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Laboratory Experiment

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Synthesizing and Characterizing Graphene via Raman Spectroscopy: An Upper-Level Undergraduate Experiment That Exposes Students to Raman Spectroscopy and a 2D Nanomaterial

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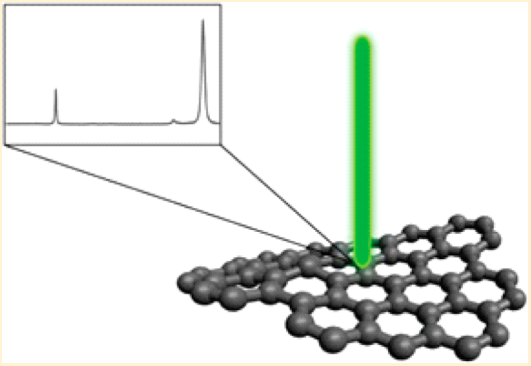
S *Supporting Information*

ABSTRACT: In this upper-level undergraduate experiment, students utilize micro-Raman spectroscopy to characterize graphene prepared by mechanical exfoliation and chemical vapor deposition (CVD). The mechanically exfoliated samples are prepared by the students while CVD graphene can be purchased or obtained through outside sources. Owing to the intense Raman signal of a few-layer graphene on a 300 nm thermal oxide silicon wafer, students can learn how different instrumental parameters used in Raman microscopy affect the quality of the measurement. This experiment gives students a first-hand experience in the production of a two-dimensional nanomaterial and exposes them to the utility of micro-Raman spectroscopy as a characterization technique.



KEYWORDS: *Upper-Division Undergraduate, Analytical Chemistry, Physical Chemistry, Materials Science, Laboratory Instruction, Hands-on Learning/Manipulatives, Nanotechnology, Raman Spectroscopy*

Nanomaterials are a prevalent topic of discussion and research owing to their potential applications from electronics[1] to agriculture.[2] In particular, in 2004 graphene was first produced and studied by Geim and Novoselov through the mechanical exfoliation of graphite. In their approach, adhesive tape was used to mechanically separate graphite sheets.[3] Through repetitive exfoliation, a single layer of graphite (i.e., graphene) consisting of sp^2 hybridized carbon atoms arranged in a hexagonal pattern was obtained. Graphene has remarkable properties, with mechanical strength 300 times that of steel,[4] near ballistic electron transport at room temperature,[3] carrier mobilities[5] up to 200,000 $cm^2\ V^{-1}\ s^{-1}$, and ∼97% optical transparency.[6] The single-layer nature of graphene yields its high structural strength and flexibility compared to graphite, which has weak out-of-plane van der Waals interactions between layers. While mechanical exfoliation produces the highest quality graphene, it does so in low quantities and without control of the size and shape of the resulting flake. Another popular method is chemical vapor deposition (CVD) on metal substrates,[7] which can create sheets of graphene from 30 in.[8] to 100 m in length.[9] This method creates large amounts of graphene, but, the quality suffers, resulting in lower carrier mobilities[8] from defects. However, we note that recently CVD graphene with carrier mobilities similar to that of mechanically exfoliated graphene has been reported.[10] As a result of the outstanding properties and the low cost of synthesis, graphene may have a wide range of potential applications such as replacing indium tin oxide (ITO), an expensive and brittle material widely used in current touch-screen electronics.

Raman spectroscopy can be used as a quick and nondestructive method[11] to determine the number of layers present in a graphene sample. The principle phenomenon behind Raman spectroscopy lies in the interaction of light with the polarizable electron density around a molecule.[12] As the impinging photons interact with the molecule, the electrons in the ground state of the molecule are excited to a higher virtual energy state. Upon relaxation of the excited electrons, photons are emitted. A majority of the photons emitted are elastically scattered, resulting in the strong Rayleigh band that has the same frequency as the impinging light. A small amount of photons that are scattered inelastically give rise to the Stokes and anti-Stokes shifts (Figure 1).

As can be seen in Figure 1, the frequency of the Stokes (anti-Stokes) shifted photons is decreased (increased) from the incoming photon (ν_{ex}) by the vibrational frequency (ν_n) of the normal mode. The intensity of the Stokes shifted peaks are greater than the intensity of the anti-Stokes shifted peaks due to a greater population in the vibrational ground state of the molecule. Due to the low cross section that is inherent to the

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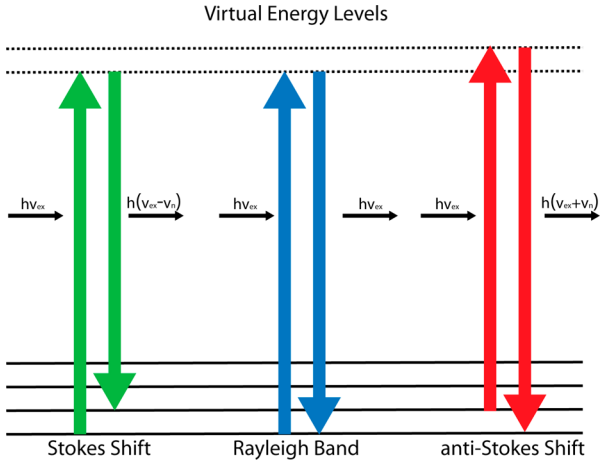

Figure 1. Diagram of an electron being excited to a virtual energy state followed by the emission of elastically scattered photons, represented by the Rayleigh band, and inelastically scattered photons represented by a Stokes and anti-Stokes shift. The solid and dashed horizontal lines represent vibrational energy levels in the electronic ground and virtual excited states, respectively.

Raman process (*ca.* 10^{-7} photons/inelastic scattering event), it is important to understand the effect of the laser power, acquisition time, and grating to maximize the signal.

Graphene has three characteristic Raman peaks critical for its identification and characterization[13,14]: a D peak at ∼1350 cm^{-1}, a G peak at ∼1580 cm^{-1}, and a 2D peak at ∼2700 cm^{-1}. The D peak is the ring-breathing mode of the hexagonal lattice and is symmetry forbidden in defect-free graphene. It is detected when there are defects present (e.g., point defect or edge).[15] The G peak is related to the stretching of the sp^2 C═C bond and the 2D peak is the overtone of the D peak. Both the G and 2D peaks are sensitive to the physical and chemical nature of the graphene sample. The peak position and line shape of all the three peaks are sensitive to graphene thickness and its charge-doping level[16,17]. Given this rich spectroscopy signature, graphene can be a good model system to study Raman spectroscopy.

In this undergraduate laboratory experiment, students follow a step-by-step procedure to produce mechanically exfoliated graphene and subsequently characterize and compare it to CVD graphene through micro-Raman spectroscopy. Students are able to make single layer graphene used for analysis in a 4-hour lab period. Thanks to the intense Raman signal from graphene supported on 300 nm thermal oxide silicon wafer, the grating, laser power, and acquisition time can all be varied to determine their role in the quality and throughput of the Raman data.

EXPERIMENTAL DESCRIPTION

Preparation of Exfoliated Graphene Samples

This method was adapted from existing literature.[3] Three to four flakes of graphite (NGS Naturgraphit GmbH, 500−600 μm size) were placed on an approximately 3 in. × 0.75 in. piece of adhesive tape (Scotch Brand, transparent). The tape was then folded and unfolded in the lengthwise direction repeatedly until it was no longer sticky and was completely covered in exfoliated graphitic material. A silicon wafer with 300 nm of thermal oxide (hereafter referenced as "Si/SiO_2 wafer"; University Wafer; 300 nm thermal oxide, (100), 625 ± 15 μm thickness) was cleaned in acetone for 20 min and dried with a gentle nitrogen flow. The tape, with the side containing the graphitic material facing down, was placed on top of the Si/SiO_2 wafer. Either a Teflon tweezer or one's finger can be used to press and stroke the graphitic materials on the tape onto the Si/SiO_2 wafer using moderate force for approximately 10 min. The tape was removed slowly, and the Si/SiO_2 wafer, which had graphitic material on it, was used in the subsequent measurements. The steps described here are shown visually in Figure 2. We note

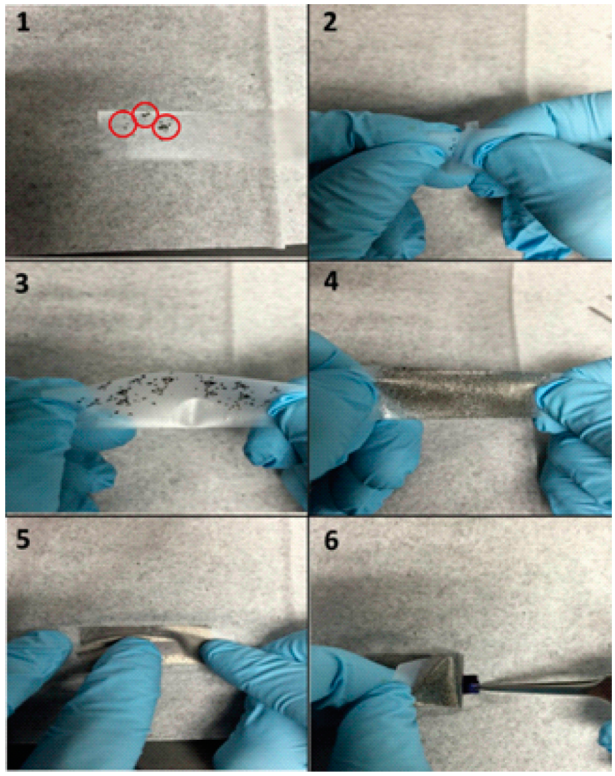

Figure 2. Graphene sample preparation procedure shown visually in a stepwise fashion. (1) Graphite pieces (highlighted in red circles) are placed onto a 3 in. × 0.75 in. piece of scotch tape. (2, 3) The tape is folded and unfolded exfoliating the graphite flakes. (4) The tape is fully covered in exfoliated graphite. (5) The tape is placed on the Si/SiO_2 wafer and either rubbed with one's finger or Teflon tweezers for 10 min. (6) The tape is removed slowly from the Si/SiO_2 wafer, producing varying thicknesses of graphene flakes on the wafer.

that others have also described preparation of exfoliated graphene in a classroom setting.[18]

If funds or resources are available, CVD single-layer graphene can be either synthesized on a metal substrate and transferred onto a Si/SiO_2 wafer or purchased ($59, Graphenea, monolayer graphene on SiO_2/Si (1 cm × 1 cm)) and used for measurements. Others have also reported the synthesis of graphene by wet chemistry and CVD methods in a classroom setting.[19,20] In this experiment, pre-prepared CVD graphene grown on a copper foil and transferred to a Si/SiO_2 wafer (see Supporting Information) was used for characterization through manipulation of the instrumental parameters.

Characterization of Graphene

The prepared graphene was characterized with micro-Raman spectroscopy using a Horiba XploRA ONE system equipped with a 532 nm laser (1 mW laser power; assuming a 1 μm beam

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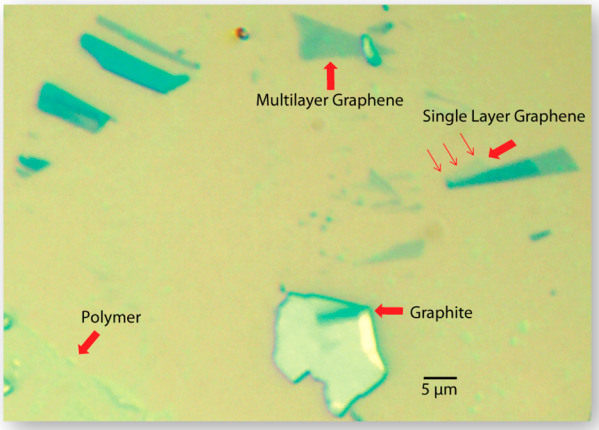

Figure 3. Optical micrograph of exfoliated graphene samples. Thicker graphene produces a larger contrast. The light blue/yellow objects on the bottom left are polymer residue left behind from the tape. Note that it is common to observe a smaller piece of single-layer graphene (outlined by the three small arrows) attached to a much larger and thicker piece of graphite, similar to the example shown here. This image was obtained using an Olympus BH2 microscope with a 50× objective and a low cost USB camera.

size, we estimated that ~3 × 10^7 carbon atoms are within the focus), a 100× objective (0.90 numerical aperture (N.A.)), and a charge-coupled device (CCD) detector. Typically, for Raman measurement a high laser power is desirable to increase signal intensity; however, here a relatively low laser power (1 mW) is used to avoid laser-induced thermal damage of graphene. For this experiment, an 1800 lines/mm (l/mm) grating was used and a spectrum range from 1200 to 2800 cm^{-1} was analyzed. A higher lines per millimeter grating will give better resolution but will reduce the spectrum range. The specific spectrum range was chosen to accommodate graphene's characteristic Raman peaks as well as to exclude the intense Si Raman peak at 520 cm^{-1} that would saturate the detector. Additional data were taken with a Witec Alpha 300 using a 100× (0.90 N.A.) objective.

Graphene was identified by optical microscopy from the contrast produced between the graphene and Si/SiO_2 wafer and verified by the presence of the G peak (1580 cm^{-1}, C═C stretching mode) and the 2D peak (2670 cm^{-1}, overtone of the ring-breathing mode) in the Raman spectrum. To observe graphene under white light illumination, it is necessary to use a silicon wafer with a 300 nm thermal oxide layer to enhance the contrast. Graphene modifies the interference effect due to the thin SiO_2 film; the change in the reflectance resulting in its visibility.[21] Note that even a 5% deviation of the SiO_2-layer thickness can reduce the contrast considerably.[22] As the thickness of the graphene layers increases, their visibility also improves. Figure 3 shows several graphene layers of different thickness. Multilayer graphene flakes appear as a dark blue color, and single-layer graphene samples are barely visible. Often times, a single layer of graphene is located at the edges of a thicker flake. Care should be taken not to confuse the polymer residue for single-layer/multilayer graphene due to similarities in contrast. Unlike graphene flakes, the polymer residues are of a lighter blue contrast with an irregular shape. Additionally, polymer will not produce the Raman signal characteristic of graphene (See Supporting Information).

HAZARDS

The laser used in Raman spectroscopy is a potential hazard if proper precautions are not observed. Follow all safety procedures suggested by the instrument vendor to avoid eye and/or skin injury. Safety glasses and gloves should be worn when in the laboratory and working with chemicals.

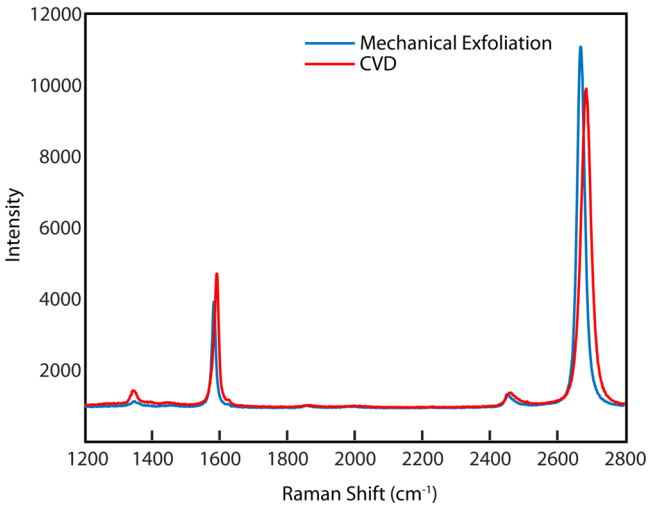

Figure 4. Raman spectra of the mechanically exfoliated (blue) and CVD (red) graphene samples.

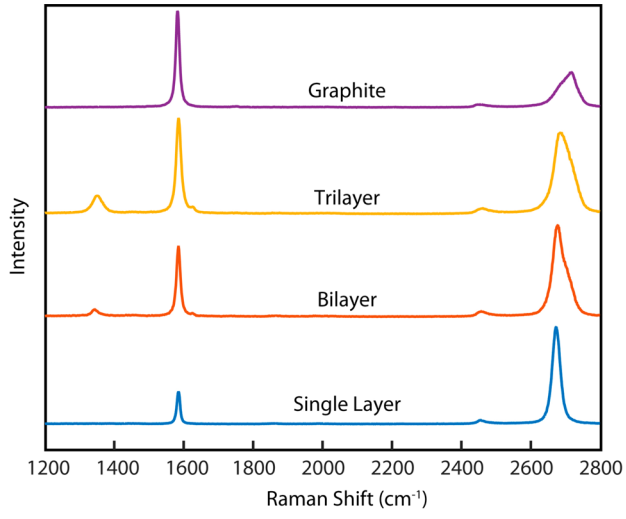

Figure 5. Raman spectra of single, bilayer, and trilayer graphene and graphite. The spectra are shifted vertically for clarity.

Table 1. Peak Intensities and Position with Calculated Ratio of I_G/I_{2D} and FWHM of 2D Peak for Mechanically Exfoliated and CVD Graphene

Sample	I_G (a.u.)	G Peak Position (cm^{-1})	I_{2D} (a.u.)	2D Peak Position (cm^{-1})	I_G/I_{2D}	$fwhm_{2D}$ (cm^{-1})
Exfoliated	3936	1583	11079	2670	0.355	30
CVD	4727	1592	9924	2685	0.476	39

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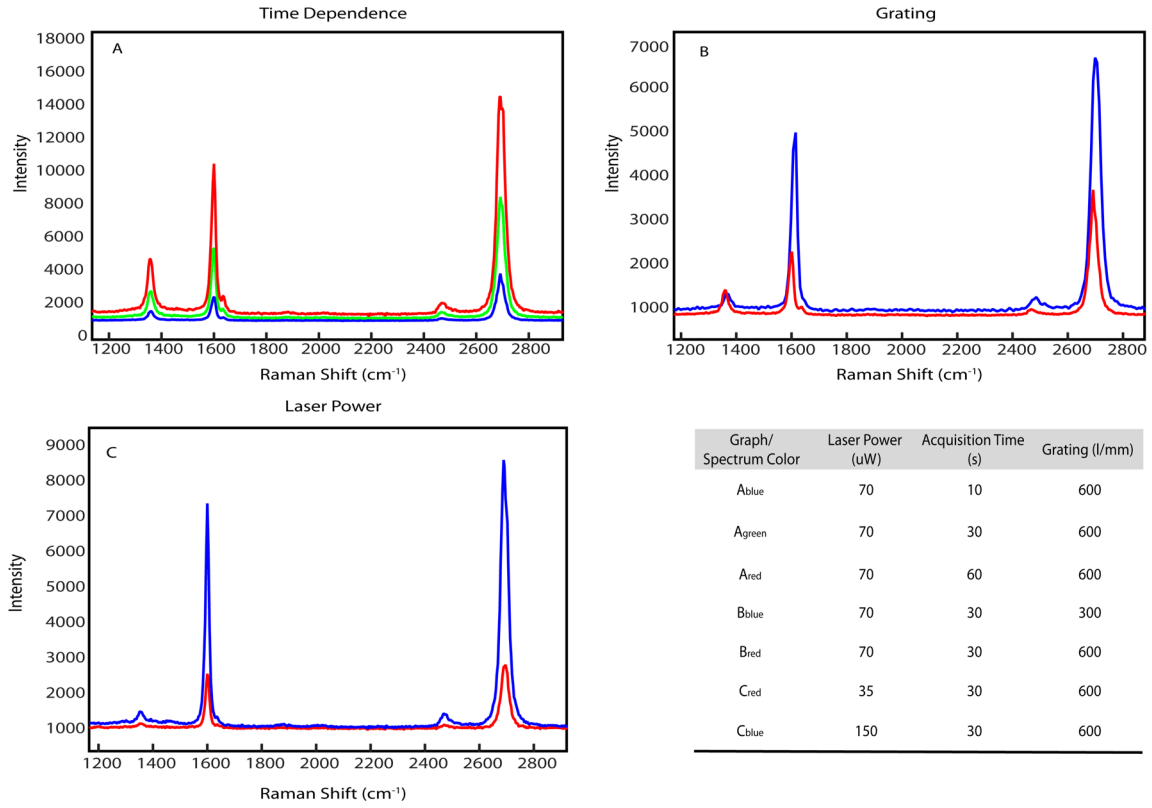
Table 2. Peak Intensities and Positions with Calculated Ratio of I_G/I_{2D} and FWHM of 2D Peak for Varying Thickness of Graphene

No. of Layers	I_G (a.u.)	G Peak Position(cm^{-1})	I_{2D} (a.u.)	2D Peak Position (cm^{-1})	I_G/I_{2D}	$fwhm_{2D}$ (cm^{-1})
1	3322	1584	8125	2673	0.41	30
2	4599	1584	5675	2678	0.81	57
3	7604	1584	6609	2688	1.15	71
Graphite	7830	1581	3471	2713	2.25	84



Graph/ Spectrum Color	Laser Power (uW)	Acquisition Time (s)	Grating (l/mm)
A_{blue}	70	10	600
A_{green}	70	30	600
A_{red}	70	60	600
B_{blue}	70	30	300
B_{red}	70	30	600
C_{red}	35	30	600
C_{blue}	150	30	600

Figure 6. Raman spectra of CVD graphene with varying instrumental parameters. (A) Time dependence of the Raman spectra varying between 10 s (blue), 30 s (green), and 60 s (red) acquisition times. (B) Difference in Raman spectra when acquired using a 600 or 300 l/mm grating (red and blue, respectively). (C) Raman spectra acquired with laser intensities of 35 and 150 μW (red, blue, respectively). These measurements were taken with a Witec Alpha 300 using a 100× (0.90 N.A.) objective; an 1800 l/mm grating was not available on this instrument.

RESULTS AND DISCUSSION

The Raman spectrum of the CVD and mechanically exfoliated single-layer graphene were compared to see the impact of the different synthetic methods. The characteristic graphene peaks at 1580 and 2670 cm^{-1} and the ratio of their intensities (I_G/I_{2D}; ca. ~0.24) along with the line shape (single Lorentzian) and full width at half-maximum (fwhm) of the 2D peak (ca. ~30 cm^{-1}) confirm that both samples are single-layer graphene[16] (Figure 4 and Table 1). The differences in the positions of the G and 2D peaks and I_G/I_{2D} values of the two samples are attributed to variations in charge-doping level and/or strain.[23] The CVD sample shows a small D peak at ca. 1360 cm^{-1}, which is due to the polycrystalline nature and/or defects in the sample. In contrast, the D peak is much weaker for the exfoliated sample, indicating its high quality.

The mechanically exfoliated sample provides flakes of varying thicknesses that allow students to see the changes in intensity, line shape, and peak position of the characteristic graphene peaks as the number of layers increases (Figure 5 and Table 2). In multilayer graphene, the 2D peak broadens and shifts to higher wavenumbers due to the electronic coupling between layers. The optical contrast and the G peak intensity increase almost linearly as the graphene thickness increases until about 10 layers;[24,25] this phenomenon can be used to differentiate bilayer and trilayer graphene by visually inspecting the optical micrograph image and analyzing G peak intensity using the Si overtone (ca. 950 cm^{-1}) peak as an internal reference. Note that both doping and strain could also change the G peak intensity (see above). The slight defect peak on the trilayer graphene sample is likely due to the laser spot being focused on the edge of the graphene flake, activating the ring-breathing mode.

CVD-grown graphene is much easier to visualize due to its larger size. The students can use this sample to acquire the Raman spectrum of a single-layer graphene using different instrumental parameters such as laser power, grating, and acquisition time. This exercise gives students hands-on exposure while maximizing their signal and provides an opportunity to learn about the trade-offs associated with each acquisition parameter. Figure 6 highlights the influence of each instrumental parameter on the intensity of the G and 2D peaks in the Raman spectrum. As can be seen, the Raman signal is proportional to the acquisition time and laser power (Figure 6A,C). Although increasing

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these parameters provides a stronger signal, it may be necessary to keep both of these parameters at a minimum to avoid photothermally induced degradation of the graphene sample. The type of grating determines the resolution and the spectral range of the measurement (Figure 6B). The 300 l/mm grating gives a higher intensity, but spectral resolution suffers. The 600 l/mm grating resolves the D′ peak near the G peak and gives a more defined 2D peak at the cost of a weaker signal.

Learning Assessments

The experiment was conducted in an upper-level Instrumental Analysis Laboratory course at the University of Pittsburgh. Formative assessment was carried out using knowledge checks (KCs). KCs are a list of questions generated by undergraduate teaching assistants (UTAs) who have completed the course and are designed to gauge the students' understanding of the experiment. During the laboratory session, the students were given three opportunities to answer two of the KC questions correctly, which 85% of the students were able to do. Upon completion of the experiment, students are also required to complete a formal laboratory report, in which they address discussion questions (Supporting Information) and present their results in written format. The discussion questions have the students identify the differences in their spectra between the CVD and mechanically exfoliated sample along with changes found using different acquisition parameters. From the students' reports, 69% were able to correctly identify the differences in the Raman spectra among the different samples and 78% of the students were able to recognize how their spectrum changed based on the adjustments in the acquisition parameters. The difficulty in the experiment arises from the challenge of discovering single-layer graphene with an optical microscope from the mechanically exfoliated sample. As seen in Figure 7, of the 53 students who

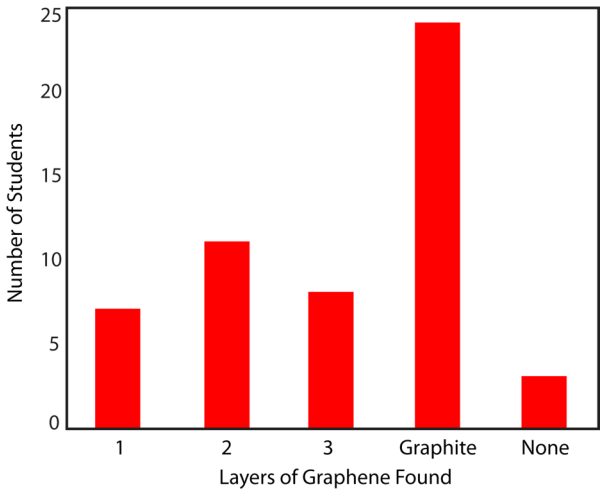

Figure 7. Distribution of students that successfully identified exfoliated graphene and graphite.

completed the experiment, 26 were able to find monolayer, bilayer, or trilayer graphene. Although 24 students found only graphene that was three or more layers in thickness, they were still able to observe the changes in the Raman spectra as a function of the instrumental acquisition parameters and confirm that their mechanically exfoliated sample was greater than three layers in thickness. Additionally, three students were unable to find any graphite or graphene on their exfoliated samples. It is likely that these students did not apply sufficient pressure to transfer the exfoliated graphite from the adhesive tape onto the silicon wafer.

CONCLUSION

In this laboratory experiment students explore Raman spectroscopy through the analysis of different graphene samples. Parameters such as laser power, grating, and acquisition time are altered so the students can learn to optimize the resolution of a spectrum while keeping a reasonable throughput. Students obtain hands-on experience in preparing a two-dimensional nanomaterial by producing their own mechanically exfoliated graphene through a step-by-step procedure and comparing it to CVD graphene with optical and micro-Raman spectroscopy. This experiment exposes students to spectroscopic analysis techniques that are commonly used in materials science and nanotechnology research. It shows the qualitative and quantitative structural information Raman spectroscopy offers and its usefulness in characterizing nanomaterials such as graphene. Additionally, students acquire first-hand experience in manipulating instrumental and optical parameters commonly found throughout instruments in the analytical sciences. The students' experiences with the complexities involved in preparing nanoscale materials using mechanical exfoliation will provide them with a practical appreciation for the value of scalability in synthesis techniques, as is found in the CVD synthesis of graphene.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.6b00198.

Detailed instructions for students and instructors (PDF)
Detailed instructions for students and instructors (DOCX)

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Notes

The authors declare no competing financial interest.

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