Synthesis and Characterisation of Gold-Silver Nanoparticles

by Stephen Ralph

Experiment Overview

There has been considerable interest in nanoparticles over the last 10 to 15 years. This has been motivated by interest in new materials for miniaturisation of electronic devices, as well as fundamental questions about how molecular properties, including electronic and optical properties, change with particle size. One of the most exciting aspects of the nanotechnology revolution is the discovery that quite unique and novel properties occur at the nano (10^{-9} m) dimension. Gold nanoparticles or "colloidal gold" have been known for many years, and can be easily prepared by using citrate to reduce a gold complex. The resulting colloidal or nanoparticle solutions are brightly coloured.

In this experiment students will prepare not only gold nanoparticles, but also a range of gold / silver alloy nanoparticles. By measuring the visible absorption spectra and particle size distribution of the resulting solutions, the effect of varying the composition of the nanoparticles on their physical properties can be elucidated.

Aims and Objectives

This experiment involves the synthesis of gold nanoparticles and a series of gold / silver nanoparticles with differing compositions. The nanoparticles are readily prepared by using citrate ion as a reductant with aqueous solutions containing either gold chloride [$HAuCl_4$] or mixtures of gold chloride and silver nitrate. They are then characterised by measurement of their visible absorption spectra and particle size distributions.

Nanotechnology involves the development of devices and structures based on ordered materials with at least one dimension in the range 1 – 100 nm. Nanoparticles are one of the most important classes of nanomaterials, and are being actively investigated for a host of applications including drug

and gene delivery, tissue engineering, optoelectronic displays, new sunscreens, magnetic storage devices, solar energy capture and biosensing. The principal aim of this experiment is to introduce students to the unique chemical and physical properties displayed by materials when prepared on a nanodimensional scale. This is achieved by the striking difference in colours between solutions of the gold chloride complex used as a starting material, and the gold nanoparticles subsequently produced. An additional aim of the experiment is to introduce students to one of several methods of particle size characterisation. The zeta-sizer is a simple to use instrument that enables the systematic variation in particle size distributions between the different solutions to be examined.

Level of Experiment

Third year undergraduate

Keyword Descriptions of the Experiment

Domain
inorganic chemistry, physical chemistry

Specific Descriptors
nanoparticles, nanotechnology, particle size measurements

Course Context

This experiment is one of six that comprise the laboratory course for our 3rd level subject "Advanced Materials and Nanotechnology". During the course students receive 12 lectures covering various classes of advanced materials including carbon nanotubes, polyelectrolytes, conducting polymers and nanoparticles. This experiment provides a simple but effective illustration of how the chemical and physical properties of a material can be dramatically altered when prepared on the nanoscale, by allowing a comparison of the colours of gold complex ions (yellow) and gold nanoparticles (red).

Prerequisite Knowledge and Skills

In order to successfully complete this experiment students must know what nanomaterials such as nanoparticles are, and understand why these materials display different physical and chemical properties to ordinary bulk phase materials. In order to complete the experimental report they must be able to describe the physical technique used in the experiment to measure particle sizes (dynamic light scattering), and the basic principles behind scanning electron microscopy. They should have a thorough understanding of redox chemistry, and sufficient knowledge of coordination chemistry to know what is a complex ion. Students will require basic pipetting skills and the ability to prepare solutions by dilution. They must also be able to measure absorption spectra using a spectrophotometer, and calculate mole fractions.

Time Required to Complete

Prior to Lab: 30 min
In Laboratory: 3 h
After Laboratory: 5 h

Experiment History

This experiment has been in operation at the University of Wollongong now for two years. It was developed by Assoc. Prof. Will Price to be part of the new subject "Advanced Materials and Nanotechnology", which was also introduced two years ago, and which is coordinated by the author of this educational analysis. The experiment is an extension of another developed by Assoc. Prof. Price which involves the synthesis of nanoparticles, and which is carried out as part of our second year physical chemistry curriculum.

Comments

An integral component of this experiment is the measurement of particle size distributions for the various nanoparticles prepared. This is achieved at the University of Wollongong using a MALVERN zeta-sizer instrument, which utilises the dynamic light scattering technique. Since not all departments will have such an instrument available, it is important to note that there are other methods for performing particle size analysis, such as transmission electron microscopy.

A related experiment in which gold nanoparticles are used as an electrolyte sensor is described in the *Journal of Chemical Education* (see reference 3). This experiment is based on the different colours displayed by gold nanoparticles when they undergo aggregation after being exposed to different concentrations of salt. It would be very easy to incorporate some aspects of this experiment into that presented here in order to provide a visual aid to students to assist them understand the importance of electrostatic forces in determining the size and stability of nanoparticles.

References

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