



Synthesis of Gold Nanospheres Using Citrate, Gallic Acid, and Glutathione/ Sodium borohydride as Reducing Agents and Stabilizers: A Comparative Study

Luane S. Soares¹ (G), Kennedy B. Gonçalves² (PG), Juliana C. S. Almeida¹ (PQ), Tiago S. Gontijo¹ (PQ),
Livia S. Gomes² (PQ), Rosimeire C. Barcelos¹ (PQ)*

¹ Federal University of São João del-Rei (UFSJ), Campus Centro-Oeste Dona Lindu, Divinópolis/MG - Brazil – 35.501-296.

² Nanomaterials and Graphene Technology Center, Federal University of Minas Gerais, BH/MG - Brazil – 31.310-260.

*e-mail: rosicbarcelos@ufsj.edu.br

ABSTRACT

The choice of reducing agent used in the synthesis of gold nanoparticles (AuNPs) is a determining factor in the efficiency and final properties of AuNPs, directly influencing the size, shape and position of the absorption peak in the UV-Vis spectrum. In this study, spherical gold nanoparticles were synthesized using different reductants — sodium citrate, glutathione/sodium borohydride and gallic acid — in order to analyze the reproducibility, homogeneity, presence of amorphous particles and consistency between samples. The syntheses were performed under standardized conditions, and the obtained gold nanospheres were characterized by UV-Vis spectroscopy. Dynamic Light Scattering (DLS) analyses are in progress. Preliminary results revealed significant differences between the reductants used, reinforcing the importance of this step to ensure the synthesis of gold nanospheres with well-defined and specific characteristics, necessary according to the desired final application for AuNPs.

Keywords: Gold nanoparticles; Reducing agents; Glutathione; Gallic acid; Sodium citrate.

Introduction

Nanotechnology is a constantly growing area that has been gaining visibility in contemporary society, driven by the understanding of the unique properties of nanoscale materials, which significantly increase their conventional forms.[1] Gold nanoparticles (AuNPs) stand out for their stability, biocompatibility, ease of functionalization and intense optical response resulting from Localized Surface Plasmon Resonance (LSPR), in addition to having potential for therapeutic applications. Their shape can vary — including spheres, rods and prisms — and is chosen according to the desired application.[2] The reducing agents used in stability ensure the retention of the gold, but also have the function of stabilizers, since they adsorb on the surface of the particles and prevent electrostatic repulsion, being of fundamental importance for AuNPs to avoid aggregation.[3]

Experimental

All syntheses were performed using the same stock gold solution, chloroauric acid (0.1 mol/L) and the reaction protocol was standardized as far as possible, so as not to favor any of the reductants. The synthesis using glutathione (GSH), took the hydrochloric acid solution and mixed it with a GSH solution (0.018 mol/L), the pH of the reaction was approximately 9, since NaOH (1 M) was used to neutralize the reaction medium. After this step, a stronger reductant NaBH₄ was added, the entire process was carried out at room temperature under stirring and remained overnight. In the citrate synthesis, the reflux procedure was used until the gold solution reached the boiling point, being mixed with a citrate solution (0.05 mol/L), the pH was approximately 5, not needing to be

neutralized, in the entire reaction the temperature was controlled and in an increasing manner. Finally, the synthesis with gallic acid was similar to that of citrate, using the reflux circuit with the mixture of the boiling gold solution with a gallic acid solution (0.0013 mol/L), its pH around 10, since it used a NaOH solution (0.5 M).

Results and Discussion

The characterization of AuNPs was performed by UV-VIS spectroscopy. Dynamic Light Scattering (DLS) analyses are in progress. We observed that there was a maximum absorbance range around 520-530 nm in the UV-VIS spectra (Figure 1) using the 3 proposed reducing agents, indicating the formation of nanospheres. Another parameter to evaluate the efficiency of the reactions is the color change (Figures 2-4) and the characteristic peaks in the graph of symmetric isotropic particles, such as spheres. Our protocol was efficient and gold nanoparticles encapsulated in GSH, citrate and gallic acid were successfully fabricated. However, from Figure 1, it can be noted that gallic acid becomes a more efficient reductant because the nanoparticles are more homogeneous. Regarding glutathione and citrate, no significant differences were observed in the reduction efficiency. However, glutathione appears to favor the formation of less homogeneous particles, with a greater proportion of amorphous structures, as suggested by the graphs, due to the greater width at half height, evidencing less homogeneous particles among themselves. Furthermore, its synthesis proved to be more complex and sensitive to experimental variations and encountered some obstacles during the centrifugation process due to the pH.

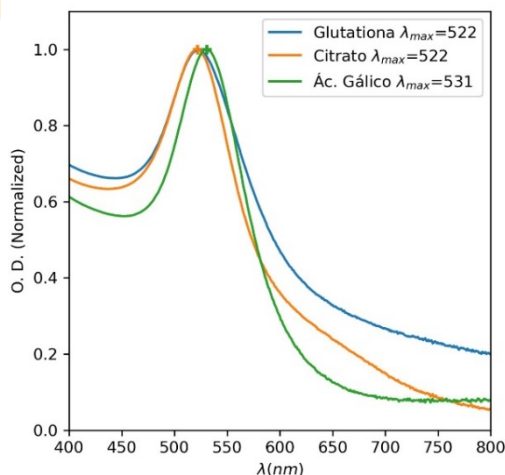


Figure 1: UV-Vis spectra of gold nanoparticles synthesized with different reductants. The smaller width at half height in gallic acid indicates more homogeneous particles.



Figure 2: Aqueous solution of gold ions before the start of the nanoparticle synthesis reactions, showing the initial state of the material before reduction.



Figure 3: Reaction after synthesis with sodium citrate, containing gold nanoparticles formed.



Figure 4: Reaction after synthesis with gallic acid.



Figure 5: Reaction after synthesis with GSH/NaBH₄.

Conclusions

The comparison between the reducing agents demonstrated that the choice of the reductant directly influences the final properties of the spherical gold nanoparticles. Although all methods enabled the formation of AuNPs, the use of gallic acid stood out for generating more homogeneous samples with a lower presence of amorphous particles. These results reinforce that the selection of the reductant should consider not only the synthesis efficiency, but also the desired final application, since different reductants affect characteristics such as reproducibility, consistency and size distribution of the nanoparticles.

Acknowledgments

CNPq, CAPES and FAPEMIG. PROPE/UFSJ, UFSJ Analytical Center/CCO, Analytical Laboratory and CTNano for support.

References

- [1] MUDASSAR, Saeed; et al. Nanomaterials: types, properties, recent advances, and applications. *Materials Today*, [S. l.], v. 47, p. 14–22, 2021.
- [2] MELÉNDEZ-VILLANUEVA, M. A. et al. Virucidal activity of gold nanoparticles synthesized by green chemistry using garlic extract. *Viruses, Basel*, v. 11, n. 12, p. 1111, 2019.
- [3] MELO JR., M.A et al. Preparação de nanopartículas de prata e ouro: um método simples para a introdução da nanociência em laboratório de ensino. *Química Nova*, v. 35, n. 9, p. 1872–1878, 2012.
- [4] FATHY, M. M. et al. An insight into synthesis and antitumor activity of citrate and gallate stabilizing gold nanospheres. *Scientific Reports*, [s.l.], v. 13, n. 2749, p. 1–11, 2023.