerin elektronik özellikleri", Doktora Tezi, Fen Bilimleri Enstitüsü, Ankara Üniversitesi, 2012.

[7] M. C. Hsiao, S. H. Liao, Y. F. Lin, C. A. Wang, N. W. Pu, H. M. Tsai ve C. C. M. Ma, "Preparation and characterization of polypropylene-grafted-termally reduced graphite oxide with an improved compatibility with polypropylene-based nanocomposite", Nanoscala, no. 3, pp. 1516-1522, 2011.

[8] H. J. Choi, S. M. Jung, J. M. Seo, D. W. Chang, L. Daic ve L. B. Baek, "Graphene for energy conversion and storage in fuel cells and supercapacitors", Nano Energy, no. 1, pp. 534-551, 2012.