Nanotechnology and Relevant Technologies Lab Development at WSU

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Abstract

*This work reports on the development of a nano-technology lab at Wichita State University. Undergraduates and graduate students learn and experience nano-technology processes with respect to ferro-fluid nano-particle fabrication, nano-composites, nano-coatings, nano-fibers, nano-porosity films, biodegradable nano-particle polymer for use in drug delivery and solar cells***.**

Introduction:

Universities strive to prepare students to be successful in the industry by providing the most advanced education possible. Nano-technology is the fastest growing and most advanced technology affecting all scholarly fields, thus preparing students in the field of nano-technology is a primary concern for universities. We present a lab that is educational and intriguing for students to learn about nano-technology. In the nano-technology lab, nano-particles, functionalization of nano-particles, nano-composite, drug delivery, electroplating, electrospinning, atomic force microscope (AFM) Lithography, nano-porous film and spin coating are studied.

In this lab, 10 nano-technology experiments are performed. In order to engage the student's intellect and enthusiasm, magnetic nano-particles are created and functionalized producing ferro-fluids that spike in the presents of a magnetic field. Students find the interactive spiking of the ferro-fluid, caused by a magnetic field that is placed near the ferro-fluid, fascinating. Nano-composites are produced by adding different percentages of magnetic nano-particles to epoxy, then observing agglomeration and magnetic attraction. In the drug delivery lab, poly (lactide-co-glycolide) acid (PLGA) nano-particles are fabricated that can be used in drug delivery and focus is placed on the potential benefit of nano-technology in medicine. Electroplating and spin-coating are frequently used methods to produce nano-coatings in both labs, these methods are performed and the benefits of both are taught. [1] An atomic force microscope (AFM) can be used to image the surface of materials on the nanometer level and can also manipulate the surface. [2] Students get the opportunity to etch the surface of a DVD with their own design via AFM, and then image the modified surface with the AFM. Nano-fibers are produced by electrospinning and students discover the important variables that can be changed to create engineered nano-fibers with desired characteristics. A nano-porous film is produced by dealloying a gold-silver alloy, and students learn the methods and difficulties in handling nano-materials, specifically an 80 nm thick nano-film. Solar cell fabrication is done by using blackberry or blueberry dye which significantly reduces the price of solar cells and has the potential to produce an important eco-friendly power source.

Magnetic Nano-Particles Lab (1)

The purpose of this lab is to show students how to make nano-particles using homogenous nucleation. When ferrofluids were first developed at NASA in the 1960's, magnetite particles were reduced to the nano-scale through a long milling process. In this lab students learn the advantages of synthesizing magnetite particles on the nano-scale using a bottom-up approach. The magnetic nano-particles will also be used in Functionalization and Nano-composite labs. The first process is to measure 25 mL of HCl solution into one beaker and 100 mL of HCl solution into the second. Then, measure out 1 g of Iron (II) Ferrous Chloride, and add this powder to the 25 mL of HCL in the fume hood. The yellow solution can be seen in Figure 1. Next, measure out 1.6 g of Iron (III) Ferric Chloride, and ground it into a fine powder by mortar and pestle. Add this powder to the 100 mL of HCl solution in the second beaker. Combine the two mixtures into a single beaker and add the magnetic stir bar and stir for 10 minutes. Once the mixtures are well combined, remove 25 mL from the beaker using a syringe and transfer it to another beaker. Place the magnetic stir bar in this new beaker, and while stirring vigorously, add 6 mL of ammonium hydroxide slowly (drop by drop) over the course of five minutes using a syringe. The mixture at this time is black due to the presence of magnetic nanoparticles (Fe_3O_4) as is seen on Figure 2. Stop the mixer and apply the strong Nd magnet to the base of the beaker. The particles immediately begin to settle by decantation. Once they are firmly held at the bottom of the beaker by the magnet, the waste fluid is poured off to be disposed. Add 25 mL of de-ionized water in the beaker containing the particles to rinse them. Separate the particles using the Nd magnet and decant the fluid again as is seen on Figure 3. Repeat this step 5 times. After completion of the last step, set magnetic nano-particles aside to dry. [3] These nano-particles will be used in the nano-composite lab.

Figure 1: Ferric and ferrous ions in solution with HCl

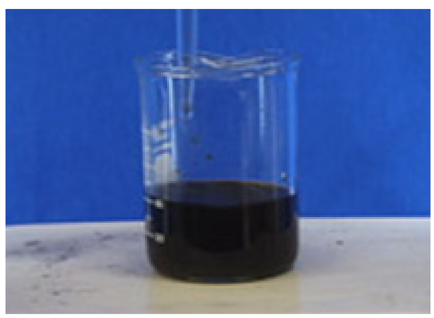

Figure 2: Ammonium hydroxide addition Increases PH

Figure 3: Washing particles

Functionalization of Magnetic Nano-particles Lab (2)

The purpose of this lab is to teach students the impact that functionalization can have on nano-materials. In this lab the students are able to see the difference between the behavior of nano-scale particles and macro-scale particles of the same material. Magnetic nano-particles are produced before lab, so each student can functionalize their own specimens. The first process is to decant the sample dish containing the nano-particles supplied by the instructor by placing the strong Nd magnet beneath the dish. Then allow the particles to settle and pour the fluid off into a waste container. Next, add 3 mL of tetramethylammonium hydroxide solution to the sample dish. Using the glass stirring rod, stir the particles into the solution to ensure that all the particles are well coated. Allow the dish to rest in the fume hood for at least 30 minutes. Open a vial, and carefully transfer the contents of the sample dish to the vial. Since there is retained water and tetramethyl ammonium hydroxide, the solution will have a low viscosity and will be easily poured into the vial. Place a strong magnet against the vial and decant the fluid and particles that are not held in place by the magnet. Lastly, pour mineral oil into the beaker using the syringe. Then, transfer mineral oil from the beaker to the glass vial filling it to the brim and cap the vial. Prove that ferro-fluid has been produce by using a Nd magnet to produce spikes from the ferro-fluid as is seen on Figure 4.

Figure 4: Ferro-Fluid spiking in the presence of a magnetic field

Nano-composite Lab (3)

The purpose of this is lab is to teach students the benefits and difficulties in producing nano-composites. In industry, students will deal with the three difficulties in composite - agglomeration, interfacial bonding and alignment. [4] This lab strives to prepare students to deal with these difficulties in relation to nano-composites. The students will produce three nano-composite (nano-magnetic particles/epoxy) specimens - one specimen without any addition of nano-particles, one with 15% nano-magnetic particles and one with 25% nano-magnetic particles. First, measure out 15 g of epoxy and 3 g of the hardener agent into a beaker and stir to combine. Place the first mold on the scale and add 6 g of epoxy. Next, place the second mold on the scale and add 5 g of epoxy and 0.9 g of magnetite. This specimen is 15% by weight magnetic nano-particles. Place the third mold on the scale and add 4.5 g of epoxy and 1.5 g of magnetite. This specimen is 25% by weight magnetic nano-particles. Allow the samples to cure in an oven at 80°C for twenty minutes to speed up polymerization. Once solidified, remove the samples from the molds. Observe color change, agglomeration, and magnetic strength of each specimen.

Electrospinning Lab (4)

This lab will expose students to electrospinning and their applications. Although the process of electrospinning has been around since the 1930's, it is now possible to spin fibers at the nano-scale. There are great advantages in being able to control the fiber diameter and surface characteristics, and many companies have begun using nano-scale fibers for filtration, sound dampening, absorbance, wound care, sensors, etc. Since the 1980's industries have increasingly begun using electorspun nano-fibers. [9] Figure 5 and Figure 6 show the apparatus used in electrospinning. First, place the solution in the syringe, and insert the platinum probe and put the syringe in the syringe pump. It should be set to a flow rate of 2.5 mL/h. Next, position the metal screen opposite the syringe at a distance of 25 cm. Activate the potential difference and set it to 20 kV. Do not touch the metal components once the field is in operation as there is high voltage going from the needle to the metal screen. Once the solution has been exhausted, stop the motor and turn off the electric field.

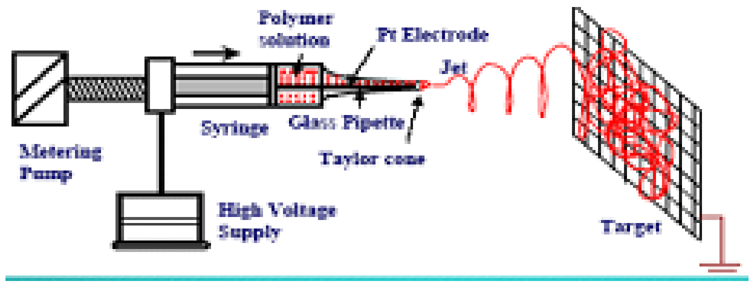

Figure 5: Schematic of the electrospinning apparatus used in electrospinning

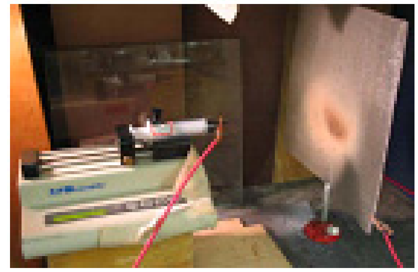

Figure 6: An electrospinning apparatus: used in electrospinning

Spin Coating Lab (5)

Many products use spin coating to put a low cost nano-coating on a substrate. For example photoresist for defining patterns in microcircuit fabrication, dielectric/insulating layers for microcircuit fabrication, DVD, CD ROM, television tube phosphor and antireflection coatings [5]. The experiment begins when the glass substrate is placed on the seal in the center of the spin coater. The vacuum pump is then activated, securing the substrate in place. The experimenter should fill the syringe with a solution of 10% polyvinyl chloride and 90% dimethylacetamide and place an excess amount of the solution on the substrate. Next, the spin coater is activated and spins at low speed for approximately ten seconds, and then increases in speed to the desired number of revolutions per minute.

AFM Lithography Lab (6)

This lab is meant to demonstrate characterization and manipulation at the nano-scale. Lithography at this scale is used in making nano-fluidic devices, nano-scale electronics, etc. An atomic force microscope (AFM) is capable of magnifications between 100 X and 100,000,000 X in the horizontal (x-y) and vertical axis while standard electron microscopes are only capable of 100,000 X [6]. This high magnification makes AFM an excellent devise for characterization. Agglomerated and dispersed carbon nano-tubes (CNT) are seen on Figure 7. AFMs also have the unique ability to manipulate the surface, and AFM lithography was demonstrated to the class. Figure 8 is an image of Wichita State University’s logo created by using AFM lithography.

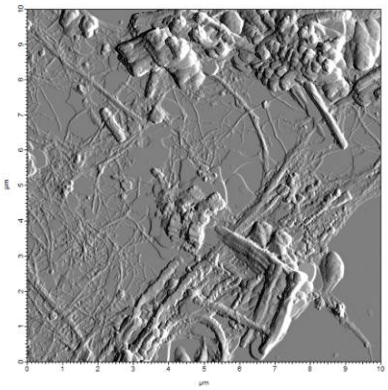

Figure 7: AFM characterization of agglomerated and dispersed carbon nanotubes

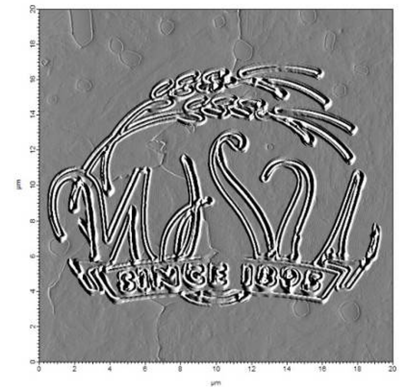

Figure 8: Wichita State University's logo etched into the surface of a DVD by use of AFM lithography

Drug Delivery (7)

This lab shows students the benefits of nano-technology in the medical field, specifically in drug delivery. Smart-drugs and targeted drug delivery systems are of great interest to researchers, and nanotechnology is key to this field. Students created biodegradable and biocompatible polymer nano-particles that can be used for drug delivery. The biodegradable nano-particles created in the lab were produced using single emulsion-solvent evaporation using oil in oil [7-8]. First, weigh out 0.0625 mg of PLGA crystals using the scale and sample tray. Next, 5 mL of ACN is placed in the Florence flask using the low volume syringe. The PLGA crystals are also added to the flask. The magnetic stirrer is put into the flask and placed on the magnetic stir plate. The chemicals are allowed to combine. Meanwhile, a second phase is created by putting 40 mL of paraffin viscous into a beaker and 0.2 mL of Span 80 into the same beaker. Next, the beaker is positioned beneath the high speed stirrer and the blades are lowered into the beaker. The magnetic stirrer (hot plate) is activated and allowed to combine the elements of the second phase. The first phase is removed from the flask using a large pipette, and is added drop-wise to the beaker. The drops are positioned so that they landed on the stirrer blades. The mixture is allowed to evaporate for one hour stirring at 5,000 rpm with a high speed stirrer. The nano-particles are later separated from solution with a centrifuge and washed. The fished nano-particles can be seen on Figure 9.

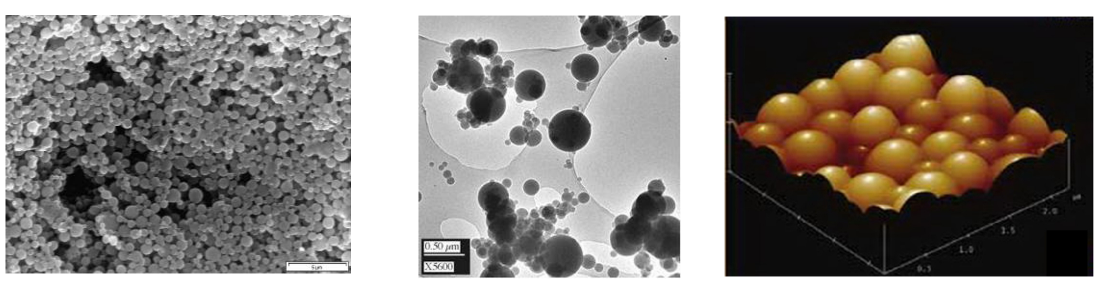

Figure 9: Images of PLGA nano-particles made with the process used in the drug delivery lab

Nano-porous Film Lab (8)

The purpose of this lab is to teach students the benefits of high surface area inherent to nano-materials; also this experiment is very good at giving the students hands on experience of handling of nano-thin films. The gold/silver alloy nano-film will be de-alloyed by the nitric acid producing a nano-porous film. First, the graphite cylinder is placed on the wooden dowel and suspended over the glass dish as is seen on Figure 10-A. The dish is filled with DI water to the height of the cylinder. A sheet of gold leaf is placed on the surface of the water as seen on Figure 10-B and C. It should be noted that placing the fragile 80 nm film on the water is very difficult. The nano-film must be shaken off the paper it is packaged on without ripping the film. The gold leaf is slowly rolled up the side of the graphite cylinder that the experimenter is closest to as seen on Figure 10-D. The graphite cylinder and gold leaves are now ready for safe transfer. The cylinder and gold leaf are removed, the water is poured out, the dish is set inside the fume hood, and nitric acid is placed in the glass dish at the height of the bottom of the graphite cylinder. The cylinder is returned to its position above the dish and the gold leaf is rolled off onto the surface of the acid as is seen on Figure 10-E. Next, the gold leaf is allowed to sit for fifteen minutes on the acid. Acid is removed and replaced with DI water to rinse the nano-porous film which results in pores smaller than 20 nm.

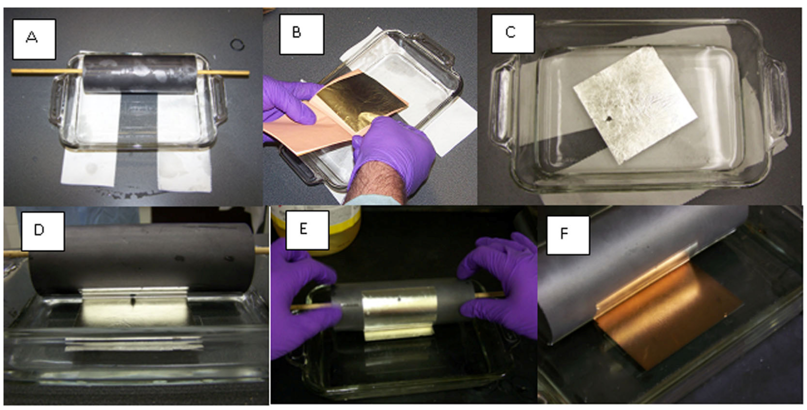

Figure 10: The process used to make a nano-porous gold film.

Electroplating Lab (9)

Electroplating is a commonly used coating process and students are taught and experience the process. The process starts out by making an electrolyte solution made of $CuSO_4 \cdot 5H_2O$, H_2SO_4, and water. Then, alligator clips are attached to the insulated copper wires. The wires are attached to the DC power source, which is plugged in and the power source is turned on. The objects that are to be electroplated should be cleaned with acetone to remove impurities that would impede the formation of the electroplated layer. The platinum bar should be attached to the positive side to act as the anode. The object to be electroplated is connected to the negative electric terminal of the DC power source and the area of the sample measured, while the other piece of metal is hooked to the positive terminal. Both objects are submerged in the solution for a short period of time. When they are removed, the negatively charged object has been electroplated. The plated object should be rinsed to remove the remaining solution.

Solar Cell (10)

Due to increased concern for the environment and a renewable energy source, solar cells have been extensively studied. The main problem with a solar cell is the cost per kilowatt. Nano-technology is presenting a possible way to produce cheap solar cells with blackberry or blueberry juice and titanium dioxide (TiO_2) nano-particles. Students learn how to make cost effective solar cells in this lab. Two grams of nano-crystalline titanium dioxide (TiO_2) is ground in a mortar and a few drops of dilute acetic acid (0.035 M) are added. Repeat that step until a slightly soupy colloidal suspension with a smooth consistency is obtained. Next, add a few drops of clear dishwashing detergent and mix until combined. Tape a conductive coated glass with the conduction coated side up on three sides of the conductive coating to serve as a spacer. Add TiO_2 past to the cell and scrap the TiO_2 off so that coating is even with the height of the tape. Remove the tape and heat the glass until the surface turns brown. Cool the sample, and immerse the coating in blackberry juice. Rinse with DI water and then ethanol. Pass a second piece of tin oxide glass with the conduction side down through a candle flame to coat the conduction side with carbon. Wipe off the carbon along the perimeter of the three sides of the carbon-coated glass plate using a cotton swab. Lastly, assemble the two glass plates with coated sides together with a slight offset and clamp the pates together. Test the solar cell using a multimeter and sunlight.

Conclusion:

Ten labs have been used to teach students about nano-technology processes; magnetic nano-particles (1), functionalization (Ferro-Fluid) (2), nano-composite (3), electrospinning (4), spin coating (5), AFM lithography (6), drug delivery (7), nano-porous film (8), electroplating (9) and solar cells (10). Students were surveyed and found this lab to be informative, enjoyable and useful for their future careers. It is expected that as technology increases the more industry will utilize nano-technology. Wichita State University is striving to prepare students for a high-tech education by providing cutting edge labs to teach theory and hands on applications. Providing this level of learning, students will have the knowledge to thrive in a high-tech industry.

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