



Master 2

Année universitaire 2024-2025



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**Unité d'enseignement**  
**HAC934C**  
**Etude Thématique**  
**SANTE**

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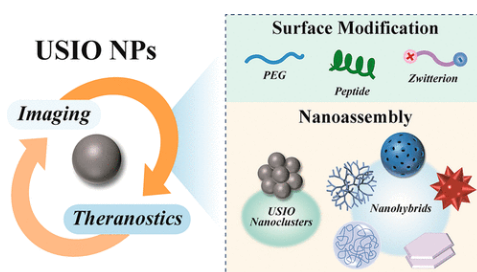
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# Synthesis of nanoparticles

The development of nanotheranostics based upon rational materials design has broadened up alternatives for personalized management of cancer and other serious diseases that plague human beings, along with improved diagnostic and therapeutic efficiency to overcome the current limitation in clinical treatments. Among the most widely investigated nano-systems in recent decades including liposomes, dendrimers, polymeric micelles, nanofibers, and inorganic metal nanoparticles (NPs) like Gold Nanorods or iron oxide (IO) NPs, especially the magnetite  $\text{Fe}_3\text{O}_4$  NPs, have attracted increasing attention because of their versatility in chemical modifications and excellent biosafety with well-studied pharmacokinetic and toxicity profiles.

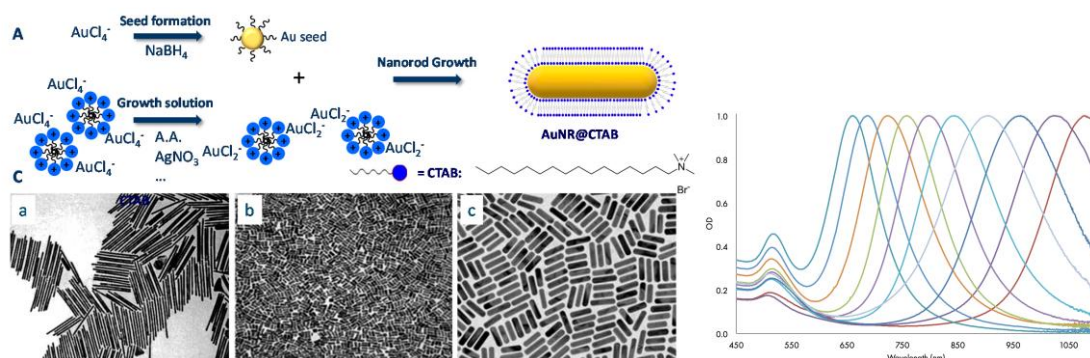
During the past decades, IO NPs have been widely applied in iron deficiency anemia treatment, magnetic separation, magnetic hyperthermia, magnetic resonance (MR) imaging, cell labeling and tracking, and drug delivery toward cancer theranostics. In addition, there are also some frontier research works exploring and revealing the potential of IONPs as reagents to mediate chemodynamic therapy of cancer through the Fe-triggered Fenton reaction. Along with rapid advances in the field of fundamental research, some of the IO NP-based products have made great achievements in clinical trials.



*ACS Appl. Mater. Interfaces* 2021, 13, 45119–45129

Although the biomedical story originated with spherical gold nanoparticles, anisotropic particles, and in particular gold nanorods (AuNRs), are increasingly dominating the field. Indeed, while spherical gold nanoparticles exhibit a unique LSPR band around 520 nm, the two dimensions of AuNRs lead to two absorption bands, the first, also located around 520 nm, is due to the transverse localized surface plasmon resonance (t-LSPR), and the second, far more advantageous, is due to the longitudinal localized surface plasmon resonance (l-LSPR). The l-LSPR band is located at higher wavelengths, matching the first (650–950 nm) or the second (1000–1350 nm) biological window. The position of the l-LSPR band can be tuned by

modulating the aspect ratio (AR) of AuNRs rather than their length. As a result, AuNRs have become the ideal candidates for a wide range of biomedical applications.



*Biosensors* **2020**, *10*(10), 146

This study aims to demonstrate the synthesis and functionalization of iron oxide nanoparticles with oleic acid and the synthesis of gold nanorods:

### **A1) Magnetic nanoparticle synthesis** (*J.Chem.Educ.*2018, *95*, 121–125)

The preparation of magnetite nanoparticles  $\text{Fe}_3\text{O}_4$  is done using a mix of  $1/3 \text{ Fe}^{\text{II}}$  and  $2/3 \text{ Fe}^{\text{III}}$  precursors: 1 mL of a  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$   $2.0 \text{ mol.L}^{-1}$  and 4 mL of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$   $1.0 \text{ mol.L}^{-1}$  both prepared in a  $2.0 \text{ mol.L}^{-1}$  HCl solution are added into a 100 mL Erlenmeyer. 50 mL of  $1.0 \text{ mol.L}^{-1}$   $\text{NH}_3$  solution are added in 4-5 min dropwise in the Erlenmeyer under fast magnetic stirring under a fume hood to prevent inhaling  $\text{NH}_3$  vapors. As a result of the addition of  $\text{NH}_3$ , the color shifts from yellow to brown and finally to black. The successful preparation of magnetite can be checked using a very strong magnet that allows the ferrite decantation. The Nanoparticles were then washed in water until the pH of the supernatants remain neutral. The nanoparticles were then stored in 5 mL of water. 1.5 mL of the suspension were dried for weight calculation of the suspension and further characterizations (IR)

### **A2) Magnetic nanoparticle functionalization with oleic acid** (*Materials Chemistry and Physics* 203 (2018) 212-222)

0.5 mL of the above suspension of the IONPs suspension were added to 1.5 mL of water. Oleic acid was added at the quantity of 50 wt % oleic acid to iron oxide weight and the mixture was sonicated for 5 minutes. The iron oxides were then washed one time with ethanol. The nanoparticles were then suspended in hexane and water is added to demonstrate the

hydrophobicity of the nanoparticles (comparison with another tube with the unmodified iron oxides in water where hexane is added). (photo)

**B) Gold Nanorods synthesis** (*J. Mater. Chem., 2011, 21, 16759-16782*)

Seed-mediated growth in the presence of the cationic surfactant CTAB has been continuously improved since its first demonstration and is now widely adopted for the preparation of GNRs.

**Seed Solution.** CTAB solution was mixed with  $\text{HAuCl}_4$ . To the stirred solution  $\text{NaBH}_4$  was added, which resulted in the formation of a brownish yellow solution. Vigorous stirring of the seed solution was continued for 2 min. After the solution was stirred, it was kept at 25 °C.

**Growth of NRs with Plasmon Bands Less Than 850 nm.** CTAB was added to  $\text{AgNO}_3$  solution at 25 °C. To this solution,  $\text{HAuCl}_4$  was added, and after gentle mixing of the solution ascorbic acid was added. Ascorbic acid as a mild reducing agent changes the growth solution from dark yellow to colorless.

The final step was the addition of the seed solution to the growth solution at 27–30 °C.

The GNRs suspension will be characterized by UV to observe the two absorption bands.

Solution Seed			
	[ ] mM	V (mL)	n (mmoles)
CTAB	100	10	1
$\text{HAuCl}_4$	10	0,5	$2,5 \cdot 10^{-3}$
$\text{NaBH}_4$	10	0,6	$6 \cdot 10^{-3}$
Solution Growth			
CTAB	100	10	1
$\text{HAuCl}_4$	10	0,5	$5 \cdot 10^{-3}$
$\text{AgNO}_3$	10	0,08	$8 \cdot 10^{-4}$
Acide asc.	100	0,055	$5,5 \cdot 10^{-3}$
		~10	
<b>Ajout Seed</b>	0,225	0,01	$2,25 \cdot 10^{-6}$

**Report** : in the form of a short article (Deadline 20/12/2024) :

- Introduction,
- Experimental Part,
- Result and Discussions,
- Conclusion,
- References

Join all figures (characterizations) in the articles; TEM images will be provided and a distribution on 50 nanoparticles will have to be done.