AC 2011-18: DEVELOPMENT AND IMPLEMENTATION OF A COMPREHENSIVE NANOTECHNOLOGY FUNDAMENTALS LAB FOR ENGINEERING STUDENTS

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Development and Implementation of a Comprehensive Nanotechnology Fundamentals Lab for Engineering Students

Abstract
Nanotechnology findings and products have exponentially increased in the last decade without any parallel development in workforce to support this increase. The gap between supply of skilled workforce and demand in nanotechnology is expected to continue in growth for a while. This paper describes efforts to help in bridging this void of workforce through the development and implementation of a comprehensive lab component for educating an interdisciplinary class of engineering and science students at the junior level. The course is introduced as part of a larger plan to bring nanotechnology into undergraduate education. The approach followed in this lab development is unique since it aims at providing the students with a comprehensive set of hands-on skills and practical knowledge that will enable them to expand and work in the field of nanotechnology, in as many directions as possible. The lab includes eleven separate activities divided into four categories. These categories include: 1) Applications of nanotechnology, 2) Synthesis of nano products, 3) Hands-on training on nanotechnology tools and equipment, and 4) Safety in the nanotechnology operations environment, particularly the clean room. This lab was implemented as part of a course entitled: "Fundamentals of Nanotechnology," at Grand Valley State University (GVSU) School of Engineering (SOE) during the spring semester of 2010. Sixteen students from multiple engineering disciplines were enrolled in this course during its first offering. Objectives of the course and lab were developed previous to the offering and outcomes were assessed during and after the offering. In particular, the lab part was distinctly assessed and the results were evaluated. This resulted in action items and conclusions which have already been integrated in the next offering for continuous improvement. The next offering is planned to take place during the spring of 2011.

Keywords
Nanotechnology education, Nanotechnology lab, Nanotechnology course

Introduction
The products and outcomes of Nanotechnology research and developments have been exponentially expanding for the last decade. This expansion is expected to continue in the next decade as well, resulting in an increased demand on a skilled workforce that can support it [1]. Multiple efforts and investments have been made in the recent years to train and provide this much needed workforce, especially by educational institutions, which are central to these efforts. Many colleges and universities are trying to do their part in providing a skilled workforce to serve the demand of nanotechnology through providing different types of education and training settings [2, 3, 4].

This paper presents the details of a comprehensive nanotechnology lab component that was developed and implemented at the school of engineering (SOE) of Grand Valley State University (GVSU), as part of Fundamentals of Nanotechnology Course. The lab was developed to provide a comprehensive hands-on component to students seeking nanotechnology education while preserving the interdisciplinary nature of the field and the audience. The goal of this approach was to provide students with fundamental skills which will come in handy in as many directions as they could possibly go into, within nanotechnology. The course is part of a plan to introduce nanotechnology into undergraduate education at GVSU serving engineering and science students. This plan includes this course and a sequel project based course in nanotechnology, as well as major components to disseminate nanotechnology education to K-12 students, master students, and the public. The lab described here was offered as part of a course during the spring semester of 2010 and because of high demand had to be capped at 16 students as a maximum limit. The paper describes the details of the lab activities, the outcomes of the lab, and the assessment results of this first offering. Some conclusions are drawn based on this experience to help in improving the next offering of this course and lab as well as the sequel course being developed.

Description of the lab activities

The lab was developed to cover a wide range of skills and tools, divided into four categories as follows:

1) Applications of nanotechnology.
2) Synthesis of nano-products.
3) Hands-on training on nanotechnology tools and equipment.
4) Safety in nanotechnology operations environment, particularly the clean room.

A total of eleven lab activities were performed by students during this lab. The lab manual was distributed as hand-outs to students and experiments were documented by students and graded. Direct and indirect assessment methods of the lab were utilized to collect data which will be used for continuous improvement. The lab objectives were developed so that by the end of this course, students will be able to:

1. Measure nanoscale materials and structures using appropriate equipment like the Atomic Force Microscope (AFM) and the Scan Electron Microscope (SEM).
2. Students will be able to synthesize nanoparticles and nanodevices.
3. Students will be able to use simulation tools to study light absorption in Quantum Dots as an example of nanotechnology products.

The lab included eleven activities which were divided under four categories and included the following:

1. Applications of nanotechnology: Activities in this category aimed at introducing nanotechnology to the students through identifying existing products and properties which utilize the technology and allowing the students to reproduce some of these products with tangible properties. The category includes two activities:

a. Hydrophobic and Hydrophilic materials: The objectives of this lab were to learn how to form a self-assembled monolayer (SAM) using thiol-containing organic molecules and to study the hydrophobic and hydrophilic behavior of the SAM modified surfaces.

Hydrophobic is caused by nonpolar molecules associating with each other while repelling the water molecules. On the other hand, hydrophilic is caused by polar molecules forming ionic or hydrogen bond with the water molecule. Water is repelled more by a surface when the hydrophobicity of the surface is increased. The surface of a solid substrate can be modified by forming a self-assembled monolayer (SAM). One of the methods to form a SAM is to use thiol-containing organic molecules. Since the exposed end of the SAM determines the surface properties of the SAM modified substrate, a hydrophobic surface can be altered into a hydrophilic surface by carefully selecting the SAM forming molecules [1].

In this lab, students learned to modify the copper surface properties using SAM of 1-hexadecanethiol and 16-mercaptohexadecanoic acid. Both of these molecules contain a thiol group that forms a covalent bond to the copper surface. However, the exposed ends of these two molecules have completely different chemical properties. 1-hexadecanethiol SAM on the copper surface renders a more hydrophobic surface while 16-mercaptohexadecanoic SAM makes the surface more hydrophilic. Students were able to see the difference of two modified copper surfaces. Hydrophobic interaction plays critical roles in the formation of the lipid bilayer of the cell membrane and the folding of proteins and nucleic acids. Hydrophobic interaction is the foundation for the existence of life.

b. Microcapsulation and smart paper: The objectives of this lab were to introduce the microencapsulation technology used in “smart” paper and to understand the different methods used to break microcapsules and benefit from nanotechnology.

Both carbonless copy paper and thermal paper are called “smart” paper. Carbonless copy paper is coated with a variety of chemicals. The coated back sheet has a layer of microcapsules that contain an invisible ink. The coated front sheet has a coating of a co-reactant, which when exposed to the colorless ink in the microcapsules reacts with them to produce color. Microcapsules keep the reactants away from each other until the microcapsules are broken. Other than the carbonless copy paper, the applications of microcapsules include delivery of drugs and scratch and sniff stickers [5].

Thermal paper is a heat sensitive medium used to produce images or designs by the application of heat energy. It has a coating containing a colorless dye (color former), a bisphenol or acidic material (the color developer), and a sensitizer. These components are sensitive to the environment (heat, humidity, and pressure) and many materials (organic solvents, cleaners, petroleum solvents, ammonia, some oils, and plasticizers). The main type of thermal printing is known as Direct Thermal printing, where heat is generated in the print head which activates the ink in the paper to develop color.

In this lab, students used pencils and soldering iron to identify and use different paper types containing microcapsules: carbonless copy paper, computer copy paper, and thermal paper. They were also given absorbent paper and lemon juice to design secret codes that can be visible either by applying red cabbage juice or a heat from a house-hold iron.

2. Synthesis of nano products: Activities in this category included synthesizing actual nano products by the students and utilizing them in applications or study their properties. The category includes three activities as follows:

a. Synthesis and application of gold nanoparticles: The objective of this lab was to synthesize gold nanoparticles and to use them as chemical or biological sensors. At the nanometer scale, gold interacts with light differently through a process called surface plasmon resonance. Depending on the specific size and shape of gold nanoparticles, they can appear red, purple, blue, or in other colors [6].

In this lab, students synthesized a solution of well-separated, 13 nm diameter gold particles. To keep the nanoparticles from aggregating, a layer of citrate anions was applied to each nanoparticle's surface to produce an electrostatic repulsion. The solution appeared to be red instead of gold color. When a strong electrolyte was added to the solution, the high concentration of ions screened the repulsive electrostatic forces between the particles. The gold nanoparticles aggregated and the solution turned blue. When a larger quantity of the electrolyte was added, large nanoparticles aggregated and the solution became clear with gold-colored particles on the bottom of the vial. When non- or weak electrolyte was added, the electrostatic repulsions between the gold/citrate particles were not disrupted and the solution remained red. Figure 1 shows the gold nanoparticle solutions with different amount of electrolyte added. Students were also assigned to research the application of gold nanoparticle as chemical or biological sensors. They were requested to answer the following specific questions:

- How could gold nanoparticles be used to detect the binding of biomolecules, such as DNA or antibodies that stick to one another or to other molecules?
- How could these molecules be used to cause aggregation of the nanoparticles?

Results were reported by the students within their lab reports for this activity.

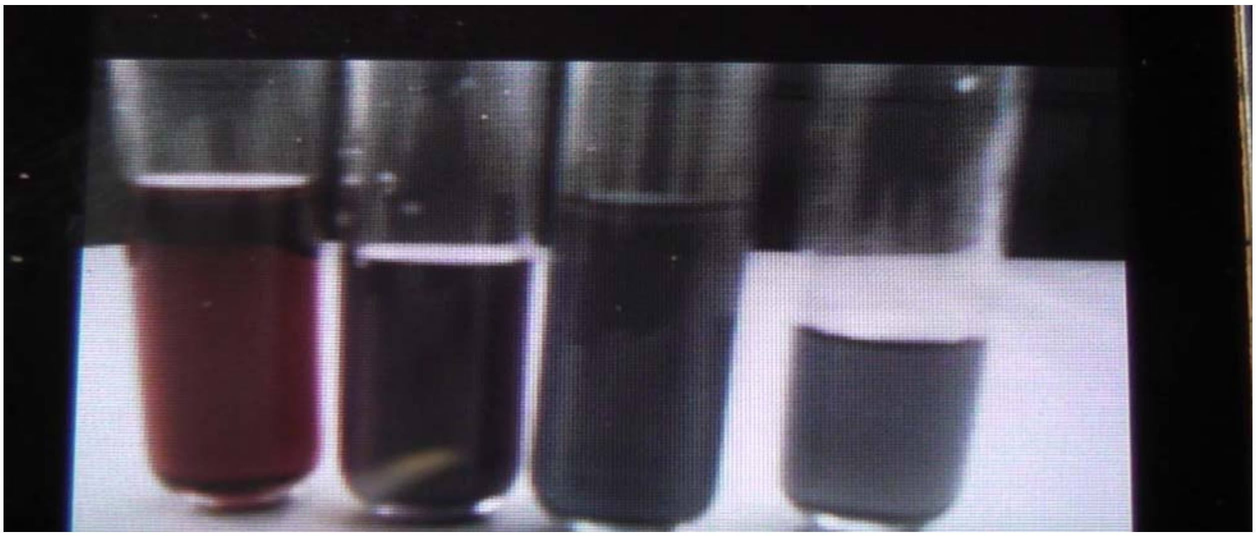

Figure 1 Gold nanoparticles synthesis and the different colors they reflect depending on the concentration of electrolyte in the mixture.

b. <u>Synthesis and application of silver nanoparticles:</u> The objectives of this lab were to synthesize yellow colloidal silver nanoparticles and to study their aggregation effects. Silver nanoparticles are on demand for several uses in medicine and technology. They can be used as an anti-microbe agent to effectively eliminate fungus, bacteria, and viruses. They also are used as an ingredient for electroconduit slurry, air purifiers, and water purifiers [7].

In this lab, students synthesized a yellow silver nanoparticle solution through a reaction of silver nitrate with ice-cold sodium borogydride. In addition, silver particles' aggregation was studied. Students learned that adsorption of borodydride played a key role in stabilizing growing silver nanoparticles and aggregation can be prevented by polyvinylpyrrolidone (PVP) solution into the silver colloidal suspension.

c. <u>Organic light emitting diode (OLED):</u> The objective of this activity was for the students to synthesize an actual nano device and study its characteristics.

In this lab, students learned to create an electrochemiluminescent ruthenium-based organic light emitting diode (OLED) and to measure the characteristics of the OLED. The process of making the OLED is depicted in figure 2. The transparent conductive glass was used as a substrate. The ruthenium (II) PVA solution was prepared and spin-coated onto the conductive side of the glass. The polymer coated glass was then heated on a hot plate to drive off moisture in the film. The process was completed by depositing gallium-indium eutectic alloy on the polymer layer using a shadow mask. When applying positive voltage to the conductive oxide and negative voltage to the metal alloy, emitted light was observed. The OLED was further explored by measuring its current-voltage characteristics.

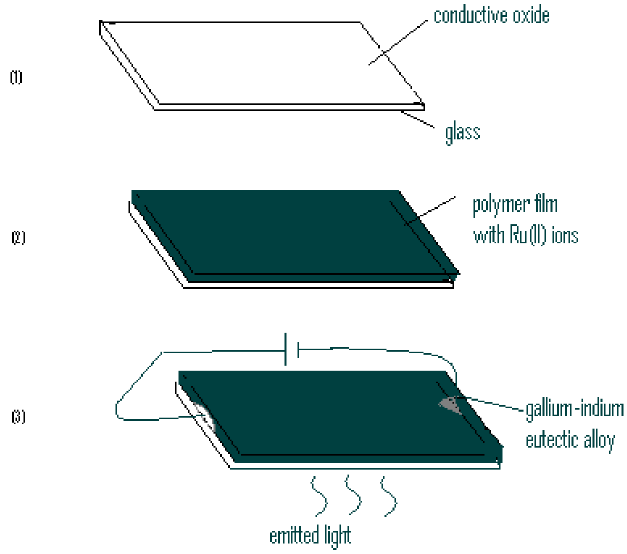

Figure 2 The process of forming an OLED

3. Hands-on training on nanotechnology tools and equipment: Activities in this category aimed at familiarizing students with tools and equipment used in the field of nanotechnology and providing them the opportunity for hands-on training on these tools of the trade. The category includes three activities:

a. <u>Scanning Electron Microscope (SEM):</u> The objective of this activity was to introduce the students to SEM and its principles of operation then provide them the opportunity to actually use the instrument for imaging samples to make conclusions based on their results.

Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. Due to the high energy carried by electrons entering the surface of the sample, SEM has higher resolution than conventional optical microscope and is one of the crucial tools for "seeing" the nanometer scale.

Two samples were imaged during this activity. The standard sample was used to demonstrate the operation of the SEM. Students were then given the opportunity to image gold nanoparticles prepared from a previous lab activity.

b. Atomic Force Microscope (AFM): The objective of this activity was to expose students to the principles of operation of this microscope and to provide them the opportunity to utilize the instrument in collecting actual images of samples.

Atomic Force Microscope (AFM) can resolve features in atomic scale. It provides high-resolution and three-dimensional information with little sample preparation. It is used in a variety of fields such as life science, material science, electrochemistry, polymer science, nanotechnology, etc.

In this lab activity, students learned the operating principle of AFM and how to generate the surface topography of a data CD as well as nanoparticles using AFM. Figure 3 shows the topography of a data CD.

c. Thermal evaporation and deposition of materials: The objectives of this activity were to teach the students, and provide them the opportunity to perform the process of physical thermal evaporation and deposition of thin film material used in many nano-scale materials. Moreover, another objective was to introduce the students to the clean room, practice gowning procedure, and safety rules. During this activity, students were provided proper gown and safety equipment and were allowed in the clean room. They used the thermal evaporator to deposit a thin layer of aluminum on 2” X 2” glass substrates.

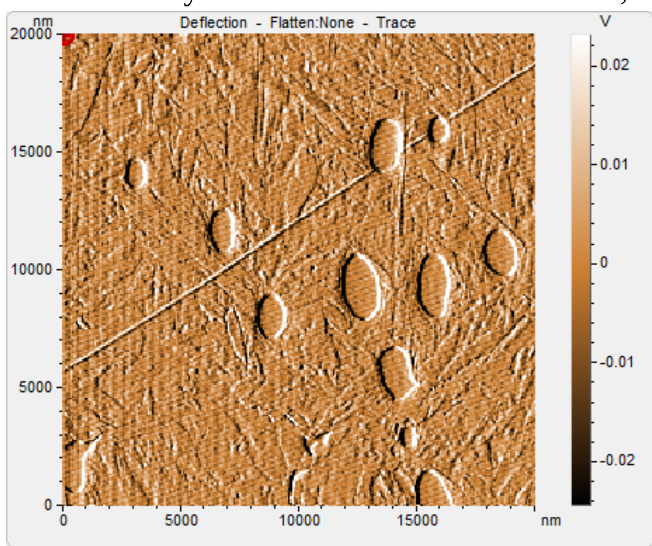

Figure 3 AFM images of a data CD.

d. Simulation and analysis tools: The objectives of this activity were to expose the students to one of the simulation tools available for nanotechnology development and analysis and allow them the opportunity to utilize this tool. In addition, students utilized the activity to learn about quantum dots, as an example of light-properties based nano-devices and apply some of the theory studied in class in this regard.

A quantum dot (QD) is a semiconductor nanostructure which is confined in all three spatial dimensions. The motion of conduction band electrons, valence band holes and excitons (electron and hole pairs) are restricted in the three spatial directions. QDs have many applications in displays, LEDs, photonics, telecommunications, and solar cells [8]. During this lab activity, students used the simulation tool to obtain the wave function and eigenenergy of an electron in a quantum dot. In addition, light absorption in quantum dots was studied.

4. Safety in nanotechnology operational environment: This category is an absolute necessity when performing any work with nanotechnology. The safety procedures and rules are a two way protection for both the practitioners and the products. The students had a lecture part on this topic before getting exposed to the actual rules and procedures and practicing them. The

documents for clean room safety training were generated and provided to the students. Related topics such as the classification of clean room, contamination, sources of contamination, and contamination control were introduced. In addition, students learned general clean-room regulations and gowning procedures.

Lab outcomes assessment and evaluation of results

1. Direct assessment:

For each lab activity, students were not only required to conduct experiments and collect data, but also to analyze data and write reports to demonstrate their understanding of the materials covered. In some labs, questions were asked to further inspire students' thinking. In lab # 2-a, after synthesizing gold nanoparticles by the students, the following questions were asked:

- How could Au nanoparticle be used to detect the binding of biomolecules, such as DNA or antibodies that stick to one another or to other molecules?
- How could these molecules be used to cause aggregation of the nanoparticles?

The lab report grades' distribution for this activity is shown in Figure 4. It shows that all students conducted the experiment successfully and understood the materials well.

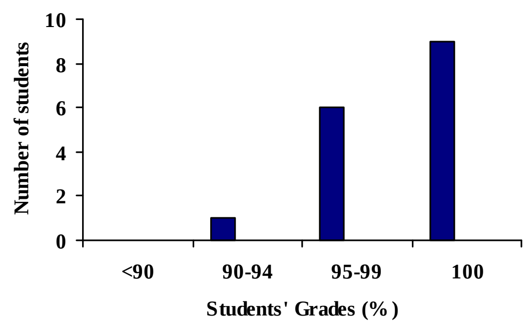

Figure 4 Report grade distribution for Lab # 2 - a.

Another example is from lab # 3-d "Light absorption in quantum dots." After the students learned how to use the simulation tool and simulated light absorption at different incident angles for different geometrical quantum dots, the following questions were asked:

- How does the absorption change as a function of incident angle?
- What if more states (> 20) are included in the design calculation?

The lab report grades' distribution for this lab is shown in figure 5. It can be seen that 81% of the students received a grade above 80 %.

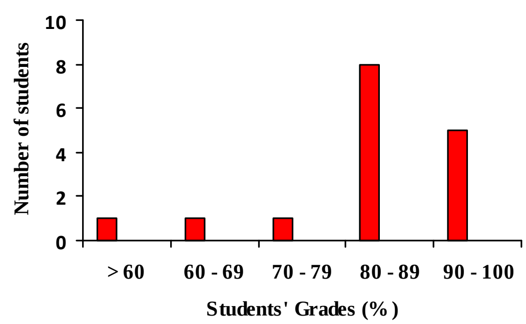

Figure 5 Report Grade Distribution for lab # 3 - d.

The lab results shown in figure 4 show higher achievement levels compared to the results shown in figure 5. The main reason for this difference is the difficulty level of the concepts and theory being applied by an interdisciplinary group of students. However, most students seem to have managed to understand and apply the concepts successfully to a satisfactory level.

2. Indirect assessment:

A survey was conducted at mid-term point of the semester. Three questions related to the lab portion of the class were asked and the response from the students is shown in figure 6, where 1 is "strongly agree" and 5 is "strongly disagree." A majority of students (75%) indicated that lab material is relevant to the course material and the quantity is reasonable. Moreover, 87.5% of the students indicated that they can synthesize nanoparticles.

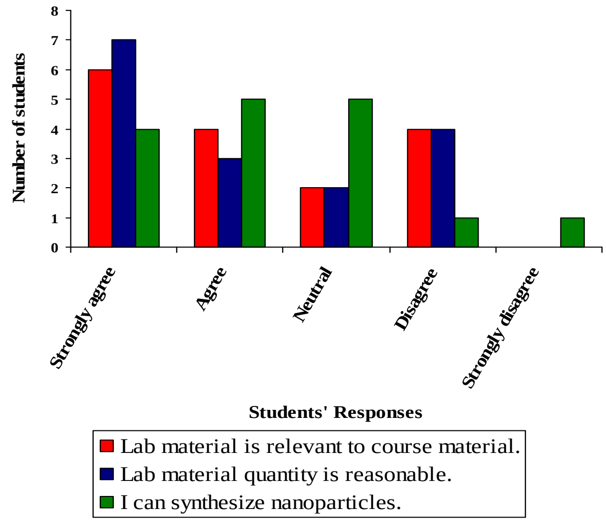

Figure 6 Mid-semester Students' Evaluation of the Lab Component.

During the end of term evaluation, 92.9% students indicated that the course was taught well. Some of the comments regarding the lab component were:

- Instructor used lab activities as an effective way to stimulate thinking about subject matter outside of the scope of the lab itself.
- The laboratory exercises coincided with the lecture portion.

Conclusions

A lab component was developed and delivered for an interdisciplinary class of engineering students as part of an introduction to nanotechnology course. The course is an activity within a bigger plan to develop undergraduate educational modules, and outreach activities, in nanotechnology. The lab activities covered multiple aspects of nanotechnology to expose the students to as many parts as possible of the spectrum of nanotechnology. This was by-design to provide the students with a practical background that will enable them to have more choices, should they decide to pursue a career in nanotechnology.

Lab activities were assessed directly and indirectly to improve the following offering of activities. Achievements by students differed depending on the level of difficulty of the lab topic, but most students managed to grasp the material very well.

In the next offering, an attempt will be made to provide more exposure to clean room equipment.

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