Experimental Nanomaterials and Nanoscience - An Interdisciplinary Laboratory Course

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1. Introduction

Nanomaterials, nanotechnology and nanoscience play a key role in the advancement of modern technologies in sensors, medicine, renewable energies, and more. Globally, governments and industries have made significant investments in this field both intellectually and economically. In the United States, federal funding agencies have invested tens of billions of dollars on R&D and commercialization of nano-products as well as understanding of the social environmental impacts.[1] The National Science Foundation (NSF) projected that the nanotechnology sector will employ 6 million workers by 2020 and over 80% of the jobs will require trained workforces in nanoscience and nanotechnology.[2] To meet the projected future demand, it will be greatly beneficial to provide students education and hands-on experience in nanoscience and nanotechnology starting from the undergraduate level. However, a majority of current workforces in the nano-sector receive their education at the graduate level, while only a few institutions have taken the initiative to train undergraduates both on fundamental principles and experimental skills in this highly specialized field.

To gain insights of materials and science at the nanoscale, physics, chemistry and engineering have all combined in a coherent manner. With the funding support from NSF, a new interdisciplinary combined lecture-laboratory (1 credit hour lecture and 2 credit hours laboratory) course, entitled *EXPERIMENTAL NANOMATERIALS AND NANOSCIENCE,* was developed and has been successfully offered two terms at Wright State University. This course is taught by a team of faculties together with graduate research students representing the Department of Mechanical and Materials Engineering, the Department of Chemistry, and the Department of Physics. This lab-intensive course covers a large breadth of research-inspired topics from fabrication and characterization, to application of nanomaterials including metal nanoparticles, quantum dots, and graphene nanosheets. The series of laboratory modules complement and reinforce the concepts students learned in lecture courses in the areas of chemistry, physics, materials, engineering, and nanotechnology. This course is highly appraised by the participating undergraduate and graduate students according to the anonymous student evaluations.

2. Course Contents

Nanomaterials are built upon four basic nanoscale blocks, i.e., 0D, 1D, 2D, and 3D, classified in dimensionality. These nanomaterials can be fabricated through either bottom-up or top-down approaches. Bottom-up refers to the approach starting with atomic/molecular reaction followed by self-assembly formation control, while top-down refers to the one using macroscopic bulk precursors to physically or chemically process nanostructured materials. The size effect and unique dimensionality of nanomaterials render their novel properties and functionalities for advanced applications. Quantum confinement will result in distinguished properties from bulk materials. Large surface area translates to a significantly great number of adsorption/reaction sites that are available to interact with other species, ideal for charge storage, sensing, catalysis, etc. There exist various microscopic and spectroscopic pathways to characterize nanomaterials' structure, morphology, and physical/chemical properties. The laboratory modules selected in this course covered the above-mentioned five key contents in the nanotechnology sector, which are comprised of materials, fabrication, characterization, properties, and applications. Table 1 summarizes the covered contents in this novel lecture-laboratory course and Table 2 lists the detailed weekly laboratory module.

Quantum dots (QDs) are 0D semiconductors in which electron movement is confined in all three dimensions. Since their electronic characteristics can be finely tuned by adjusting the size and shape of the individual crystal, QDs are being developed for a wide variety of applications including sensors and biomedical imaging.[3-5] Fabrication and manipulation of metallic nanoparticles, e.g., silver nanoparticles (AgNPs), is an extremely active research area due to their numerous applications to catalysis, photonics, electronics, SERS-based sensing, and pharmaceuticals.[6-10] QDs and AgNPs can be fabricated through typical low-cost bottom-up colloidal dispersion reactions. The 2D-structured graphene exhibits many astonishing properties such as high intrinsic mechanical strength, high thermal conductivity, high specific surface area, and high electron mobility. The dramatic advancements in graphene science and technology have opened up unlimited opportunities for the development of nano-enhanced products and manufacturing innovations. [11-13] Graphene-based nanocomposites have found extensive applications in the field of renewable and clean energy, serving as high-performance electrode materials or supporters in Li-ion batteries, supercapacitors, fuel cells, and solar cells.[14-18] Graphene nanosheets (GNSs) can be mass-produced from graphite through a series of oxidation/intercalation, exfoliation, and reduction processes. For this course, the QD specimens were purchased from commercial vendors. Colloidal AgNPs were synthesized through the widely-used Creighton and Lee-Meisel methods.[19-21] GNSs were synthesized through the modified Hummers method.[13-15] Besides learning approaches of fabricating AgNPs and GNSs, students further gained hands-on experience in operating various modern characterization instruments like FES, UV-VIS, ICP-OES, RS, AFM, SEM, and XRD. Additionally, students were taught to use computational methods for modeling nanoscale physical phenomena. Moreover, students were introduced to the industrial process of manufacturing Li-ion batteries and gained hand-on experience with an environmentally controlled glove box, tape-cast film coater, button-cell crimping machine, and battery testing station to fabricate and assess button-cell Li-ion batteries.

Table 1. Summary of contents covered in this lecture-laboratory course, which is categorized based on the five key components in the nanotechnology sector.

Materials	0D: nanoparticles (Si quantum dots and Ag nanoparticles) 2D: nanosheets (Graphene nanosheets)
Fabrication	Bottom-up: colloidal dispersion Top-down: chemical exfoliation
Characterization	Scanning Electron Microscope (SEM), Atomic Force Microscope (AFM) Raman Spectroscopy (RS), UV-VIS Transmission Spectroscopy(UV-VIS), Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), Fluorescence Emission Spectroscopy (FES), X-Ray Diffraction (XRD)
Properties	Electrical, optical, chemical/electrochemical
Application	Health care (biomedical imaging and sensing); Energy (Li-ion battery)

Table 2. A list of weekly laboratory modules and a brief description of each module.

Lab #	Lab Contents
1	Fabrication of noble metal colloidal nanoparticles Silver nanoparticles (AgNPs) were synthesized using two well-established, wet-chemistry method by the reduction of Ag^+ in silver nitrate with sodium borohydride and sodium citrate at low and high temperatures, respectively (Creighton and Lee-Meisel methods). Afterwards, students were encouraged to explore their own novel approaches using a "green" method, which is energy-efficient and uses non-toxic and renewable reducing reagents such as glucose, starch, honey, coffee, tea and fruit extracts.
2	"Green" manipulation of AgNPs by tangential flow ultrafiltration Students learned to use tangential flow ultrafiltration to perform the size-selection, concentration and purification of AgNPs with minimal aggregation.
3	Characterization of colloidal AgNPs concentration by UV-VIS and ICP-OES Students became familiar with the theoretical and experimental aspects of UV-VIS and ICP-OES for the quantification of the particle size and concentration of silver nanoparticles.
4	Estimating the SERS enhancement factors of noble metal nanomaterials Students learned to obtain Raman spectra and then to estimate the SERS (Surface Enhanced Raman Spectroscopy)-sensing efficiencies of colloidal AgNPs by calculating their analytical and surface enhancement factors.
5	SERS molecular imaging of cells using noble metal nanomaterials This lab demonstrated that AgNPs can serve as SERS-based nanosensors for various subcellular compartments at a low dose.
6	Silver nanoparticle adsorption to solid surfaces Students were taught the basic principles of AFM and later learned to apply AFM to characterize the extent of adsorption and other spatially-dependent characteristics of nanoparticle-surface interactions.
7	UV-VIS and FES characterization of quantum dots Students became familiar with UV-VIS and FES to estimate the size and concentration of QDs.
8	Terahertz spectroscopy of nanomaterial composite systems Students were introduced to the operation and utilization of a commercial terahertz time-domain spectroscopy system and to determine how the bulk optical properties, i.e. refractive index, absorption, conductivity, change with nanoparticle concentration and size.
9	Using computational methods to model the nano-world Students were taught the basic quantum effect concepts and perform simulations and calculations, using pre-developed softwares, to model nanoscale physical phenomena.
10	Fabrication of graphene nanosheets and electrode coating Students fabricated GNSs via the modified Hummers approach, which basically entails three distinct sequential procedures: oxidation/intercalation, exfoliation, and separation. Afterwards, they conducted electrode coating manually and automatically, which is a necessary process for GNSs electrochemical performance assessment.
11	Microstructural characterizations of GNSs and Swagelok half-cell assembling Students were taught to operate on XRD and SEM and to compare the structural and morphological difference between graphite and GNSs. Additionally, students learned to assemble Swagelok half cells in an environmental-controlled glove box and then set the electrochemical tests on a battery testing station.
12	Electrochemical performance analyses of GNSs and Li-ion button-cell assembling Students were taught to quantify a series of electrochemical performances of GNSs, based on the obtained experimental data, such as Li-storage specific capacity, columbic efficiency, rate capability, and charge/discharge cycling behavior. The results were compared with those of graphite. Students independently fabricated actual button-cell Li-ion batteries.
13	Guidelines for data analysis and writing a journal article-like laboratory report Students were introduced basic scientific writing process of journal article-like laboratory reports and scientific data analysis process.
14	Final Presentation

Undergraduate students usually have a very limited experience with the scientific writing process, data analysis, and preparation of high-quality figures. Within this course, one laboratory module was entirely dedicated to teach students the writing process of journal article-like laboratory reports and scientific data analysis. In this context, faculty elaborated in detail the structure and content of a journal article-like lab report, the preparation of a professional oral presentation, and provided explicit examples related to the experiments. Further, students were introduced to various software packages available for the analysis of the collected data (*e.g.* Microsoft Excel and Origin 8) and were walked through several exemplar data obtained from their experiments. During the computer-based demonstrations, students learned how to analyze and to plot their own laboratory data.

The course is suitable for meeting either once or twice per week. In fall 2012, the course was offered once per week. To accommodate students' schedules and to provide students with more hands-on time on the state-of-the-art instrumentation, the course was offered twice a week in two sessions in fall 2013. The lab sequence schedule listed in Table 2 is grouped based on the contents but they can be varied if necessary. For instance, Lab #13 in Table 2 was actually offered in week 3, after students completed a few lab practicums and before they started to write their first journal article-like lab report. In the last week of the semester, students will give a 10-minute presentation on the selected laboratory module/topic that matches better with their future research/career interest.

3. Exemplary Experimental Results

Due to the page limitation, the experimental results of the three-week lab series on GNSs were selected for presentation in this paper (see figures 1-4). Laboratory results on AgNPs related contents have been published in the Journal of Chemical Education. [22,23] Laboratory results from the QDs experiment will be submitted for publication elsewhere.

Figure 1 displays qualitative changes in color of the reaction suspension that students observed during the fabrication of GNSs in Lab #10. The initial black graphite suspension turned into purple color after harsh oxidation with the addition of $KMnO_4$. Later, H_2O_2 was added to cease the oxidation process, and the solution changed to yellow. With the progress of reaction at 135^oC, the solution gradually changed to light brown graphite oxide suspension and eventually to black suspension indicative of the formation of graphene oxide nanosheets. The graphene oxide was then filled and later, thermally reduced in a box furnace at 250^oC to obtain the desired GNSs. After chemical exfoliation reaction, the 3D-structured graphite completely transformed into the 2D-structured reduced graphene oxide nanosheets. Figure 2 presents XRD profiles and SEM images of graphite and GNSs powders students obtained in Lab #11, which manifest the distinguished structure and morphology between graphite and GNSs. The obtained GNSs powders were coated on a copper foil and the coated film was cut into $1cm^2$-sized discs to be used as the electrodes in both lithium half-cell and Li-ion button cell. The cells were assembled in an environmentally-controlled glove box with water and oxygen concentrations less than 5ppm (see figure 3). In Lab #12, both the half-cells and Li-ion cells students assembled were subjected to performance evaluation. Based on the obtained voltage-time-current values, which were automatically recorded on battery testing stations, students calculated Li-storage specific capacity, columbic efficiency, charge/discharge cycling behavior of GNSs, and compared with the characteristics of graphite. Figure 4 shows the discharge/charge profiles of graphite and GNSs at two different current densities, i.e., 50μA/cm^2 and 200μA/cm^2. Apparently, GNSs has a reversible Li-storage capacity of 820mAh/g. This value is over 3 times higher than the capacity of graphite (260mAh/g). Moreover, GNSs retains its high capacity at the increased current density, which is significantly better than graphite, rendering its potential high power density when it is applied in Li-ion batteries.

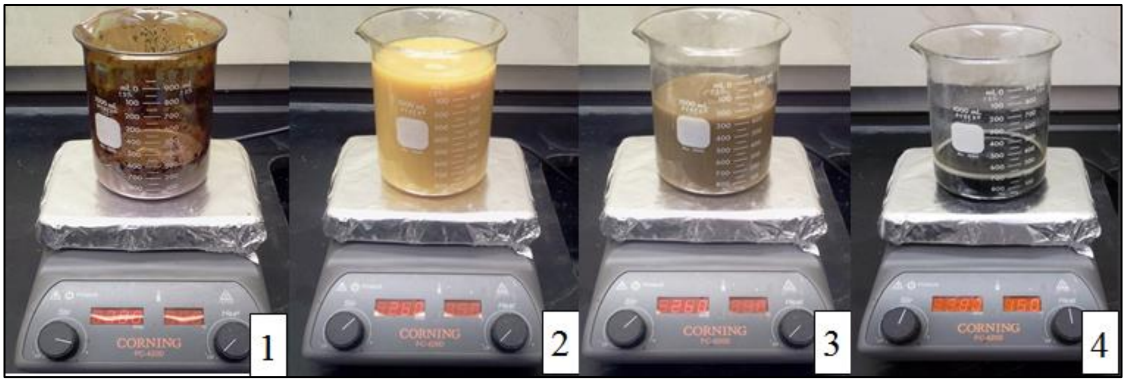
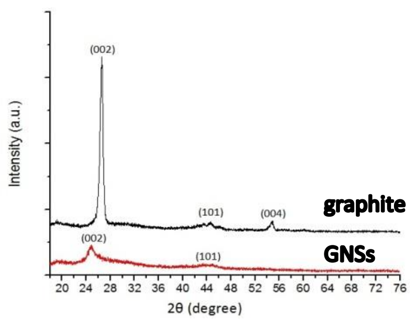


Figure 1. Qualitative changes observed during the fabrication of GNSs: (1) purple suspension after harsh oxidation with $KMnO_4$, (2) yellow suspension after the addition of H_2O_2, (3) light brown suspension indicative of formation of graphite oxide, and (4) black suspension indicative of formation of GNSs.

(a)



(b)

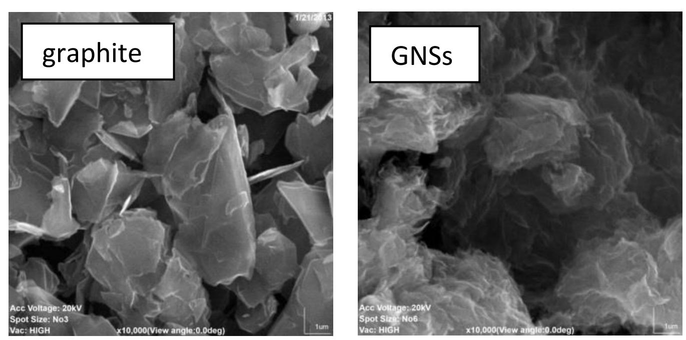


Figure 2. (a) XRD profiles and (b) SEM images of graphite and GNSs powders showing their distinguished structure and morphology.

(a)

(b)

(c)

Figure 3. (a) Swagelok cell used for assessing lithium storage performances of GNSs and graphite; (b) practical button-cell Li-ion battery containing GNSs anode after crimping process; and (c) the environmental controlled glove box where the cells were assembled.

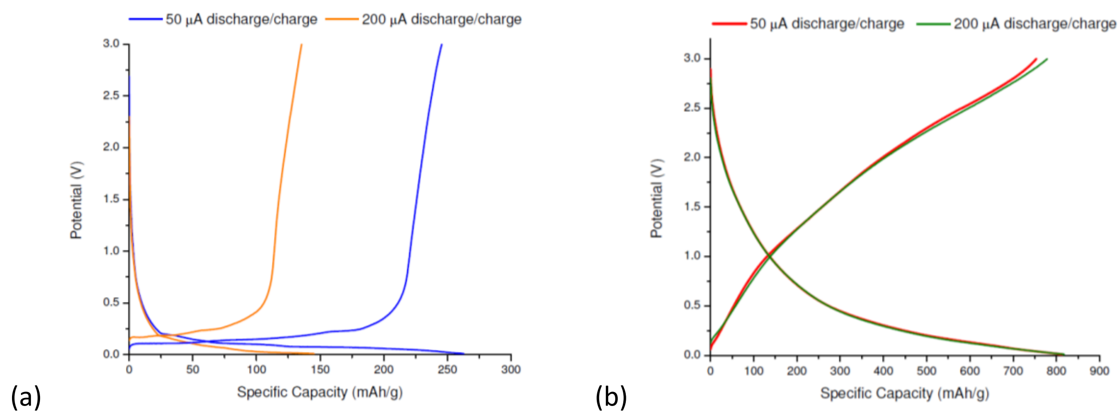


Figure 4. Discharge/charge profiles of graphite (a) and GNSs (b) at two different current densities, i.e., 50 $\mu A/cm^2$ and 200$\mu A/cm^2$.

From this set of laboratory modules, students gained not only basic knowledge of graphene-based nanomaterials and their benefits for Li-ion batteries but also extensive hands-on experiences in fabrication, characterization, and performance assessment of GNSs. According to the student evaluation results (see in the following section), students enjoyed their unique experiences in the fabrication of the state-of-the-art GNSs nanomaterials and actual Li-ion batteries in the glove box. This set of laboratory modules will also be beneficial to develop students' scientific awareness of the nanomaterials contribution to renewable and clean energy.

4. Student Survey Evaluations

A course evaluation form, with emphasis on the students' learning interest before (Q1) and after (Q2) their completion of the experiment, the overall laboratory experiences (Q3), and the laboratory content ratings (Q4), was anonymously administered for each laboratory module on the 12 hands-on laboratory modules during fall 2012 and fall 2013 semesters. In average, thirteen students voluntarily participated in the evaluation per semester. The evaluations were based on a scale from 1 to 10, with 1 being extremely poor and 10 being excellent.

Table 3 summarizes the evaluation results based on the average of all twelve hands-on experiments in terms of Q1-Q4 obtained in two semesters. The student evaluations indicated their learning interest increased significantly after their completion of the experiments. The students truly enjoyed the course and rated their experiences consistently high during both academic years. Students' evaluations together with their comments are also very helpful for the improvement of the laboratory design. Based on the students' feedback received in fall 2012, the investigators improved the Power Point presentations and standard operating procedures (SOPs), and further made some adjustments on laboratory planning and scheduling. As a result, the ratings were improved in fall 2013.

Figure 5 displays the histogram showing the class ratings of each laboratory module obtained in fall 2013. Eight out of the twelve labs for Q2-Q4 were rated around 9.5 out of 10 manifesting the students' true interest and good experiences with the novel, high-quality nanolab course. According to students' comments, the relative low rating on Lab #6 is most probably due to the limited hands-on access to the AFM equipment. Students also commented that Lab #9 would have been more enjoyable if the software on the nanohub website was working properly, which might be attributed to the relative low rating of Lab #9. It is anticipated that the class rating will be further improved with the purchase of more instruments, softwares, and supplies.

Table 3. Students' evaluation results based on the average of all twelve hands-on experiments in terms of Q1-Q4 obtained in two semesters.

	Fall 2012	Fall 2013
Q1: Overall interest before the experiment	8.36	8.49
Q2: Overall interest after the experiment	9.02	9.21
Q3: Overall experience	9.12	9.26
Q4: Overall rating	9.14	9.31

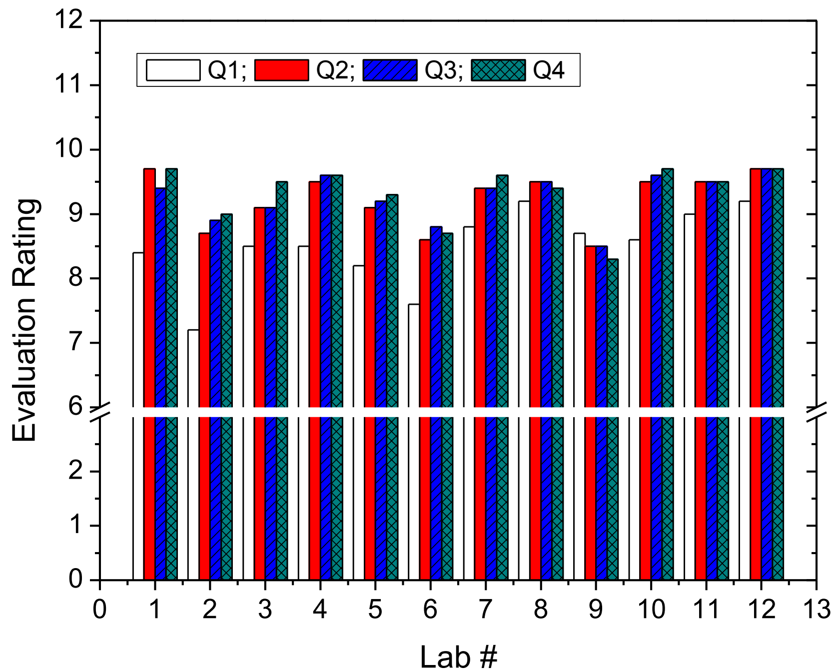


Figure 5. The histogram showing the overall class ratings of the twelve hands-on laboratory modules in the aspects of Q1-Q4 obtained in fall 2013.

5. Conclusion

At Wright State University, we have developed an interdisciplinary laboratory-intensive course: Experimental Nanomaterials and Nanoscience. This course has been successfully offered two terms and is highly appraised by the participating students. It is hopeful that the presented course can be disseminated nationwide to benefit the development of the next- generation nanotechnology engineers and scientists. Courses focusing on nanoscience and nanotechnology present a unique educational platform in that they allow for teaching interdisciplinary principles, while simultaneously covering the cutting–edge developments that are tied closely to applications. Such novel courses will enhance intellectual enthusiasm and passion for STEM studies and research. Meanwhile, they can provide faculty and students with new research and education collaboration opportunities that cross traditional disciplinary and departmental boundaries.

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