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RESEARCH PAPER

# Fast and Facile Synthesis of Graphene Oxide Nanosheets for Commercial Purposes

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**ABSTRACT**

Everyday growth of the application of Graphene, Graphene oxide, and other materials based on graphene causes a huge demand for finding commercial and straightforward methods for synthesizing this valuable compound. Today, there are several laboratory methods for synthesizing graphene oxide nanosheets that researchers around the world use. The ability to produce on a large scale and industrially with many of these methods is a significant challenge due to the use of expensive raw materials or the production of toxic substances in the production process. The presence of these poisonous substances in the production cycle creates environmental issues. This study aimed to find an easy and inexpensive way to produce graphene oxide on an industrial scale that does not produce toxic substances, or the amount of their occurrence is minimal during the production process. In this study, graphene oxide nanosheets have been synthesized successfully by a straightforward and nontoxic method that produces no hazardous materials during the synthesizing process. The samples were analyzed using several techniques: X-ray diffraction (XRD), Raman spectroscopy, UV-Visible spectroscopy, Fourier-transform infrared spectroscopy (FTIR), and transmission electron microscopy (TEM). Comparing the results with the other researchers' works confirmed synthesizing high-quality graphene oxide nanosheets. Due to the simple and environment-friendly method, which produces no hazardous materials during the production process, the method may quickly develop for the commercial production of graphene oxide. In other words, the research proposes a new and ideal method for large-scale synthesizing graphene oxide nanosheets for commercial purposes with the lowest environmental issues.

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## INTRODUCTION

Graphene and its derivatives have received a lot of attention in recent years. These valuable materials have been used extensively, from optoelectronic devices to pharmaceuticals. Due to its extraordinary optical properties, especially its ability to absorb light in a wide range from ultraviolet (UV) to terahertz (THz), graphene, with its ultrathin two-dimensional (2D) structure, can be used in applications such as optoelectronics and nan photonics in a wide range of spectra[1]. Graphene oxide is one of the derivatives of graphene. It has attracted much attention in recent years due to its unique 2D structure, extremely large surface area, excellent stability, facile modification of surface characteristics, environmental

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compatibility, and high industrialization potential [2]. Graphene oxide is a single layer of a polycyclic hydrocarbon lattice. The graphene oxide band gap is more than 1.5 volts and depends on its oxidation measure[3]. Graphene oxide surfaces contain the functional groups of epoxy, carboxylic, hydroxyl, and carbonyl. Functional groups in the basal and peripheral areas cause a loss of order due to strong σ bonds, localized electrons π, decrease in density, and stimulation of charge carriers, generally leading to fundamental changes in the electronics properties and surface structure [4-6].

The use of graphene oxide is expanding daily because of its unique properties, such as its easy dissolving in solvents, dielectric properties, transparency, adjustable electronic properties, and superior mechanical properties [7]. Graphene oxide is beneficial for the adsorption and entrapment of drug molecules at the surface due to the single-atomic structure of the layer and increasing the hydrophilicity of insoluble drugs; it improves the drugs' better performance [8].

Graphene oxide is a good choice in superconductors due to its good performance, stability, and low cost [9]. In addition to being hydrophilic, this nanomaterial is suitable for absorbing various ions of heavy metals because of its high negative charge density and surface interaction. This property is used to remove many-colored contaminants, heavy metal ions, and oily liquids [10-12]. With better oxidation and creating more band-gap, the interaction of graphene oxide with a wide range of minerals and organics is facilitated, and applied composites with remarkable properties can be easily synthesized. By reducing graphene oxide, oxygenated functional groups lose their chemical bonds, and they rise from the surface and gradually change their insulating properties to semiconducting and semi-metal graphene-like characteristics [13-15].

In addition, many research groups have attracted nonlinear optical properties in recent years due to the π bonding of carbon atoms sp2 in graphene and its derivatives, including graphene oxide, and their use as saturated absorbers has been reported in ultra-plastic lasers. Functionalization of graphene oxide regulates the energy gap, making it a better candidate for nonlinear optical applications [13].

In order to make composites with nonlinear optical properties, the presence of graphene oxide functional groups allows them to be easily combined with nonlinear optical materials such as metal nanoparticles [16] or organic colors [17]. Graphene oxide has also been used in catalysts and sensors [18-19]. Due to the extraordinary properties and widespread use of this nanomaterial, finding high-quality, affordable synthesis methods on a large scale is very important. One of the best ways to obtain graphene oxide, without the complexity and high energy consumption and industrial-scale production, is to oxidize graphite. Graphite oxide was first produced in 1855 by Brody et al. To synthesize, potassium chlorate was added to a concentrated suspension of graphite in nitric acid vapor. It was found that the modified graphite crystals were composed of oxygen, carbon, and hydrogen and could be dispersed in water. Then, to improve the oxidation process, the amount of potassium chlorate was increased [20]. In 1950, Hammers suggested another method for synthesizing graphene oxide. This method was performed at low temperatures for approximately two hours. In this method, a mixture of potassium permanganate, potassium nitrate, sodium nitrate, and sulfuric acid was used to oxidize graphite. The Hammers' approach, with a few minor modifications called modified Hammers, is widely used in the production of graphite oxide. In this method, the use of hydrogen peroxide is critical to remove excess permanganate ions acting as contaminates [21-23].

Recently suggested a new method for the synthesis of graphene oxide. In this method, removing sodium nitrate increased the amount of potassium consumed [24-25]. Research is ongoing to produce high-quality graphene oxide. In 2020, Muniyalakshmi et al. synthesized graphene oxide nan sheets by increasing potassium permanganate and removing one of the oxidants. In this method, more permanganate ions were produced by increasing the oxidant, requiring more hydrogen peroxide to remove the contaminants, causing the reaction to be extremely exothermic [25]. In 2019, Aydin et al. succeeded in producing a nan composite based on graphene oxide. They synthesized graphene oxide using nitric acid oxidizer by modified hammers method. This oxidant produced toxic gases $NO_2$ and $N_2O_4$ [26]. In 2018, Yoo et al. examined the role of hydrogen peroxide and its effects on graphene oxide and found that hydrogen peroxide could better enhance the synthesis of graphene oxide[27]. In 2020, Zlaoui et al., after synthesizing graphene oxide by using the modified Hammers' method,

investigated this nanomaterial's electrical conductivity and dielectric properties [28].

An easy and fast method is used to synthesize graphene oxide nanosheets in the present work. In order to oxidize pure graphite and remove the vapors of the toxic gases $NO_2$ and $N_2O_4$, phosphoric acid was replaced instead of nitric acid as an oxidizer. In this way, graphene oxide is synthesized using relatively cheap materials without having a high temperature by removing the oil bath in a relatively short time.

In other words, the main novelty of this research is developing a nontoxic, fast, and facile method of synthesizing graphene oxide nanosheet with the lowest environmental issues, which makes the technique ideal for large-scale synthesizing of this material for commercial purposes.

## MATERIALS AND METHODS

Graphite powder with particles sizes of less than 50 micrometers with a purity of %99/9 (104206), sulfuric acid with a purity of more than %98 (100731), hydrogen peroxide %35 (108600), and hydrochloric acid with a purity of %37 (822287) was prepared from Merck Germany company, and potassium permanganate (105080) was prepared from Sigma Aldrich. Graphene oxide nanosheets were synthesized using the modified Hammers' method. In order to do this, the pure graphite was exposed to a mixture of strong oxidants for a short time. The use of phosphoric acid prevents the formation of toxic fumes of sodium nitrate. For graphite oxidation and rapid synthesis of graphene oxide, 0.25gr of pure graphite was added into a mixture of 9ml sulfuric acid and 1ml phosphoric acid. Then 1.5gr of potassium permanganate was added to the acidic mixture in the ice bath for 2 hours. Due to the thermal condition, potassium permanganate was poured carefully and slowly. At this stage, with the development of graphite oxidation, the color of the solution changed from light green to dark green. After ensuring that the graphite was oxidized, 6ml of deionized water and 2ml hydrogen peroxide were added to the solution to dilute the solution and remove the extra potassium permanganate. The solution was then placed in an ultrasonic bath for 30 minutes to produce a single-layer graphene acid. Finally, the solution was washed using a centrifuge to remove metal ions with deionized water and %10 hydrochloric acid. Finally, the sample was dried for 20 minutes at 90 degrees Celsius in an oven (Fig. 1).

X-ray diffraction spectrum was taken from the synthesized sample using an X-ray diffraction spectrometer (Bruker-D8 Advance) in the range of 5 to 70 degrees. Raman spectrum was taken to determine the chemical structure and identify materials with the help of vibration spectrum of a sample synthesized by Raman spectroscopy device

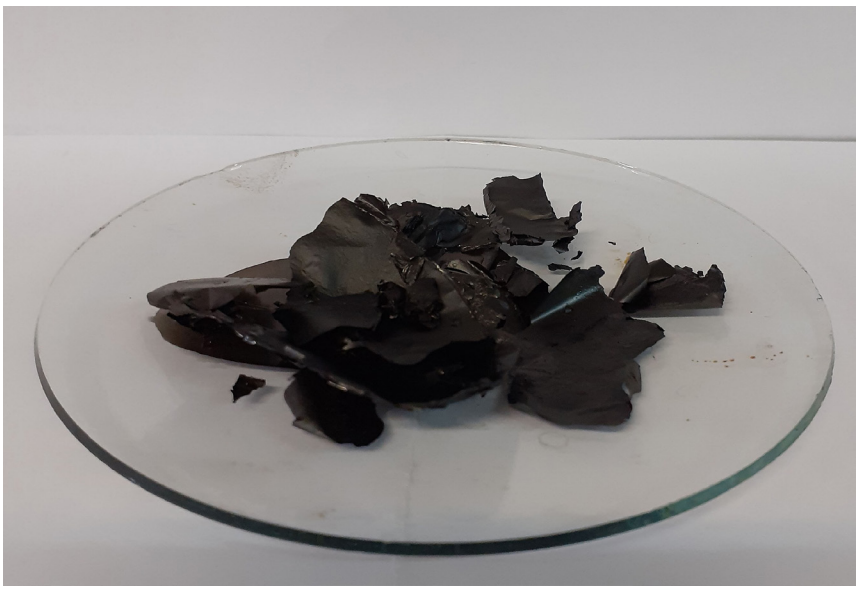

Fig. 1. Dried sample of graphene oxide.

manufactured by TakfamSazan Noor spectrum using a laser with a wavelength of 532nm. Using the UV-VIS spectrophotometer model T92+ product of PG Instruments Company, the absorption spectra of the synthesized sample were examined in the wavelength range of 200 to 900. Also, the FTIR device made by PerkinElmer, the RXI model, was used to study the infrared spectrum of the sample.

## RESULTS AND DISCUSSION

Powder X-ray diffraction studies were done using an X-ray diffract meter having Cu Kα radiation as the X-ray source. The scanning was done in the 2θ range of 5° to 70° with a step size of 0.01°. In the pattern of the scattering spectrum obtained (Fig. 2), the three main peaks related to crystal plates (001), (002), and (200) were observed at angles (2θ) 10.9, 25.8, and 43.6 degrees, respectively. The comparison of the X-ray diffraction pattern obtained was completely consistent with the standard card. Therefore, the results of X-ray diffraction data confirmed the successful synthesis of graphene oxide. Also, the presence of X-ray diffraction peaks in the XRD pattern, graphene oxide, is consistent with previous reports. The XRD spectrum of the synthesized sample was compared with the work of other researchers. Similar reports are provided of the X-ray diffraction pattern. This spectrum is in good agreement with the report of Sanglakpam et al. and shows that not all graphite is oxidized in both spectra.

If the report of Sharma et al. shows complete oxidation of graphite and it is clear that the synthesized sample has good crystallization [29-32].

The results from the Raman spectrum showed the synthesized samples of the two peaks (Fig. 3). The location of these peaks was consistent with the previous results, which could be another confirmation of the synthesis of graphene oxide. The D and G peaks are wavelengths of Raman 1327cm$^{-1}$ and 1587cm$^{-1}$, respectively. Graphite mode (SP2) and D band are due to diamond mode (SP3). The ratio of peak intensity D to peak G indicates the number of oxygen groups in the structure, and proximity to 1 indicates fewer defects in the carbon lattice. Similar reports have been made by Yadav et al. and Wang et al., And compared to the Raman spectrum of the synthesized sample, the peak locations have changed only slightly, which may be due to the synthesis method and crystal defects. [33-36]

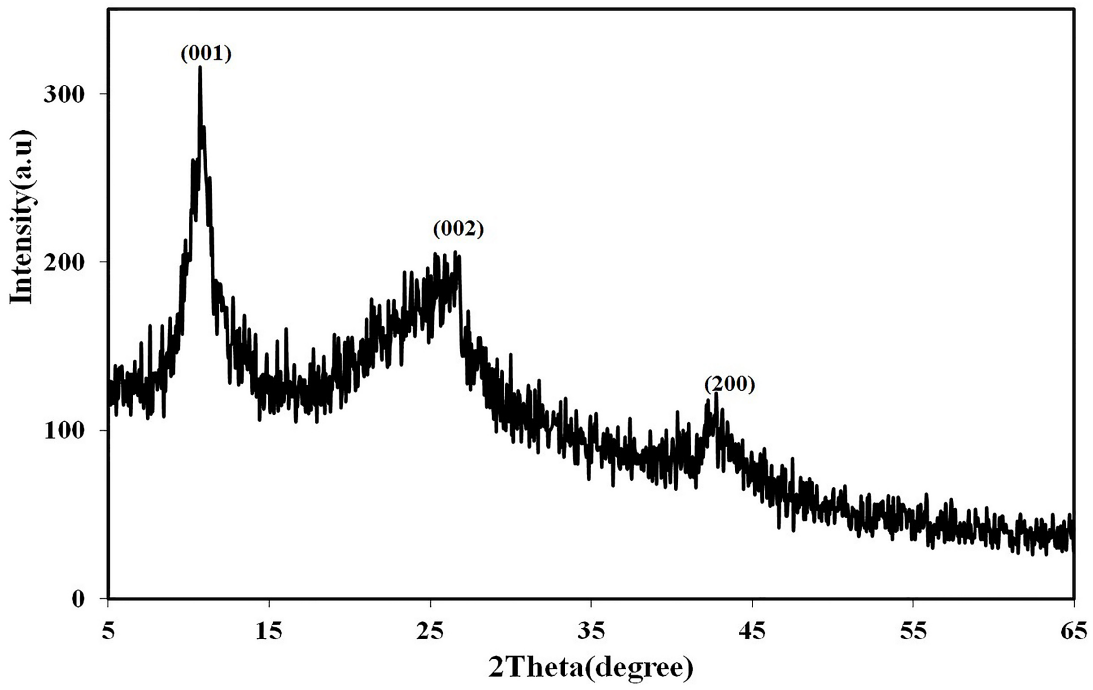


Fig. 2. X-ray diffraction spectrum of the synthesized sample.

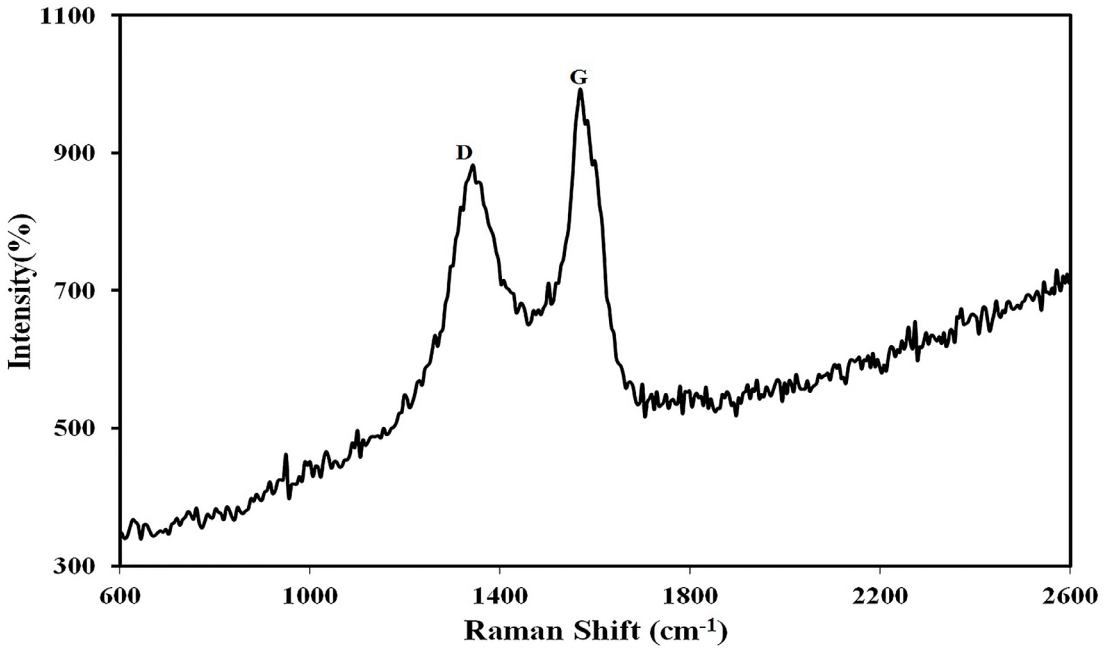


Fig. 3. Raman spectrum of the synthesized sample.

From the diluted suspension of the sample was taken a visible-ultraviolet absorption spectroscopy ultraviolet spectrum (Fig. 4). In the absorption spectrum of the samples, two absorption peaks were observed at wavelengths 231 and 293. The first peak is related to the transfer of the π-π* aromatic ring C-C and the second peak is related to the transfer of n-π* carbonyl group C=O. These

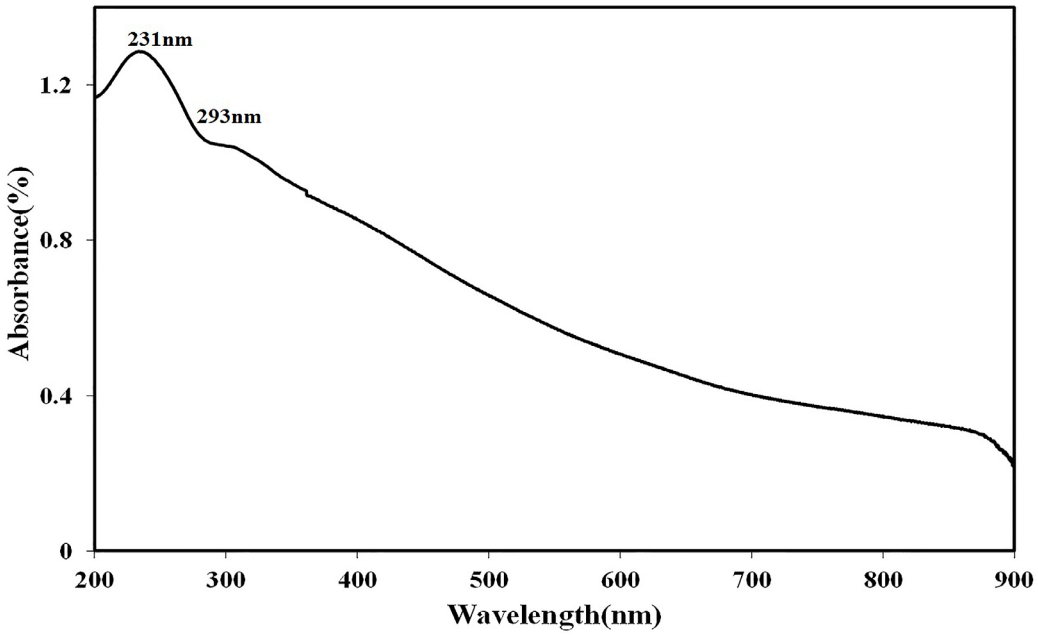


Fig. 4. The absorption spectrum of the synthesized sample.

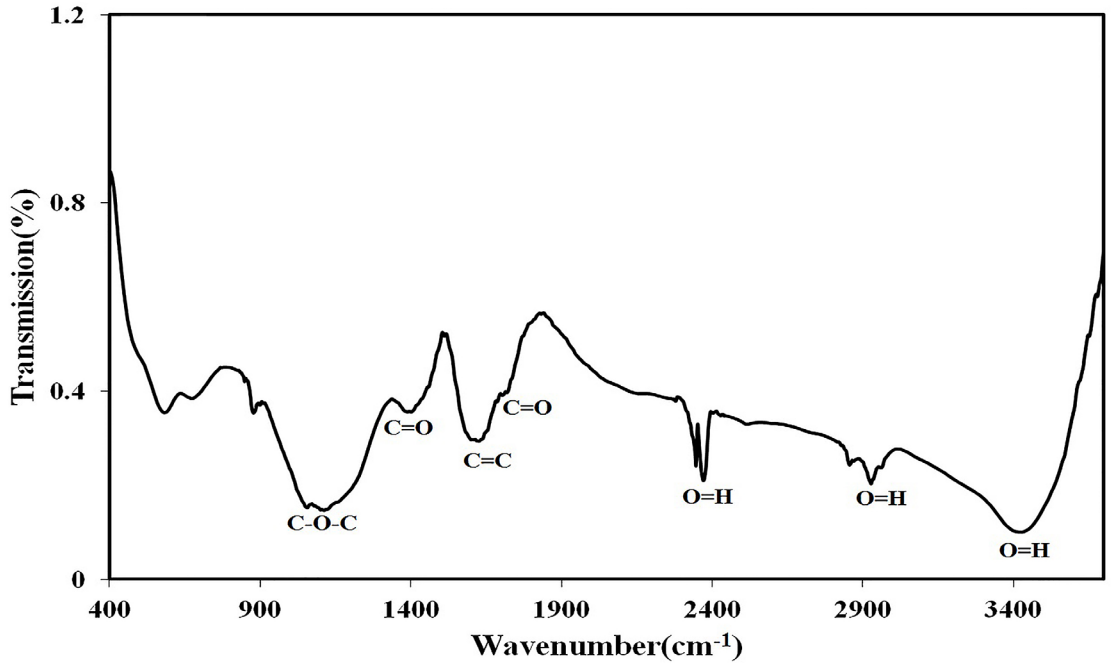


Fig. 5. FTIR spectrum of the synthesized sample.

peaks are characteristic of graphene oxide. It is clear that the sample is highly transparent, and in the visible light area, it has minimal absorption, and the absorption diagram decreases with increasing wavelength. These results are in agreement with the results obtained from the synthesis of graphene oxide by other researchers, , and we can refer to a similar report of the UV-Vis spectrum presented in the article by Yang et al [37-40].

FTIR analysis was performed to determine the oxygen groups on the surface of the synthesized sample. The Fourier transform infrared spectroscopy is a powerful method to determine functional groups, including carbonyl (C = O), carboxyl (COOH), epoxy (C-O-C), and hydroxyl (O-H) [29]. In the infrared spectrum of the samples (Fig. 5), the absorption frequency was observed at 3423 cm-1. This strong absorption is related to the tensile vibration of the O-H bond. In addition, the peaks observed in 2927 $cm^{-1}$ and 2370 $cm^{-1}$are related to the tensile vibration of the O-H bond. Also, in the infrared spectrum of the samples, other absorptions were observed in 1726 $cm^{-1}$ and 1400cm-1, and this adsorption frequency is related to the tensile vibrations of the carbonyl group (C = O). Other absorptions were observed at 1112$cm^{-1}$ and 1621$cm^{-1}$, related to epoxy tensile vibrations (C-O-C) and tensile vibrations related to the C - C bond belonging to unoxidized carbons, respectively. The presence of oxygenated functional groups in this spectrum confirms the synthesis of graphene oxide. The FTIR spectrum was also compared with that of other researchers. Compared to other researchers' reports, including Chen et al. and Rahmanian et al., Graphene oxide functional groups were confirmed. Also, in the spectrum reported by Chen et al., More graphite is oxidized, and fewer carbon groups are formed. [3-6].

For the structural analysis of graphene oxide, transmission electron microscope (TEM) images were taken (Fig. 6). These images have been reported at different magnifications. Diffraction electron shows that, on average, the underlying carbon grid maintains the order and distances of the graphene lattice. In these images, dark and clear areas are seen. The dark areas are related to the carbon bonds of graphite, which have not been completely oxidized, while the light areas with a thin layer are related to graphene oxide with some oxygen functional groups, which shows that some of the graphite has been oxidized. In fact, these transparent areas are graphene monoxide plates

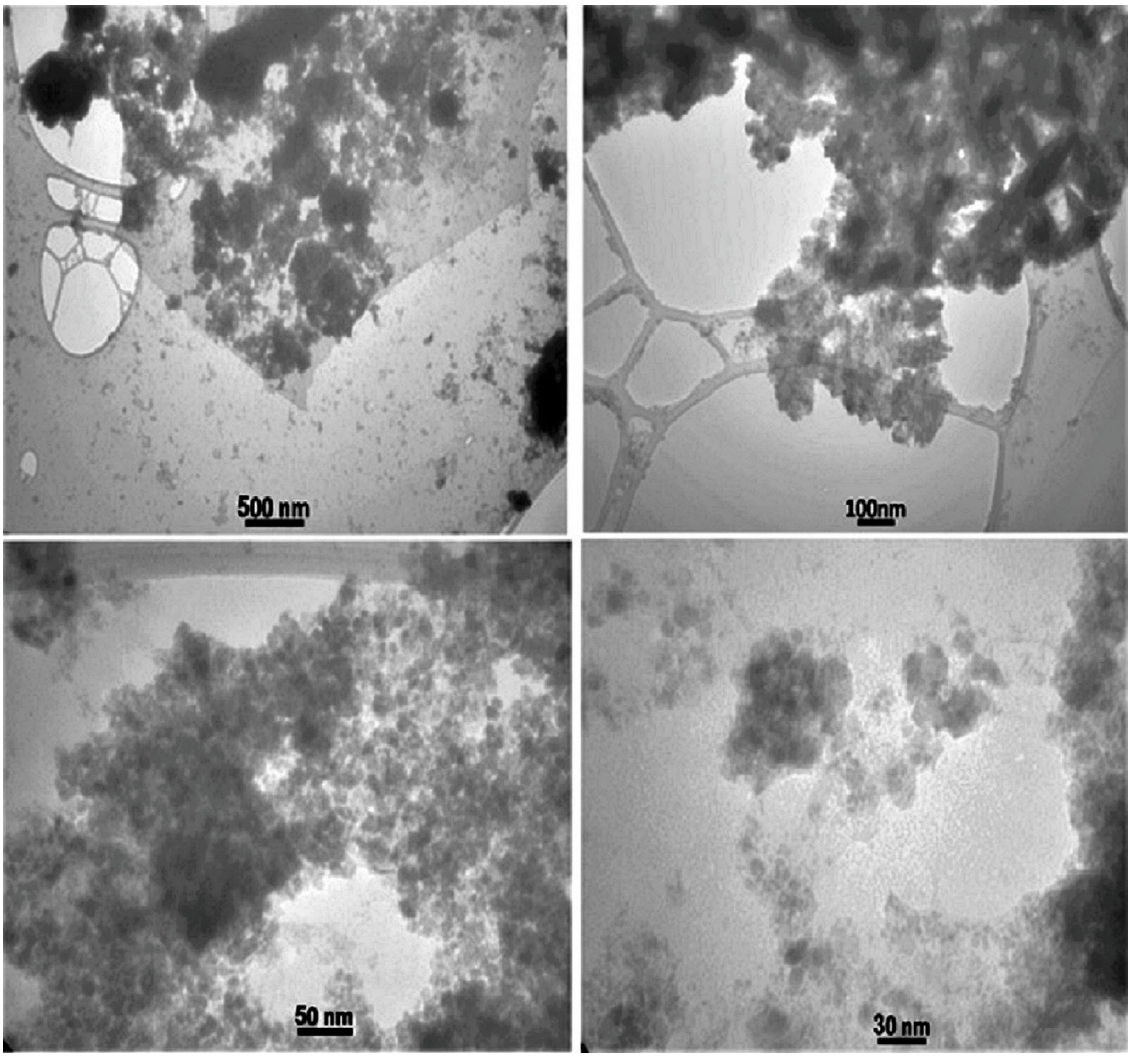


Fig. 6. The transmission electron microscope (TEM) images of samples in different magnifications.

and are ideal for studying nanoparticles. In this case, similar reports from the work of Stobinski et al., and Wilson et al., Show that in this paper, several layers of graphene oxide overlap [41-42].

## CONCLUSION

In this study, graphene oxide was successfully administered easily. By using this method, graphite powder was exposed to a mixture of sulfuric acid, phosphoric acid, and potassium permanganate powder. Graphene oxide synthesis was performed in a short time by graphite oxidation. In this method, the formation of toxic gases was minimized.

After relatively rapid synthesis, the sample was analyzed using various analysis devices such as XRD, TEM, Raman spectrometer, visible-ultraviolet spectrometer, and FTIR spectrometer. The results agreed with the work of researchers who synthesized graphene oxide in other ways.

In addition to confirming the results of X-ray diffraction of graphene oxide synthesis, the results of FTIR spectroscopy also showed the formation of oxygenated functional groups and confirmed graphite oxidation. Raman spectroscopy also confirmed the synthesis of graphene oxide, although comparisons between the peak intensity of the peaks revealed defects in the carbon lattice. The proposed method, thus, seems to be an excellent way to produce graphene oxide on a

large scale.

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## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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