

Introducing “Green” and “Nongreen” Aspects of Noble Metal Nanoparticle Synthesis: An Inquiry-Based Laboratory Experiment for Chemistry and Engineering Students

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S Supporting Information

ABSTRACT: In recent years, nanoscience and nanotechnology have been drawing enormous attention due to the numerous applications of nanomaterials. In an attempt to nurture interest towards these areas in young minds and to develop the next generation of environmentally conscious scientists and engineers, this new laboratory module focuses on the green and nongreen aspects of noble metal nanoparticles (NPs) synthesis. The element of novelty is represented by the guided, inquiry-based exploration of alternative, green fabrication methods using environmentally friendly reducing agents (e.g., tea extracts, coffee, honey, coconut oil, and banana peel). The inquiry-based learning was developed according to the five essential features laid by the National Research Council (NRC), and was successfully implemented after science and engineering students gained theoretical knowledge and hands-on experience with conventional, nongreen fabrication methods of colloidal silver and gold NPs (i.e., the Lee-Meisel and Turkevich methods). The student assignments and evaluations demonstrated that this inquiry-based laboratory increased students' interest in green nanochemistry, provided them with new laboratory skills, and stimulated their critical thinking.

KEYWORDS: Upper-Division Undergraduate, Graduate Education/Research, Environmental Chemistry, Interdisciplinary/Multidisciplinary, Laboratory Instruction, Hands-On Learning/Manipulatives, Inquiry-Based/Discovery Learning, Colloids, Green Chemistry, Nanotechnology



INTRODUCTION

Since the first recorded use of nanoscience in the fourth century, man has been intrigued with the unique properties of noble metal nanoparticles (NPs).¹ By 2015, the nanoscience and nanotechnology sectors are expected to contribute over \$2.4 trillion to the global economy and to impact our society, much like information technology has influenced the economy and the way we live.² This growth is related to the increased demand for nanotechnology-enabled products in the health-care, electronic, semiconductor, chemical and automotive markets.³ Thus, there is a great demand for qualified scientists, engineers and technicians in the field of nanoscience and nanotechnology. In fact, a recent study by the National Science Foundation (NSF) indicated that by 2020, over 6 million nanotech employees will be needed worldwide, with over 2 million of jobs being located in the U.S. alone.⁴

As of March 2013, the Nanotechnology Consumer Product Inventory comprised 1628 registered products or product lines.³ Over 380 of these products contain silver nanoparticles (AgNPs) and around 19 gold nanoparticles (AuNPs). Generally, colloidal noble metal nanoparticles are synthesized in a nongreen manner, by the reduction of a metallic salt.⁵ For example, one of the most widely used fabrication methods for colloidal AgNPs and AuNPs involves the high temperature, energy consuming reduction of silver nitrate (by Lee-Meisel^{6,7}) and tetrachloroauric acid (by Turkevich^{8,9}),

respectively, by trisodium citrate. As an alternative, green fabrication methods have been raising increased research and environmental interest. To address this need, a few groups have proposed traditional, undergraduate laboratory experiments that focus on the fabrication of noble metal nanostructures using reducing agents of low environmental impact and enhanced biodegradability such as green tea¹⁰ and plants.¹¹ Adding to the ongoing efforts, the main goal of this laboratory module is to help develop the next generation of environmentally conscious chemists and engineers by introducing them to both the green and nongreen aspects of noble metal nanoparticle synthesis. In comparison to the previous nanolaboratory educational work,^{12–20} the proposed laboratory module brings an additional element of novelty by using guided, inquiry-based learning. Briefly, undergraduate and graduate students from multiple disciplines (chemistry, physics, mechanical and materials engineering, and environmental sciences) were given a common research problem and were allowed to come up with their own, educated solution. In the first segment of the laboratory activity, students enrolled in an *Experimental Nanomaterials and Nanoscience laboratory course* synthesized AgNPs and AuNPs using the nongreen Lee-Meisel and Turkevich methods. In the second segment of the laboratory activity, students were requested to identify and

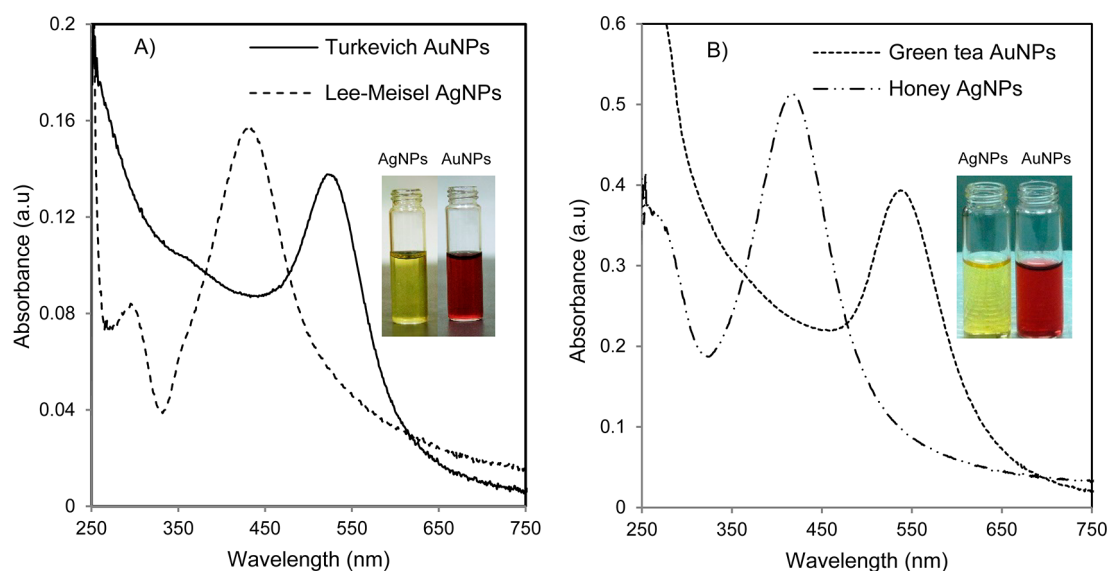


Figure 1. UV-vis absorption spectra and characteristic colors of the colloidal AgNPs and AuNPs synthesized by (A) nongreen and (B) green methods.

implement a green fabrication method based on their level of knowledge and experience.

The inquiry-based learning is a novel pedagogy that emphasizes students and teachers as learning partners.²¹ In real scientific scenarios, a researcher is exposed to several unanswered issues, and needs to use his cognitive abilities and scientific reasoning to address the experimental questions. A mere knowledge from text books as traditionally taught in instructional classrooms may not be sufficient to master the technical field. The inquiry-based learning provides room for students to question themselves and to explore the given topic of interest. In the past decade, this educational approach has been successfully encompassed in several elementary and higher grade level schools,^{20,22} as well as in the undergraduate curriculum.^{14,23,24} Thus, learning by guided scientific inquiry is expected to increase students' enthusiasm in nanochemistry as well as to broaden their understanding and acquisition abilities compared to conventional laboratory experiments.

MATERIALS AND METHODS

Reagents

All the reagents utilized in the experiment were obtained from Fisher Scientific. High purity water ($>18\text{ M}\Omega\text{ cm}$) was employed as solvent throughout the laboratory activity.

Nongreen Fabrication of Colloidal AgNPs and AuNPs

In the first segment of the laboratory activity, students performed test tube level syntheses of colloidal AgNPs and AuNPs using the Lee–Meisel⁶ and Turkevich^{8,12,25} methods, respectively. In this regard, a standard operating procedure (SOP) was prepared and distributed to students beforehand. Briefly, 20 mL of silver nitrate (AgNO_3 , 1 mM) and 20 mL of tetrachloroauric acid ($\text{HAuCl}_4\cdot 3\text{H}_2\text{O}$, 1 mM) were added to precleaned test tubes, which were labeled AgNPs and AuNPs, respectively. The test tubes were then placed in a hot water bath preheated to boiling for about 10 min. This was followed by the addition of 2 mL of trisodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7\cdot 2\text{H}_2\text{O}$, 34 mM) in each of the noble metal salt solutions. Shortly after (~ 15 min), notable color changes were observed in the test tubes, namely, wine red for AuNPs and dark yellow

for AgNPs (Figure 1A). To ensure the completion of the reduction reaction, heating was continued for another 10–15 min. It should be noted that the original synthesis procedures from literature were scaled down and adapted to fit the allotted laboratory time and resources.

Green Fabrication of Colloidal AgNPs and AuNPs

In the second segment of the laboratory activity, students performed synthesis of colloidal AgNPs and AuNPs using a series of environmentally friendly reducing reagents (e.g., phytochemicals in tea extracts,^{26,27} polyphenols in coffee,²⁷ glucose in honey,^{28,29} triglycerides in coconut oil³⁰ and cellulose, pectin in banana peel³¹), while the noble metal salt was kept the same (i.e., AgNO_3 or $\text{HAuCl}_4\cdot 3\text{H}_2\text{O}$). The solvent of choice was in most cases water, and the reactions were performed in test tubes under the instructor's supervision. The above-mentioned procedures were tested for reproducibility by the teaching assistants after the laboratory completion. Two sample procedures, which were adopted and employed by students for their inquiry-based learning assignment, are outlined below:

Natural Honey-Reduced AgNPs.²⁹ In this procedure, 1 mM of an aqueous AgNO_3 solution was reduced using a honey extract. The extract was prepared by dissolving 20 g of honey in 80 mL of water. Twenty milliliters of the AgNO_3 solution was dispensed into 15 mL of the honey extract, and the mixture was stirred well while heating at $80\text{ }^\circ\text{C}$ for 10 min. The appearance of a stable, dark yellow color (in ~ 10 min) marked the formation of AgNPs (Figure 1B). Stirring was continued for another 30 min to ensure the completion of the reduction reaction.

Lipton Green Tea-Reduced AuNPs.¹⁰ In this procedure, 100 mM of an aqueous $\text{HAuCl}_4\cdot 3\text{H}_2\text{O}$ solution was reduced using a green tea extract. The tea extract was prepared by magnetically stirring 200 g of tea leaves in 12 mL of water for 15 min at room temperature. One hundred microliters of the $\text{HAuCl}_4\cdot 3\text{H}_2\text{O}$ solution was added to 6 mL of the tea filtrate, and a wine red color characteristic to the formation of AuNPs was observed in ~ 10 min (Figure 1B). Stirring was continued for another 30 min to ensure the completion of the reduction reaction.

UV–Vis Absorption Spectrophotometry of Colloidal AgNPs and AuNPs

To confirm the formation of NPs, small colloid aliquots were collected at the end of each synthesis for further characterization using a Cary 50 UV–vis-NIR spectrophotometer (Varian Inc.), at 1 nm intervals. Prior to the UV–vis measurements, samples were diluted with high purity water (1:10 volume ratio) in disposable cuvettes of a total volume of 3.0 mL and a path length of 10.0 mm (Fisher Scientific).

HAZARDS

Both $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ and AgNO_3 (a strong oxidizer) have the potential to cause irritations upon contact with skin, eyes and respiratory tract. $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ is corrosive and can cause burns by all routes of exposure. All chemical reactions must be performed in a chemical safety hood. Personal protective equipment (PPE) must be worn at all times during the course of the experiment (laboratory coats, ANSI-certified safety goggles and gloves).

RESULTS AND DISCUSSION

Guided Scientific Inquiry

This laboratory module was developed according to five essential features laid by the National Research Council (NRC) for inquiry-based teaching and learning.³² In this approach, learners:

- (1) “Are engaged by scientifically oriented questions,
- (2) Give priority to evidence, which allows them to develop and evaluate explanations that address scientifically oriented questions,
- (3) Formulate explanations from evidence to address scientifically oriented questions,
- (4) Evaluate their explanations in light of alternative explanations, particularly those reflecting scientific understanding, and
- (5) Communicate and justify their proposed explanations”.³²

To incorporate these inquiry-based features into the proposed laboratory, the following steps were undertaken:

- (1) Students were posed a scientific problem, namely, the identification and implementation of a green fabrication method for colloidal AgNPs or AuNPs.
- (2) To guide the selection process, a few examples of nongreen and green bottom-up approaches were carefully discussed during the recitation period of the first segment of the laboratory activity, and the process of performing a scientific literature search was demonstrated. This was in particular helpful to the students, who had no previous experience with inquiry-based learning. Afterward, students successfully synthesized colloidal AgNPs and AuNPs using “cook-book” procedures for the well-established Lee–Meisel and Turkevich methods, respectively.
- (3) During the second segment of the laboratory activity, students were requested to review the scientific literature in order to identify a method for the fabrication of green NPs. Graduate students and upper-level undergraduate students were also encouraged to explore new, creative routes of fabrication. Some students actually adapted methods to include the use of materials not listed in scientific literature. For example, several students attempted to synthesize AuNPs using multiple roasts of

coffee or different types of tea from local grocery stores as green reducing agents.

- (4) The selected green methods were then closely assessed and modified (if necessary) together with the instructor to ensure the successful completion of the reactions within the allocated time period and access to all needed resources. Building upon the laboratory experience gained through the nongreen syntheses, students implemented the proposed green syntheses, characterized the colloidal products by absorption spectroscopy, and carefully collected the experimental observations.
- (5) Following the completion of the laboratory activities, students were invited to compare the results of the green and nongreen fabrication methods, and to justify their selection of a green method in a journal-article like laboratory report. Students were also encouraged to discuss the reactions involved in the reduction process and to provide future experimental measures for improving the green products.

Comparing the Optical Properties of Green and Nongreen Noble Metal NPs

A widely explored optical property of NPs, the localized surface plasmon resonance (LSPR), was utilized in this experiment to confirm the formation of AgNPs and AuNPs. LSPR arises from the collective oscillation of free electrons confined to the surface of nanomaterials (i.e., the so-called surface plasmons) upon the incidence of plane polarized light, at resonance wavelengths.³³ As a result, AgNPs and AuNPs exhibit characteristic LSPR peaks in the visible region of electromagnetic spectrum (Figure 1). The LSPR peaks and the colors exhibited by the colloidal NPs are interrelated: larger NPs exhibit red-shifted LSPR peaks. Representative UV–vis absorption spectra and the characteristic color of the colloidal NPs, which were fabricated by students using the nongreen and green methods, are shown in Figure 1. Stable AgNPs exhibited yellow color due to the absorption in the blue region corresponding to an LSPR peak at 420–440 nm. Similarly, AuNPs appeared wine red due to the absorption of green light corresponding to an LSPR maximum at 520–540 nm in the visible spectrum. The observed LSPR peaks were reproducible and were in good agreement with literature.^{6,12}

In-Class Assessments

To assess the overall comprehension of basic fabrication concepts and the correct execution of the experiments, a set of predefined laboratory skills (LS#1–4) were anonymously evaluated by the instructors (Table 1). These evaluations were based on the application of the theoretical knowledge and the experience gained through this laboratory. Table 1 shows that students completed the experiments with excellent laboratory practices and successfully applied the green chemistry principles underlying the proposed synthesis methods.

Because this laboratory targeted a multidisciplinary audience of science and engineering students, a small set of multiple choice questions (Q#1–5) was posed to students during recitation to evaluate their understanding of basic nanoscience concepts prior to the laboratory. The questions interrogated the nanoscale dimensions (Q#1), the Tyndall effect (Q#2), the type of nanofabrication methods commonly utilized by chemists (Q#3) and engineers (Q#4), and the color of colloidal nanoparticles associated with the LSPR effect (Supporting Information). At the beginning of the first segment

Table 1. Results of the Anonymous Assessments of Laboratory Skills

Laboratory Skill (LS)	Assessment Results ^a	
	Class Average (n = 15)	Standard Deviation
LS#1: Students were able to perform nongreen synthesis of NPs with the instructor's assistance.	1.1	0.2
LS#2: Students were able to independently extract the green reducing agent.	1.1	0.1
LS#3: Students were able to complete the green synthesis of NPs.	1.2	0.3
LS#4: Students were able to correctly collect the UV-vis absorption spectra on the diluted colloidal samples of NPs	1.1	0.2

^aLaboratory skills were assessed on a three point system: 1, *Always performing the skill*; 2, *Sometimes performing the skill*; and 3, *Never performing the skill*.

of the laboratory, students were invited to answer a similar set of questions. The student responses (Figure 2), which were

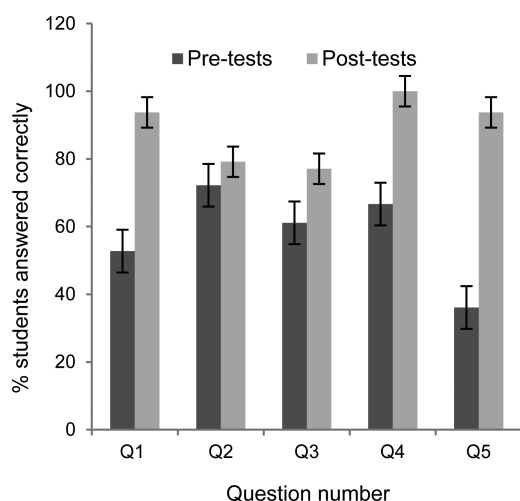


Figure 2. Histograms portraying the contribution of this laboratory module to the overall procurement of nanochemistry knowledge. Students ($n = 15$) volunteered to anonymously answer a set of scientific questions (Q#1–5) before and after the recitation period. For each question, the percentage of students that provided a correct answer was instantly evaluated by the Turning point software. Error bars represent the standard errors of the results.

recorded by means of Power Point polling (Turning Point Clicker technology), demonstrated that the introductory recitation material improved students' basic understanding and acted as a bridge for the students from various disciplines. As an additional benefit, this activity also increased class interactivity and improved student engagement. The completion of laboratory experiments culminated with a written assignment, which required students to compare the advantages and disadvantages offered by the nongreen and green fabrication methods of noble metal NPs in a journal article-like laboratory report.

Pre- and Postlaboratory Surveys

To ensure the overall success of the new laboratory experiment, anonymous surveys were administered to students. These evaluations were aimed at collecting feedback on the overall design of the laboratory, the students' interest and experience

before and after the completion of the experiments (SQ#1–4). On a comparative scale of 1–10, with 1 being the lowest, the survey results (Table 2) showed that the overall interest in this

Table 2. Results of the Anonymous, Pre- and Postlaboratory Student Surveys

Survey Question (SQ)	Survey Results ^a	
	Class Average (n = 15)	Standard Deviation
SQ#1: Overall interest in the proposed experiments prelaboratory	8.4	2.4
SQ#2: Overall interest in the proposed experiments postlaboratory	9.7	0.5
SQ#3: Overall experience in this laboratory	9.4	0.6
SQ#4: Overall rating of the laboratory module	9.7	0.6

^aStudents' responses to the survey questions were evaluated on a 10 point scale, with 1 being the lowest and 10 being the highest.

experiment increased from a class average of 8.4 prelaboratory to 9.7 postlaboratory. The comments from the postsurveys also showed that students' enthusiasm about nanoscience had increased. Here are a few examples of students' comments: "This laboratory is interesting, I would like to pursue a career in nanotechnology", "Employing green methods to make nanoparticles is cool", and "This nanotechnology lab is awesome".

CONCLUSIONS

The proposed laboratory module and the associated student assignments were successful in providing students of diverse academic backgrounds with hands-on experience and basic knowledge in the green and nongreen aspects of noble metal NP syntheses. Students' creativity and critical thinking were stimulated through a guided, inquiry-based learning approach. As a result, the overall interest and enthusiasm for green nanochemistry increased after the completion of the experiments as indicated by the student assignments and anonymous evaluations.

ASSOCIATED CONTENT

Supporting Information

Additional material for instructors, students, and lesson plan recommendations. This material is available via the Internet at <http://pubs.acs.org>.

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Notes

The authors declare no competing financial interest.

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