

## Graphene Oxide Effect on the Properties of Nickel Oxide-Graphene Oxide (NiO-GO) Composite

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### Abstract

Nickel oxide matrix graphene oxide (NiO-GO) composite was prepared and properties were studied to observe the effect of GO content. Here, Graphene oxide was synthesized by using modified Hummers method and NiO-GO composite was prepared by Hydrothermal method with the use of graphene oxide solution,  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and urea as raw materials. Composites were prepared in different NiO-GO ratio (NiO: GO = 1:1, 2:1) and comparative studies were done with pure nickel oxide. The synthesized composites were characterized by SEM, XRD and Impedance analyzer. The results demonstrated that GO particles are well oriented on the NiO layer. The XRD peaks confirmed GO with diffraction angle at  $10^\circ$ . The particle size was just above the nanometer range so purely nanoparticles were not obtained. Incorporation of GO content in the composites had improved the properties. The capacitive reactance is better in the composite than pure NiO. The conductance of the composites were higher in NiO-GO composite. Increasing GO content has increased the conductance. Resistance decreased to a large extent with increasing GO content. Susceptance of the composite was higher than pure NiO. This composite can be used as a capacitor with high capacitance instead of traditional capacitor and also can be used as an electrode as a replacement of carbon cathode in a battery. Depending on the application, the GO content may vary in the composite.

Keywords: Graphene oxide, Capacitor, Nickel oxide, Carbon cathode, Electrical Properties.

### 1. Introduction

Graphene first made noise among scientific community in 2004 when two researchers of Manchester University, Professor Andre Geim and Professor Kostav Novoselov, reported the observation of electric field effect in atomically thin two dimensional (2D) material referred to as few layers Graphene (FLG). Graphene is a rapidly rising star on the horizon of materials science and condensed-matter physics. This strictly two-dimensional material exhibits exceptionally high crystal and electronic quality. Graphene offers new inroads into low-dimensional physics that has never ceased to surprise and continues to provide a fertile ground for applications [1]. A lot of scientists said that graphene would be a competitive material for energy storage applications like batteries, solar cells and super capacitors etc. [2-3]. Graphene Oxide has emerged as a precursor offering the potential cost-effective, large scale production of graphene-based materials [4]. At present, the researchers find great interest in adding GO with different metallic oxide, especially transitional elements to enhance properties such as graphene-NiO, graphene-MnO<sub>2</sub>, graphene-Mn<sub>3</sub>O<sub>4</sub>, graphene-Bi<sub>2</sub>O<sub>3</sub>, graphene- Co<sub>3</sub>O<sub>4</sub> and graphene- ZnO have been exploited and improved performance of pseudo-supercapacitor [5]. Supercapacitors can be generally classified into two categories following the charge storage mechanism, of which they are electric double layer capacitors (EDLC) and pseudocapacitors. This is because EDLC only store charges at the electrode and electrolyte interface, it provides a low energy density compared with their theoretical value, which dramatically limits their widely applications [6-7]. In contrast, pseudocapacitors delivers 3-4 times higher capacitance [8-9]. The electrochemical method has an advantage of controllability but is not suitable for mass production [10]. NiO electrode has high resistivity, which is a serious drawback to apply for practical applications to supercapacitors. It is crucial to enhance the electrode conductivity in order to improve the energy density and power density of electrodes. Moreover, the specific surface area of electrodes is directly related to the specific capacitance. However, the specific surface area of the NiO is in general not high enough for high capacitance. Graphene has been known to yield high conductivity and large specific surface area [11-12]. In this experiment, the main focus is to see the effect of GO in NiO in various ratio and to observe the difference in various properties.

### 2. Experimental procedure

Graphene oxide was synthesized using Modified Hummers Method. Initially graphite (10g) and  $\text{NaNO}_3$  (5g) were mixed in 250 mL of  $\text{H}_2\text{SO}_4$  (98%) in a volumetric flask and kept under continuous stirring for 25 minutes and cooled to  $0^\circ\text{C}$  by putting it in an ice bath.  $\text{KMnO}_4$  (30g) was added to this mixture at a rate so that the temperature remained under  $6^\circ\text{C}$ . The solution was continuously stirred using a glass rod. Ice bath was removed then and stirred for 30 minutes until the mixture became pasty brown color. Then maintaining a temperature of  $70^\circ\text{C}$  on a hot plate, it was heated for 1 hour. Deionized water (200 ml) was added slowly and carefully. The exothermic reaction produced much heat. The temperature was kept to  $98^\circ\text{C}$  for 20 minutes. The mixture became brown. The solution was diluted with water (500 ml). 40 ml  $\text{H}_2\text{O}_2$  (30%) was added to this solution and stirred at  $70^\circ\text{C}$  for 2 hrs.  $\text{H}_2\text{O}_2$  was added for complete removal of  $\text{KMnO}_4$ . The solution was filtered to form a solid cake and then washed with water. The solid cake was separated from the filter paper. Centrifugation was done at 8000 rpm for 15 minutes. Obtained precipitate was mixed with Deionized (DI) water. The mixture was ultrasonicated for 45 minutes to form homogeneous dispersion of solute and to remove additional impurities. Then it was dried at  $70^\circ\text{C}$  for 27 hours to get hard solid graphene oxide (GO). Finally grinding was performed to obtain desired GO powder.

The composites were produced using hydrothermal method. 1.5g  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  was dissolved in DI water and added into 20 ml graphene oxide (1.5g) and stirred for 30 minutes. 20 ml urea was added to maintain the pH. Then the solution was heated for 5 hours at  $120^\circ\text{C}$ . The precursor was taken out and washed with DI water for 3 times to remove the probable absorbed ions, metal salts and remained raw material, then dried at  $80^\circ\text{C}$  for 12 hrs. Obtained precursor was heated then at  $400^\circ\text{C}$  for 4hrs in the furnace. The NiO-GO nanocomposite was obtained as powder after grinding. It was heated in such way that protects the contamination in the furnace.

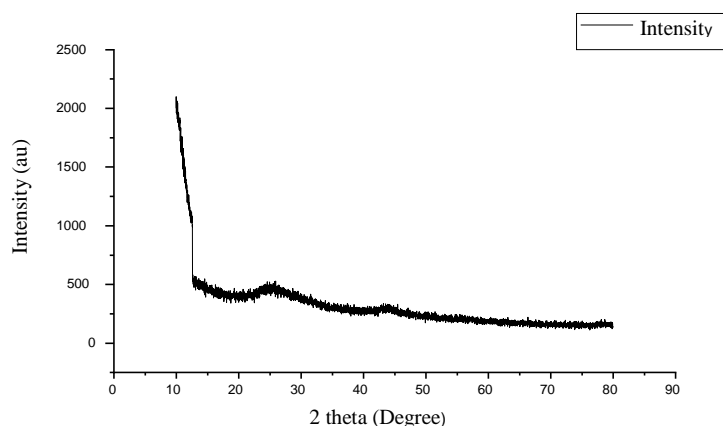
Amount of NiO was calculated here as the amount of Nickel Nitrate Hexahydrate. This composite contained 50% graphene oxide. Another composite were prepared with 33.3% GO content. Hydrothermal method was used here only changing the amount of Nickel nitrate hexahydrate.

### 3. Characterization

The obtained samples are characterized by X-Ray Diffraction (XRD), Capacitance test, Resistance test, Conductance test and Field Emission Scanning Electron Microscopy (FESEM).

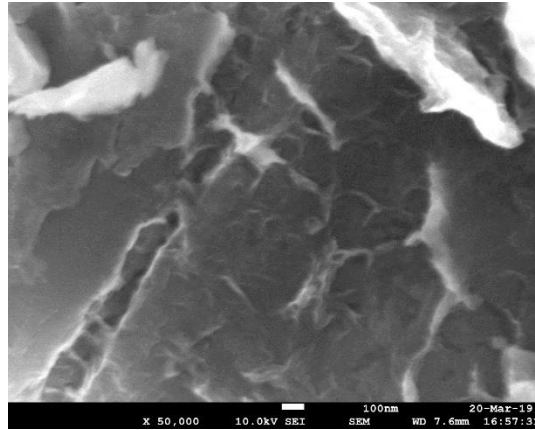
### 4. Results and discussion

X-Ray Diffraction analysis is done to know the crystal phase and structure of the synthesized samples. From Fig.1 the peak is seen of the XRD spectra of pure GO. The characteristics picks of pure GO to be found diffraction angle  $2\theta = 10$  degree. As the angle has started from 10 degree, the previous portion from 10 degree in the graph is not seen unfortunately. From the graph it is clear that, the particles in pure graphene oxide is well oriented as there is no other sharp peak here. And the crystal structure is as expected. The spectra isn't smooth everywhere because of moisture.



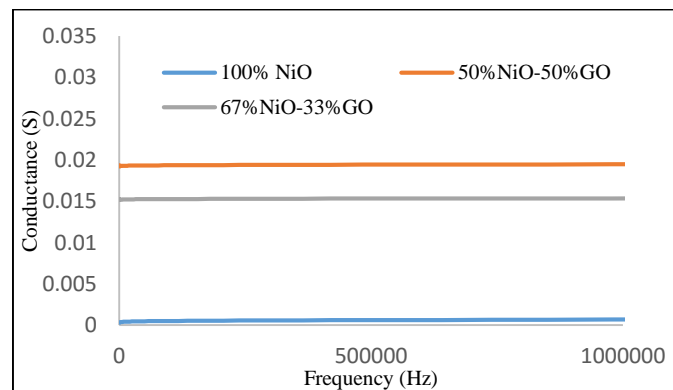
**Fig. 1.** XRD spectra of Graphene Oxide

The microstructure, morphology and particle size of synthesized NiO-GO composites are observed by SEM. In Fig.2, the SEM images of the particles show that the particles are well oriented and NiO-GO particles are aggregated together.



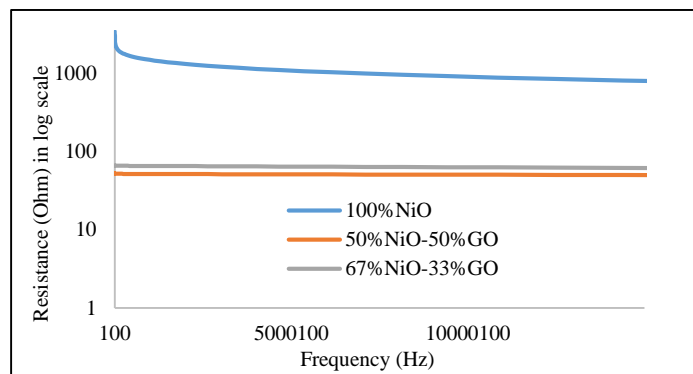
**Fig. 2.** SEM image of NiO-GO composite.

From the concept of conductance, we know that, higher the conductance, better the capacitor. In Fig.3 we can see that the conductance of the pure NiO is the lowest. With the increasing in GO content, the conductance of the composites are increased. With 50% GO content, it shows the highest conductance value whereas pure NiO has the lowest value.



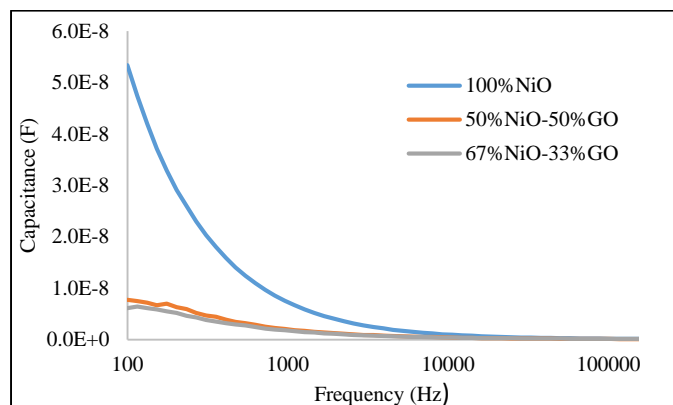
**Fig. 3.** Conductance vs Frequency graph

The resistance is a material property. So it should remain constant under any condition for a specific material at a fixed temperature. Form Fig.4 we can see that, pure NiO has the highest resistance whereas composite with 50% GO has the lowest. With increasing GO content, the resistance is decreasing.



**Fig.4.** Resistance vs Frequency graph

Capacitance decreases with increasing frequency. From Fig.5, it is clear that the capacitance is decreasing with increasing frequency. The capacitance is lowest for the composite containing NiO: GO=2:1. With increasing GO content, the capacitance is decreasing with increasing frequency.



**Fig.5.** Capacitance vs Frequency graph

## 5. Conclusion

Synthesis of Graphene oxide was done by using modified hummers method and NiO-GO composites have been effectively prepared by hydrothermal method. XRD analysis specified that the produced precursor is GO. Impedance analysis specified that presence of GO has increased the properties of the composites and percentage of GO content in the NiO-GO composites plays an effective role in increasing the properties.

## 6. Acknowledgement

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