
AC 2011-620: NANOTECHNOLOGY IN UNDERGRADUATE EDUCATION: DEVELOPMENT OF EXPERIMENTAL MODULES

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Nanotechnology in Undergraduate Education: Development of Experimental Modules

Introduction

This paper discusses the development of experimental modules to provide hands-on experience for undergraduate students interested in nanoscale science and technology in the College of Engineering and Applied Science (CEAS) and the College of Arts and Sciences (A&S) at the University of Cincinnati. The modules were integrated into two new courses that were developed as part of a grant from the National Science Foundation *Nanotechnology in Undergraduate Education (NUE)* program to enhance undergraduate education in nanotechnology and engineering at UC. Four modules were developed for the new course *Nanoscale Devices*, which addresses important contemporary issues including design, construction, and emerging applications of nanoscale devices; three additional modules were developed for the new course *Environmental Aspects of Nanotechnology*, which discusses environmental applications of nanotechnology as well as the environmental impact of nanotechnology. The new courses build on the background that students have gained in existing courses entitled *Introduction to Nanoscale Science and Technology* and *Experimental Nanoscale Science and Technology* and provide students at UC with an outstanding educational experience in nanoscale science and engineering. The new and existing courses support UC students participating in the *Engineering Research Center (ERC) for Revolutionizing Metallic Biomaterials* in which UC partners with lead institution North Carolina Agricultural and Technical State University and the University of Pittsburgh. They also address the need for a technologically advanced workforce in the areas of nanomaterials and nanotechnology as expressed by Ohio's *Third Frontier Project*¹ and *Deloitte Study*² and by employers in UC's internationally acclaimed mandatory co-op engineering program. All four courses are offered yearly, are cross-listed by CEAS and A&S to make them available to students in many different disciplines, and will eventually form the basis for a minor focused on interdisciplinary nanoscale science and technology.

Initially, three modules were developed for the course *Nanoscale Devices* and were entitled "Synthesis of Nanoparticles as Sensor Building Blocks," "Making Wireless Biosensors Using a Nanomanipulator," and "Electrochemical Sensors in Fluidic Channels." However, a fourth module entitled "Introduction to Carbon Nanotube Applications" was subsequently developed. Three additional modules were developed for *Environmental Aspects of Nanotechnology*; they were entitled "Magnetic Separation of Toxins by Applied Fields," "Magnetic Measurement of Nanoparticles," and "Active Nanosystems for the Destruction of Toxins in Water." Details of the modules as well as results obtained during the first presentation of *Nanoscale Devices* during the autumn quarter of 2010-2011 are discussed.

The experimental modules were adapted for presentation to students in the Summer Institute (SI) conducted by CEAS as part of an outreach program. SI is a five-week program designed to increase the awareness and interest of underrepresented ethnic students in STEM fields. The program targets students from high schools and junior high schools throughout the

greater Cincinnati area. Results obtained from presentation of the modules in SI during the summer of 2010, including extensive feedback from students, are also discussed.

Experimental modules for the new courses

A brief description of the modules is given below; modules M1-M4 are intended for students in the course *Nanoscale Devices* while modules M5-M7 are intended for those in *Environmental Aspects of Nanotechnology*. Most of the facilities required are available in UC's *Nanoworld* laboratory which is comprised of three separate laboratories, including a Nanotube Synthesis Laboratory for carbon nanotube production, a Nanostructured Materials Processing Laboratory where nanomaterials are modified and incorporated into other materials such as polymer nanocomposites, and a Smart Materials and Devices Laboratory where nanomaterials are manipulated, assembled, and processed into sensors, actuators, devices, which are then evaluated for practical applications.

Equipment available in the Nanotube Synthesis Laboratory for the experimental modules includes EasyTube Model 1000 and Model 3000 nanofurnaces from FirstNano, Inc. for the CVD computer controlled synthesis of nanotubes including liquid-vapor delivery system. The Nanostructured Materials Processing Laboratory includes a vacuum processing glove box, vacuum oven, other ovens, balances, molds for casting nanocomposite materials, and a custom built machine for spinning of carbon nanotube arrays into threads. The Smart Materials and Devices laboratory includes nanomanipulators for processing nanotubes and an environmental scanning electron microscope (ESEM) with a stage for incorporating the nanomanipulators. Several instruments were purchased for this project, including a microgripper system, a force measurement system, and a rotational tip, which are all accessories for the nanomanipulators. A computer system to support the use of the nanomanipulators was also purchased, as were an arc lamp system, particle size analyzer, and potentiostat system.

Module M1: Introduction to carbon nanotube applications. In this module, students learn the physics of carbon nanotubes (CNTs) and their applications through demonstrations and hands-on experiments that are organized into four sub-modules that provide the students with an understanding of CNTs and introduce them to basic engineering skills. The four sub-modules are described below.

a. Measuring the resistance of CNT threads using robots. Students measure the resistance of CNT threads and copper wires in this sub-module using a robotic manipulator to simulate working under a microscope while manipulating nanotube materials. The students obtain an understanding of robot manipulation and compare resistance measurements made by hand with a multi-meter to measurements made using a robot and the multi-meter. Joysticks are used to move probes so as to contact screws that are connected to copper wires or nanotube threads. The robots are the same as those used to manipulate nanotubes in an electron microscope, providing a unique opportunity for students to use sophisticated instrumentation for nanotechnology research.

b. Building an electromagnetic solenoid. The objective of this sub-module is to use fine copper wire to build an electromagnet solenoid. In the future, the copper wire may be replaced by CNT thread to make a smaller solenoid and eventually a micro-manipulator for nanomedicine applications. Students obtain a basic understanding of electromagnetics and how a linear

actuator can be used to build a manipulator. Students use copper wire, a switch, battery, breadboard, and iron needle core to make a solenoid or manipulator with one degree of freedom.

c. *Spinning carbon nanotubes into thread.* In this sub-module, the spinning of CNTs into thread is demonstrated so as to provide students with a basic understanding of how carbon nanotube threads are made. CNT thread is a bulk intermediate material that has many potential applications; it would be the strongest and lightest bulk material in the world and this would be a game-changing material for many applications. A lecture about spinning of yarn is given as background before the spinning of CNTs into thread from a CNT array is demonstrated. Several CNT ribbons are pulled from an array and then twisted and pulled by a spinning machine. The threads can have different twist angles depending on the speed of pulling and twisting. One aspect of the demonstration is to spin thread at a different twist angle to vary the strength of the thread.

d. *Concepts for nanomedicine devices.* This sub-module includes a demonstration of tools that are being used to make nanomedicine devices or “tiny machines.” Nanomedicine is a new research area that requires specialized tools with at least one dimension that is at the micro- or nano-scale to handle nanoparticles. The use of a micro-manipulator system to handle CNT materials inside of an environmental scanning electron microscope (ESEM) is demonstrated. There are four robots in the system; two robots are outside of the ESEM to do coarse jobs. There are also two robots inside the ESEM to do fine jobs that are impossible to do by hand, such as picking up a small bundle of CNTs with diameter only 100 nm.

CNT materials can be used as building blocks for tiny nanomedicine devices which could be sensors or robots. Sensors, actuators, and nano-robots built from CNT material can be small enough to go into the human body for medical applications. One example is a linear actuator (nano-solenoid) with one moving part that could be used as a surgical tool to remove plaque or treat stenosis in arteries or remove cancerous tissues. The concept nano-robot is sub-millimeter in size; it will move attached to a catheter (a thin tube) and perform sensing and treatment in the human body. The nanorobot would be actuated by an electric current in select regions of the body and would have greater precision and capability than any other robot.

Module M2: Synthesis of nanoparticles as sensor building blocks. In this module, students use the chemical vapor deposition technique (CVD) to grow carbon nanotubes (CNTs) needed for building sensors in Module M3. In addition, the students produce Mg, Ni, Cu, and Au metal nanowires (NW) which will also be used as building blocks for the sensors and for other devices. Students synthesize CNT posts by CVD³⁻⁶, Ni nanowires⁷ by electrochemical deposition using an alumina template, and Mg nanowires by physical vapor deposition (PVD). The students then observe the morphology and the chemical composition of the nanoparticles using scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) as well as transmission electron microscopy (TEM).

Module M3: Making wireless biosensors using a nanomanipulator. In module M3, students are trained to use a unique nanomanipulator installed inside an environmental scanning electron microscope (ESEM). Training is done using two identical microscopes equipped with nanomanipulators and the students work in groups. Students within each group rotate so that

each student in the class has an opportunity to use the instrument and assemble nanoparticles. Students are able to use different attachments to the robots, including a rotational attachment and tweezers. They become familiar with using the nanomanipulators in this module and employ the nanomanipulators to build sensors using nanoparticles synthesized in Module M2. In the same module, students develop a wireless biosensor (WB) to monitor chemical and biological species in the environment (e.g., heavy metals, acids, microbes and bacteria). The WB proposed is label-free⁸⁻¹² and responds directly to the analyte, which is especially useful for applications where it is impractical to send samples to a laboratory for testing¹³⁻²¹. The sensor responds to environmental and external stimuli through wireless communication, and uses EIS²²⁻²⁷. UC has expertise in nanoparticle synthesis^{5,28,29} and device fabrication³⁰⁻³⁷. Each group of students in the class assembles part of the sensor in stages to arrive at the final sensor. The sensor is calibrated using an EIS system. Modeling of the sensor is discussed in the lecture part of the course.

Module M4: Electrochemical sensor in a fluidic channel. In this module, students receive patterned micro-size electrodes prepared in advance by the conventional photolithographic technique, which is available in the UC clean room. Gold is patterned on Si or glass wafers and students employ cyclic voltammetry to characterize the performance of the sensor. As a second step, they also assemble the patterned electrodes with a previously prepared polydimethylsiloxane (PDMS) structure which accomplishes the fabrication of a micro-fluidic channel. For creating certain sensor selectivity, specific antibodies or receptors are immobilized on the electrodes³⁸⁻⁴². This generic sensor performs as a device and is used to detect toxins or nanoparticles in water. The module requires one lab session. During the lecture part of the course, students design the sensor. The lab session is dedicated to assembling the sensor, immobilization of specific molecules, and detection of selected analytes in water solutions using electrochemical impedance spectroscopy.

Module M5: Magnetic separation of toxins by applied fields⁴³⁻⁴⁷. In this module, the surfaces of Fe₃O₄ nanoparticles suspended in water are functionalized with a toxin-specific antibody. The surface-functionalized magnetic particles are then removed from the water by application of an external magnetic field. Due to surface functionalization, toxins bind to their surfaces. By application of an external field, driven by magnetic force, these nanoparticles, carrying the toxins on their surfaces, will be attracted to the region near the magnet within 5-10 sec. Purified water is then separated from the toxin-concentrated magnetic particles. Continuous toxin removal mechanisms can be developed to treat large quantities of water. A particle size analyzer is used to study aggregation and kinetics of aggregation of these nanoparticles. The aggregates are expected to be in the micrometer range. The role of water conditions (pH, ionic strength, types of inorganic salts) is also examined on the aggregation kinetics of the nanoparticles and the size of aggregates formed.

Module M6: Magnetic measurement of nanoparticles⁴³⁻⁴⁷. In this module, students learn to use a magnetometer to characterize surface-functionalized nanoparticles. This experiment is designed for the students to have hands-on experience in several aspects of physical property characterization, including cryostat, low temperature physics, magnetic measurements, temperature control, and computer data acquisition. They will learn a great deal about behavior changes as the particle size of a material is reduced to the nanoscale. For instance, nanoscale

Fe₃O₄ exhibits so-called superpara-magnetic behavior, particularly required in magnetic separation. Students also learn about several key applications of magnetic nanoparticles in environmental monitoring of toxins in drinking water. In this lab module, students obtain magnetization curves of various nanomaterials including oxides, thin films, and single crystals. They also analyze the magnetic property differences of these materials and relate them to environmental applications such as magnetic separation and bioassay reading. For instance, the students develop correlations between particle size and magnetic driving force, a key quantity in separation rate, using magnetization measurements.

Module M7: Active nanosystems for the destruction of toxins in water. In this module, students gain hands-on experience in the application of visible-light activated TiO₂ nanostructured films immobilized on appropriate support materials for the purification of water contaminated with toxic organic chemicals⁴⁸⁻⁵¹. The experiments are carried out using bench-scale solar systems for water purification and a dye as the contaminant (i.e., safe to use in the laboratory and also easy to visualize). Students visualize dye degradation since the dye is progressively decolorized and ultimately fully decomposed during photocatalytic oxidation. Visual de-coloration of the dye is accompanied with the analysis of dye concentration, total organic carbon, and carbon/nitrogen mineralization (i.e., fraction of carbon and nitrogen that has been completely oxidized) using appropriate analytical techniques. Synthesis of the nanocatalyst using environmentally friendly methods⁵¹, selected nanocatalyst characterization, development of solar-powered water purification systems, and evaluation of system performance is included.

As indicated above, *Nanoscale Devices* was given for the first time during the autumn quarter of 2010. There were eight (8) students in the course and they were asked to fill out a questionnaire that is used in many courses in CEAS using a five-point Likert scale (strongly disagree = 1, disagree = 2, neutral = 3, agree = 4, and strongly agree = 5). The results are shown in Table 1 where the response for the students in *Nanoscale Devices* is compared to the responses by students in many other courses in CEAS. It can be seen that the responses of the students in *Nanoscale Devices* were quite positive, especially when compared to responses for other courses in the CEAS. For example, students responded favorably when asked to rate the course (question 5). Students were less favorable regarding the planning of the course (question 1) but that was expected for such a new and ambitious course being taught for the first time. The students also responded favorably when asked how the course contributed to their professional skills. Thus, they responded favorably when asked if the course contributed to their ability to design and conduct experiments; work in teams; use techniques, skills or modern engineering tools necessary for engineering practice; and use written communication effectively.

Experiments for Summer Institute

Several of the experiments developed for the new courses in nanotechnology were adapted for presentation to students in the Summer Institute conducted by the College of Engineering and Applied Science (CEAS) as part of our outreach program. Summer Institute is a five-week program designed to increase the awareness and interest of underrepresented ethnic students in STEM fields. The program targets high school and junior high school students in Cincinnati Public Schools but is open to students from schools throughout the greater Cincinnati

		Average Response	
		Course	College
1.	The course was well planned.	3.7	3.9
2.	The professor was approachable to discuss problems related to the course.	4.5	4.1
3.	Class assignments and exams were relevant to the course material.	4.7	4.1
4.	The grading was fair.	4.3	4.0
5.	Overall, how do you rate this course?	4.7	3.7
6.	Overall, how do you rate this professor?	4.3	3.9
7.	Please enter any comments about the professor in the box below:	N/A	N/A
8.	... apply my knowledge of math, science, and/or engineering	4.7	4.0
9.	... design and conduct experiments	5.0	3.3
10.	... analyze and interpret data	4.7	3.8
11.	... design a system, component, or process to meet desired needs within realistic constraints	3.7	3.7
12.	... work as a team member	4.7	3.6
13.	... identify, formulate, and solve engineering problems	4.7	3.9
14.	... understand professional and ethical responsibility	4.3	3.6
15.	... communicate (orally) effectively	3.3	3.5
16.	... communicate (written) effectively	4.7	3.8
17.	... understand the impact of engineering solutions in a broad context	4.7	3.8
18.	... use techniques, skills or modern engineering tools necessary for engineering practice	5.0	3.9

Table 1. Summary of responses by students in Nanoscale Devices to questionnaire.

area. Summer Institute seeks to improve students' understanding of science and mathematics as they relate to engineering through hands-on classroom and lab experiences.

During the mornings, students in Summer Institute participated in math and science classes to prepare for their upcoming academic year. A pre-test in science and math was used to

assess the proficiency level of the students and to assign them to their appropriate math/science classes. In the afternoons, students performed experiments related to nanotechnology. The experiments were similar to those conducted in the courses *Nanoscale Devices* and *Environmental Aspects of Nanotechnology* but were modified somewhat to make them appropriate for the age groups involved in Summer Institute. A brief description of each module for SI is given in Table 2.

Module Number	Module Description
S1	Introduction to Carbon Nanotube Materials
S2	Introduction to Carbon Nanotube Applications
S3	Electrochemical Sensing of Lead in Water and Sensor Fabrication
S4	Magnetic Separation
S5	Magnetic Hyperthermia
S6	Active Nanosystems for the Destruction of Contaminants in Water

Table 2. Brief description of the experimental modules in nanotechnology presented to students in Summer Institute.

There were 52 students in Summer Institute in 2010. They were placed into two groups based on the math/science pre-test referred to above. Group A consisted of 26 students with lower math/science proficiency while Group B comprised 26 students having greater proficiency. Generally speaking, students in Group A were younger than those in Group B. Each group was further divided into four teams of six or seven students. As shown in Table 3, each team performed one experiment per week for four weeks. Although there was some overlap, students in Group A carried out experiments that were somewhat less demanding than did students in Group B.

		Week 1	Week 2	Week 3	Week 4
Group A	Team 1	S1	S5	S4	S6
	Team 2	S1	S5	S4	S6
	Team 3	S1	S4	S5	S6
	Team 4	S1	S4	S5	S6
Group B	Team 1	S1	S6	S2	S3
	Team 2	S1	S6	S2	S3
	Team 3	S1	S6	S3	S2
	Team 4	S1	S6	S3	S2

Table 3. Schedule for experimental modules in Summer Institute.

At the beginning of each week, students were given a pre-test to determine their initial knowledge of the experiment they would be doing that week. They were also given a presentation explaining the background of the experiment and the procedures they would use to conduct the experiment. During the middle of the week, the students conducted their experiments and collected information. At the end of the week, the students prepared reports using a standard format, completed a post-test to determine their final knowledge of the experiment, and completed a questionnaire to determine their reactions to the experiment. A five-point Likert scale was again used to evaluate the responses of the students to statements on the questionnaire. When the results of the questionnaires for the individual modules were combined, the results shown below were obtained.

Overall results indicated that the modules were well received and were rated 4.1 out of 5 overall. Module S6, "Active Nanosystems for the Destruction of Contaminants in Water," received the highest overall rating, 4.33 out of 5. Module S2, "Introduction to Carbon Nanotube Applications," received the lowest overall rating (3.18 out of 5). The students in SI strongly agreed that the modules were different from most science activities they did in their school, that the modules were *more hands-on*, and required them to *work at a higher level*. They were also very positive with regard to the organization of the modules and the ability of the instructors to answer their questions.

	Mean	Std. Dev.
1. Overall, I would rate this lesson as...	4.10	.893
2. I liked the activities we did in this module.	4.04	.929
3. The module was very well organized.	4.23	.849
4. The instructor was able to explain the subject very easily.	4.28	.884
5. The instructor encouraged us to ask questions.	4.10	.942
6. The instructor was very good at answering our questions.	4.32	.788
7. The group work was very interesting.	3.95	1.004
8. I learned a lot from this module.	4.15	.886
9. I learned a lot from the instructor.	4.22	.868
10. This module made me interested in learning more about engineering.	3.87	1.143
11. This module helped me feel more confident about studying math.	3.61	1.161
12. This module helped me feel more confident about studying science	3.75	1.171
13. The module was different from most science activities I do during school.	4.43	.956

Table 4. Summary of responses by students in Summer Institute to questionnaire.

The students were somewhat less sure that the modules helped them feel more confident about studying math, science, or engineering. One of the most frequent negative comments concerned students with high levels of interest working on teams with students having little interest.

Future developments

Modules M5 – M7 will be introduced in the course *Environmental Aspects of Nanotechnology* during the winter quarter of 2011. Oral communication will be emphasized along with written communication by requiring each team of students to make a presentation of their results at the end of the quarter. We will also increase the amount of time available for students to complete each module when UC converts from a quarter-based academic calendar to one that is semester-based in the autumn of 2012.

Students in Summer Institute work more effectively in smaller teams; therefore, we will limit the team size to no more than five students. We will also introduce the students to public speaking by requiring each team to make an oral presentation at the end of Summer Institute in which each team member has a prominent role.

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