# CHEMICALEDUCATION

# Sweet Nanochemistry: A Fast, Reliable Alternative Synthesis of Yellow Colloidal Silver Nanoparticles Using Benign Reagents

Jason Cooke,\* Dominique Hebert, and Joel A. Kelly

Department of Chemistry, University of Alberta, Edmonton, Alberta T6G 2G2, Canada

**Supporting Information** 

**ABSTRACT:** This work describes a convenient and reliable laboratory experiment in nanochemistry that is flexible and adaptable to a wide range of educational settings. The rapid preparation of yellow colloidal silver nanoparticles is achieved by glucose reduction of silver nitrate in the presence of starch and sodium citrate in gently boiling water, using either a hot plate or domestic microwave oven as the heat source. During the experiment, the students are encouraged to consider the role that each reagent plays in the synthesis. If desired, the experiment can be repeated with the systematic elimination of individual reagents to more rigorously investigate the importance of each in the optimized reaction conditions. The benign reagents used in the preparation are commonly found in most educational science storerooms. The optimized synthesis has proven to be



quite reliable in student hands and may also appeal as an alternative method to instructors who already include the preparation of silver nanoparticles in their laboratory curriculum.

**KEYWORDS:** First-Year Undergraduate/General, Second-Year Undergraduate, Inorganic Chemistry, Laboratory Instruction, Hands-On Learning/Manipulatives, Colloids, Metals, Nanotechnology, UV-Vis Spectroscopy

Tanoscience is of continuing interest to the chemical education community at large. Four of the top 20 publications in the *Journal* for 2012 involved nanoscience,<sup>1</sup> and nanotechnology was the theme for National Chemistry Week in 2012.<sup>2</sup> The synthesis of silver nanoparticles is a popular and fairly inexpensive experiment in many educational laboratory courses that deal with nanochemistry. Indeed, an excellent experiment published in 2007 by Solomon et al.<sup>3</sup> remained one of the most-read articles in the Journal of Chemical Education in 2012.<sup>1</sup> Silver nanoparticles are of interest to the wider scientific community for a variety of reasons, ranging from their historical application in stained-glass windows<sup>4,5</sup> to more modern interests exploiting the characteristic surface plasmon resonance that gives metal nanoparticles their characteristic colors.<sup>6–8</sup> The use of benign reagents and experimental methods is also of increasing interest,  $9^{-14}$  particularly when the end use of the silver nanoparticles has a possible medical application such as antimicrobial or antibacterial activity.<sup>15–19</sup>

Several experiments exist in the chemical education literature, with sodium borohydride being the preferred reducing agent to produce silver nanoparticles.<sup>3,20–22</sup> Although we can verify that these syntheses are generally successful and certainly pedagogically useful, a potential disadvantage for some instructors is that sodium borohydride is a flammable, corrosive solid in its pure state that slowly decomposes in solution, the latter necessitating the preparation of a fresh solution for each laboratory period.<sup>3,20,21</sup> The evolution of hydrogen gas also requires the use of a fume hood or other well-ventilated environment and the cautioning of students to not tightly seal

their reaction flasks so as to avoid potentially dangerous buildup of pressure.<sup>20</sup> Concerns relating to the reproducibility of silver nanoparticle syntheses employing sodium borohydride have also been expressed, given the spontaneous decomposition of the reagent in solution and subsequent inaccuracy of active borohydride concentration.<sup>23</sup>

Other alternatives for educational settings have been reported, with geranium extract,<sup>11</sup> glucose<sup>24</sup> or household products (instant coffee, teas, juices)<sup>25</sup> serving as the reducing agent. In these cases, polydisperse silver nanoparticles form (with diameters varying from below 10 to over 70 nm). However, syntheses of monodisperse samples are often viewed more favorably as greater control over particle dimensions is achieved, which in turn allows greater insight into the physical, chemical, optical and electronic properties of these materials.<sup>9,14</sup> For example, NaBH<sub>4</sub> reduction mostly produces nanoparticles with 10-14 nm diameters; these monodisperse samples have characteristic yellow coloration with UV-vis absorption maxima centered near 400 nm and peak widths at half maxima (PWHM) of 50-70 nm.<sup>3</sup> Conversely, samples that are polydisperse show a pronounced shift to longer wavelength and broader peaks, with PWHM approaching or exceeding 200 nm.<sup>11,24,25</sup> From a practical perspective, producing the yellow sol is advantageous because the visual change from colorless to yellow immediately indicates a successful reaction.

The synthesis described below reproducibly generates high quality colloidal yellow silver nanoparticle sols using benign



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reagents widely available in most science storerooms. The experiment is flexible in design and should be adaptable to a wide range of educational laboratories, either as a new exercise or as an alternative synthesis if silver nanoparticles already feature in the curriculum. If desired, the experiment can be performed as a single synthesis or can be expanded to include a systematic study of the role played by each of the reagents.

# EXPERIMENTAL DETAILS

Colloidal yellow silver nanoparticles will form rapidly and reproducibly when an aqueous mixture containing about 2.5 mg/mL soluble starch,  $3 \times 10^{-4}$  M silver nitrate,  $2 \times 10^{-3}$  M sodium citrate, and  $5 \times 10^{-3}$  M D-glucose is heated to boiling. Soluble starch, D-glucose, trisodium citrate dihydrate, and silver nitrate ( $\geq$ 99%) were all reagent grade. Although we use Type 1 Millipore grade water,<sup>26</sup> we have verified that the experiment works well in good quality distilled water.

Several heating methods are possible. A conventional approach that combines all reagents in one flask and uses a hot plate with magnetic stirring typically produces the desired color change within a few minutes once gentle boiling has been achieved, but the total time taken is dependent on the rate of heating of the hot plate. Alternately, use of a domestic microwave oven (or one modified for microwave-assisted  $reflux)^{27}$  can reduce the heating time frame to 1 min or less. If available, the microwave oven approach reduces the time necessary for the overall synthesis significantly because heating is essentially instantaneous. If microwave ovens are not available, using a preheated hot plate and having a total solution volume of 10 mL reduces the induction time to achieve boiling to only 5-10 min. Overall, most students using the microwave approach can produce the yellow sol in about 15 min, whereas an additional 10 min is commonly needed if a preheated hot plate is employed.

After the reaction flask is cooled to room temperature, subsequent characterization of the sol is readily achieved by UV–vis spectroscopy. A laser pointer can also be used to demonstrate the Tyndall effect for colloidal suspensions.<sup>23</sup> If available, more sophisticated methods, such as transmission electron microscopy (TEM),<sup>3,11,14</sup> scanning tunneling microscopy (STM)<sup>24</sup> or atomic force microscopy (AFM)<sup>11</sup> could be applied.

If desired, the experiment can also be conducted in the absence of starch, with the less stable sol being compared and contrasted with the starch-protected one. The experiment can also be run eliminating first sodium citrate and then glucose to demonstrate the role played by each. Full details, including detailed student and instructor notes, are provided in the Supporting Information.

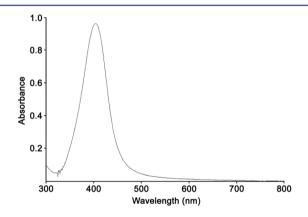
# HAZARDS

Silver nitrate is a corrosive solid that can stain and burn the skin and eyes; students and instructors working with solid silver nitrate should take appropriate precautions including the wearing of eye protection, appropriate gloves and a lab coat or apron. If provided as a dilute aqueous solution, the risk posed by silver nitrate is markedly reduced, but appropriate best practices for laboratory safety should still be followed. Insulated gloves or tongs should be used to manipulate flasks that are removed from hot plates or microwave ovens. All silver waste should be collected and disposed of according to local regulations.

# RESULTS AND EVALUATION

The synthesis has been successfully performed by six classes each of 35-39 third year undergraduate students in a course titled "Inorganic Materials Chemistry". The student body was diverse, including students who were chemistry majors and also nonspecialists who had enrolled to fulfill an option toward a general science degree. However, the relative ease of the experiment would make it appropriate for freshman undergraduate students in any course featuring nanochemistry, or potentially even senior high school students in a suitably equipped laboratory. Although we do not employ the rigorous glass-cleaning methods that are suggested by other workers,<sup>3,7</sup> a vast majority of students produce a yellow sol on their first attempt, and the few who do not are typically able to do so by repeating the procedure with a new reaction mixture. We have concluded that student error (for example, cleaning flasks with acid or laboratory detergent beforehand) is the most likely explanation for the infrequent failures. In some semesters, we have used D-glucose that is over 30 years old and silver nitrate of undetermined purity that was obtained through our chemical recycling program; these results also demonstrate the robust, flexible and reliable nature of the optimized reaction conditions.

The UV-vis spectra of the student samples typically show a single, narrow band centered between 400 and 405 nm with a PWHM of approximately 60–80 nm (Figure 1).



**Figure 1.** Typical UV–vis spectrum for the yellow sol produced by the glucose reduction of silver nitrate in the presence of starch and sodium citrate in a domestic microwave oven.

With initial  $[AgNO_3]$  of  $3.0 \times 10^{-4}$  M and microwave heating times of 30-60 s (or conventional gentle boiling for 3-5 min), the absorbance of the sol is usually between 0.8 and 1.2. The sols are stable and retain their yellow coloration indefinitely. Addition of an electrolyte such as NaCl has no visible effect; this contrasts with silver nanoparticles produced by borohydride reduction, which typically aggregate into larger particles with a color change to gray/brown unless a stabilizer such as polyvinylpyrrolidone (PVP) has been added.<sup>3</sup> When a laser pointer is directed through the sol, the laser beam is visible (Figure 2); this is a manifestation of the Tyndall effect which is observed for colloidal dispersion.<sup>23</sup>

Higher starting  $[AgNO_3]$  (up to ~1 × 10<sup>-3</sup> M) and/or longer heating times can produce more concentrated sols, but brown precipitates may form. Following dilution, the UV-vis spectra of such sols show a 10–15 nm shift to longer



Figure 2. Visualization of a laser beam directed through the yellow sol of silver nanoparticles.

wavelength and increased PWHM. Similar observations were made for samples where boiling was vigorous, but was not usually seen for syntheses performed in a microwave oven, which probably is due to better control over heating. Where facilities exist, TEM,<sup>3,11,14</sup> STM<sup>24</sup>, or AFM<sup>11</sup> could be

Where facilities exist, TEM,<sup>3,11,14</sup> STM<sup>24</sup>, or AFM<sup>11</sup> could be used. The TEM image of a representative sample from our optimized synthesis is shown in Figure 3, which conforms closely to results from similar syntheses in the literature<sup>9,13</sup> and also to the popular experiment employing NaBH<sub>4</sub> reduction.<sup>3</sup>

To demonstrate the role of each reagent in the synthesis, a series of experiments were carried out in which one or two reagents were omitted from the reaction mixture. A summary of these efforts is provided in the Supporting Information. The overall conclusion was that omission of any of the ancillary reagents resulted in poorer sol quality and/or poorer reproducibility. More specifically, eliminating starch led to yellow sols that were less stable to storage and aggregated when an electrolyte such as NaCl was added. Removing glucose caused either no or very slow silver nanoparticle formation in the short time frame allowed, and leaving citrate out resulted in few successful preparations. Thus, reliable generation of the desired yellow sol is best achieved by the stated combination of silver nitrate, soluble starch, D-glucose and sodium citrate.

The success of the experiment and its impact on the student educational experience was gauged informally by the teaching assistants who facilitated the activity in the lab. The impressions from the various instructors were discussed, with suggested improvements implemented during the subsequent year. Perhaps not surprisingly, the students were reported to be more satisfied when they were asked to perform syntheses that were reliable and reproducible, and felt frustrated if their work did not result in the production of the desired yellow sol. Accordingly, the primary focus of this work was to achieve the optimized synthetic conditions described earlier.

# DISCUSSION

Nanoparticles (<100 nm diameter) are primarily of interest because of their different physical properties when contrasted with small particles (>100 nm diameter) of the bulk solid. It is therefore interesting to discuss how size control is achieved in systems that successfully produce stable nanoparticles. It follows naturally that superior size control and the production of monodisperse samples is desirable, as this in turn implies greater control over the physical, chemical, optical and electronic properties of the materials. For example, when sodium borohydride is employed as the reducing agent, it is argued that an adsorbed protective surface of excess BH<sub>4</sub>anions effectively encapsulate the nanoparticles and that electrostatic repulsion prevents aggregation into larger particles. In this case, the reliance on electrostatic repulsion can be demonstrated by adding an electrolyte such as NaCl, which disrupts the adsorbed borohydride layer and leads to aggregation of the nanoparticles.

Starch has been identified as being able to form a protective template for the production of nanoparticles,<sup>13,28</sup> and it has been suggested that starch plays a role in controlling particle size.<sup>9</sup> Starch is the second most abundant biomolecule on earth and is predominantly composed of the branched amylopectin and linear chain amylose polymers of glucose (Figure 4).

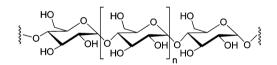
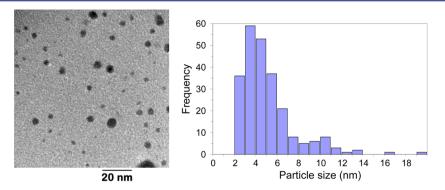


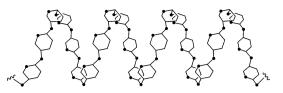
Figure 4. Structure of amylose comprising repeating glucose units.

Amylose adopts left-handed helical structures in the solid state (Figure 5), but whether dissolved starch retains this structure or adopts a more random coiled arrangement has been a subject of some controversy.<sup>29</sup>

The largest central cavity available in the amylose helices has a diameter of about 0.4 nm, which is large enough to accommodate small molecules such as iodine and n-butanol.<sup>29</sup> However, this is at least an order of magnitude less than the



**Figure 3.** TEM image of a representative sample of silver nanoparticles produced by glucose reduction in the presence of starch and sodium citrate in a domestic microwave oven. Mean particle size = 5.1 nm and  $\sigma$  = 2.5 nm (*n* = 241 particles). The TEM image was obtained on a JEOL-2010 microscope, with a lanthanium hexaboride filament and 200 keV accelerating voltage.



**Figure 5.** A depiction of the helical structure of amylose adopted in the solid state. For clarity, only ring atoms are shown. Oxygen atoms are represented by solid circles  $(\bullet)$ .

diameter of the smallest silver nanoparticles that are observed (Figure 3), and so it is clearly not possible for the nanoparticles to form and remain within the tight helices. Rather, it has been suggested that the disordered, flexible arrangement of the amylose chains in solution and abundance of hydroxyl groups create protected areas that facilitate silver ion complexation and encapsulate the nanoparticles to protect against further aggregation and particle growth.<sup>9,10</sup> Referring to Figure 5, it is not difficult to imagine the amylose helix unwinding in solution to surround the larger silver nanoparticles. In other systems, a similar encapsulation can be achieved by adding PVP.<sup>3</sup>

It is also possible to produce colloidal yellow silver nanoparticles from mixtures containing silver nitrate, sodium citrate and glucose, but these sols were less stable, especially toward added electrolyte. Thus, our results suggest that while dissolved starch may not necessarily play a role in controlling nanoparticle size, it does clearly have a beneficial protective influence over the suspended nanoparticles.

A connected topic is the importance of the other reagents in the reaction mixture, and how they contribute to controlling the size and monodispersity of the formed nanoparticles. As starch is fundamentally a polymer comprised of glucose units, it is logical to include the monomeric sugar as the reducing agent in the synthesis, particularly as it has been successfully used as such in other related applications.<sup>13,15,24,28,30</sup> Although nanoparticle formation in hot starch solutions has been reported to be comparatively slow to the present procedure, it has been suggested that starch hydrolysis forms the glucose that is required to reduce Ag<sup>+</sup>.<sup>19</sup> Also, while sodium citrate is widely used as an independent reducing agent in silver nanoparticle formation,<sup>31,32</sup> we have been unable to achieve similar results under the present reaction conditions unless starch was also present (and then, not reliably); it is likely that much longer heating periods are necessary to achieve nanoparticle formation in reaction mixtures containing only Ag<sup>+</sup> and citrate ion. Accordingly, glucose is the preferred reducing agent, as it promotes highly reproducible and very rapid nanoparticle formation.

The reduction of  $Ag^+$  by glucose is represented by the equation:

HO  
HO  

$$HO$$
  
 $HO$   
 $HO$ 

The function of the included citrate ion as a near-neutral or slightly basic buffer<sup>20</sup> becomes apparent, as the reduction of silver produces an equal molar quantity of acid. In the absence

of added base, it seems likely that the acidic solution could etch or redissolve any nanoparticles that might form. This holds with observations that silver nanoparticle formation in glucose/ starch systems is dependent upon maintaining neutral or slightly basic pH, which is not achieved in the absence of an added weak base.<sup>28</sup> The citrate anion can also act as a capping agent, and it has been used in this role in the synthesis of gold nanoparticles,<sup>33–36</sup> silver nanoprisms,<sup>20</sup> and polydisperse silver nanoparticles.<sup>19,24,31</sup> Excess glucose has also been suggested to function as a capping agent for the nanoparticles,<sup>24</sup> but our results cannot conclusively confirm this as the reactions attempted on solutions containing only AgNO3 and glucose frequently failed to produce the desired yellow sol. However, given the similarity of the sols prepared in the presence and absence of starch, it can be stated that a combination of glucose and citrate effectively controls the size and initial stability of the silver nanoparticles that are formed, while starch acts as an additional protecting agent to enhance the longevity of the sample.

# SUMMARY

We have described an accessible and reliable nanochemistry experiment that uses benign reagents commonly found in science storerooms. The synthesis is suitable for a wide range of educational settings, and characterization of the silver nanoparticles that are produced can vary from a simple assessment of color to the application of sophisticated imaging techniques. The procedure is suitable as a "stand alone" exercise, or can be incorporated as part of more complicated experiment that includes other tasks from previously published works in this *Journal*. Additionally, it can be carried out as a simple verification experiment or can be made more open-ended with the students carrying out an investigation of the role that each reagent plays in the synthesis.

#### ASSOCIATED CONTENT

#### Supporting Information

Detailed information regarding the experimental procedure, reagents used, and instructor notes. This material is available via the Internet at http://pubs.acs.org.

#### AUTHOR INFORMATION

#### **Corresponding Author**

\*E-mail: jason.cooke@ualberta.ca.

#### Notes

The authors declare no competing financial interest.

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