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## Current methods for synthesis of gold nanoparticles

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### Abstract

Metal nanoparticles, such as nanoparticles synthesized using gold, have numerous uncommon chemical and physical properties due to the effects of their quantum size and their large surface area, in comparison with other metal atoms or bulk metal. Gold nanoparticles (GNPs), in particular, are very attractive because of their size and shape-dependent properties. Metal nanoparticles have gathered extensive attention due to their uncommon properties and promising applications in photonics, electronics, biochemical sensing, and imaging. This review covers recent advances in the synthesis of GNPs.

**Keywords:** gold nanoparticles, metal nanoparticles, photonics, quantum size

### Introduction

Nanotechnology generally refers to a field of science and engineering dedicated to materials of sizes ranging from 1–100 nm (Mody et al. 2010, Salata 2004). The term ‘nano’ is extracted from the Greek word ‘dwarf’, which means ‘extremely small’. When used as a prefix, it means  $10^{-9}$  or 0.000000001. A nanometer (nm) is one billionth of a meter (Thakkar et al. 2010).

Nanoparticles are a unique group of materials with exceptional features and broad applications in various fields (Geethalakshmi and Sarada 2012, Matei et al. 2008).

In recent years, researchers have become increasingly interested in the synthesis of gold nanoparticles (AuNPs), because of their unique physical and chemical properties in wide areas like catalysis, biolabeling, nonlinear optical devices, and in the field of drug delivery (Pal et al. 2013, Daniel and Astruc 2004).

AuNPs have exceptional optical properties due to surface plasmon resonance (SPR) effects. It is an optical phenomenon occurring from the interaction between an electromagnetic wave and the conduction of electrons in a metal. (Hu et al. 2006) The SPR properties of AuNPs makes them quite useful in the fields of bioimaging and biomedical therapeutics, and as biodiagnostic tools (Verma et al. 2014, Jain et al. 2006).

AuNPs are being widely used in a variety of biomedical applications because of their compatibility of synthesis and functionalization, less toxicity, and facility of detection (Tiwari et al. 2011).

AuNPs can accumulate in the tumor cells and show optical scattering; thus, these nanoparticles can play an important role as a probe for the microscopy study of cancer cells. In addition, these nanoparticles can be used in chemotherapy and for the diagnosis of cancer (Tomar and Garg 2013, Cai and Chen 2007). The AuNPs also provide an applicable and promising scaffold for drug and gene delivery (Ghosh et al. 2008, Paciotti et al. 2004, 2006).

This method comprises the assembly of atoms (produced by the reduction of ions) into desired nanostructures. Top-down techniques, such as photolithography and electron beam lithography (Shah et al. 2014, Sun et al. 2006, Schaal et al. 2012), involve the removal of matter from the bulk material to get the desired nanostructure.

### Synthesis of gold nanoparticles

A variety of techniques, including chemical, thermal, electrochemical and sonochemical pathways, have so far been introduced for the synthesis of GNPs (Mandal 2014, Porta and Rossi 2003, Yu et al. 1997, Nakanishi et al. 2005).

### Chemical method

Generally, the preparation of AuNPs by the chemical reduction method includes two main parts: (1) reduction by agents, for instance borohydrides, aminoboranes, formaldehyde, hydrazine, hydroxylamine, polyols, citric and oxalic acids, sugars, hydrogen peroxide, carbon monoxide, sulfites, hydrogen, acetylene, and onto electronic reducing agents including electron-rich transition-metal sandwich complexes; (2) stabilization using agents, for instance trisodium citrate dihydrate, sulfur ligands (in particular thiolates), phosphorus ligands, oxygen-based ligands, nitrogen-based ligands (including heterocyclic compounds), dendrimers, polymers and surfactants (in particular, cetyltrimethylammonium bromide (CTAB)). To

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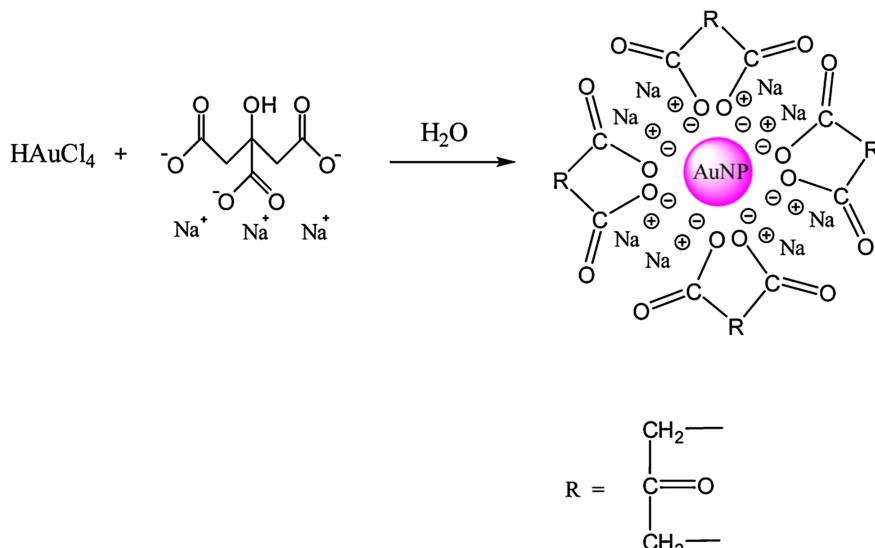


Figure 1. AuNP synthesis using the Turkevich method. Zhao, Pengxiang, Na Li, and Didier Astruc. "State of the art in gold nanoparticle synthesis." *Coordination Chemistry Reviews* 257, no. 3 (2013): 638–665.

avoid the aggregation of the particles, some kind of stabilizing agent is usually added (Zhao et al. 2013).

### Turkevich method

One of the most well-known techniques for the synthesis of AuNPs is based on the reduction of  $\text{HAuCl}_4$  by citrate in water, which was first designed by Turkevich in 1951. In this method, the  $\text{HAuCl}_4$  solution is boiled, and the trisodium citrate dihydrate is then rapidly added into the boiling solution under vigorous stirring. After a few minutes, the color of the solution changes from light yellow to wine red. This method results in AuNPs measuring about 20 nm in diameter. In this technique, citrate ions play a double role, as both stabilizing and reducing agents (Hu et al. 2006, Turkevich et al. 1951).

The schematic route for synthesis of AuNPs by the Turkevich method is shown in Figure 1.

The Turkevich method was modified by Frens, in 1973, to obtain AuNPs with diameters ranging from 15 to 150 nm, by controlling the ratio of reducing agent/stabilizing agent (trisodium citrate/gold). The Turkevich-Frens method has been further modified by several research groups (Frens 1973).

Kimling et al. demonstrated that a high citrate concentration more quickly stabilizes AuNPs of smaller sizes, while a small concentration of citrate will lead to the aggregation of the small particles into larger particles (Zhao et al. 2013, Kimling et al. 2006).

Recently, the important role of sodium citrate on the pH of the solution, and its role in controlling the size of the nanoparticle, were indicated based on a theoretical model and experimental results (Li et al. 2011, Ji et al. 2007, Yang et al. 2007, Kumar et al. 2007).

Puntes et al. have reported that the addition of reagents in the inverse sequence (addition of  $\text{HAuCl}_4$  into a boiling sodium citrate solution) leads to the production of AuNPs with small size and a narrow size distribution (Figure 2) (Ojea-Jiménez et al. 2011).

A considerable amount of literature has been published on the effect of temperature (Link and El-Sayed 1999), pH (Patungwasa and Hadak 2008), citrate concentration (Volkert et al. 2011), and gold chloride concentrations (Zabetakis et al. 2012), on the characteristic properties of AuNPs synthesized using citrate as reductant.

### The Brust-Schiffirn method

The Brust-Schiffirn method was discovered by Brust and Schiffirn in 1994. This method allowed an easy approach to the synthesis of thermally stable and air-stable AuNPs of controlled size and low dispersity. In this technique,  $\text{AuCl}_4^-$  was transferred to a toluene phase from an aqueous solution using tetraoctylammonium bromide (TOAB) as the phase-transfer agent, and reduced by  $\text{NaBH}_4$ , in the presence of dodecanethiol. Addition of the reducing agent causes a color change of the organic phase, from orange to deep brown. This clearly indicates the formation of AuNPs (Brust et al. 1994).

### Electrochemical method

The electrochemical production of nanoparticles was first studied by Reetz et al., in 1994 (Reetz and Helbig 1994, Reetz et al. 1995). Their studies showed that size-selective nano scale of transition metal particles could be

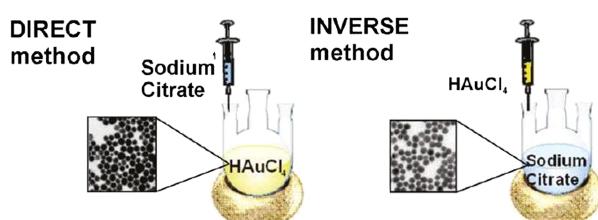


Figure 2. Direct and inverse sequence of reagent addition. Ojea-Jiménez, Isaac, Neus G. Bastús, and Victor Puntes: "Influence of the sequence of the reagent addition in the citrate-mediated synthesis of gold nanoparticles." *The Journal of Physical Chemistry C* 115, no. 32 (2011): 15752–15757.

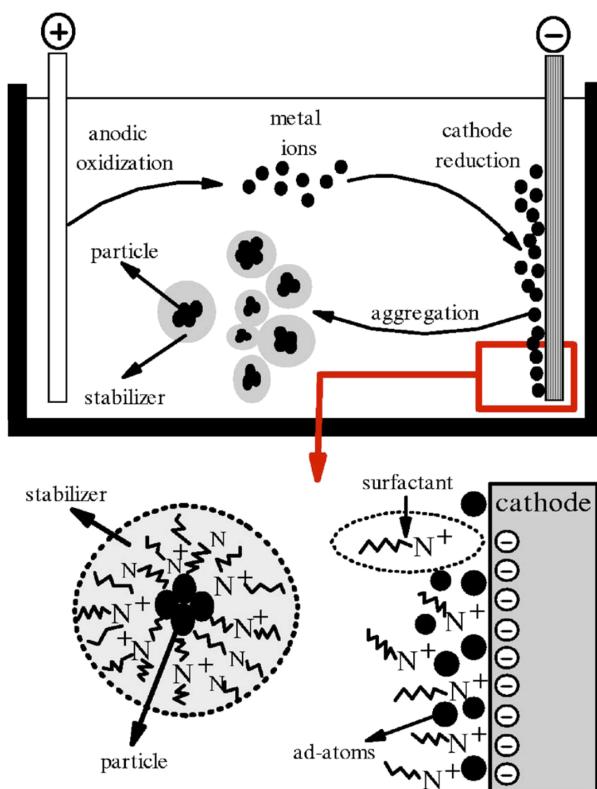


Figure 3. Scheme of the electrochemical system for synthesizing gold nanoparticles. Huang, Chien-Jung, Pin-Hsiang Chiu, Yeong-Her Wang, Kan-Lin Chen, Jing-Jenn Linn, and Cheng-Fu Yang. "Electrochemically controlling the size of gold nanoparticles." *Journal of The Electrochemical Society* 153, no. 12 (2006): D193-D198.

set electrochemically, using tetra alkyl ammonium salts as stabilizers of metal clusters in a nonaqueous medium.

Gold nanoparticles on the surface of multi-walled carbon nanotubes with glassy carbon electrodes can be prepared using the electrochemical synthesis technique (Song et al. 2013).

The gold nanoparticles were prepared electrochemically using a simple two-electrode cell, with oxidation of the anode and reduction of the cathode. Figure 3 schematically depicts the electrochemical apparatus (Huang et al. 2006).

The electrochemical process has been verified to be superior to other methods of nanoparticle production, due to its modest equipment, low cost, lower processing temperature, high quality, and ease of controlling the yield (Freeman et al. 1995, Chen and Yang 2002, Haruta and Daté 2001, Kuge et al. 2000, Kamat et al. 1998).

### Seeding growth method

Another method that has also been reported for the synthesis of gold nanoparticles is the seeding growth method. According to the seeding growth process, gold nanoparticles of diameters 5–40 nm and a narrow size distribution were synthesized. Particle size can be controlled by the changeable ratio of seed to metal salt, and therefore every size in the range 5–40 nm can be prepared (Jana et al. 2001). This method has the advantage of being a simple, quick, and low cost process; while trisodium citrate was used as a source of  $\text{OH}^-$  ions in the seeding step, sodium borohydride ( $\text{NaBH}_4$ ) was used as a reducing agent (Siti et al. 2013) (Figure 4).

### Biological method

Although chemical methods are the most common approach for the synthesis of metallic nanoparticles, the use of expensive and toxic reagents as reducing and stabilizing agents limits their applications. In addition, these nanoparticles may have harmful effects in biomedical applications (Noruzi et al. 2011, Shankar et al. 2004). Hence, there is a growing need to develop eco-friendly and cost-effective procedures for the synthesis of nanoparticles that do not use any toxic chemicals. Biological synthesis of nanoparticles has been at the center of attention as a green and eco-friendly method in current years. In biological methods, nanoparticles are synthesized by microorganisms, enzymes, and plants or plant extracts (Mohanty et al. 2008, Singh et al. 2013).

Recently, the use of plants for the synthesis of nanoparticles is gaining importance, because of their availability, low cost, eco-friendliness and non-toxic nature. In recent years, the biosynthesis of AuNPs using plants such as *Azadirachta indica* (Shankar et al. 2004), *Medicago sativa* (Gardea-Torresdey et al. 2002), *Aloe vera* (Chandran et al. 2006), *Cinnamomum camphora* (Huang et al. 2007), *Pelargonium graveolens* (Shankar et al. 2004), *Coriandrum sativum* (Narayanan and Sakthivel 2008), *Terminalia catappa* (Ankamwar 2010), and lemongrass (Shankar et al. 2004), have been reported (Smithaa et al. 2009, Parida et al. 2011).

Many papers have been published, reporting the synthesis of AuNPs using plant extracts (Vadlapudi and Kaladhar 2014), such as *Memecylon umbellatum* (Arunachalam et al. 2013), *Macrotyloma uniflorum* (Aromal et al. 2012), *Brevibacterium casei* (Kalishwaralal et al. 2010, Mittal et al. 2013), *Citrus limon*, *Citrus reticulata* and *Citrus sinensis* (Sujitha and Kannan 2013), *Piper pedicellatum* (Tamuly et al. 2013), *Terminalia chebula* (Kumar et al. 2012), *Memecylon edule* (Elavazhagan and Arunachalam 2011), *Nyctanthes*

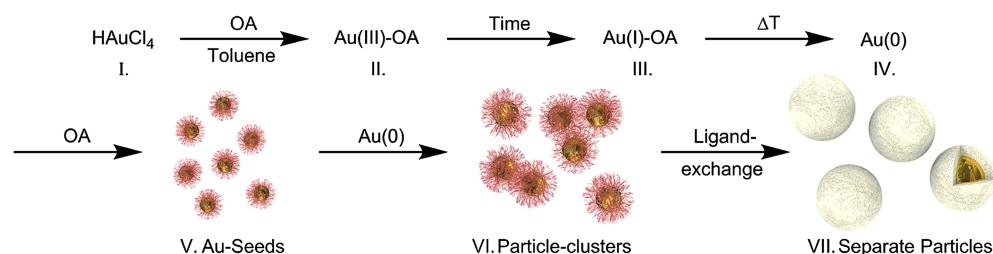


Figure 4. Growth mechanism of gold nanoparticles. Stanglmair, Christoph, Sebastian P. Scheeler, and Claudia Pacholski. "Seeding Growth Approach to Gold Nanoparticles with Diameters Ranging from 10 to 80 Nanometers in Organic Solvent." *European Journal of Inorganic Chemistry* 2014, no. 23 (2014): 3633–3637.

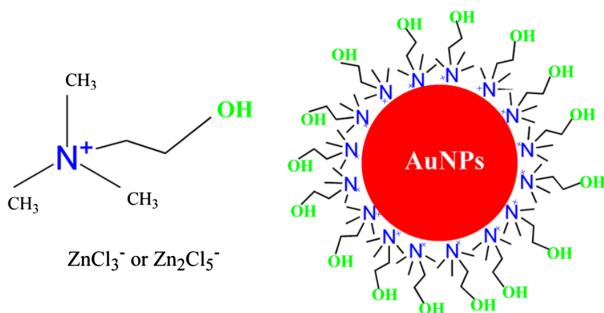


Figure 5. Molecular structures of QAILs and stabilized gold nanoparticles (Huang et al. 2011).

*arbor-tristis* (Das et al. 2011), *Murraya koenigii* (Philip et al. 2011), *Mangifera indica* (Philip 2010), Banana peel (Bankar et al. 2010), *Cinnamomum zeylanicum* (Smitha et al. 2009), and *Cochlospermum gossypium* (Vinod et al. 2011).

Kumar et al. synthesized AuNPs, with a particle size in the range of 5–15 nm, using the extract of *Zingiber officinale*, which plays a double role as a reducing and stabilizing agent (Kumar et al. 2011).

Umesh Kumar et al. reported the green synthesis of gold nanoparticles using onion (*Allium cepa*) extract as the reducing agent. The reduction of AuNPs occurs due to the activity of vitamin C in onion extract (Parida et al. 2011).

### Ionic liquids and gold nanoparticles

In recent years, ionic liquids (ILs) have been discovered to be remarkable and unique media for the synthesis and stabilization of metal nanoparticles (Richter et al. 2013, Dupont et al. 2002, Mudring et al. 2009, Jacob et al. 2006).

ILs are salts with low melting points, which are becoming progressively more important as solvents because of their unique properties such as nonvolatility, thermal stability, and designable miscibility with cosolvents (Kumar et al. 2007, Welton 1999, Gao et al. 2008).

ILs can act not only as the solvents, but can also serve as capping agents (Mu et al. 2005), templates (Fechler et al. 2013), and even precursors of the materials (Paraknowitsch et al. 2010, Zhang et al. 2014).

ILs have been broadly used in a variety of chemical reactions (Cassol et al. 2005, Leitner 2003), separations (Arce et al. 2007), and electrochemical applications (Gao et al. 2008, Enders Dickinson et al. 1999).

### Synthesis of gold nanoparticles in ionic liquids

Kim et al. described a one-phase synthesis of AuNPs using thiol-functionalized ILs. Thiol-functionalized ILs acted as the stabilizing agents for the synthesis of gold nanoparticles. They reported that the size and stability of the nanoparticles

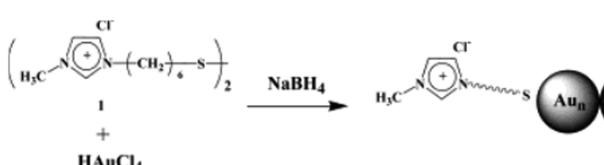


Figure 6. Synthesis of AuNPs modified with ionic liquid based on the imidazolium cation (Itoh et al. 2004).

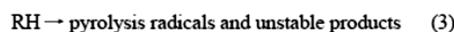
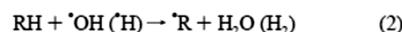


Figure 7. Sonochemical reduction of Au(III) in the presence of an organic additive (Okitsu et al. 2005). Okitsu, Kenji, Muthupandian Ashokkumar, and Franz Grieser. "Sonochemical synthesis of gold nanoparticles: effects of ultrasound frequency." *The Journal of Physical Chemistry B* 109, no. 44 (2005): 20673–20675.

were affected by the number and position of thiol groups in the IL (Kim et al. 2004).

In 2006, Kim et al. reported an easy one-pot method of synthesis of AuNPs using *N*-(2-hydroxyethyl)-*N*-methylmorpholinium tetrafluoroborate. The alcohol ionic liquids function both as the reducing agent and as a stabilizer, thus considerably simplifying the procedure of nanoparticle preparation (Kim et al. 2006).

Stable gold nanoparticles were synthesized in quaternary ammonium ionic liquids (QAILs) by simple heating. The QAILs can not only be used in the role of the reducing agent because of the reactivity of their hydroxyl groups, but can also function as the stabilizing agent due to their chelating ability. The molecular structures of QAILs and stabilized gold nanoparticles are shown in Figure 5 (Huang et al. 2011).

### Synthesis of gold nanoparticles modified with ionic liquid

Itoh et al. have demonstrated the synthesis of GNPs modified with IL, based on the imidazolium cation (Figure 6). They found the application of the aggregation-induced color changes of the gold nanoparticles in aqueous solutions as an optical sensor for anions, via anion exchange of the ionic liquid moiety (Itoh et al. 2004).

The sonochemical method is a unique technique for the synthesis of metal nanoparticles. The benefits of using this method for the synthesis of metal nanoparticles include the capability to form very small metal nanoparticles, and a rapid reaction rate (Park et al. 2006).

However, metal nanoparticles synthesized by sonochemical reduction usually have wide size distributions. In order to eliminate these difficulties, surfactants and alcohols are generally used in the sonochemical method, to control the particle size and shape (Cao et al. 1995, Fujimoto et al. 2001, Okitsu et al. 1996, Yeung et al. 1993, Nagata et al. 1996).

The sonochemical reduction of Au(III), in the presence of an organic additive, occurs in the following steps (Okitsu et al. 2005, Yeung et al. 1993, Nagata et al. 1996, Okitsu et al. 2002, Caruso et al. 2002).

Okitsu, Kenji et al. proposed a method to synthesize gold nanoparticles deposited on chitosan powder using the sonochemical method. The average size of the prepared AuNPs was measured to be 22 nm (Okitsu et al. 2007).

Highly stable AuNPs, with small particle size and uniform distribution, were successfully synthesized using the sonochemical method in thiol-functionalized ILs (Ebrahimnezhad et al. 2013) (Figure 7).

## Conclusion

As we have seen, the synthesis of AuNPs is an important area of research in nanotechnology. Generally there are two approaches for the synthesis of AuNPs: the “bottom-up” approach and the “top-down” approach (Eustis and El-Sayed 2006). The bottom-up approach consists of nanosphere lithography, templating, chemical, photochemical, electrochemical, sonochemical, and thermal reduction techniques (Haes et al. 2004, Pileni 1997, Okitsu et al. 2005, Hall et al. 2001, Magnusson et al. 1999).

## Authors' contributions

AA conceived of the study and participated in its design and coordination. RH, and EA participated in the sequence alignment and drafted the manuscript. All authors read and approved the final manuscript.

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## Declaration of interest

The authors have no declaration of interest. The authors alone are responsible for the content and writing of the paper.

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