Floating synthesis with enhanced catalytic performance via acoustic levitation processing

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**ABSTRACT**

Acoustic levitation supplies a containerless state to eliminate natural convection and heterogeneous crystal nucleation and thus provides a highly uniform and ultra clean condition in the confined levitating area. Herein, we attempt to make full use of these advantages to fabricate well dispersed metal nanoparticles. The gold nanoparticles, synthesized in an acoustically levitated droplet, exhibited a smaller size and improved catalytic performance in 4-nitrophenol reduction were synthesized in an acoustically levitated droplet. The sound field was simulated to understand the impact of acoustic levitation on gold nanoparticle growth with the aid of crystal growth theory. Chemical reducing reactions in the acoustic levitated space trend to occur in a better dispersed state because the sound field supplies continuous vibration energy. The bubble movement and the cavitation effect accelerate the nucleation, decrease the size, and the internal flow inside levitated droplet probably inhibit the particle fusion in the growth stage. These factors lead to a reduction in particle size compared with the normal wet chemical synthetic condition. The resultant higher surface area and more numerous active catalytic sites contribute to the improvement of the catalytic performance.

**1. Introduction**

Gold nanoparticles exhibit fascinating application potentials in the fields of catalysis [1–5], biology [6,7], and medical testing [8,9]. Various methods have been employed to synthesize gold nanoparticles, including liquid phase synthesis, chemical vapor deposition, solid phase method, etc. Among these, the liquid phase reduction has proven to be a convenient and practicable method to obtain nano-sized particles [10–12]. In order to improve the catalytic behaviors, gold nanoparticles (AuNPs) with smaller particle size and better dispersibility are required in order to obtain higher surface area and more numerous active catalytic sites [13–16]. The particle size of AuNPs synthesized by liquid phase reduction is related to the species and quantity of the reducing agent and chemical ligand [17,18]. However, the size limitation of AuNPs with normal method is about 2 nm. In two phase method, the size of AuNPs can reach 2 nm, but it requires a complicated protocol and phase transfer reagent, or immediately thiols functionalization of AuNPs which leads to the loss of the opportunity of the post-functionality [19,20]. The contact between the container and chemical agent in normal wet chemistry method causes heterogeneous nucleation, and the containerless processing technology can eliminate this influence. Acoustic levitation technology supplies a containerless state to avoid the heterogeneous nucleation caused by container and change liquid flow by sound field effect. The levitated sample can interact with sound field in a well dispersed and ultra clean condition. It’s an emerging extraordinary physical environment producing numerous novel phenomenon in the fields of material solidification [21,22], material preparation [23,24] and analytical chemistry [25,26]. Therefore, it is expected to verify the improved catalytic performance of gold nanoparticles synthesized in acoustic levitation.

Here we synthesized the gold nanoparticles in acoustic levitation condition (Fig. 1), and the size of AuNPs based on the TEM images (see Fig. 3b) is down to ~2 nm. The gold acid chloride trihydrate (HAuCl\(_4\)·3H\(_2\)O) is reduced by sodium borohydride (NaBH\(_4\)) in a Polyvinylpyrrolidone (PVP) solution, which is the same as the normal protocol to produce gold nanoparticles. The gold nanoparticles prepared in

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2. Experimental section

2.1. Gold nanoparticles synthesis

In the case of acoustic levitation, 2.5 mL HAuCl₄·3H₂O solution (5 mM) was added to the 2 mL PVP solution (62.5 mM), and the beaker was vigorously stirred to make it mixed uniformly. The ultrasonic frequency was 21.3 kHz and the ultrasonic power was 350 W. A pipette was used to transfer 45 µL of the PVP and HAuCl₄·3H₂O mixture into the levitation position, and the tuning handle was turned to change the interval between the reflector and emitter achieving the levitation (Fig. 1). 5 µL NaBH₄ solution (75 mM) was added to the stable levitated mixture with a pipette (avoiding artificial introduction of bubbles), and keep stable acoustic levitation for 10 min. Finally a pipette and a beaker was used to remove the sample and store it. During the synthesis, the shape of acoustically levitated droplet was like a disk and its volume was 50 µL (Fig. 2). The phenomenon of droplet evaporation existed in acoustic levitation, and the synthesis process was finished in the 10-min levitation to avoid the evaporation of water. Sometimes, the acoustic radiation force was enhanced to restore the tiny vertical vibration of droplet during the synthesis.

In the case of normal condition, 2.5 mL HAuCl₄·3H₂O solution (5 mM) was added to 2 mL PVP solution (62.5 mM), and the beaker was vigorously stirred to make it mixed uniformly. 0.5 mL NaBH₄ solution (75 mM) was added to the mixture. A beaker was used to store the sample.

2.2. Physical characteristics and catalytic performance of gold nanoparticles analysis and test

The UV-8000S spectrophotometer of Shanghai Metash Instruments Co. Ltd. was used to analyze and monitor the size evolution of gold nanoparticles, and the surface plasmon resonance peak of its wavelength near 520 nm was normalized as 1 for each spectra. The FEI Talos F200X TEM was used to characterize the morphology and particle size of the gold nanoparticles. The measurement statistics software was used to perform statistical analysis on TEM images. We statistically analyzed the frequency distribution in different particle size ranges with 1 nm length as the interval. The particle size peak was normalized as 1 for each sections, and the particle size distribution and average particle size were obtained (Fig. S3). A single peak fitting with Gaussian function on the frequency distribution histogram was performed and the fitting curve was obtained (Fig. 3d).

The catalytic reaction is performed as follow. 1 mL 4-nitrophenol (4-NP) solution (0.2 mM) is diluted in 1.4 mL H₂O mixture into the levitation (Fig. S5). Then 100 µL NaBH₄ solution (100 mM) was added into the 4-NP solution. After that, 40 µL gold nanoparticles was added to initiate the reaction. The time when the gold nanoparticles added were considered as the zero point in catalytic reaction kinetic characterization, and the UV–vis spectra was continuously collected for measurement within 30 min.

2.3. Simulation of the sound field during the synthesis of gold nanoparticles under acoustic levitation

The Pressure Acoustics, Frequency Domain module in COMSOL software was used for simulation. A downward vibration of specific amplitude to the emitter was applied to produce the ultrasound. The vibration amplitude of the emitter is 6 µm, which was measured by an OptoMET Nova laser Doppler vibrometer and the geometric parameters were set according to the experimental situation and shown in the Table 2. The shape of the droplet was approximated by a flat cylinder. In order to provide a closed solid area to set material properties in the COMSOL software, a closed cylindrical calculation solution area (excluding emitter and reflector) was artificially established and their boundary was set as radiation boundary condition (Fig. S5). The dilute solution material droplet and the closed solution area material except acoustic levitation condition have the characteristics of smaller particle size and better catalytic performance compared with the wet chemistry method in normal condition. The theoretical model is further proposed to understand the acoustic assisted size decreasing phenomenon during nanoparticle synthesis, combining sound field analysis and crystal seed growth model. The acoustic levitation synthesis of gold nanoparticles provides a promising potential to use the sound field effect to influence the nucleation growth process of metal nanoparticles, and guides us to achieve fascinating properties for metal nanoparticles, such as smaller size and better catalytic performance for metal nanoparticles, obtained in acoustic levitation.
for droplet were approximately regarded as water and air, respectively. The hard sound field boundary was used for the emitter reflector end and the calculation solution area boundary, and the impedance boundary condition was used for the droplet boundary. Finally, regular element size free tetrahedral elements were used for meshing, the number of degrees of solving freedom was 105626.

3. Results and discussion

The synthetic route of gold nanoparticles in acoustic levitation and normal condition is based on wet chemistry method. PVP is used as stabilizer, and NaBH₄ is selected to reduce HAuCl₄⋅3H₂O in normal and acoustic levitation conditions to obtain the gold nanoparticles. The ultrasonic waves form a standing wave field between the emitter and reflector to balance the weight of the sample to achieve levitation. The general procedure for the synthesis of gold nanoparticles in acoustic levitation is to realize the stable levitation of the mixture of PVP and HAuCl₄⋅3H₂O solution firstly (Fig. 2a), and then NaBH₄ is rapidly pipetted into the levitated droplet to obtain gold nanoparticles by reducing the gold salts (Fig. 2b). A high speed Charge Coupled Device (CCD) was used to photograph the bottom of the levitated droplet (The setting and optical path of CCD to take the photos is shown in Fig. 1), and the magnified figure is shown in Fig. 2. (1–2 light spots in the figure are formed by polishing not part of sample itself). More bottom-view CCD photographs of the levitated droplet during the synthesis of gold nanoparticles is shown in Fig. S1.

The UV-Vis spectra of gold nanoparticles synthesized in two conditions exhibit a significant difference of SPR (surface plasmon resonance) position, which is located at 511.0 nm and 537.5 nm, respectively (Fig. 3c). The SPR wavelength of the gold nanoparticles synthesized in acoustic levitation shows a blue shift compared with the typical value of 520 nm. It is indicated that the particle size of the gold nanoparticles synthesized in acoustic levitation condition is smaller [27,28]. In order to obtain more intuitive evidence, high resolution TEM images were used to obtain the accurate size distribution by statistical analysis. The TEM images are shown in Fig. 3a and b and Fig. S2, and the particle size distribution of the gold nanoparticles obtained through the two approaches are plotted in Fig. 3d. (More details in Fig. S3) The morphologies of the gold nanoparticles synthesized in both conditions show a similar spherical shape. In Fig. 3d, according to the Gaussian fitting curve, the particle size distribution range is narrower and more concentrated, the average particle size and its standard deviation were 2.6 ± 0.8 nm in acoustic levitation. However, the size of normal condition synthesized gold nanoparticle is 4.9 ± 1.3 nm. The average particle size of the synthesized gold nanoparticles in acoustic levitation condition is almost half of that in normal condition.

The molar ratio of NaBH₄ to HAuCl₄⋅3H₂O is a crucial factor influencing the gold nanoparticle size. Here, the UV-Vis spectra are first collected at different ratios for two conditions (in Fig. 4a and c) to trace the rough size evaluation at specific condition. The Gaussian fitting curve of particle size distribution (Fig. 4b and d) is obtained based on the statistical analysis of TEM images. (More details in Table 1 and Fig. S4) Increasing the ratio of NaBH₄ to HAuCl₄⋅3H₂O from 0.5 to 10, the surface plasmon resonance peak wavelength of the AuNPs synthesized in both conditions has a continuous blue shift, indicating the smaller AuNPs with higer ratios of the reductant, which is consistent with the statistical result of TEM. When the ratio of NaBH₄ to HAuCl₄⋅3H₂O is 10, the nucleation completes instantaneously, so that reducing agent plays a major role in regulating the particle size evolution during synthesis of gold nanoparticles. Therefore, the reducing agent influence on particle size in the two conditions is approximately equal.

The universally acknowledged method of decreasing the particle size of AuNPs is increasing the quantity of reducing agent or using stronger reducing agent. Various chemical ligands also result in the different particle size [17]. Furthermore, the particle size of AuNPs obtained directly in solid polymer films by thermal decomposition of the [O(Au

![Fig. 3. Experimental results of two synthetic routes of AuNPs. (a) and (b) TEM of AuNPs synthesized in two conditions. (c) UV-Vis spectra. (d) Particle size distribution.](image-url)
(PPh$_3$)$_3$][BF$_4$] precursor molecularly dispersed in the polymer is similar to those prepared in diluted solutions [29]. The particle size of AuNPs synthesized by sonochemical reduction of aqueous solution of HAuCl$_4$ in 1-propanol at 21.3 kHz ultrasound is 15.5 nm [30]. The gold nanoparticles synthesized in acoustic levitation is 53% of normal condition when all other conditions are kept constant. It should be noted that the ultrasound frequency of the sonochemical and acoustic levitation is incomparable because of different ultrasound transmission medium (directly in liquid or from gas into liquid). Most of the sound energy is unable to enter the acoustically levitated droplet due to the impedance of gas-liquid interface, and the gentle sound energy inside the droplet does not cause excessive damage to the material. The particle size is related to the unique sound field characteristics of acoustic levitation.

The catalytic performance of gold nanoparticles is depended on its morphology and particle size [13,14], and the better catalytic performance of gold nanoparticles synthesized in acoustic levitation is incomparable because of different ultrasound transmission medium (directly in liquid or from gas into liquid). Most of the sound energy is unable to enter the acoustically levitated droplet due to the impedance of gas-liquid interface, and the gentle sound energy inside the droplet does not cause excessive damage to the material. The particle size is related to the unique sound field characteristics of acoustic levitation.

The catalytic reaction is carried out gently in aqueous solution, it is easy to monitor the reaction kinetics by UV-Vis spectra. Therefore the reaction has been widely used to evaluate the catalytic performance of metal nanoparticles. The characteristic absorption peaks of reactants and products were detected continuously over a period of time, and the extent of catalytic reaction can be revealed through their concentration changes. The UV-Vis spectra of the chemical catalytic reaction of gold nanoparticles synthesized in two conditions respectively are shown as Fig. 5a and b. The characteristic absorption peak located at 400 nm

![UV-Vis spectra and particle size distribution of AuNPs synthesized with different molar ratios of NaBH$_4$ to HAuCl$_4$·3H$_2$O. (a) and (b) Normal condition. (c) and (d) Acoustic levitation condition.](image-url)
represents the 4-NP and the 300 nm indicates the freshly generated 4-AP, while the values of the peak can be converted into their respect concentration according to the Beer–Lambert law. The peak intensities at 400 nm and 300 nm keep decreasing and increasing, respectively, with the time going. The change of the characteristic absorption peak value reflects the variation of the reactant concentration, and the kinetic of the gold nanoparticles catalyzed reaction can be further characterized based on the reactant concentration variation. Take the characteristic absorption peak value of 400 nm at 0 s as \( A_0 \), and the characteristic absorption peak value at \( t \) s as \( A \). A kinetic plot is shown in Fig. 5c, using \(-\ln(A/A_0)\) as \( Y \) and \( t \) as \( X \). In kinetic plot, the slope of the two straight lines in the figure represents the reaction rate constant \( k \), which is \( 2.86 \times 10^{-3} \) s\(^{-1} \) in normal condition and \( 3.15 \times 10^{-3} \) s\(^{-1} \) in acoustic levitation condition. The equilibrium time for the catalytic reaction of normal condition is around 1027 s, and that of the acoustic levitation is around 873 s, which exhibits 85.0% reaction time in normal condition. The catalytic performance of gold nanoparticles synthesized in acoustic levitation has been significantly improved. The catalytic reaction takes place on the catalyst’s surface after adsorption of reactants onto the active area of catalyst’s surface [31]. Therefore, the catalytic efficiency enhanced with decreasing size of nanoparticles due to growth of total surface area in the reaction system.

The sound field during the synthesis of gold nanoparticles in acoustic levitation has been simulated using COMSOL software based on the finite element method. The sound field between the emitter and the reflector is numerically simulated according to Helmholtz equation as Eq. (1).

\[
\nabla \cdot \left( -\frac{1}{\rho_0} \nabla p \right) - \frac{k^2}{\rho_0} p = 0
\]

in which \( p \) is the sound pressure, \( \rho_0 \) is the medium density of air, and \( k = \omega/c_0 \) is the wave number (\( \omega \) is the angular frequency and \( c_0 \) is the sound velocity in air). The nonlinear effects of ultrasound waves will impose an acoustic radiation pressure and force [32,33] on the object in the sound field, which will levitate the substances in gaseous medium. The acoustic radiation force \( F_r \) is derived from the integral of the acoustic radiation pressure \( p_r \) acting on a surface, which is described as Eqs. (2) and (3).

\[
\rho_r = \frac{\langle p^2 \rangle}{2\rho_0 c_0^2} \rho_0 \langle u \cdot u \rangle
\]

\[
F_r = \int p_r n dS
\]

Where \( u \) is the medium particle velocity, and the angle brackets mean the time average over a period of acoustic oscillation. A downward vibration of specific sound amplitude \( A \) to the emitter was applied to produce the ultrasound, and the normal acceleration is described as Eq. (4).

\[
a_n = A(2\pi f)^2
\]

The interface of the droplet and air is set as impedance boundary condition, and it’s shown as Eq. (5).

\[
n \left( -\frac{1}{\rho_0} \nabla p \right) = \frac{i\omega}{Z_1} p
\]

which \( Z_1 = \rho_1 c_1 \) is the impedance of the droplet, and \( \rho_1 \) is the medium density of water and \( c_1 \) is the sound velocity in water. The related physical and geometric parameters are shown in the Table 2. Details of the simulation have shown in the experimental section.

We have calculated the acoustic radiation force \( F \) acting on the concave reflector as a function of interval between emitter and reflector \( H \) (inset in Fig. 6a). The resonant intervals are slightly longer than an integer multiple of the half-wavelength in the air, and we chose the third resonance mode \( (H = 27 \text{ mm}) \) to stimulate experimental condition. In
this case the acoustic radiation force $F$ acting on the bottom and top of the droplet is $5.11 \times 10^{-4}$ N and $5.76 \times 10^{-6}$ N, respectively. The difference between them is approximately equal to the droplet’s weight $4.90 \times 10^{-4}$ N. This is consistent with the experimental results of force equilibrium, and verified the validity of our model.

The calculated sound pressure field distribution is displayed in Fig. 6b. To show the details of the sound field in the droplet in acoustic levitation, the magnified droplet’s figure and its internal sound pressure field is displayed (Fig. 6c). The calculated sound pressure level applied on the droplet interface is shown in Fig. S6. The balance of the several forces acting on the levitated droplet determines the shape of the droplet. The radiation pressure acting at the poles and the suction pressure acting at the equator cause the droplet to flatten. On the other hand, surface tension and internal pressure oppose the flattening [34]. The droplet flattens as the sound pressure level increases applied on the droplet interface. According to the acoustic radiation pressure of the top and bottom surface of the droplet (Fig. 6d), the center of the droplet is positive and the edge is negative, which makes the center surface of the droplet concave inward. This strengthens the effect of sound field on the surface of droplet and promotes the nucleation of the droplet surface attributed to the vibration energy.

The bubbles produced by the reduction reaction and the internal flow inside the levitated droplet are also worthy of attention. Adding NaBH$_4$ to the stable levitated mixture of PVP and HAuCl$_4$·3H$_2$O solution, the reduction reaction produces hydrogen bubbles inside the droplet (Fig. S1). The bubble movement and the cavitation effect provide more nucleation sites and promote the cracking of water under the action of acoustic cavitation. The collapse of cavitation bubbles generates extreme temperatures within cavitation bubbles that ultimately result in the formation of highly reactive radicals [35]. The generation of hydrogen and hydroxyl radicals would facilitate the gold precursor reduction and decrease the size of gold nanoparticles [36–38]. Immediately after droplet levitation, the droplet external flow field direction was toward the droplet, with a circulating vortex forming near the droplet surface. As evaporation progressed, the external flow transitioned toward the opposite direction, while the circulation vortex expanded [39]. The acoustic streaming around the droplet surface and the gradient of sound pressure inside the droplet cause the internal flow of the levitated droplet. The internal flow of acoustically levitated water droplet was investigated experimentally, and a kind of vortex flow which rotates in the meridional plane of the levitated droplet was revealed. The magnitude of fluid velocity is nearly vanishing at the droplet center, whereas it increases toward the free surface of a levitated droplet until the maximum value of about 80 mm/s [40]. The chemical reactions in the levitated droplet trend to proceed in a better dispersed state, causing the gold salt to be reduced continuously. Consequently the instantaneous nucleation of gold nanoparticles is induced in acoustic levitation and brings the solution below supersaturation. According to Lamer’s theory [41], this causes the monomer concentration to drop sharply. The final phase of the Lamer’s theory is growth of the newly formed particles either from the remaining monomer species left over in the solution or from newly generated monomers from continued precursor conversion [42]. Therefore the insufficient supplyment of precursors during the growth stage eventually leads to the reduction of the size of gold nanoparticles. In Ostwald ripening theory, larger nanocrystals grow at the expense of smaller nanocrystals, which are progressively dissolved [43,44]. Nanocrystal growth by aggregation and coalescence has been directly observed in real time by in situ TEM studies [45]. In most instances, the shear rate in the flow field induces the shear aggregation but there are exceptions: the flow-induced
alignment of small gold nanorods in aqueous sucrose solutions is reported [46]. Optical absorption spectra have been measured over a range of shear rates and the observed optical changes are reversible, indicating that the nanorods don’t undergo aggregation during measurement. Molecular dynamics simulations have been conducted to study the effect of shear flow on polymer nanocomposite systems [47], and shear has been shown to significantly slow down nanoparticle aggregation. In the acoustic levitation condition, the internal flow of acoustically levitated droplet probably hinder the particle coalescence and fusion in the growth stage, thus inhibiting the further increase of the size of gold nanoparticles than the normal synthesis with glassware. In addition, the evaporation was tried to avoided in the whole experimental process, and it’s about within 10%. The existence of droplet evaporation caused the real-time change of concentration and may affect the particle size synthesized in acoustic levitation. The evaporation degree depends on different solvents, droplet volume, sound pressure level and other factors. The excessive evaporation deformed the droplet severely and deviated it from its original shape and size, resulting in the unstable levitation state and even the atomization of droplet.

4. Conclusion

In conclusion, the gold nanoparticles synthesized in acoustic levitation have more uniform and finer particle sizes with different molar ratios of NaBH₄ to HAuCl₄·3H₂O. The mechanism is elaborated to optimize the synthesis of gold nanoparticles in acoustic levitation. Besides the advantage of containerless characteristic, the acoustic also supplies a continuous vibration energy, limiting the fast growth of nucleus. Meanwhile, fresher nucleus generated simultaneously, dominated in the reduction of gold salt reduction and decreased the particle size because the highly reactive radicals produced by acoustic cavitation. The internal flow of levitated droplet probably inhibits the particle fusion in the growth process, resulting in a smaller size than that of the normal synthesized nanoparticles with glassware. The catalytic performance in 4-nitrophenol reduction of gold nanoparticles synthesized in acoustic levitation is improved compared to normal condition. It is expected to be applied to the pollution clean-up of the phenols compounds in water. Our work can predict that other nano-sized metal alloys, metal-organic frameworks, organic polymer materials especially of ultrasonic response characteristic synthesized in acoustic levitation may be endowed with more fantastic properties, and encourage more exploration of extensive material containerless synthesis mechanism.

CRediT authorship contribution statement

Yuhang Zheng: Conceptualization, Methodology, Software, Investigation, Formal analysis, Visualization, Writing – original draft. Qiang Zhuang: Validation, Formal analysis, Writing – review & editing. Ying Ruan: Conceptualization, Funding acquisition, Investigation, Resources, Supervision, Writing – review & editing. Guangyao Zhu: Software. Wenjun Xie: Methodology, Investigation. Yanyan Jiang: Formal analysis, Writing – review & editing. Hui Li: Investigation. Bingbo Wei: Supervision, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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