JOURNAL OF CHEMICAL EDUCATION
Cite This: J. Chem. Educ. XXXX, XXX, XXX−XXX
Laboratory Experiment
pubs.acs.org/jchemeduc

Reduced Graphene Oxide Joins Graphene Oxide To Teach Undergraduate Students Core Chemistry and Nanotechnology Concepts

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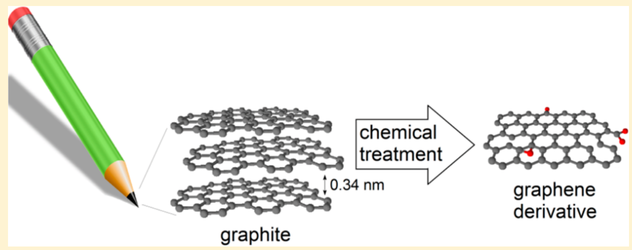
Supporting Information

ABSTRACT: Novel carbon nanomaterials such as reduced graphene oxide (rGO) and graphene oxide (GO) can be easily incorporated into the undergraduate curriculum to discuss basic chemistry and nanotechnology concepts. This paper describes a laboratory experiment designed to study the differences between GO and rGO regarding their physicochemical properties (e.g., color, hydrophobicity, type of functional groups, electrical conductivity, etc.). In this course, students carry out the chemical reduction of GO using ascorbic acid, a mild and environmentally friendly reducing agent. The differences between GO and rGO can be spotted by the naked eye and can be further evaluated by spectroscopic methods, as Fourier transform infrared and UV−vis spectroscopy and X-ray diffraction. Simple and applicable in all laboratories, use of the multimeter to measure resistance was proposed to reveal the different electrical properties of GO and rGO. Moreover, the proposed laboratory experiment is an ideal pretext to discuss the definition of graphene in the context of the overuse of this term in the literature.



KEYWORDS: *Second-Year Undergraduate, Laboratory Instruction, Interdisciplinary/Multidisciplinary, Hands-On Learning/Manipulatives, IR Spectroscopy, Nanotechnology, UV−Vis Spectroscopy, Physical Properties*

■ INTRODUCTION

Graphene refers to a monolayer of carbon atoms arranged in a hexagonal, honeycomb-like lattice. It is the first two-dimensional atomic crystal available to us, which makes it an excellent example of a nanomaterial. As a Nobel prize winning material, graphene has attracted much attention due to its peculiar properties. Graphene exhibits high electrical conductivity (electron mobility up to 200 000 $cm^2\ V^{-1}\ s^{-1}$ at room temperature) and thermal conductivity (above 3000 $W\ m^{-1}\ K^{-1}$). It has a large surface area and at the same time shows outstanding mechanical properties, such as mechanical stiffness, strength, and elasticity. In addition, it is almost transparent because it absorbs only 2.3% of light in the visible region. In terms of these properties, graphene has enormous potential to be used in a wide range of future applications, including electronics, energy storage, and medicine.[1−3] The synthesis of graphene and its derivatives is one of the hottest topics in modern science. Graphene can be produced by mechanical exfoliation of graphite, chemical vapor deposition, or epitaxial growth, and some examples of synthesis methods can be found in previous papers.[4,5] However, among many methods, the chemical approach is recognized as a simple, cost-effective, and accessible way for the preparation of graphene derivatives.[6−15] This method uses graphite to produce graphene oxide (GO), which is a single layer of graphene with oxygen-containing functional groups, such as hydroxyls, epoxy, or carboxylic groups. These functionalities can be removed from GO in the reduction reaction, leaving a material with a graphene-like structure containing defects and residual oxygen groups. However, the properties of the obtained material are different from those of pristine graphene; therefore, the term "reduced graphene oxide" (rGO) should be used for this type of graphene derivative. Reduction of GO can be performed by means of chemical, physical, and even biological methods.[7−15] Most commonly used chemical reducing agents are hydrazine and ascorbic acid (AA). In the experiment, students carry out the reduction of GO with AA, which in contrast to hydrazine, is not toxic and can be safely used by students. This project is ideal for undergraduate students to introduce them to the chemistry lab, as it is easy and requires no complicated equipment. This is an interesting exercise for students who can be involved in the latest research in nanoscience conducted in many laboratories around the world. This fact can increase their motivation and improve their engagement. By participating in this course, students can strengthen their laboratory as well as teamwork skills.

The proposed experiment shows the properties of rGO in comparison to those of GO in relation to their structure. This laboratory experiment can be combined with the previously

Received: July 28, 2017
Revised: April 27, 2018

DOI: 10.1021/acs.jchemed.7b00568
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published article on GO production[6] or can be employed as a separate exercise.

EXPERIMENTAL OVERVIEW

This project can be divided into six experimental sessions, each described in detail in the Supporting Information file. The project is designed for undergraduate students of Nanotechnology or related fields (2nd or 3rd year) within the "Nanochemistry"/"Chemistry of Nanomaterials" laboratory. The class of 12−15 students is divided into groups of 3−4 with one team leader and is supervised by one or two teaching assistants. The introductory class of general chemistry is required for all students participating in this experiment. The whole laboratory experiment takes 15 h. Before the laboratory work, students are taught about graphene as well as the structural properties of graphene, GO, and rGO (about 1 h lecture). Students should also write a short theoretical introduction for their report (maximum one A4 page long) prior to the experiments (see section "Before you start" in Supporting Information). The experiment consists of GO chemical reduction using AA and the characterization of the material using infrared (IR) and UV−vis spectroscopy, along with X-ray diffraction (XRD).

Recording IR spectra for both GO and rGO samples, students were able to recognize the oxygen-containing functional groups in GO, like hydroxyls, epoxy, or carboxylic groups, and to observe their disappearance in the case of rGO, which is consistent with the chemical reduction of GO to rGO. In addition to IR, UV−vis spectroscopy was used to verify the creation of rGO by the reduction of GO by monitoring the red shift of the absorption band, which is due to increased conjugation length. Analysis of XRD results can give information about the crystal size, number, and distance between graphene layers (interlayer spacing) in GO and rGO. Decreasing the interlayer spacing for rGO, compared to that of GO, is connected with the changes in surface chemistry, occurring upon GO reduction. Finally, the electrical properties of rGO and GO are determined using a digital multimeter. It provides a great teaching opportunity to discuss the electronic band structure of solids.

The analysis of the results and the report writing can be done partially at the end of each session or, alternatively, after all sessions. In the report, the experimental observations and conclusions should be documented, and the questions provided by a teacher should be answered. Students are highly encouraged to collaborate with other groups and to discuss the results and talk about the theory hidden in the experiment. As a result of this experiment, students are able to describe the properties of GO and rGO, including its dispersion behavior in different solvents, the level of reduction, and electrical properties.

Materials and Equipment

Graphene oxide was purchased from Sigma-Aldrich or synthesized by students according to our previous article[6]. Ascorbic acid, *N*,*N*-dimethylformamide (DMF), tetrahydrofuran (THF), dichloromethane, 1,2-dichlorobenzene, and propan-2-ol were purchased from Sigma-Aldrich.

Logistics

Depending on a block schedule of the whole course, the experiment might be organized arbitrarily, but the authors would like to point out the crucial steps that should be taken into consideration. The tasks to be done, with timeframes, are given in Table 1.

Table 1. Experimental Design and Suggested Timeline

task	time required, h
theoretical introduction to graphene materials and characterization methods	1
reduction of GO	2−3
removing byproducts (filtering or centrifuging)	1
drying of rGO	overnight at rt or 1−2 h in vacuum dryer at 40 °C
dispersion tests	1
IR spectroscopy analysis	1
UV−vis spectroscopy analysis	1
XRD analysis	1−2
measuring the electrical properties	1
report writing	1−2 h or partially at the end of each session

REDUCTION OF GRAPHENE OXIDE

The synthesis and characterization of GO were reported by us previously.[6] The synthesized or commercially available GO can be reduced under benign conditions. The typical procedure is as follows: 200 mg of ascorbic acid was added to the graphene oxide water suspension (50 mL, 1 mg mL^{-1}). The vial was heated to 90 °C and magnetically stirred for ∼2 h. During the reaction, the color of the suspension turned black and the rGO was obtained. Students washed the precipitate with distilled water and ethanol, centrifuged it, and left it to dry at room temperature for the next lab session. If possible, the rGO can be dried in a vacuum dryer.

Different chemical reducing agents were proposed in the literature, including hydrazine, HI, citric acid, glucose, plant extracts, and more.[8−15] Ascorbic acid, used to reduce GO to rGO, is a nontoxic, mild, and freely available reducing agent, thus it can be safely used by students. GO is an oxidized form of graphene possessing hydroxyl, carboxyl, and epoxide functional groups. During the reduction, oxygen functionalities are removed from GO, and evolution of gases occurs, with CO_2 being the main component. Simultaneously, AA is oxidized to dehydroascorbic acid, guluronic acid, oxalic acid, and finally to CO_2 and H_2O.[14,16,17] The scheme of the reaction is presented in Figure 1.

Chemical reduction is frequently used to reduce GO in order to obtain rGO. However, it is important to emphasize that chemical reduction cannot completely remove the oxygen functional groups from GO, thus the rGO is still very different from the ideal graphene. Moreover, during the oxidation of graphite, the hexagonal carbon network can be partially destroyed, and therefore, the finally obtained rGO can still possess structural defects. Despite that, the chemical reduction method is frequently used for the rGO fabrication, as it is simple, requires noncomplicated apparatus, and allows copious amounts of rGO to be produced in one step.

HAZARDS AND PRECAUTIONS

Personal protective equipment (PPE) must be worn during the experiment: safety glasses, protective gloves, lab coat, long trousers, and shoes that cover the foot. The organic solvents used in this experiment are harmful to human beings and the environment, and all experiments with them should be done

DOI: 10.1021/acs.jchemed.7b00568
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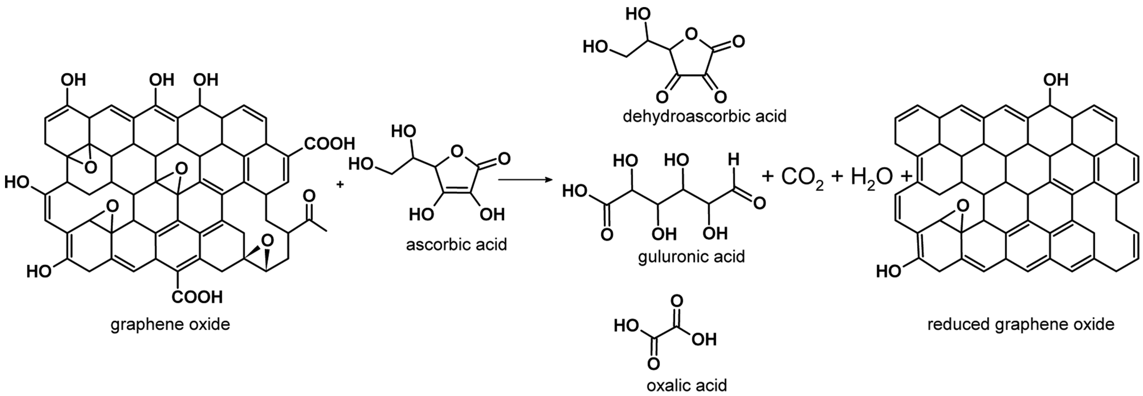

Figure 1. Scheme of GO reduction using AA.

under a fume hood. After usage, the wastes should be disposed in the proper containers, taking special attention to chlorinated hydrocarbons. Graphene oxide, reduced graphene oxide, and ascorbic acid are not classified as hazardous substances (Regulation (EC) No. 1272/2008 of the European Parliament). Before the laboratory session, students must take part in a safety and chemical handling course. Students should also be familiar with the chemicals they are going to use and be aware of all hazards and precautions.

RESULTS AND DISCUSSION

Properties of Reduced Graphene Oxide

Upon chemical reduction of GO, students can observe visual changes in properties of rGO; for example, the color turned from yellow to black (Figure 2). At the same time, the properties changed from hydrophilic to hydrophobic.

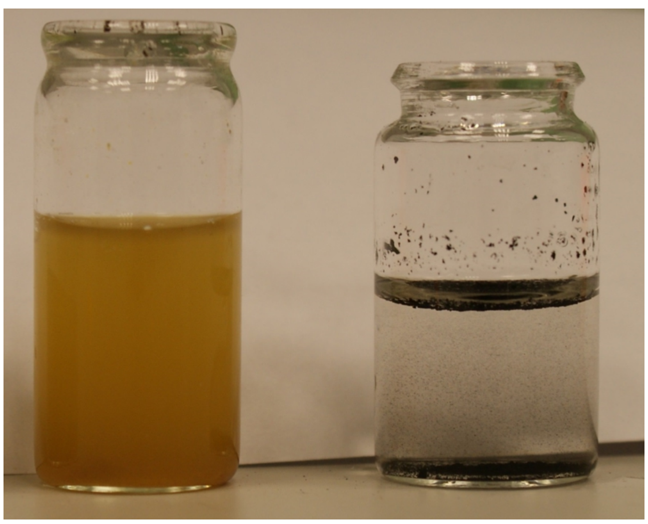

Figure 2. Water dispersions of GO before reduction and produced rGO directly after synthesis.

Students prepared rGO suspensions in different solvents: water, DMF, THF, dichloromethane, and 1,2-dichlorobenzene. The picture illustrating the results of the experiment is presented in the Supporting Information (Figure S3B). By analyzing the polarity of the solvents, students could better understand the connection between dispersion behavior of rGO and polarity. Different solvents can be used, depending on their availability in the lab; however, for the best results, the polarity of chosen solvents should vary. It is also good to compare aromatic and nonaromatic solvents. The comparison of the results for GO and rGO is also presented in the Supporting Information (Figure S3A,B).

Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopy allows students to determine chemical groups that are present in GO and rGO and helps them to examine the level of reduction of rGO prepared using ascorbic acid. Figure 3 shows the FTIR spectra of GO and rGO. The

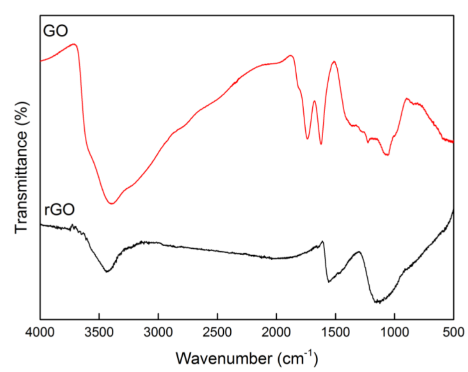

Figure 3. FTIR spectra of GO and rGO.

GO spectrum shows the characteristic bands that are consistent with previously reported results.[6,9,14,16] FTIR bands at 1735 and 1360 cm^{-1}, corresponding to C=O and O=**C−OH**, respectively, confirm the presence of carboxylic groups in GO. Bands at 1225 cm^{-1} (C−OH stretching vibrations) and 1060 cm^{-1} (C−O−C stretching vibrations) were also detected, indicating the presence of alcohol and epoxide functional groups in GO. Characteristic wide bands arising from O−H stretching vibrations were centered at 3422 cm^{-1} for both samples, showing, however, lower intensity for rGO. Moreover, other bands characteristic for oxygen functional groups disappeared in the rGO spectrum, except for the low-frequency band at ~1100 cm^{-1}, ascribed to epoxides. The C=C band is present in the GO spectrum at 1615 cm^{-1} and is shifted toward lower wavenumbers (1544 cm^{-1}) for rGO. It is consistent with the rule that C=C bonds coupled with C=O (as it is in GO) give rise to bands at higher wavenumbers compared to C=C in aromatic molecules (as it is in rGO). Summarizing, the FTIR spectroscopy results confirmed the successful reduction of GO.

DOI: 10.1021/acs.jchemed.7b00568
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UV–Vis Spectroscopy

IR analysis revealed that GO contains oxygen functional groups connected to C═C bonds that are also known to absorb light in the UV range. Thus, the UV–vis spectroscopy can be used to surveil the changes occurring in the GO structure upon reduction. The UV–vis spectra of GO and rGO aqueous suspensions are presented in Figure 4. The main maximum absorption band in the GO spectrum is observed at 230 nm, and the second, smaller band is positioned at 310 nm. The origin of these bands was discussed in our previous paper.[6] Briefly, the first band is due to $\pi \rightarrow \pi^*$ electron transition in conjugated carbon–carbon double bonds in the GO plane, and the second, smaller band is connected to $n \rightarrow \pi^*$ transition of nonbonding electrons in oxygen atoms connected with double C═C bonds (see Supporting Information). Upon reduction, the removal of oxygen functionalities in GO occurs, and the structure of conjugated double bonds is restored. Therefore, the UV–vis spectrum of rGO is different than that of GO. The small band at 310 nm, connected with $n \rightarrow \pi^*$ electron transition, disappeared in the rGO spectrum. Moreover, the 230 nm band is red-shifted to 260 nm. It is consistent with the general rule that addition of conjugated bonds into the structure causes the bathochromic shift of the band position in the UV–vis spectrum. As it was mentioned earlier, after the reduction, most of the carbon–carbon bonds were restored, increasing the overall number of conjugated bonds in rGO.

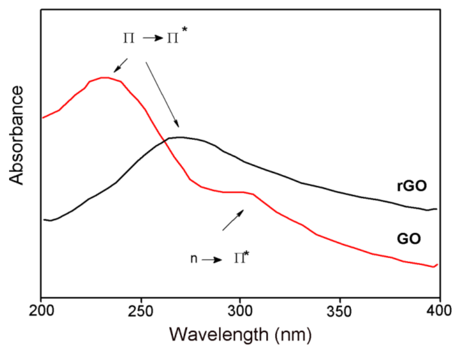

Figure 4. UV–vis spectra of GO and rGO.

XRD Analysis

X-ray diffraction analysis was performed on both GO and rGO in order to measure variations in the interlayer spacing (d-spacing) and the height of the stacking layers (H). Such variations come from the fact that, during the oxidation and reduction processes, different species are intercalated between and removed from individual graphene sheets. As described in our previous works concerning GO,[6,16] the oxidation of graphite causes the introduction of oxygen functionalities between the graphene layers, increasing the interlayer spacing of graphite sheets. In contrast, subsequent reduction removes most of these oxygen-containing groups, resulting in a restacking of graphene sheets and decreasing the distance between them. XRD patterns of GO and rGO are shown in Figure 5. A typical diffraction reflex for pure graphite is observed at $2\theta = 26.5°$ and corresponds to a d-spacing of about 0.336 nm.[16] The XRD spectrum of GO shows a sharp (002) diffraction peak at $2\theta = 10.3°$. Interlayer spacing calculated from Braggs' law was about 0.863 nm. The average height of GO stacking layers was equal to about 22.3 nm, and the number of stacked layers was about 25, as determined by Scherrer's equation (see Supporting Information). A minor diffraction peak observed at $2\theta = 20.1°$ refers to the (002) graphite plane and reveals the trace presence of unoxidized graphite flakes. The diffraction line for rGO occurs at $2\theta = 23.9°$, which corresponds to $d = 0.375$ nm, $H = 2.5$ nm, and, consequently, $n = 6–7$. From the XRD diffractograms, this is clear that the interlayer spacing decreases for rGO, so that the oxygen groups are removed during the AA-assisted reduction of GO.

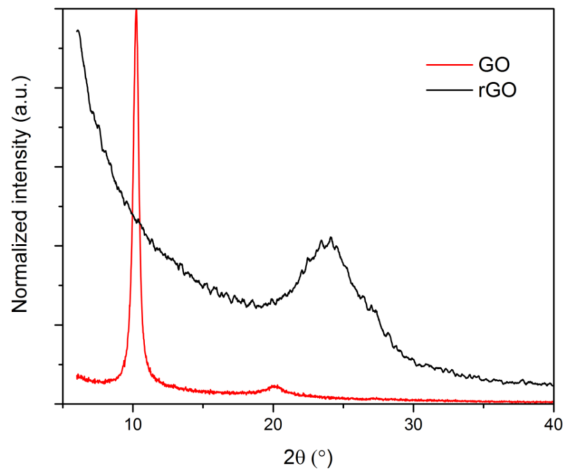

Figure 5. XRD patterns of GO and rGO.

Electrical Measurements

The most distinct difference between GO and rGO is their electrical conductivity. Graphene oxide is an electrical insulator, whereas rGO is a semiconductor with variable band gap energy.[2,9,11,18] The more detailed description of that phenomenon is given in the Supporting Information. In order to examine the electrical properties of GO and rGO, students prepared the strips of paper soaked with GO and rGO. They prepared dispersions of GO and rGO (equally concentrated) and, using a syringe or pipet, soaked the filter papers with the same amount of the material. After drying, the resistance of the prepared strips of paper was measured by using a simple digital multimeter. Alternatively, the GO and rGO pellets were prepared in the powder press and also tested using a multimeter. Usually, the resistance of the strip soaked with GO was not measurable by a multimeter (values out of scale) compared to the rGO-soaked strip (about 700–900 kΩ). In the case of GO and rGO pellets, resistance of GO was measured to be about 5–8 MΩ, whereas resistance of the rGO pellet decreased significantly to about 13–16 Ω. The step-by-step procedures with photographs are given in the Supporting Information.

■ LEARNING OUTCOMES AND ASSESMENT

After completion of this course students will be able:

- to list examples of graphene-type materials;
- to list examples of protic and aprotic, polar and nonpolar solvents;
- to describe differences between GO and rGO;
- to identify the relationship between chemical structure and properties of GO and rGO;
- to perform rGO synthesis;
- to apply known spectroscopic methods (FTIR, UV–vis) to identify functional groups present on GO and rGO;

DOI: 10.1021/acs.jchemed.7b00568
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- to analyze X-ray diffraction results obtained for GO and rGO;
- to perform and interpret the results of GO and rGO electrical properties studies.

Well-organized laboratory experiments should cover a wide spectrum of learning domains, which is realized in the proposed experiment. Next to the lowest levels of learning (first two in the list above), higher levels of learning could be accomplished (other listed learning goals). Students needed to complete the final test and prepare the report, including introduction, results, and discussion. The correct responses to the final report indicated that students were able to apply knowledge to new situations. For example, all students were able to identify the relationship between chemical structure and properties of GO and rGO. They correctly used previously known spectroscopic methods such as FTIR and UV−vis for analyzing and interpreting new data. Having basic knowledge of electronic band structures, they were able to predict and explain the electrical behavior of new materials. In the final assessment, students were asked a series of constructed-response (open-ended) questions, encouraging them to explain the phenomena, construct arguments, and find connections between different disciplines (such as chemistry and nanotechnology). Students received a separate score for the report and the final test (examples are given in the Supporting Information). These scores were averaged to give an overall result for the course. In Figure 6, the results (as a percent of the highest possible score)

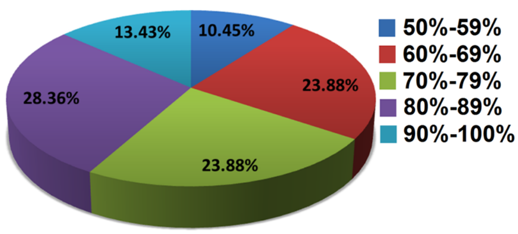

Figure 6. Pie chart presenting the results, as a percent of the highest possible score, obtained by 60 students studying in Gdansk University of Technology in the years 2015−2017.

obtained by 60 students are presented in the form of a pie chart. In order to pass, the total score needed to be at or above 50% of the highest possible score, and all of the students exceeded the point required for passing. As can be seen, more than 65% of students exceeded 70% of the highest possible score, and only ∼10% of students gathered the number of scores in the lowest range. Therefore, the overall results are more than satisfactory.

CONCLUSIONS

Graphene oxide and reduced graphene oxide are versatile and useful teaching materials because of their differences that can be studied using many characterization techniques, including FTIR, UV−vis, or XRD. Moreover, synthesis methods of GO and rGO are simple, safe, and easily adaptable to every undergraduate teaching laboratory. Until now, 20 groups of 3−4 students of Nanotechnology carried out this experiment in the Nanochemistry course. All of them completed the experiment with satisfactory results. During this experiment, all of the student groups in the Nanochemistry class at Gdańsk University of Technology successfully reduced graphene oxide using ascorbic acid. At the same time, they gained hands-on experience with sample preparation for different characterization techniques, as well as with the analysis of the spectra and the use of a digital multimeter. Most of them correctly identified features characteristic for GO and rGO. Moreover, students appreciated the possibility of being involved in the latest research in modern science. We envision that GO and rGO materials can be used in most of the teaching laboratories to encourage students to familiarize themselves with the currently active research field of nanotechnology and nanochemistry, which may benefit their future scientific and professional careers.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.7b00568.

Material list, preparation instructions, results description, examples of exam questions and additional information (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

The project was partially financed by National Science Centre, Poland (Grant No. 2016/23/D/ST5/02800).

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