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Preparation of Gold Nanoparticles Using Tea: A Green Chemistry Experiment

R. K. Sharma,* Shikha Gulati, and Shilpa Mehta

Green Chemistry Network Centre, Department of Chemistry, University of Delhi, Delhi-110007, India

Supporting Information

ABSTRACT: Assimilating green chemistry principles in nanotechnology is a developing area of nanoscience research nowadays. Thus, there is a growing demand to develop environmentally friendly and sustainable methods for the synthesis of nanoparticles that utilize nontoxic chemicals, environmentally benign solvents, and renewable materials to avoid their adverse effects. A simple, economic, and environmentally benign experimental route to synthesize gold nanoparticles using tea leaves in an aqueous media at room temperature is described with a goal to introduce chemistry students to the concept of green chemistry as well as nanotechnology. The single-step method circumvents the use of surfactant, capping agent, or template and follows several principles of green chemistry. The experiment can be conducted in a typical laboratory session and is suitable for incorporation into the undergraduate introductory chemistry laboratory curriculum and constitutes an influential example of green chemistry in action.



KEYWORDS: First-Year Undergraduate/General, Laboratory Instruction, Physical Chemistry, Hands-On Learning/Manipulatives, Green Chemistry, Nanotechnology, Plant Chemistry, UV–Vis Spectroscopy

reen chemistry, the design of chemical products and **U** processes that reduce or eliminate the use and generation of hazardous substances, is a subject of emerging importance in chemical industry, research laboratories, and teaching laboratories because of environmental, health, and economic concerns.^{1–3} The American Chemical Society Green Chemistry Institute (ACS-GCI) is a foremost force in intriguing the concept and the practice of green chemistry around the world. The Indian International Chapter of ACS-GCI thrives by promoting and fostering the persistence of green chemistry through research,⁴ education,⁵ and outreach activities in India.⁶ In recent times, the green chemistry task force created by the Department of Science & Technology (Government of India) invited the head of this chapter (R.K.S., one of the authors of this paper) to develop a Green Chemistry Experiment handbook for undergraduate and postgraduate chemistry laboratories.7

Integration of green chemistry principles to nanotechnology is one of the key issues of nanoscience research. There is a growing need to develop environmentally friendly and sustainable methods for the synthesis of nanoparticles that utilize nontoxic chemicals, environmentally benign solvents, and renewable materials to circumvent adverse effects in medical applications. Thus, the efficacy of plant-based phytochemicals in the overall synthesis and architecture of nanoparticles and various nanoparticle-embedded products is extremely attractive as it brings an imperative symbiosis between plant sciences and nanotechnology. This connection between plant sciences and nanotechnology provides an inherently green move toward nanotechnology referred to as "green nanotechnology".⁸

In general, the synthesis of nanoparticles involves the reduction of metal salt by a reducing agent, followed by protection or coating of the freshly formed nanoparticles by a capping agent to prevent their agglomeration. Most of the "nongreen" procedures reported in the literature use organic solvents, sodium borohydride⁹ or hydrazine¹⁰ as reducing agents, and surfactants or polymers as stabilizer agents for the synthesis of gold and other metallic nanoparticles.^{11,12} All these substances are considered to be noxious and environmentally unfriendly. Moreover, most of these methods require intricate controls or nonstandard conditions making them quite expensive. We proposed an easy experimental procedure to synthesize gold nanoparticles (AuNPs) using tea leaves in an aqueous media at room temperature to introduce chemistry students to the concept of green chemistry as well as nanotechnology. There are numerous educational objectives incorporated in this experiment. The single-step green method uses no surfactant, capping agent, or template, and physicochemical characteristics are demonstrated, such as the color change of nanogold compared to the bulk gold. The experiment allows students to prepare gold nanoparticles using natural resource (tea leaves) and to study the effect of the tea concentration on the size of nanoparticles, which produces color changes that can be monitored by UV-visible spectroscopy. The synthesis and measurements of AuNPs are

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conducted in 1- or 2-h laboratory sessions, which is appropriate for an undergraduate green chemistry laboratory curriculum.

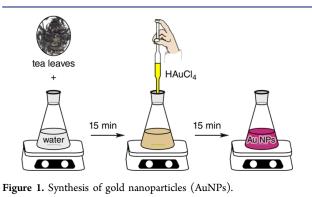
EXPERIMENTAL PROCEDURE

In designing an experiment involving the synthesis of metal nanoparticles from a green chemistry perspective, the choices of green solvent, environmentally benign reducing agent, and nontoxic material for the stabilization of nanoparticles were essential considerations. In the present protocol, water is utilized as an environmentally benign solvent throughout the preparation, and polyphenols and various phytochemicals present in tea (Table 1) are used as the reducing agent as well as the stabilizers on nanoparticles and, thus, provide robust shielding from agglomeration.

Table 1. Phytochemicals Present in Black Tea Leaves

Catechins	Theaflavins	Thearubigins
Catechin	Theaflavin	
Epicatechin	Theaflavin-3-gallate	
Epicatechin-3-gallate	Theaflavin-30-gallate	
Epigallocatechin	Theaflavin-3,30-gallate, etc.	
Epigallocatechin-3-gallate, etc.		

The synthetic approach for the production of well-defined gold nanoparticles involves simple mixing of an aqueous solution of tetrachloroauric(III) acid with the stock solution of tea leaves (Figure 1). The tea stock solution was prepared by



vigorous stirring of tea leaves in water over a magnetic stir plate for 15 min and filtering with Whatman filter paper. Darjeeling tea leaves were used. A small volume of the pale yellow gold solution was added to the tea solution with stirring and the solution turned purple red within 5 min indicating formation of gold nanoparticles. The reaction mixture was stirred for an additional 15 min, after which the stirring was stopped and the stir bar was removed. Production of AuNPs in this phytochemically mediated process was completed at room temperature within 30 min. The applications of AuNPs are dependent on their size, shape, and morphology, so the effect of different concentrations of tea (10%, 5%, and 1% stock solution) on the size and dispersity of gold nanoparticles was examined. The amount of gold added was fixed. Students characterized the gold nanoparticles by UV-vis absorption spectroscopy. The experimental details are fully documented in the Supporting Information. The techniques of transmission electron microscopy (TEM) analysis and Dynamic light scattering (DLS) could also be used to characterize gold

nanoparticles and instructor results are presented in the Supporting Information.

HAZARDS

Tetrachloroauric(III) acid is corrosive and will stain any contacting surface.

RESULTS

The resultant color of the nanoparticle solutions is dependent on the tea concentration. Because the size of nanoparticles can be varied on varying the quantity of capping agent, the solution color becomes more intensely purple red as the tea concentration increases (Figure 2), that is, wherein the greater

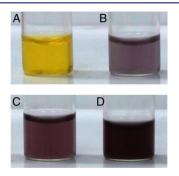


Figure 2. The solution color becomes more intensely purple red as the tea concentration increases: (A) 0% tea, (B) 1% tea, (C) 5% tea, and (D) 10% tea.

the quantity of tea used, smaller is the particle size formed. When dispersed in liquid media, the nanoparticles exhibit a strong UV-vis absorption band that is not present in the spectrum of the bulk metal. This band results when the incident photon frequency is resonant with the collective excitation of the conduction electrons and is known as the surface plasmon resonance (SPR). Because the SPR depends on the size, shape, and aggregation of AuNPs, UV-vis spectroscopy is a useful technique to estimate nanoparticles size, concentration, and aggregation level. The UV-vis spectra of the dispersions of different-sized nanoparticles produced by using 1%, 5%, and 10% tea stock solution, respectively, in this experiment are shown in Figure 3. As the concentration of tea leaves decreases, $\lambda_{\rm max}$ changes from 530 to 563 nm. The observed shift could be due to an increase in the particle size. In other words, the wavelength shift observed in the plasmon bands is a consequence of different concentrations of tea added to the gold ions solution.

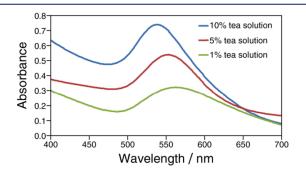


Figure 3. The UV-vis spectra of the dispersions of different-sized nanoparticles produced by using 1%, 5% and 10% tea.

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DISCUSSION

Our aim in designing this experiment was twofold. We sought to teach students the synthetic strategy of nanoparticles as well as the benefits of an approach that uses greener reagents, reaction conditions, and products. In the synthesis of metal nanoparticles by reduction of the corresponding metal ion salt solutions, there are three potential areas to engage in green chemistry: (i) choice of solvent, (ii) the reducing agent employed, and (iii) the capping agent (or dispersing agent) used. Several of the currently used nanoparticles processes utilize toxic chemicals either in the form of reducing agents to reduce various metal salts to their corresponding nanoparticles or as stabilizing agents to stop nanoparticles from agglomeration.¹³ But with the use of tea leaves, the synthesis of AuNPs colloids is so simple that no surfactant, photoirradiation, or heat is required. A well accepted scientific consensus emanating from several scientific investigations is that the phytochemicals present in tea serve a dual role as effective reducing agents to reduce gold and also as stabilizers to provide a robust coating on the gold nanoparticles in a single step.^{14,15} As the gold ions are reduced, gold atoms begin to aggregate, forming welldefined nanoparticles in the presence of tea. Therefore, using different concentrations of tea in the reaction media enables variation of the size of gold nanoparticles and, as a result, the colors of their dispersion.^{16,17}

CONCLUSION

This experiment fulfils all the criteria we set forth for a successful green chemistry experiment. In addition to the primary goals of teaching green chemical concepts and techniques, we found that inclusion of green experiments into the curriculum provides a platform for discussion of environmental problems in the classroom. The use of green reagents and solvent (water) in this experiment reduces the use of hazardous toxic chemicals. The experiment is expedient, economical, and can be carried out effectively in the teaching laboratory, and spare more time for students in the lab for other activities such as spectroscopy and analysis.

ASSOCIATED CONTENT

S Supporting Information

Experimental details; materials; equipment; and solution preparation instructions. This material is available via the Internet at http://pubs.acs.org.

AUTHOR INFORMATION

Corresponding Author

*E-mail: rksharmagreenchem@hotmail.com.

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