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Synthesis and Characterization of Few-Layer Reduced Graphene Oxide Nanosheet by Modified Hummer's Method

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The present study reveals that synthesized Graphene oxide (GO) by using a modified Hummer's method and then by applying heat treatment yielded reduced Graphene oxide (rGO). The phase purity and crystal structure of the synthesized rGO was determined using X-ray diffraction (XRD). Which confirms the production of reduced graphene oxide. Also, Scherrer's equation was used for average crystallite size calculation. Fourier transform infrared spectroscopy (FTIR) was used to identify the molecular vibrations and functional groups which confirms the presence of C=C and C-O bonds in the sample. The surface morphology of the produced samples was investigated using a field emission scanning electron microscope (FESEM), which revealed that the surface of the rGO sheet is smooth. The optical characteristics are measured using a UV-Visible spectrometer. The band gap is calculated by the tauc plot analysis. the band gap of the sample is found to be 1.92 eV. In this study, efforts were attempted to manufacture reduced graphene oxide nanosheet by modified hummer's method.

Keywords: X-ray diffraction, Hummer, Band gap.

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1. INTRODUCTION

Due to outstanding features such as high surface area (2630 m^2gm^{-1}), transparency, thermal conductivity, and good interface contact with absorbents, graphitic nano-carbons such as graphene, graphene oxide, and reduced graphene oxide have garnered substantial attention in recent decades [1-2]. A small number of electrons produce a remarkable change in the conductivity of graphene because of its high electrical conductivity, high carrier mobility, and extremely low electrical noise [3]. The most frequent technique of graphite exfoliation used powerful oxidizing chemicals to produce a non-conductive hydrophilic carbon material called graphite oxide (GO) [4]. The two-dimensional hexagonal honeycomb is unique in a structure similar to that of graphene and consists of a single atom layer of sp^2 hybridized carbon atoms [5-7]. Mechanical exfoliation (scotch-tape technique) of bulk graphite initially generated transferable single-layer graphite nanosheets. In 1958 firstly Hummers developed an alternative technique for the synthesis of graphene oxide by using $KMnO_4$ and $NaNO_3$ in concentrated H_2SO_4 [8]. This method can be used to prepare large graphitic films.

2. MATERIAL AND METHOD

2.1 Chemicals and Reagents

Graphite powder, $NaNO_3$, NaOH, H_2SO_4, $K_3Fe(CN)_6$, H_2O_2, and $KMnO_4$ were purchased from Himedia and Sigma-Aldrich. All the chemical compounds were of analytical reagent grades (purity 99.9 %) and used without further purifications.

2.2 Synthesis of Reduced Graphene Oxide (RGO)

A modified Hummer's technique was used to make graphene oxide (GO) from graphite powder. 2 gm of graphite and 1 gm of sodium nitrate were combined, then 23 ml of concentrated sulphuric acid was added while constantly stirring using a magnetic stirrer. To avoid overheating and explosion, 3 g of $KMnO_4$ was progressively added to the aforementioned solution after 1 hour, while keeping the temperature below 20 °C. The solution was diluted by adding 500 ml of water to the mixture and stirring vigorously for 12 hours at 35 °C to confirm that the reaction with $KMnO_4$ was completed. The suspension was treated with 5 ml of H_2O_2 solution and HCl & H_2O were used to wash the resultant mixture via centrifugation respectively. After several times washing by centrifuge machine, filter, and drying in a hot air oven, thus graphene oxide sheets were obtained. From Graphene oxide, reduced Graphene oxide nanosheet is formed by heat treatment using a microwave oven.

3. RESULT AND DISCUSSION

XRD pattern of Reduced Graphene oxide (rGO) nanosheet is shown in Fig. 1 confirms the hexagonal graphitic structure of the carbon material according to the JCPDS Card No. 75-1621. The diffraction lines at the angle (2θ) of 26.37, 42.30, 44.47, and 54.29 are indexed to the corresponding crystalline planes of (002), (1-10), (1-11), and (400). After analyzing the most intense peak at a 26.37 angle give the average crystallite size of rGO nanoparticles by using Scherrer's equation.

$$D = \frac{K\lambda}{\beta \cos\theta}, \tag{3.1}$$

where K is a shape factor having a value of 0.94, λ is the wavelength of X-ray (1.5406 Å), β is the FWHM of the most intense peak of the XRD pattern, and θ is the diffraction angle.

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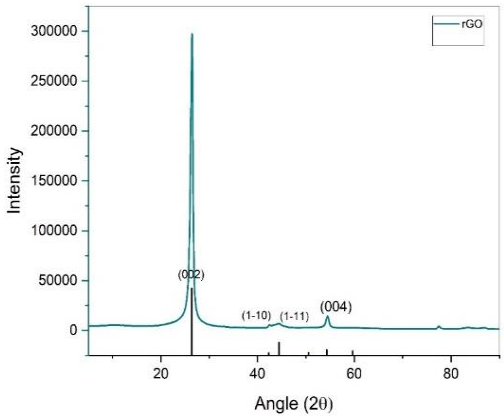

Fig. 1 – XRD pattern of rGO nanosheet

The surface morphology of rGO by FESEM analysis is shown in Fig. 2. the surface of the rGO nanosheet is found to be highly smooth. As represented in Fig. 3. Shows the EDX analysis confirms the presence of Carbon and Oxygen in the sample.

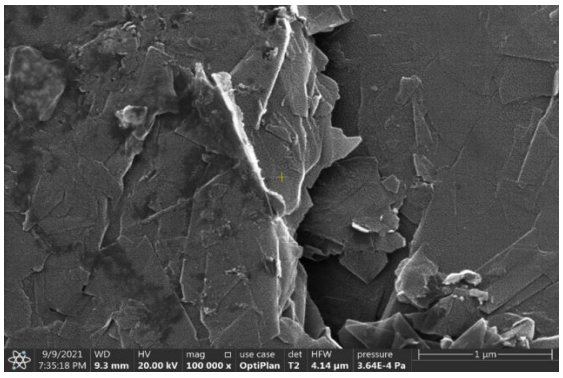

Fig. 2 – FESEM analysis of rGO nanosheet

FTIR analysis was used to identify the atomic vibrational mode of the rGO nanosheet plotted in Fig. 4. In The measured peak, the position is Tabulated in table 1. their functional group and peak position. The rGO nanosheets are covered by functional groups such as hydroxyl (-OH) [10], epoxy (C-O-C), and carboxylic (-COOH) groups.

The UV-Vis spectra for the absorption of rGO nanosheets are shown in Fig. 5. The absorption peak of the rGO nanosheet was found to be 300 nm.

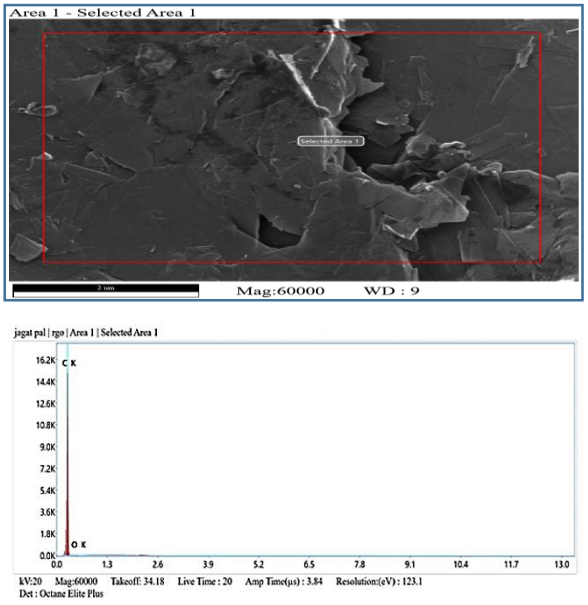

Fig. 3 – EDX analysis for confirmation of the presence of carbon and oxygen in the prepared rGO nanosheet

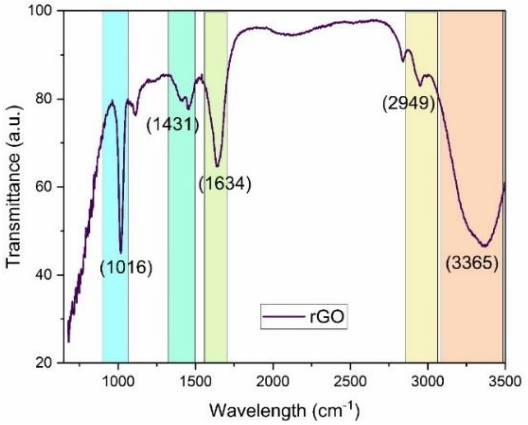

Fig. 4 – FTIR analysis of rGO nanosheet

From the absorption spectra direct optical band gap is calculated by using tauc plot analysis using the formula

$$(\alpha h\nu)^2 = N(h\nu - E_g). \qquad (3.2)$$

Where α is an absorption coefficient, h is planks constant, ν is the frequency and $h\nu$ is the incident photon energy.

Table 1 – rGO vibrational mode by FTIR analysis

Peak Position (cm^{-1})	**Functional group**	**Vibrational Mode**
1016	Bonding C-O	Stretching
1431	Alcohols (-OH)	Bending
1634	Bonding C=C	Stretching
2949	$-CH_2$	Stretching
3365	Water (C-OH)	Stretching

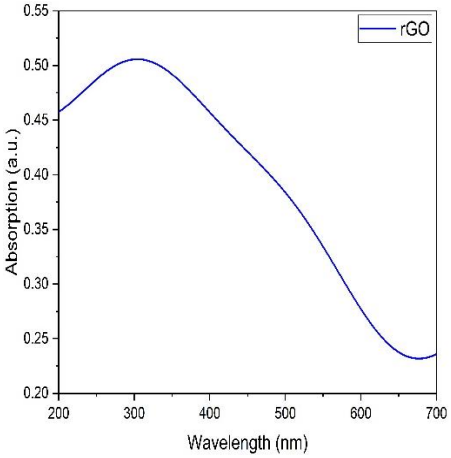

Fig. 5 – UV-Vis spectra of rGO nanosheet

The plot $(\alpha h\nu)^2$ vs. $h\nu$ extrapolating the straight line to intercept on the horizontal photon energy axis was found to be 1.92 eV as shown in Fig. 6.

4. CONCLUSION

The reduced graphene oxide (rGO) was prepared using the modified Hummer's method. The structure, morphology, and optical properties of the sample were investigated. The XRD pattern confirmed the hexagonal structure and the average crystalline size of rGO was found to be 12 nm. FESEM analysis revealed that the morphology of the rGO nanosheet is smooth. The EDS mapping showed the distribution of carbon and oxygen in the rGO nanosheets. FTIR characterization was used to substantiate the formation of possible C-O and C=O bonds to identify the functional oxygen-containing group. Tauc plot analysis gives a direct optical band gap of 1.92 eV of rGO.

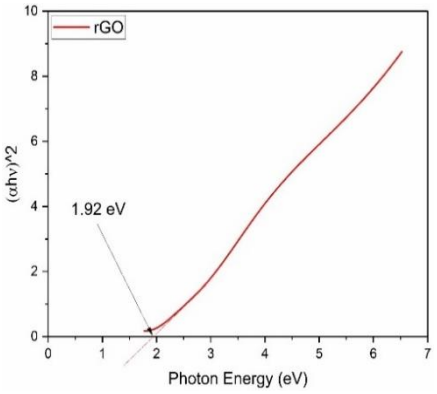

Fig. 6 – Direct optical band gap calculation by tauc plot analysis

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Синтез і характеристика кількашарового відновленого та модифікованого методом Хаммера нанолиста оксиду графену

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Дослідження показують, що синтезований за допомогою модифікованого методу Хаммера, а потім за допомогою термічної обробки оксид графену (GO) дав відновлений оксид графену (rGO). Фазову чистоту та кристалічну структуру синтезованого rGO визначали за допомогою методу рентгенівської дифракції (XRD), який підтвердив утворення відновленого оксиду графену. Крім того, рівняння Шеррера було використано для розрахунку середнього розміру кристалітів. Інфрачервона спектроскопія з перетворенням Фур'є (FTIR) була використана для ідентифікації молекулярних коливань і функціональних груп, що підтверджує наявність зв'язків C=C і C-O у зразку. Морфологію поверхні отриманих зразків досліджували за допомогою скануючого електронного мікроскопа (FESEM). Результати досліджень вказують на те, що поверхня листа rGO є гладкою. Оптичні характеристики вимірювались за допомогою УФ-видимого спектрометра. Ширина забороненої зони обчислюється за допомогою tauc plot аналізу. Ширина забороненої зони становила 1,92 еВ. У цьому дослідженні було зроблено спробу виготовити відновлений нанолист оксиду графену модифікованим методом Хаммера.

Ключові слова: Рентгенівська дифракція, Метод Хаммера, Заборонена зона.