

Synthesis of gold nanoparticles



**ASSIGN
BUSTER**

Gold ions are reduced to neutral gold atoms, where citrate ions act as both a reducing agent and a capping agent. This formation of gold nanoparticles can be observed by a change in color since small nanoparticles of gold are red. The size and concentration of gold nanoparticles can be calculated by a specific equation. The layer of absorbed citrate anions on the surface of the nanoparticles keep the nanoparticles separated. Switching to a smaller anion allows the particles to approach more closely and another color change is observed.

Introduction

Gold was one of the first metals discovered by humans, and the history of its study counts several thousands of years. Gold nanoparticles (AuNPs) have found widespread applications in different areas. While synthesis of monodispersed AuNPs has been fairly convenient by using chemical reduction of hydrogen tetrachloroaurate by trisodium citrate dihydrate, the AuNPs of high quality and high concentrations were not readily obtained via this method. As an example, the monodispersed around 15-17 nm AuNPs were readily synthesized at relatively low concentrations (e. g. 5. 8 nM); in contrast, 13 nm AuNPs of 10. 8 nM obtained by the direct reduction method were irregularly shaped and not well dispersed. For some works, the AuNPs of high concentration could be prepared by a two-step approach, i. e. chemical reduction at low concentrations and subsequent centrifugation. Compared to the direct reduction method, this new two-step method led to AuNPs with high salt resistance and high stability, which are essential for the preparation of DNA-AuNPs conjugates for mercuric ions detection. In certain

cases, the AuNPs prepared by chemical reduction is suited for detecting mercury even the concentration is relatively low.

Experiment Progress

Materials and Equipment

Chemicals

Gold(III) Chloride trihydrate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$) – (Hydrogen tetrachloroaurate)

Sodium citrate dihydrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$) – (Trisodium citrate dihydrate)

Stock Solutions

1. 0 mM hydrogen tetrachloroaurate: The solid is hygroscopic and based on its molecular weight 393. 83 g/mol so use $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ in 0. 4 g quantities. Dissolve 0. 4 g $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ in 1. 0 mL filtered Milli-Q water to make a 1. 04 M stock solution of gold(III) ions that stored in a centrifuged tube. Dilute 47. 87 μL of stock to 50. 0 mL to make the 1. 0 mM concentration for this experiment.

38. 8 mM trisodium citrate: Dissolve 1. 1 g $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (sodium citrate) in 100 mL filtered Milli-Q water.

The water (Milli-Q water) used to dilute was filtered beforehand to remove dust or impurities.

Equipment

- Reflux (Three-necked bottle and Condenser)
- Stirring hotplate

- Pipettes
- 50 mL beaker or volumetric flask
- Glass bottle and centrifuged tubes
- Magnetic stir bar

Experimental Procedures

Before the experiment, all the glassware used must be marinated by nitrohydrochloric acid in order to digest and remove dust or impurities.

Pale yellow 1.0 mM $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ and colourless 38.87 mM trisodium citrate were prepared using filtered Milli-Q water.

Set up the reflux.

Add 50.0 mL of 1.04 mM HAuCl_4 to a three-necked bottle on a stirring hot plate. Add a magnetic stir bar and bring the solution to a rolling boil. (Figure 1)

To the rapidly-stirred boiling solution, quickly add 5.0 mL of a 38.8 mM solution of trisodium citrate dihydrate, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$. The gold solution gradually forms as the citrate reduces the gold(III). Remove from heat when the solution has turned deep red or 10 minutes has elapsed. (Figure 2)

The solution was cooled down to room temperature, and a wine red solution was obtained as product. (Figure 3)

Result

After this experiment, a wine red solution of gold nanoparticles was obtained. During the preparation of AnNPs solution, the color of the solution

was change from black gray, oxford blue, modena, and to deep red (wine red). By the UV-Vis spectrometer measurement, the highest peak of absorbance of gold nanoparticles was presented at 520nm wavelength. The followings are the absorbance spectra of AuNPs solutions in the first and second preparations.

According to the absorbances, the size of gold nanoparticle was calculated by a specific equation. For the first preparation of AuNPs solution, the size and concentration of gold nanoparticles are 15.3 nm and 7.20nM, respectively. Whereas, the size and concentration of second run AuNPs are 16.5 nm and 5.79nM, respectively. These results may not suitable for determine mercuric ions. Generally, the size of AuNPs is around 13 nm. And the concentration of AuNPs for these two run preparations is lower.

Discussion

A variety of chemical methods can be employed to produce monodisperse gold nanoparticles. However, three procedures have become the most common for making particles that fall into predictable size ranges. In all three processes, hydrogen tetrachloroaurate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$) in aqueous solution is reduced by an agent in order to produce spheroidal gold particles. The greater the power and concentration of the reducing agent, generally, the smaller the resultant gold particles in the suspension

To create large-sized gold nanoparticles, an aqueous solution of hydrogen tetrachloroaurate is treated with trisodium citrate in aqueous solution. This results in particles sized 15-150 nm, the final range depending on the concentration of the citrate used in the reduction process. Medium-sized gold

particles with diameters between 6 and 15 nm and an average size of 12 nm are formed by reducing the hydrogen tetrachloroaurate solution with another agent solution, such as sodium ascorbate. The smallest particles, measuring less than 5 nm in diameter, are produced by reduction with either white or yellow phosphorus in diethyl ether.

A variety of physical parameters affect the quality of the final gold nanoparticles that is produced by the reaction of aqueous hydrogen tetrachloroaurate with an aqueous solution of trisodium citrate. Key factors worthy of consideration are the concentration of reactants, mixing of the reactants, the order in which reactants are added, operating temperature and so on.

Conclusion

The gold nanoparticle was prepared by reduction method. The size of gold nanoparticles is around 16 nm in diameters. Before the addition of the reducing agent, gold ions exist in solution. Immediately after the reducing agent is added, gold atoms start to form in the solution, and their concentration rises rapidly until the solution reaches supersaturation.

All gold nanoparticle display a single absorption peak in the visible range between 510 and 550 nm. With increasing particle size, the absorption maximum shifts to a longer wavelength, while the width of the absorption spectra relates to the size range. The smallest gold colloids (2-5 nm) are yellow-orange, midrange particles (10-20 nm) are wine red, and larger particles (30-64 nm) are blue-green. Smaller gold particles are basically

spherical, while particles in the range of 30-80 nm show more shape eccentricity related to the ratio of major to minor axes.

Future Work

The objective of this project is to detect the mercuric ions in varied samples by DNA functionalized gold nanoparticles. The preparation of gold nanoparticles through reduction of hydrogen tetrachloroaurate by trisodium citrate dihydrate was done in the first step. The next step is to assemble the functional gold nanoparticles with mercaptopropionic acid (MPA) first and DNA capped gold nanoparticles subsequently. After work, the measurement of selectivity and detection limit should be done, and the functionalized by MPA and DNA gold nanoparticles will use to determine the mercury content in real sample. By doing the determination using gold nanoparticles, the silver nanoparticles will be use as the probes to mercuric ion detection based on the progress of experiment.