Experiment 2: Preparation of Gold Nanoparticles

Rami Wail 201600112 – Ahmed Khaled 201601309 – Mennatllah Khaled 201601533
ZEWAIL CITY: UNIVERSITY OF SCIENCE AND TECHNOLOGY

INTRODUCTION

The objective of this experiment is the synthesis of and characterization of citrate-capped gold nanoparticles. Karunanayake et al. [1] explains that nanoscience is the study of matter at the nanometer scale, that is, where at least one dimension is below 100 nm in length. It is worth mentioning, even though physical and chemical properties of bulk materials are generally unaltered with size, they exhibit size-dependent characteristics at the nanoscale. This is caused by remarkably greater surface to volume ratios. Essentially, gold nanoparticles (AuNPs) are produced by reduction of chloroauric acid ($H(AuCl_4)$, hydrogen tetrachloroaurate) in an aqueous solution. Moreover, after dissolving in deionized water, the solution is rapidly stirred while a reducing agent, such as trisodium citrate, is added. Furthermore, formed Au^{3+} ions are reduced to neutral gold atoms. Accordingly, gold atoms gradually increase and aggregate to form AuNPs in a colloidal solution. It is worth mentioning, that the nanoparticle size can be regulated by the concentration of the citrate solution. This solution acts as both the reducing agent and stabilizer. Increasing citrate concentration results in smaller AuNPs and vice versa. This is because the Au^{3+} ions are limited and increasing citrate results in nucleation of more AuNPs initially which leads to lower average AuNPs size. On the other hand, AuNPs are used in many applications in various fields of study and research. Not only have they recently gained favor in various biomedical fields such as their use in therapeutic agent delivery, or in photodynamic therapy (cancer treatment), but they also have many uses in other fields including electronics, sensory, probing and catalysis. Accordingly, a wide array of characterization methods is used for these highly size-dependent nanoparticles, including: -

- UV−Vis spectrophotometry
- Transmission electron microscopy (TEM)
- Dynamic light scattering
- Scanning electron microscopy (SEM)

Materials

Used Chemicals	Used Tools
Hydrogen tetrachloroaurate(III)	Two closed bottles
Sodium citrate	A sensitive balance to weigh sodium citrate.
Deionized water	Graduated cylinders to quantify deionized water and hydrogen tetrachloroaurate(III)
-	A magnetic stirrer and heater

Methods

The final target of the procedures is to obtain gold nanoparticles. Firstly, 32.5 mL of previously prepared hydrogen tetrachloroaurate(III) solution was transferred to a closed bottle. The solution was subjected to heating at 140 degrees Celsius and magnetic stirring at 300 rpms at the same time. Secondly, 53 mg of sodium citrate was dissolved in 7.5 mL of deionized water. They were put in a closed bottle and shaken gently to ensure adequate dissolving. After the hydrogen tetrachloroaurate(III) solution starts boiling, the sodium citrate solution is added to it. Then, the heating temperature was raised to 200 degrees Celsius. The mixture was left on the heater for 5 minutes while stirring. Finally, the heater is turned off and the mixture underwent magnetic stirring for 15 minutes.

Results

1. When the Tri-sodium citrate solution (colorless) was added in one shot to the boiling solution of hydrogen tetrachloroaurate (yellow), the yellow color suddenly disappeared as shown in figure 2.
2. Then, during the next five minutes of stirring and heating, the color was observed to turn grey, then black. See figure 3.
3. After turning the heat off and keeping the stirring, the color was observed to change from black to dark wine-red color, as in figure 4

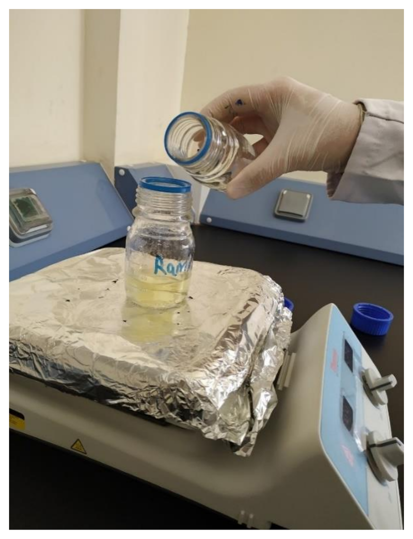

Fig 1: The sodium citrate solution is added to the hydrogen tetrachloroaurate(III) solution.4

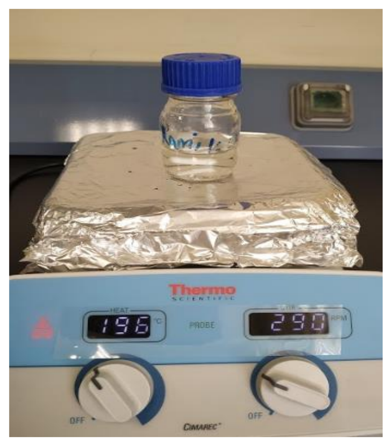

Fig 2: The yellow color disappeared

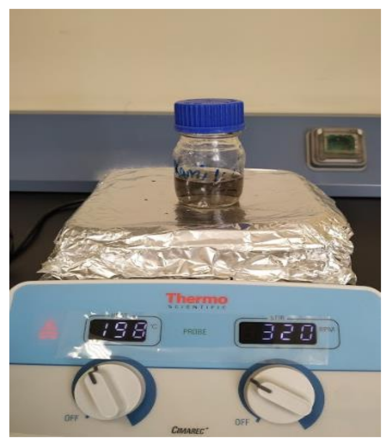

Fig 3: The color of the solution is turning black

Discussion

1. The disappearance of the yellow color at the beginning was due to the reduction of gold ion into gold atoms.
2. The change of the solution color to black was due to the formation of gold nanoparticles that could absorb the light.
3. As the size of the nanoparticles gets larger, it could absorb light with relatively larger wavelength and thus reflecting smaller wavelengths.
4. Higher concentration of citrate produces smaller nano particles. This is because larger concentration of citrate causes more nuclei to be formed and since we have a fixed amount of gold, the larger the number of particles, the smaller their size is.

Chemical properties [2]

Chemical symbol	Au
CAS No.	7440-57-5
Group	11
Electronic configuration	[Xe] $4f^{142}$ $5d^{10}6s^{1}$

Applications [3]

Electronics	Gold nanoparticles are designed and utilized as conductors from printable inks to electronic chips.
Photodynamic Therapy	Near-IR absorbing gold nanoparticles produce heat when excited by light at wavelengths from 700 to 800 nm. Used to eradicate targeted tumors.
Therapeutic Agent Delivery	Therapeutic agents can also be coated onto the surface of gold nanoparticles.
Sensors	Gold nanoparticles are used in a variety of sensors.
Probes	The scattered light colors of gold nanoparticles under dark-field microscopy are currently used for biological imaging applications.
Diagnostics	Gold nanoparticles are also used to detect biomarkers in the diagnosis of heart diseases, cancers, and infectious agents.
Catalysis	Gold nanoparticles are used as catalysts in a number of chemical reactions

Conclusion

The goal of this experiment was to prepare gold nanoparticles in the bottle. The procedure was to put 32.5 mL of hydrogen tetrachloroaurate (III) in a bottle. Secondly it was subjected to magnetic stirring while also heating at 140 degrees Celsius. After the solution started boiling and sodium citrate dissolved in deionized water was added which changed the solution color from yellow to transparent. After stirring with heating at 200 degrees Celsius the solution for 5 minutes then 15 without heat. The solution was gradually getting darker from transparent to grey then black, finally dark red wine color was formed. The red wine color indicate the formation of gold nanoparticle successfully. The dark red wine color indicate absorption of small wavelength light.

Fig 4: Colors of various sized monodispersed gold nanoparticles [3]

References

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