Chapter 10

Advanced Materials, Artificial Intelligence, and Sustainable Technologies for Energy and Environmental Engineering
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Investigation of new synthesized graphene oxide and use in the purification of waste water

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ABSTRACT

This paper shows a technique for the synthesis of graphene oxide with the improvement of an existing method and further oxidation of graphene oxide to obtain several multifunctional layers. The resulting graphene oxide was analyzed by various methods to determine the possibility of further thoughtful functionalization of active sites by organic molecules. GO particles have been characterized by transmission electron microscopy (TEM), scanning electron microscope (SEM), and X-ray diffraction (XRD). An X-ray tube with a copper anode (Cu-Kα radiation, 30 kV and mA) was used to plot the diffraction spectra at room temperature. Based on the results of EDX analysis, was determined that the resulting graphene oxide is a layered material. The optical properties of the graphene oxide layer were also determined. The GO was used in the purification of water from organic toxic substances.

Keywords: SEM; TEM; XRD; GO; Analysis.

INTRODUCTION

Currently, one of the most important problems of the era is environmental protection, given that all materials that are obtained and synthesized by materials scientists and chemical engineers are guided by the multifunctionality of the constituent groups and the spatial arrangement of these groups in relation to each other in composite materials.(1) For any synthesis, the mechanophysical properties of the materials must be taken into account in order to accurately determine their application. Graphene and nanoparticles of graphene oxide layers and all allotropic modifications of carbonate widely used in engineering and technology, biomedicine for the modification of proteins and enzymes, the creation of antibacterial composite materials, and the textile industry.(2,3)

Graphene oxide can contain different amounts of oxygen, on average, variations are known with an oxygen content from 3 % to 40 % by weight, so an accurate determination of the number of functional oxygen groups is mandatory.(4,5) Also, one of the problems is that the composition changes on each new surface area. functional groups can form various covalent and non-covalent bonds on the surface of the graphene oxide plane.(6,7) Of interest is the use of graphene oxides by researchers at Fuzhou University (China), who created a sorption system based on GO, which made it possible to create an inexpensive method for determining protein-protein interactions.(8)

For water purification, graphite oxide (GO) has recently been used. GO is a graphite oxidation product having a group of carbonyl and carboxyl groups. as well as epoxy and hydroxyl groups at the edges of its layers.[8] GO is a very valuable membrane material due to its low cost and ease of production, good chemical stability, mechanical strength, and high ability to remove pollutants. Studies have shown that GO membrane has very good ionic and molecular selectivity and water permeability. GO is widely used in many reactions.[9,10] The use of GO/Al_2O_3 composite membranes for water purification is a subject of growing research interest due to the simple and effective approach they support. On this composite obtained, it was possible to purify phenol from wastewater to 99,9 %.[11,12]

Graphene oxide belongs to a group of substances widely used in the chemical industry. It is environmentally friendly and readily available. At the same time, it has good adsorption properties. Based on nanotechnological approaches, it is widely used in water purification from toxic substances. From an ecological point of view, the role of graphene oxide in environmental protection, especially in wastewater treatment from organic toxic substances, is irreplaceable. In this regard, analyzes were carried out to study the properties of the graphene oxide synthesized by us.

Experimental part

Materials and reagents

Distilled water, sodium nitrate, potassium permanganate, sulfuric acid (98 %), hydrogen peroxide (3 %), ice.

Synthesis of graphene oxide

The work was done based on a modified Hammer method that to obtain functional centers for further modification with organic molecules. So, three parallel portions of 3 g of graphite, 1 g of NaNO3 and 6 g of KMnO4. 46 ml of 95-98 % sulfuric acid (H2SO4) were weighed and were placed in a 250 ml graduated cylinder and an ice bath was used to cool the mixture. Cooling was carried out to 0°C. Further, at 0°C, the cooling bath was stopped and observed until a temperature of 20°C was reached.

Potassium permanganate was added in portions over 2 hours, 20°C was maintained by cooling and stirred for another 4 hours while maintaining a temperature of 20 - 25. Since the temperature increased by 90°C when adding water (92 ml), cooling was needed to lower it. further washed with 280 ml DW to reduce product loss. the next step in the preparation is the addition of hydrogen peroxide for stronger oxidation, and held for twelve hours. further began decanting and filtration.

METHOD

Analysis methods: FTIR analysis, TEM analysis, XRD analysis, optical properties, SEM analysis.

FTIR analysis

According to FTIR results, we can say that GO has been synthesized completely. The FTIR results of GO has been compared with FTIR data base spectras from the other research papers. Result showed that the predicted functional groups could be obtained completely.

FTIR analysis is useful for the study of intermediate compounds formed after the oxidation process, as GO was oxidized using H_2O_2. If you look at the result of FTIR analysis in figure 2, you can notice that the wide peak between 3000-3750cm^{-1}, corresponds to the stretching vibration of the groups. This fact proves to us that water molecules were adsorbed during the reaction.

Table 1. Result showed that the predicted functional groups	
3200,1 cm^{-1}	OH group [7]
2883,6 cm^{-1}	-C-H [7]
1710,6 cm^{-1}	C=O [7]
1620,1 cm^{-1}	C=C [7]
1040,7 cm^{-1}	C-O [7]
970,06 cm^{-1}	Vinyl [7]

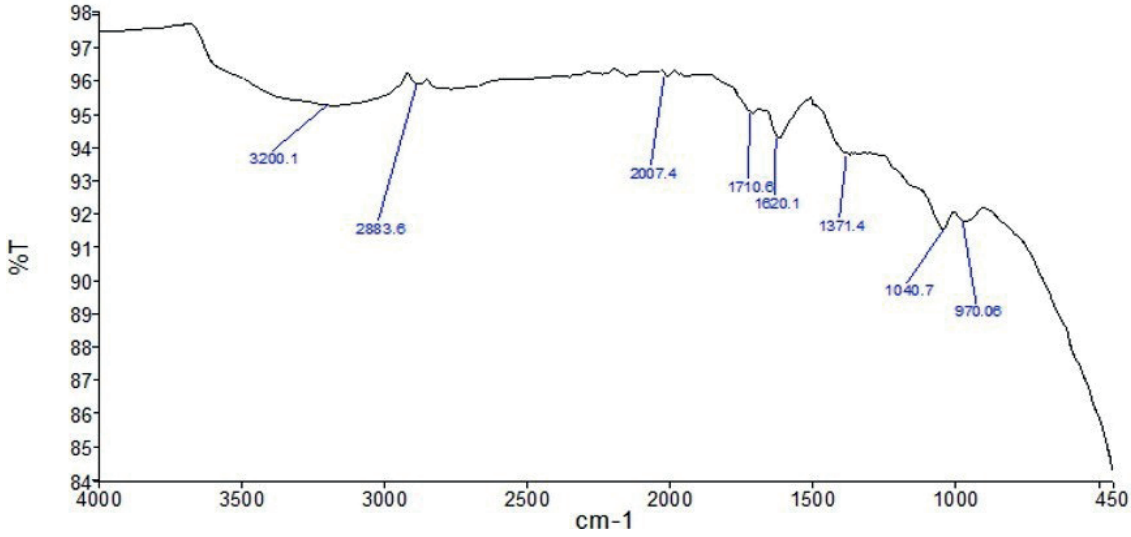


Figure 1. FTIR result of graphene oxide

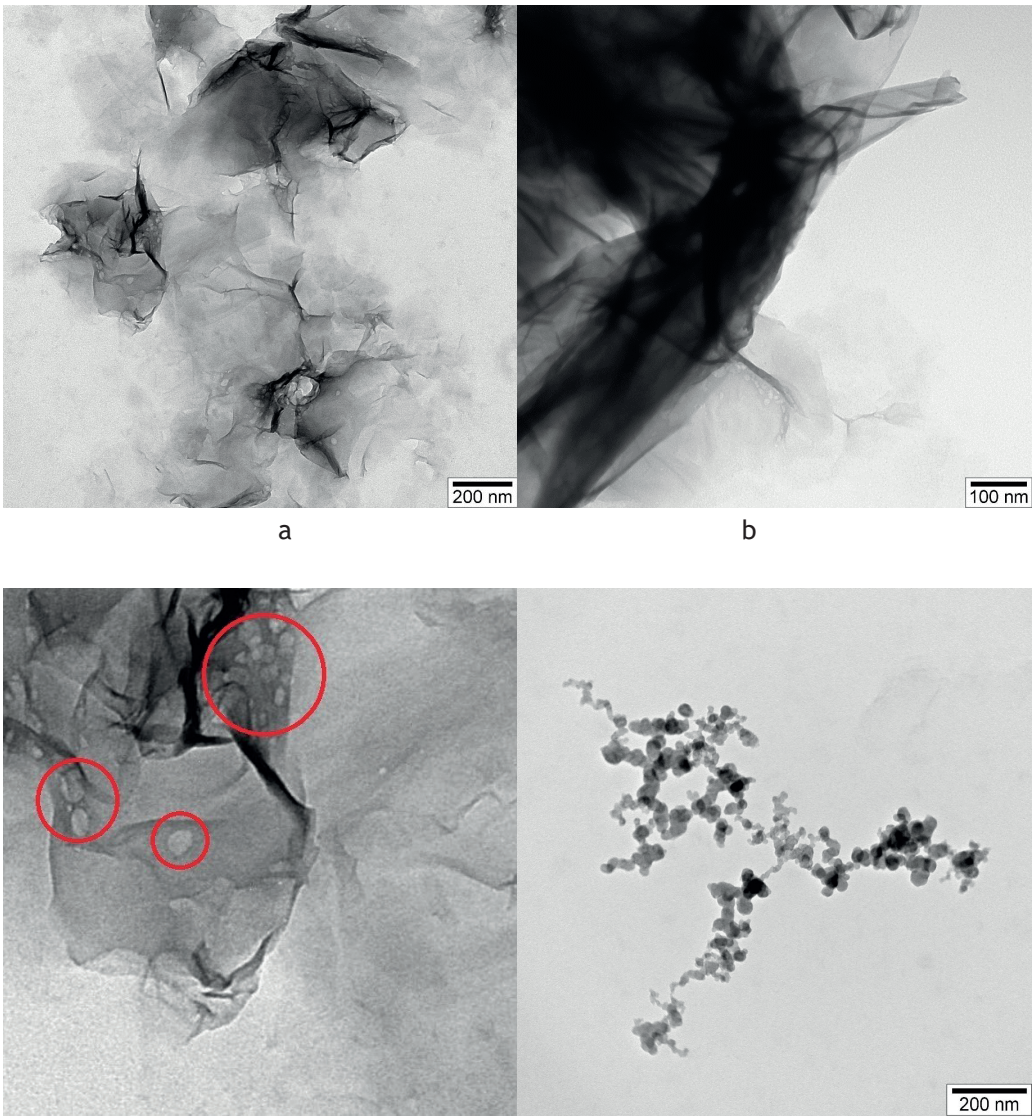
Peak 2883,6 cm^{-1} which is the absorption peak providing information for vibration of stretching C-H, while the peak of the struggle at 1710,6 cm^{-1} may be associated with the bending vibration of deformation for communication C=O. Also 1620,1 cm^{-1} can be associated with bending vibration of deformation for a double bond C=C, since GO is layered material and the presence of double C=C relationships. Finally, absorption peaks at 1040,7 cm^{-1} and 970,06 cm^{-1} correspond to vibration of deformation related to C-O and vinyl groups. The above oxygen groups, or rather their presence, proves that this reaction was successful and the graphite was oxidized. The formation of hydrogen bonds between graphite and water molecules is observed. The formation of these bonds is provoked by surface and polar hydroxyl groups. This fact additionally explains to us the hydrophilic nature of GO. The FTIR results of GO has been compared with other FTIR spectra in other research papers. Result showed that the predicted functional groups could be obtained completely.

TEM analysis of GO particles

Due to the fact that carbon and oxygen are heavy atoms, can be observed in figure 2 that these atoms are shown grey. Also, can be observed that in figure 2 there are some holes on the layered surface of the material. The larger the electronic beam deviates, the darker the part in this figure is indicated, and in our case, these dark parts are GO-lists. The presence of minimal errors in the synthesis of this product (GO) is determined by the presence of gray in this figure and this color tells us that the layer is very thin, or rather atomic. The presence of black parts

in the figure tells us that, as a layer, it has the ability to be wrinkled, and you can also observe a hole on this layer.

When synthesizing nano layers of GO, carbon nanostructures of different sizes were also obtained. On the table, can be observed the variability of the size of the agglomeration of these nanostructures, such as agglomeration "A" with a size of 428,6 nm. In figure 3, we can clearly consider that these agglomerations have amorphous ("Bα") which is located around the agglomeration "B". These results, as well as clearly marked the boundaries of nanostructures, give us the right to reason that our product is semicristalic.



a b

c d

Figure 2. TEM images of GO(a,b), TEM images of GO (mentioned holes) (c), TEM images of Carbon Nanoparticles (d)

Table 2. The variability of the size of the agglomeration of these nanostructures

Carbon Nps agglomeration type	Sizes (nm)
A	428,6
B	250
C	250
D	214,3
E	142,8
F	142,8
G	250
H	214,3
I	178,6
J	214,3
K	214,3
L	200

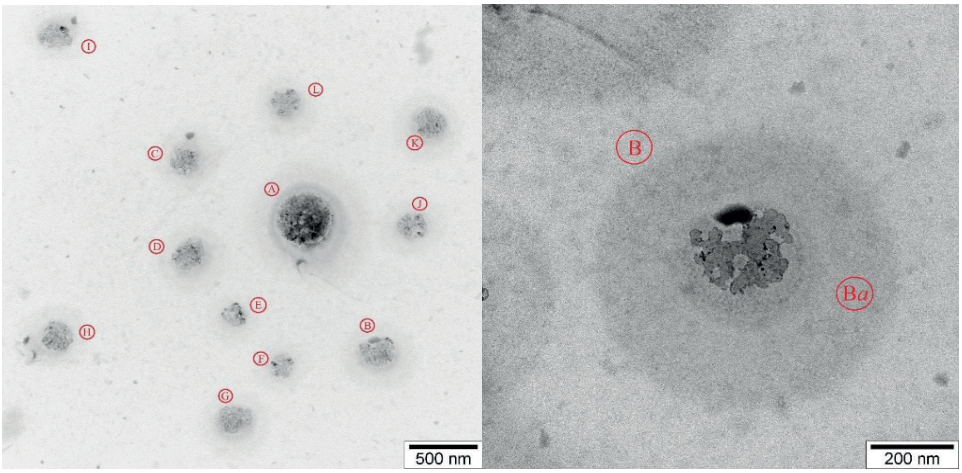


Figure 3. TEM images of Carbon Nanoparticles Agglomerations

XRD analysis of GO particles

The purity properties of the GO particles were investigated by powder X-ray diffraction (XRD) method. Figure 5 shows the XRD patterns of the synthesized GO nanoparticles. XRD peaks were well defined and corresponded to GO at amorphous phase. X-ray structure analysis graphs of the studied GO particles were recorded on the Rigaku Mini Flex 600 spowder diffractometer. Its features are given below:

X-ray tube with copper anode (Cu-Kα radiation,30 kV and mA) was used to draw the diffraction specters at room temperature. At 2θ = 20˚-80˚ with discrete growth mode these specters were obtained as Δ2θ = 0,05˚and the exposure time was τ = 5 seconds (figure 4).

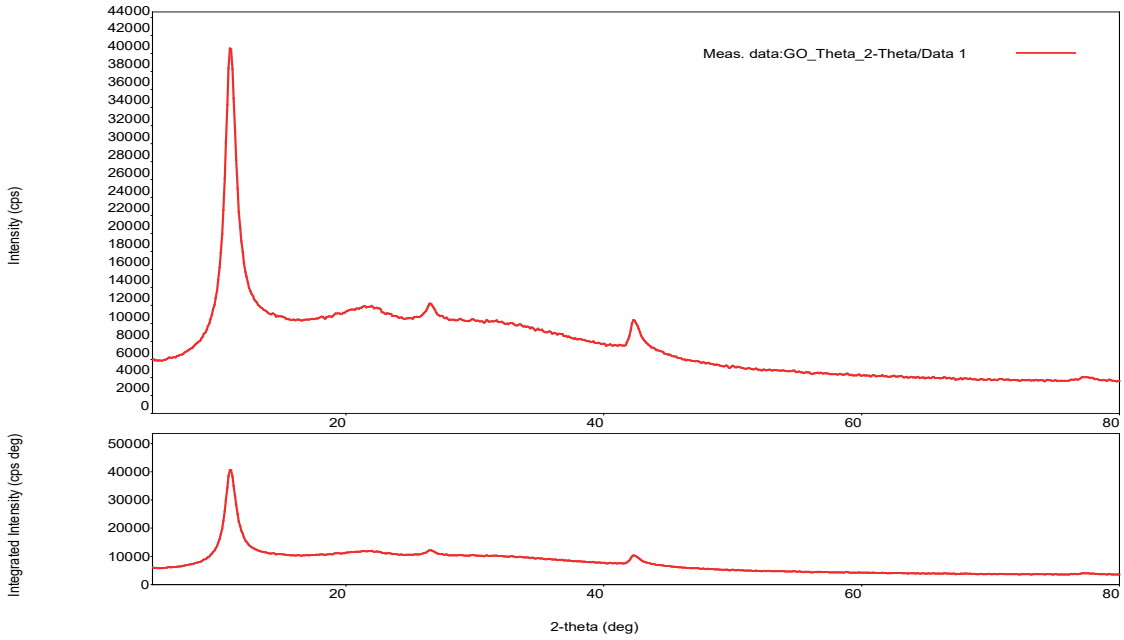
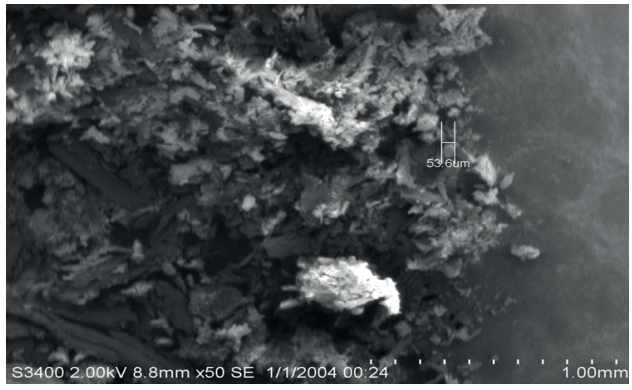


Figure 4. *XRD analysis of GO particles*

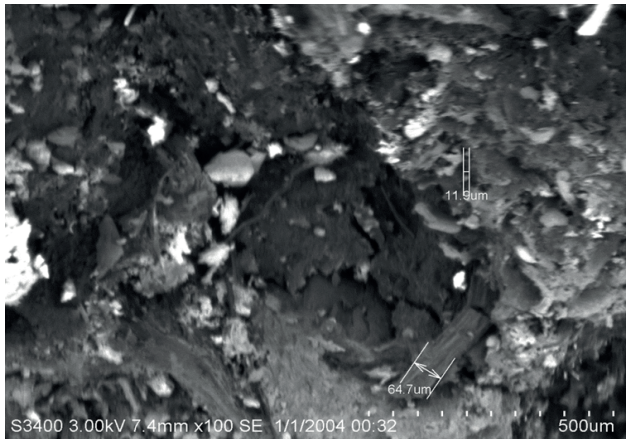
SEM analysis of GO particles

The SEM analysis of the GO particles which has been prepared for this process was performed on the S-3400N Scanning Electron Microscope.

Electron microscopic images of GO at 50x and 100x magnification were obtained using a scanning electron microscope (SEM) S-3400N and are presented in Figure 5.As can be seen from the images, the GO sample mainly consists of irregular accumulations of carbon particles with a layered structure. As a result of such combinations, various shaped parts with a porous surface with dimensions of 53,6 microns (a), 11,9 microns, 64,7 microns, larger and smaller were formed. At the same time, there are also different sizes of different nanotube-like particles. It should be noted that larger aggregates of various shapes also formed in the sample. The presence of such aggregated structures in the synthesized GO does not adversely affect the claimed scope of its application.



(a)



(b)

Figure 5. (a) Electron microscopic image of GO, magmified 50 times (b) Electron microscopic image of GO, magnified 100 times

Optical properties of GO particles

The optical properties of graphene oxide were also determined. Graphene oxide has optical properties. This is due to its absorption or reflection of light. The absorption spectrum of GO particles is shown below (figure 6).

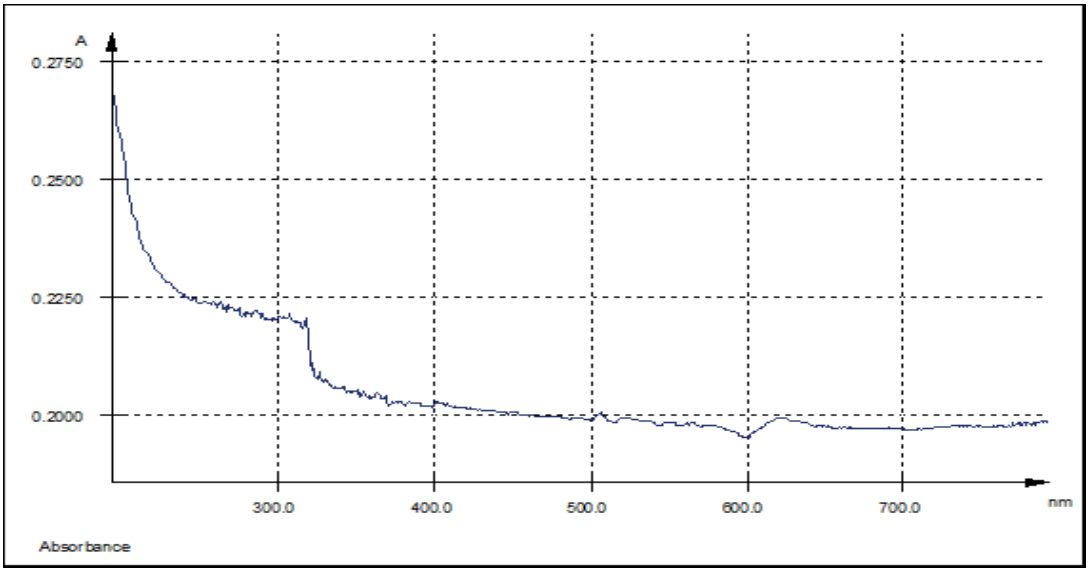


Figure 6. Emission spectrum of GO particles

The emission spectrum of GO nanoparticles manifested itself as peak curves in the range of 2000–3000 fields. The adsorption curve was plotted on the absorption spectrum of Specord 250 plus UV-Vis.

There are many scientific papers on the adsorption of activated carbon in the literature. [13] It should be noted that the adsorption properties of the obtained GO were also studied.

Adsorption of GO was studied on the "Varian Cary 50" spectrophotometer. The adsorption process continued for two hours with periodic stirring. In this case, 0,05 g of GO and 20 ml of a 1 mgL^{-1} phenol solution were taken and the process was carried out at a temperature of 25^0 C.

The graph below shows the dependence of the absorption coefficient of a solution of phenol with a concentration of 1 mgL^{-1} on the wavelength (figure 7). Adsorption has not yet occurred; therefore curves compatible with phenol were obtained in the area of 270 nm.

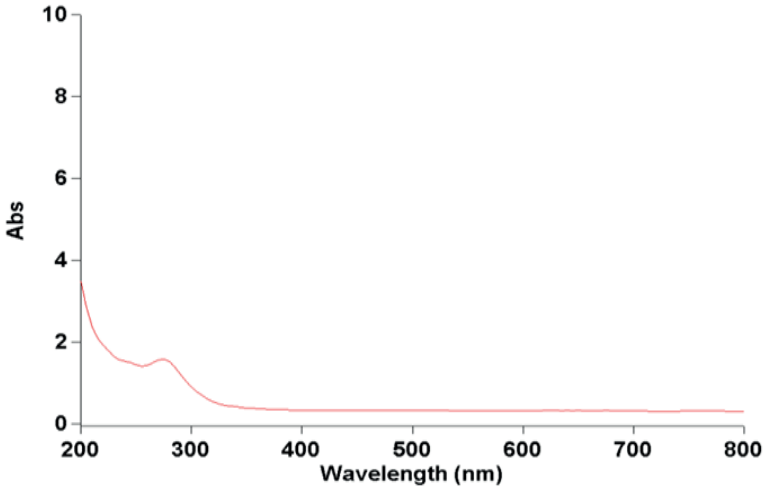


Figure 7. Graph before adsorption of phenol solution 1 mgL^{-1}

As can be seen in figure 7 the curves obtained for phenol were recorded at 200-300 nm. It is also known from the literature that the curve obtained at a wavelength of 270 nanometers corresponds to the curve of phenol. Below is a graph taken after a 2 hour adsorption process.

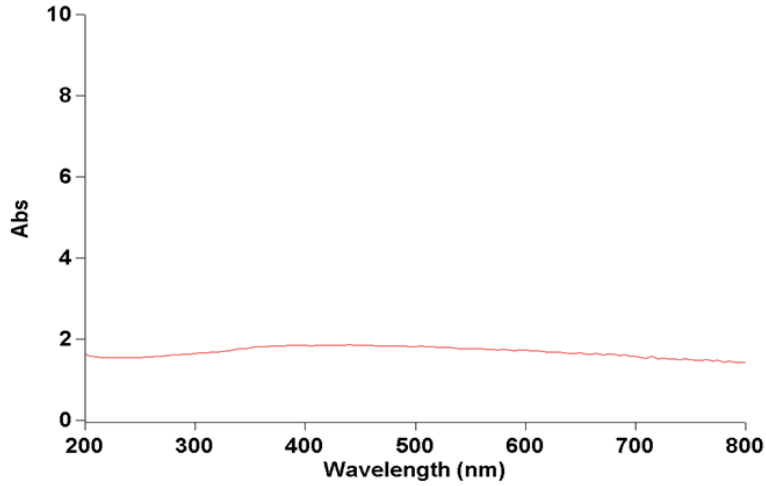


Figure 8. Graph after adsorption of phenol solution 1 mgL^{-1}

According to the curves drawn two hours after the adsorption of the solution at room temperature there are no corresponding curves in the area of 270 nm of the spectrum

corresponding to phenol (figure 8), which indicates the completion of adsorption. The process was repeated. It should be noted that the adsorption process weakened with an increase in the concentration of phenol in the solution, but accelerated with an increase in the amount of graphene oxide.

Thus, the synthesis of GO and its study were carried out in the article. GO is a very important raw material from an environmental point of view. It is used in many reactions, and also has adsorption properties that are used in the treatment of wastewater from toxic substances.[14,15,16]

In the future, research work in this direction will be continued in order to protect the environment.

CONCLUSIONS

1. GO was synthesized by the Hammer method.
2. The structural properties of the synthesized GO were studied.
3. X-ray structure analysis graphs of the studied GO particles were recorded on the Rigaku Mini Flex 600 diffractometer.
4. The SEM analysis of the GO particles was performed on the S-3400N Scanning Electron Microscope.
5. The TEM analysis of the GO particles was performed on the 120 kV JEOL JEM-1400 transmitter electron microscope.
6. The optical properties of GO were also studied on the Specord 250.
7. It was determined that the resulting graphene oxide is a layered material.
8. It was found that functional groups containing an oxygen atom in various functional groups are located on the surface of graphene oxide.
9. The graphene oxide was used in the purification of water from organic toxic substances.
10. The course of adsorption was studied on the device “Varian Cary 50”.

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FINANCING

None.

CONFLICT OF INTEREST

None.

AUTHORSHIP CONTRIBUTION

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Drafting - original draft: Elmina Gadirova, Narmina Guliyeva, Tarek Ganat, Fatma Bassyouni.
Writing - proofreading and editing: Elmina Gadirova, Narmina Guliyeva, Tarek Ganat, Fatma Bassyouni.