SYNTHESIS OF GRAPHENE - NEW NANOMATERIAL FOR ELECTRONIC'S PURPOSE

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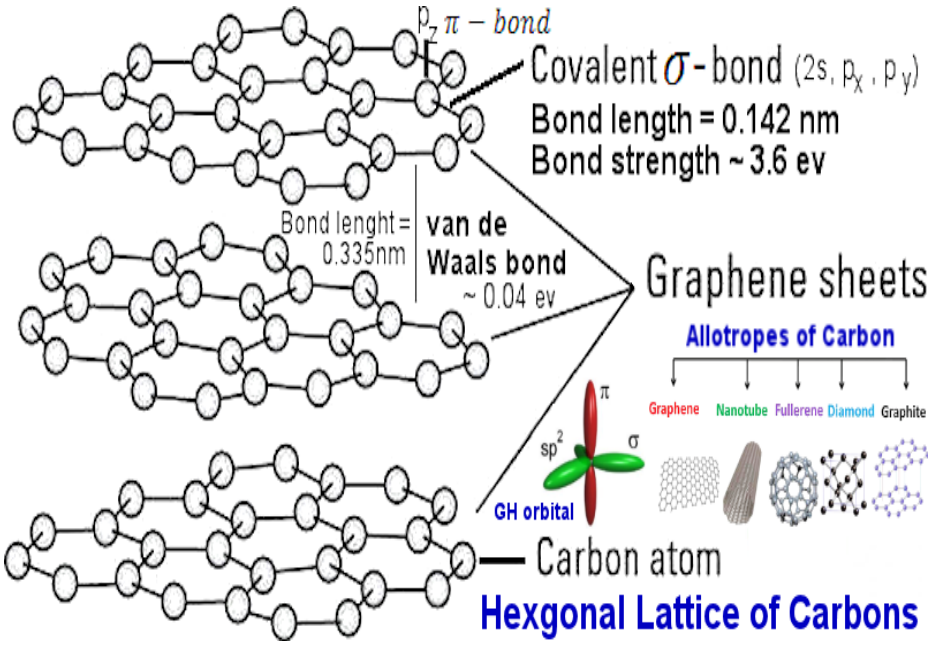
Abstract: Graphene is an amazing material, with many potential applications, that continues to impress scientists around the world with its structure and composition. Graphene also opens up many possibilities in electronics. One of the areas for which graphene is studied and developed is to be used in the supercapacitor's manufacture. Graphene is known as the first two-dimensional nanomaterial. Graphene displays remarkable electron mobility at room temperature. This makes it very suitable for coating the electrodes of supercapacitors. The supercapacitor is an energy storage device with a high power density that can charge / discharge in quick time and has long term cyclic stability. It is believed in the near future will be developed supercapacitor to store more energy than battery. One direction in which research is going to improve the capacitive characteristics of capacitors is the use of nano-graphene structures as coating on capacitor electrodes. The graphene-based supercapacitor thin coating would be able to be fully charged in a matter of minutes. There are various methods to synthesize graphene and its derivatives. The main difficulties in obtaining graphene are related to: the inability to obtain a high quality sample in significant quantities; adjusting the number of layers. Novoselov's method (micromechanical exfoliation of graphite layers) does not give us high product quality or high yield. It is necessary to overcome the energy of the connection of the Van der Waals interactions between the layers without disturbing the first, second and subsequent layers, which is difficult. In this connection in the present work was synthesized graphene from very pure, finely dispersed graphite (99.9%) by applying electrolysis and ultrasound. Graphene is obtained as a result of a combination of chemical and physical treatment. Sulfuric acid was used as the electrolyte. It loosens the weak Van der Waals bonds and, together with the acoustic action of ultrasound, contributes to the cleavage of the individual layers of graphene. As a precursor for graphene synthesis fine graphite (> 99% purity) have used, which burns without residue when have been heated. 200 ml of deionized water is poured into a beaker, 8 ml of 4N H_2SO_4 and 5 g of pure fine graphite G0 were additionally added, then the mixture is well homogenized. Electrolysis and ultrasound treatment take place simultaneously. The experiment was performed at a temperature of 17 - 30 ° C and an ultrasound frequency of 40kHz. The samples were then filtered and the solid residue was dried at 110 ° C for 5 hours. The time of ultrasound treatment is the same for all samples - 30 minutes, while the time of electrolysis is with different duration - 5, 10, 15 and 30 minutes. The obtained graphene was examined mainly by using microscope methods.
Keywords: *graphene, ultrasonic method, electrolysis, SEM*

1. INTORDUCTION

Graphene is a new material the discovery of which was awarded by the Nobel Prize in Physics in 2010 (Novoselov et al, 2004). Graphene is an amazing material, with many potential applications, that continues to impress scientists around the world with its structure and composition. It is one of the lightest and strongest materials on the planet, consisting of only one atom (Kuten, et al, 2020). Due to its remarkable mechanical and chemical properties, graphene is considered one of the future substitutes for silicon in the semiconductor industry. Due to its unique structure, graphene can easily bind to other molecules, which is one of its many advantages.

The structure of graphene is a two dimensional version of the carbon allotropes (https://universe-review.ca/R13-16-graphene.htm). Each individual atom possesses three sp^2 orbitals which interact with the other ones in its neighborhood to form covalent sigma bonds; while the fourth orbital maintains a weaker Van de Waals pi - bond for stacking up the layers to form graphite (Fig 1). In single layer, the electrons in the pi- bonds link up together to form valence and conduction bands. The bands at the edge of the sheet make contact with each other in six points (https://universe-review.ca/R13-16-graphene.htm).

Figure 1. Structure of graphene (https://universe-review.ca/R13-16-graphene.htm).



Graphene has many applications in electronics (Anithaa, Shankar & Vijayakumar, 2020). Until recently, the existence of two-dimensional material was considered impossible. With the discovery of graphene, countless possibilities open up for the engineering world. One of the directions for which graphene is studied and developed is so that it can be applied in the production of supercapacitors (Su & Wu, 2021). They can be powered very quickly and will also be able to store more electricity. Graphene-based micro-supercapacitors are likely to be developed for use in smartphones, portable computing devices, and may be commercially available over the next 5-10 years. Graphene-reinforced lithium-ion batteries could be used in electrically powered vehicles, as well as again in smartphones, laptops and tablets, but with much smaller sizes and weights (Zhan et al, 2020). So far, only the expensive cost of production hinders its wide application.

The present work proposes a method for the synthesis of graphene from very pure, finely dispersed graphite by simultaneous application of electrolysis and ultrasound. Graphene is obtained as a result of a combination of chemical and physical treatment. Sulfuric acid was used as the electrolyte. It loosens the weak Van der Waals bonds and, together with the acoustic action of ultrasound, contributes to the cleavage of the individual layers of graphene.

2. MATERIALS AND METHODS

Materials

As a raw materials in the experiments were used: pure finely divided graphite (GO) with a purity of 99.9%, sulfuric acid (H_2SO_4) and deionized water.

Methods

The morphology of the samples was characterized mainly by SEM and optical microscopy

SEM – The microstructure of graphene was examined by scanning electron microscopy (SEM). The electron microscope photographs were taken using scanning electron microscope "Philips SEM525M/EDAX9900" with attached X- ray microanalyst. The microphotograhps were made in a regime of secondary electrons at acceleration of 20 kV.

Optical Microscopy. The analyses were performed using a Celestron 5 MP LCD Deluxe Digital Microscope light microscope.

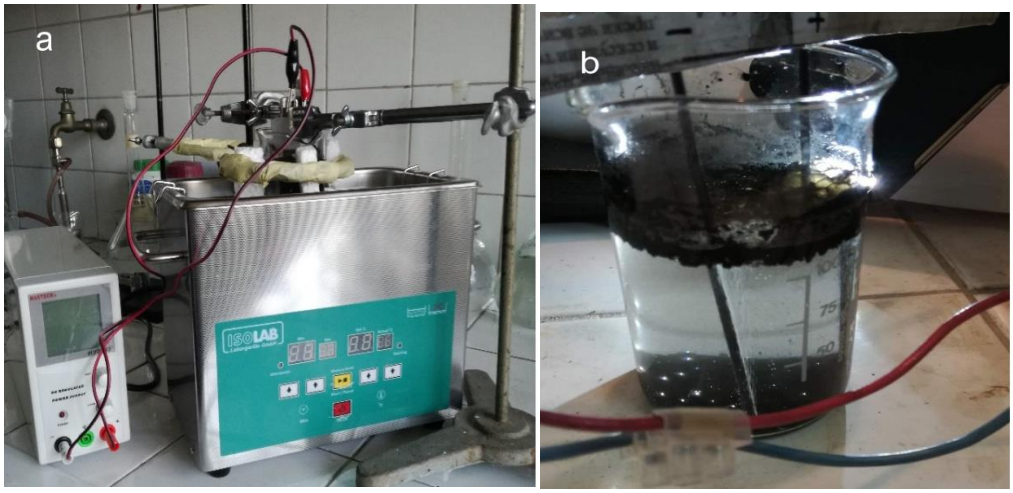
Table 1 presents the synthesized compositions and experimental conditions.

Table 1. Synthesized compositions and experimental conditions

№ of the sample	G0, g	4N H_2SO_4, ml	H_2O, ml	electrolysis, · min	ultrasound, min	magnitude of the current, A	Voltage, V
G 1	5	8	200	5	30	1	5,6
G 2	5	8	200	10	30	1	5,6
G 3	5	8	200	15	30	1	5,6
G 4	5	8	200	5	30	3,1	10,6
G 5	5	8	200	30	30	3,1	10,6

The method used for graphene synthesis involves the simultaneous application of ultrasound and electrolysis. As a starting material for graphene a fine graphite (> 99%) is used, which burns without residue. For all compositions, 8 ml of 4N H_2SO_4 and 5 g of pure fine graphite (GO) is added in beaker with 100 ml deionized water. Then the mixture is well homogenized. The beaker is placed in an ultrasonic bath, which is filled about 2/3 with deionized water (Fig. 2). The electrolysis acts between 5 and 30 minutes. Ultrasound treatment in all samples is 30 minutes. The magnitude of the current is 1 or 3.1 A, and the voltage is 5.6 or 10.6 V.
A picture of the experimental installation with combined application of ultrasound and electrolysis is shown in fig. 2.

Figure 2. a) Photograph of the experimental installation with combined application of ultrasound and electrolysis, b) conducting electrolysis.



Graphite electrodes are placed in a beaker. For samples G1, G2 and G3 the electrodes are in the form of plates, will for samples G4 and G5 – in form of cylindrical. The distance between the electrodes is 10 mm. The experiment was performed in the ultrasonic bath at room temperature for 4 hours at an ultrasonic frequency of 40kHz.
An aqueous solution of H_2SO_4 was used as the electrolyte. Sulfuric acid dissociates according to the equation: $H_2SO_4 \leftrightarrow 2H^+ + SO_4^{2-}$.

3. RESULTS AND DISCUSSION

Investigation on graphene synthesized

Scanning electron microscopy (SEM)

By scanning electron microscopy the structure of the samples was examined (fig.3).

Figure 3. SEM on sample G2.

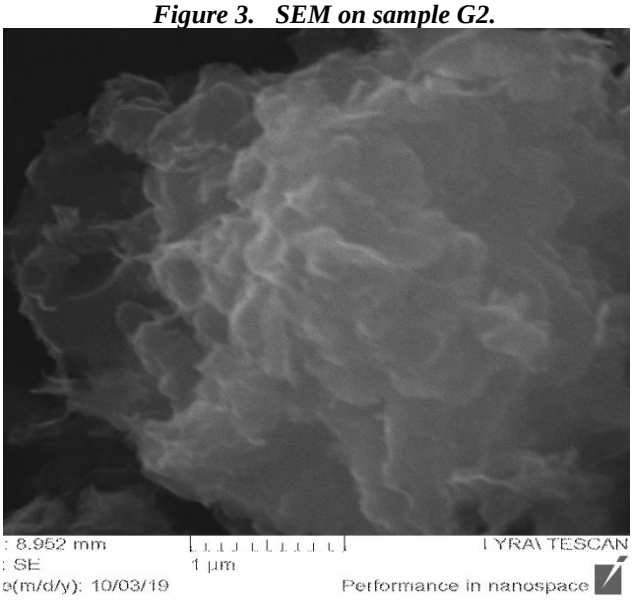


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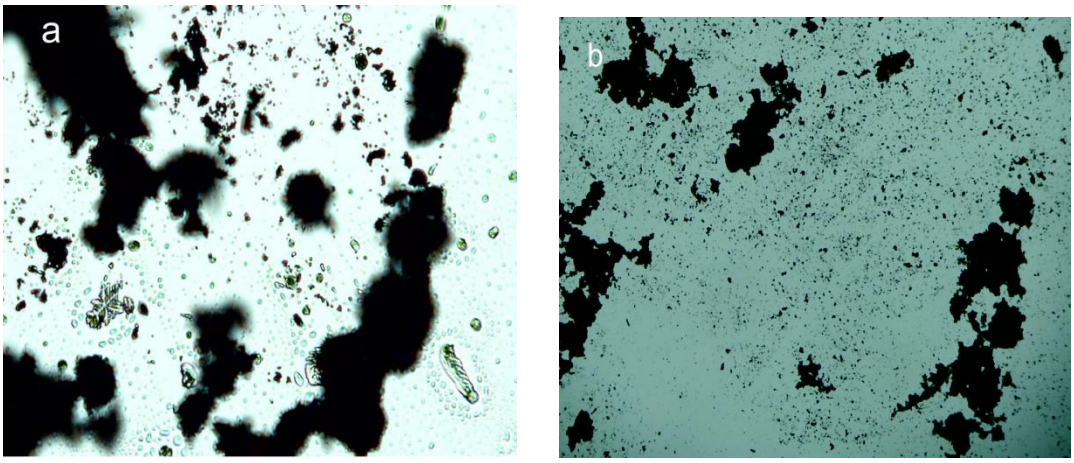
Figure 3 presents SEM image of graphene sheets which appeared as similar thin sheets randomly aggregate, with wrinkled surfaces. The photograph (fig.3) shows shaped hexagonal areas that are relevant to graphene. Due to the insufficient magnification, these areas are not clearly shaped like hexagonal honey- comb.

Optical Microscopy

The microscopic analyses of graphene are presented in fig. 4(a,b). By the optical Celestron 5 MP LCD Deluxe Digital Microscope two samples with composition G4 were examined in order to evaluate the differences in the morphological characteristics (wet (4a) and dry (4b) samples).

Figure 4. Optical image of graphene samples with compositions G4 - wet (a) and dry (b) sample.



Samples with composition G4 represent clusters of graphene particles scattered on the glass.

Electrical resistivity measurements

The electrical resistivity of samples G1, G2, G3, G4, and G5 was examined (fig. 5 and fig. 6).

Figure 5. Graphic relationship between the time for treatment with electrolysis, ultrasound and the resistance of the obtained material.

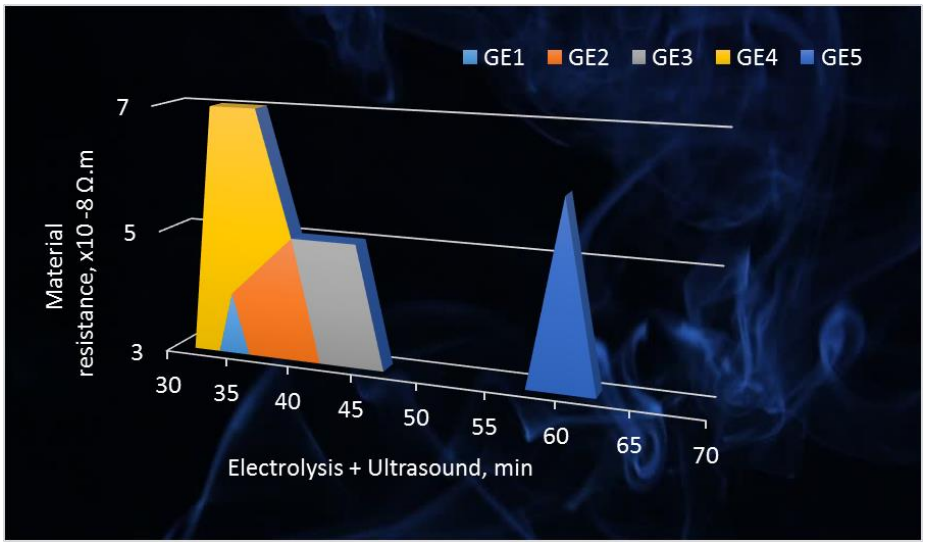
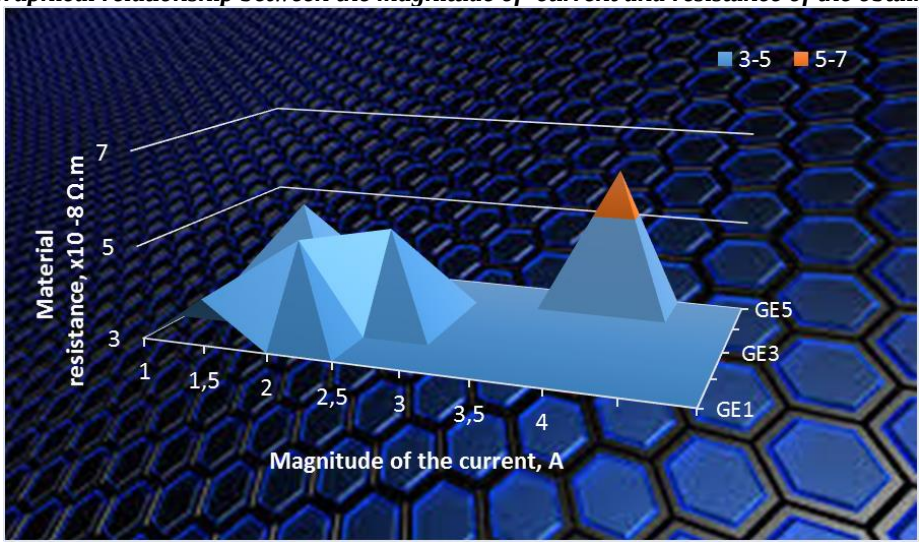


Figure 6. Graphical relationship between the magnitude of current and resistance of the obtained material.



The results of fig. 5 and 6 show that the lowest value of electrical resistance is measured in sample G1 and the highest in sample G5.

4. CONCLUSIONS

A new nanomaterial - graphene was synthesized by simultaneous application of ultrasound and electrolysis. As a precursor for graphene synthesis fine graphite (> 99% purity) have been used. The electrolysis acts between 5 and 30 minutes. Duration of ultrasound treatment for all samples was 30 minutes. The magnitude of the current is 1 or 3.1 A, and the voltage - 5.6 or 10.6 V. The structure of the material was studied by electron microscopy - SEM. The electrical resistance of all samples was examined. Graphene electrical resistance has been shown to be lower than that of pure graphite precursor.

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