NANOSCALE SCIENCE AND ENGINEERING EXPERIMENTS IN A GENERAL CHEMISTRY FOR ENGINEERS LABORATORY COURSE

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ABSTRACT

A key to developing a fundamental understanding of nanotechnology is the introduction of nanoscale science and engineering activities early in the curriculum. The Southern Illinois University Edwardsville (SIUE) School of Engineering has experienced tremendous growth in enrollment in the past few years, and the large number of engineering students taking general chemistry prompted the creation of new *Engineering Chemistry* (CHEM 131) and *Engineering Chemistry Laboratory* (CHEM 135) courses. These new courses provide a unique opportunity to integrate nanoscale science and engineering concepts into the first year curriculum. Student-centered, nanotechnology-enriched resources, available at the University of Wisconsin-Madison MRSEC website (http://mrsec.wisc.edu/nano), are being integrated into CHEM 131 and CHEM 135 at SIUE. The laboratory course, CHEM 135, consists of eight traditional chemistry experiments and eight nanoscale experiments. The nanotechnology-related experiments include: 1) *Synthesis of Ferrofluid*, 2) *Synthesis of a Nanocrystalline Phosphor*, 3) *Disassembly of a Liquid Crystal Watch*, 4) *Solid State Structure and Properties*, 5) *Periodic Properties and LEDs*, 6) *A Shape Memory Alloy, NiTi*, 7) *A Nanocrystalline Solar Cell*, and 8) *Template Synthesis and Magnetic Manipulation of Nickel Nanowires*. Details of the nanoscience experiments, preliminary assessment results, and challenges encountered in the implementation process will be discussed.

1. ENGINEERING CHEMISTRY AT SIUE

Many departments within the School of Engineering at Southern Illinois University Edwardsville (SIUE) require students to take one semester of chemistry, but the first course in the two-semester *General Chemistry* sequence (CHEM 121a) does not address much of the content that is important to engineering students. Informal surveys in 2003 indicated that approximately one third of students enrolled in CHEM 121a were engineering students, so the Department of Chemistry developed a separate course, *Engineering Chemistry* (CHEM 131) to suit their needs. The course is designed to cover core topics in chemistry with an emphasis on ties to engineering applications. The authors of this paper met with the Associate Dean and Chairs of the School of Engineering, and received unanimous support for *Engineering Chemistry*. CHEM 131 was first taught in Fall 2004, with an enrollment of 135 students. The required companion course, *Engineering Chemistry Laboratory* (CHEM 135), was also offered in Fall 2004.

2. NANOTECHNOLOGY

Modern science is becoming increasingly interdisciplinary. One example is materials science, a broad, chemically oriented view of solids that results from the combined viewpoints of chemistry, physics, engineering, and for biotechnology, the biological sciences (Ellis, *et al.*, 1993). An exciting emerging field is nanotechnology, the manipulation of the material world at the scale of atoms and molecules (Schulz, 2000). It is projected that important advances in engineering will come from an understanding of the properties of matter constructed from building blocks whose size and shape is uniform and on the 1-100 nm scale. These consequences include technologies to be used for medicine (bone/tissue replacement, gene therapy, "noninvasive" surgery) (Rawls, 2003), advances in computer technologies (molecule-sized circuit elements) (Halford, 2004), defense (portable chemical and biological hazard detection) (Wilson, 2003a, b), and everyday applications (self-cleaning paints!) (Schulz, 2003). Leaders in the chemical industry currently believe that the current "hype" over nanotechnology will quiet down, not because we will lose interest in nanotech, but because in the next 20 years nanotechnology will become a ubiquitous part of our society (Thayer, 2003).

3. NANOSCALE EXPERIMENTS

3.1 Engineering Chemistry Laboratory

Creation of the new *Engineering Chemistry* courses provided a unique opportunity to incorporate nanoscale science and engineering laboratory experiments and lecture demonstrations into the first year curriculum. Many examples of nanoscale experiments have appeared recently in the chemical education literature (Nedeljkovic, *et al.*, 1993; Smestad and Gratzel, 1998; Berger, *et al.*, 1999; Keating, *et al.*, 1999; Kippeny, *et al.*, 2002; Bolstad and Diaz, 2002). Chemists and engineers at the University of Wisconsin-Madison (where co-author Voss spent a 2003 sabbatical leave) have already introduced student-centered, nanotechnology-enriched activities into introductory college chemistry courses for engineering students (Condren, *et al.*, 2002). With support from a National Science Foundation Nanotechnology Undergraduate Education (NUE) grant, we have implemented eight nanoscale experiments in CHEM 135 at SIUE.

3.2 Lab Manual for Nanoscale Science and Technology

The *Video Lab Manual* (http://mrsec.wisc.edu/edetc/nanolab/index.html), is a "living online document" designed to deliver value-added content to students in chemistry and engineering laboratory courses. Maintained by the *Interdisciplinary Education Group* of the NSF funded MRSEC on Nanostructured Materials and Interfaces, the *Video Lab Manual* contains experiments based on cutting-edge nanoscale research that have been designed to showcase visible and exciting applications of the research. Five of the CHEM 135 nanoscale experiments are already part of the *Video Lab Manual*, and we are currently filming the other three experiments (*Solid State Structure and Properties*, *Periodic Properties and LEDs*, and *A Shape Memory Metal Alloy, NiTi*) for future inclusion.

3.3 Brief Descriptions of Selected Nanoscale Experiments

<u>Synthesis of Aqueous Ferrofluid</u>: In this experiment a suspension of magnetite nanoparticles is formed by carefully mixing iron(II) chloride, iron(III) chloride, and aqueous ammonia (Berger, 1999). Addition of a surfactant results in a magnetic fluid that "spikes" along the magnetic field lines of a strong magnet, Figure 1. Chemical concepts such as stoichiometry, balancing equations, and oxidation states are reinforced in this highly popular lab.

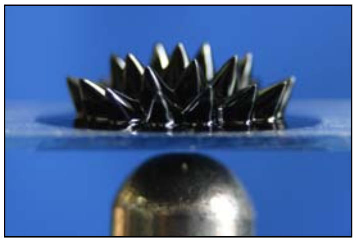

Figure 1: Aqueous ferrofluid "spikes" when brought close to a cow magnet.

<u>Synthesis of a Nanocrystalline Phosphor</u>: Nanocrystalline particles of $Y_2O_3:Eu^{3+}$ are prepared from an aqueous solution of the metal nitrates and urea in a combustion reaction at 500 °C (Bolstad and Diaz, 2002). The resulting product is a red-emitting phosphor under ultraviolet light, Figure 2. Heat treatment of the sample overnight at 850 °C results in larger particle size (measured by powder X-ray diffraction) and better phosphorescence (qualitatively observed under ultraviolet light). Combustion reactions, diffraction, phosphorescence, and ultraviolet spectroscopy are concepts that are included in this experiment.

Figure 2: Nanocrystalline phosphor viewed under room light (left) and ultraviolet light (right).

<u>Disassembly of a Liquid Crystal Watch</u>: An inexpensive liquid crystal display (LCD) watch is disassembled, and the optical, thermal, and electrical properties of the LCD panel are tested (Condren and Lisensky, 2004). Students were given the challenge to try to reassemble the watch after the experiment was completed, Figure 3.

Figure 3: Internal components (top) and assembled liquid crystal display watch (bottom).

Template Synthesis and Magnetic Manipulation of Nickel Nanowires: Nickel nanowires are grown inside the pores of an alumina filter, (Bentley, *et al.*, 2005). The filter is removed by dissolving it in sodium hydroxide to yield magnetic nanowires that can be viewed under an optical microscope or in a scanning electron microscope. This experiment is introduced when the class is studying electrochemistry, oxidation, reduction, and electrolysis, Figure 4.

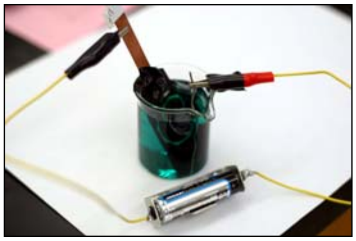

Figure 4: Simple electrochemical cell used for the synthesis of nickel nanowires.

4. ASSESSMENT

Student learning objectives for these experiments include excitement and enthusiasm for scientific thinking, awareness of nanotechnology and nanoapplications in science, 3-D manipulation, and improved quantitative reasoning, problem solving, analysis, and synthesis. Our assessment plan includes formative and summative evaluation, involving mixed (qualitative and quantitative) data collection methods.

4.1 Pre-Survey and Post-Survey

The same 30-question survey was given to CHEM 135 students during the second week of classes, and again during the final week of classes in Fall 2004. Based on collected demographic data, the typical student is a 19-year old Caucasian male freshman. The median student had 8 semesters of math in high school, 2 semesters of chemistry, and 2 semesters of physics. Interestingly, a significant portion of these engineering students answered that they were "neutral" or "uncomfortable" with science and technology related material, and this distribution did not change after a semester of college. These students had little exposure to nanoscience or nanotechnology in a previous class, although after the CHEM 131 course it is possible that a few of them realized that they had seen nanoscience topics before. Performance on exams indicated a single population rather than a "bimodal" distribution that we typically observe in general chemistry classes.

In both surveys a similar number of students were able to correctly identify the number of nanometers in a micrometer (pre = 49/94, post = 49/98). This number did not increase as one would expect. However, the students' perceived comfort levels and familiarity with nanoscience and nanotechnology increased. For example, 90% of students in the pre-survey were aware that nanotechnology is already in use, and this number increased to 97% in the post-survey.

Entering engineering students certainly are able to make connections between nanotechnology and traditional science fields. In the pre-survey, 65 students (69%) said that the fields of biology, chemistry, engineering, medicine, and physics would be affected by nanoscience, and 14 more (15%) chose four of the five fields. In the post survey, 65 students (66%) picked all five fields, and 18 more (18%) picked four of the five fields.

4.2 Laboratory Experiment Evaluation Forms

Evaluation forms were distributed to students and teaching assistants at the end of each lab period in which a new nanoscale experiment was performed. Statistical data (17 questions with a 5 point scale from "strongly disagree = 1" to "strongly agree = 5") and short comments (3 questions) were collected. For all of the labs, students consistently gave the highest ratings for the question, "My teaching assistant was helpful during the experiment." This reinforces our strong belief that proper TA training and preparation is essential to the success of a laboratory course such as CHEM 135. Informal discussions with teaching assistants during TA meetings indicated that they also had a high regard for students enrolled in CHEM 135.

Not surprisingly, the statement, "The *Video Lab Manual* helped me accomplish the experimental procedure," received relatively low ratings for the experiments that are simple to perform (students didn't need the videos to understand what to do), but received considerably higher ratings for the *Nickel Nanowire* experiment that involved many complicated steps. One student commented that (s)he wished (s)he could have watched the combustion reaction in the *Phosphor* experiment "live" (without closing the furnace door). The advantage of the *Video Lab Manual* is that a video clip of this dramatic reaction can be viewed without endangering students.

For all of the nanoscale experiments, students gave high ratings to the questions, "I understand the chemistry concepts related to this experiment," "This experiment was interesting," and "This experiment was fun." This is good news, since traditional chemistry laboratory experiments are often perceived as being unrelated to real life and boring.

5. CONCLUSIONS

In general, the nanoscale experiments in CHEM 135, *Engineering Chemistry Laboratory*, have been well received by students and appear to be an exciting way to convey chemical concepts. A few problems arose during implementation (bad solutions in the *Ferrofluid* experiment, wrong use of multimeters in the *Solar Cell* experiment), but were quickly discovered and resolved. We plan to continue assessment of the effectiveness of our efforts to integrate nanoscale science and engineering topics into the *Engineering Chemistry* courses.

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REFERENCES

Bentley, A. K., Farhoud, M., Lisensky, G. C., Ellis, A. B., and Crone, W. C. (2005). Synthesis and magnetic manipulation of nickel nanowires. *J. Chem. Educ*., in press.

Berger, P., Adelman, N. B., Beckman, K. J., Campbell, D. J., Ellis, A. B., and Lisensky, G. C. (1999). Preparation and properties of an aqueous ferrofluid. *J. Chem. Educ.*, **76**, 943-948.

Bolstad, D. B., and Diaz, A. L. (2002). Synthesis and characterization of nanocrystalline Y_2O_3: Eu^{3+} phosphor. *J. Chem. Educ.*, **79**, 1101-1104.

Condren, S. M., Breitzer, J. G., Payne, A. C., Ellis, A. B., Widstrand, C. G., Fuech, T. F., and Lisensky, G. C. (2002). Student-centered, nanotechnology-enriched introductory college chemistry courses for engineering students. *Int. J. Engng. Ed.*,**18**, 550-556.

Condren, S. M., and Lisensky, G. C. (2004). Disassembly of a liquid crystal watch. http://www.mrsec.wisc.edu/edetc/nanolab/watch/index.html.

Ellis, A. B., Geselbracht, M. J., Johnson, B. J., Lisensky, G. C., and Robinson, W. R. (1993). *Teaching general chemistry: a materials science companion*. American Chemical Society, Washington, D. C.

Halford, B. (2004). Advancing toward finer circuitry. *Chem. Eng. News,* **82**, (2), 5.

Keating, C. D., Musick, M. D., Keefe, and M. H., Natan, M. J. (1999). Kinetics and thermodynamics of Au colloid monolayer self-assembly: undergraduate experiments in surface and nanomaterials chemistry. *J. Chem. Educ.*, **76**, 949-955.

Kippeny, T., Swafford, L. A., and Rosenthal, S. J. (2002). Semiconductor nanocrystals: a powerful visual aid for introducing the particle in a box. *J. Chem. Educ.*, **79**, 1094-1100.

Nedeljkovic, J. M., Patel, R. C., Kaufman, P., Joyce-Pruen, C., and O'Leary, N. (1993). Synthesis and optical properties of quantum-size metal sulfide particles in aqueous solution. *J. Chem. Educ*., **70**, 342-345.

Rawls, R. L. (2003). Taking the pulse of chemical science. *Chem. Eng. News*, **81**, (09), 39.

Schulz, W. G. (2000). Nanotechnology: the next big thing. *Chem. Eng. News*, **78**, (18), 41.

Schulz, W. G. (2003). Nanotech business takes flight. *Chem. Eng. News*, **81**, (13), 21.

Smestad, G. P., and Gratzel, M. (1998). Demonstrating electron transfer and nanotechnology: a natural dye-sensitized nanocrystalline energy converter. *J. Chem. Educ*., **75**, 752-756.

Thayer, A. M. (2003). Nanomaterials. *Chem. Eng. News*, **81**, (35), 15.

Wilson, E. K. (2003a). Clear and present superparamagnet. *Chem. Eng. News*, **81**, (13), 29.

Wilson, E. K. (2003b). News of the week. *Chem. and Eng. News*, **81**, (21), 10.