Synthesis and optical properties research of gold nanoparticles with different morphologies

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Abstract: This paper presents an approach to synthesis of gold nanoparticles with different morphologies and investigation of the relationship between morphologies and their optical properties. Spherical gold nanoparticles with different sizes are synthesized via reduction method. Using seed-mediated solution growth method, gold nanoparticles with shuttle, star and stick shapes can be obtained. The sizes and morphologies of the gold nanoparticles are characterized by transmission electron microscopy (TEM). The characterization results illustrate the growth process of the gold nanoparticles with different morphologies. Absorption spectroscopy and Raman spectroscopy measurements are performed to demonstrate the relationship between the morphologies and optical properties. The results of Raman characterization show that the gold nanoparticles with different morphologies can be used to probe molecules with different concentrations.

Key words: star-shaped gold nanoparticles; spherical gold nanoparticles; surface-enhanced Raman scattering (SERS); seed-mediated solution growth method

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Owing to unique features in electrical, physical and optical properties at nanoscale, noble metal nanomaterials have great potential in many research fields, such as photography, optoelectronics, information, biosensors, surface-enhanced Raman scattering (SERS) and catalysis[1-4]. Optical and thermodynamic properties of mental particles deviate from bulk properties, while the size of the particles decrease from 100 to 1 nm[5-7]. Moreover, the optical properties of gold nanoparticles are closely related to their shapes. There exists an increasing demand for finding an efficient way to synthesize gold nanoparticles with different sizes and morphologies.

This paper presents our recent development of a simple and efficient method of fabricating gold nanoparticles with different patterns. Spherical gold nanoparticles are obtained using Na_3-citrate and $NaBH_4$ as reducers. The size of nanoparticles can be regulated by controlling the amount of the reducers. Using seed-mediated solution growth method, gold nanoparticles with different shapes, such as star, shuttle and stick shapes, can be obtained. The star- and the shuttle-shaped gold nanoparticles are fabricated by controlling the amount of growth solution, whereas stick-shaped gold nanoparticles can be obtained by controlling the component of the growth solution. Gold nanoparticles with different patterns are characterized by transmission electron microscopy (TEM). The results illustrate growth process of the gold nanoparticles with different morphologies. Absorption spectroscopy and Raman spectroscopy measurements are performed to demonstrate different optical properties of the gold nanoparticles with different sizes and morphologies. During the growth process, the shifts of absorption spectra are related to different sizes and morphologies of the gold nanoparticles. Raman spectroscopy measurement results indicate that the star-shaped gold nanoparticles exhibit more promising optical features because of their special patterns.

The unique contribution of this paper is that, with the proposed method, gold nanoparticles with different patterns can be fabricated using the same process. In this way, different performances of gold nanoparticles with different patterns can be compared under the same conditions, such as temperature, air pressure, noise, etc. Gold nanoparticles

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exhibit magic optical properties because of their localized surface plasmon resonance (LSPR)[8-11]. LSPR can enhance the local electric field around the gold nanoparticles. The absorption spectra and SERS of the gold nanoparticles change during their growth process. Considering that the local electric field around star-shaped gold nanoparticles is large, these gold nanoparticles can better enhance the Raman signal than those with other patterns[12-16]. SERS of spherical and star-shaped gold nanoparticles was characterized in this study. The results indicate that the patterns of gold nanoparticles are correlated with LSPR. Due to unique advantages of largely enhancing Raman signal, the gold nanoparticles can be applied to the design of micro-sensors for various chemical and biological applications.

1 Methodology

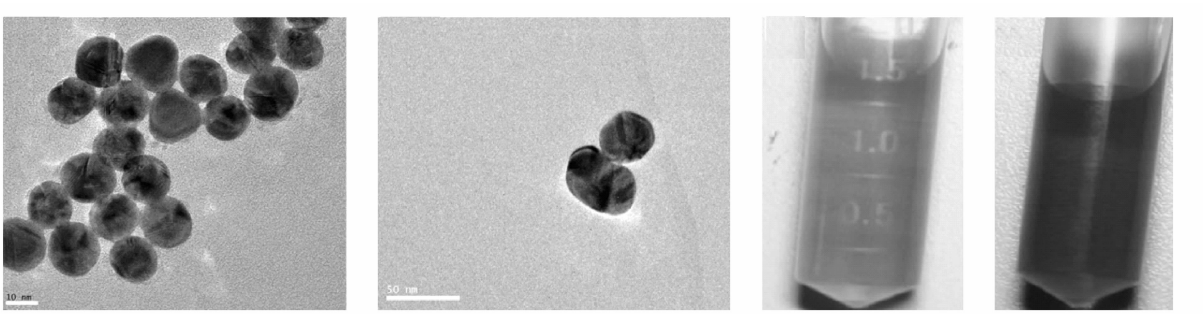
1.1 Synthesis of spherical gold nanoparticles

The sodium citrate reducing method was employed in this study. The procedures for preparing small-size gold nanoparticles are described below. Firstly, 100 mL of 0.25 mmol/L auric chloride (analytical reagent (AR)) solution was put into a 250 mL flask, and then heated to boiling by constant temperature water bath (MPL-HWS). Secondly, 3.5 mL of 1% sodium citrate (AR) was added rapidly into an 800 r/min stirring solution while heating continued. Multifunctional magnetic stirrer (MPL-CJ-88) was used to stir and heat the solution. Around 3 min thereafter, the solution turned to gray, and then to dark red. After heated for another 15 min, the solution was cooled down to room temperature, and 18.25 MΩ deionized water, which was purified through the ultra-pure (UPR) system, was added until the volume was back to 100 mL. Finally, 10 nm gold nanoparticles were obtained.

The procedures for preparing large-size gold nanoparticles were slightly different from those for small-size gold nanoparticles. Firstly, 100 mL of 0.25 mmol/L auric chloride solution was put into a 250 mL flask, and then heated to boiling. Secondly, 1.5 mL of 1% sodium citrate (AR) was added rapidly into an 800 r/min stirring solution while heating the solution continually. Around 3 min thereafter, the solution turned to gray, and then to dark red. After heated for another 15 min, the solution was cooled down to room temperature. Then, deionized water was added until the volume was back to 100 mL. Finally, 50 nm gold nanoparticles was obtained.

Transmission electron microscope (JEOLJEM-1200EX) was used to characterize patterns and sizes of the nanoparticles. The fabricated gold nanoparticles were characterized by TEM.

Fig.1 shows the TEM images and pictures of spherical gold nanoparticles with different sizes. The 10 nm spherical gold nanoparticles were obtained by adding 3.5 mL sodium citrate, as seen in Fig.1(a). The 50 nm spherical gold nanoparticles were obtained by adding 1.75 mL sodium citrate, as seen in Fig.1 (b). The light red 10 nm spherical gold sol is shown in Fig.1(c), and the dark red solation with 50 nm gold nanoparticles is shown in Fig.1(d). These results demonstrate that dark red gold sol with 10 nm spherical gold nanoparticles could be obtained by keeping the volume ratio of chloroauric acid to sodium citrate less than 1:3.5. By reducing the amount of sodium citrate, the purple gold sol with 50 nm spherical gold nanoparticles was obtained.



(a) TEM image of 10 nm spherical gold nanoparticles

(b) TEM image of 50 nm spherical gold nanoparticles

(c) 10 nm spherical gold sols

(d) 50 nm spherical gold sols

Fig.1 TEM images and spherical gold nanoparticles with different sizes

1.2 Synthesis of shuttle-, star- and stick-shaped gold nanoparticles

A total of 10 mL of 0.25 mM auric chloride solution was put into a beaker. After adding 0.35 mL of 1% sodium citrate, the beaker was shocked gently for 3 min. Then, 0.3 mL fresh sodium borohydride (99%) was added to the solution, which was then shocked for 5 min. After maintaining the process

for 2 h, the seed solution was obtained.

The procedures for preparing the growth solution are described below. A total of 7.28 g hexadecyl ammonium bromide (AR) and 1.36 mg silver nitrate (AR) were put into a beaker, which had 200 mL of 0.25 mM auric chloride solution. The beaker was kept at 40 ℃ for half an hour until hexadecyl ammonium bromide was dissolved completely. After the solution was shocked for 5 min, it turned into a clear dark red color. Then, 0.1 mL seed solution was put into a beaker. Next, 0.006, 0.6, and 6 mL of 10 mM ascorbic acid were put into three beakers with 1, 10, and 100 mL growth solution, respectively. Three portions of the growth solution were finally obtained. The three portions of the growth solution were then put into the seed solution every 30 s.

The shuttle-shaped gold nanoparticles were obtained before adding the second portion of the growth solution. The star-shaped gold nanoparticles were obtained after adding all the three portions and after maintaining the process for several hours. The stick-shaped gold nanoparticles all the procedures of synthesizing were the same as that of synthesing star-shaped gold nanoparticles, but no silver nitrate in the growth solution.

Fig. 2 illustrates gold nanoparticles obtained after adding each portion of the growth solution during the synthesis of star-shaped gold nanoparticles. Fig. 2(a) illustrates the TEM image of gold nanoparticles in the seed solution, where 10 nm spherical gold nanoparticles were obtained. Fig. 2(b) illustrates that, after adding the first portion of the growth solution, 50 nm shuttle-shaped gold nanoparticles were obtained. After adding the second portion of the growth solution, small tips occurred on the gold nanoparticles, as shown in Fig. 2(c). Owing to the aeolotropic growth mode of particles, 80 nm star-shaped gold nanoparticles were obtained 5 h after the third portion of the growth solution was added, as shown in Fig. 2(d). Fig. 2(e) shows that stick-shaped gold nanoparticles were fabricated when there was no silver nitrate in the growth solution. The seed solution with 10 nm spherical gold nanoparticles was red, as shown in Fig. 2(f). The blue star-shaped gold sols are shown in Fig. 2(g). The above results demonstrate that shuttle-shaped, star-shaped and stick-shaped gold nanoparticles could be fabricated by controlling the components of the growth solution.

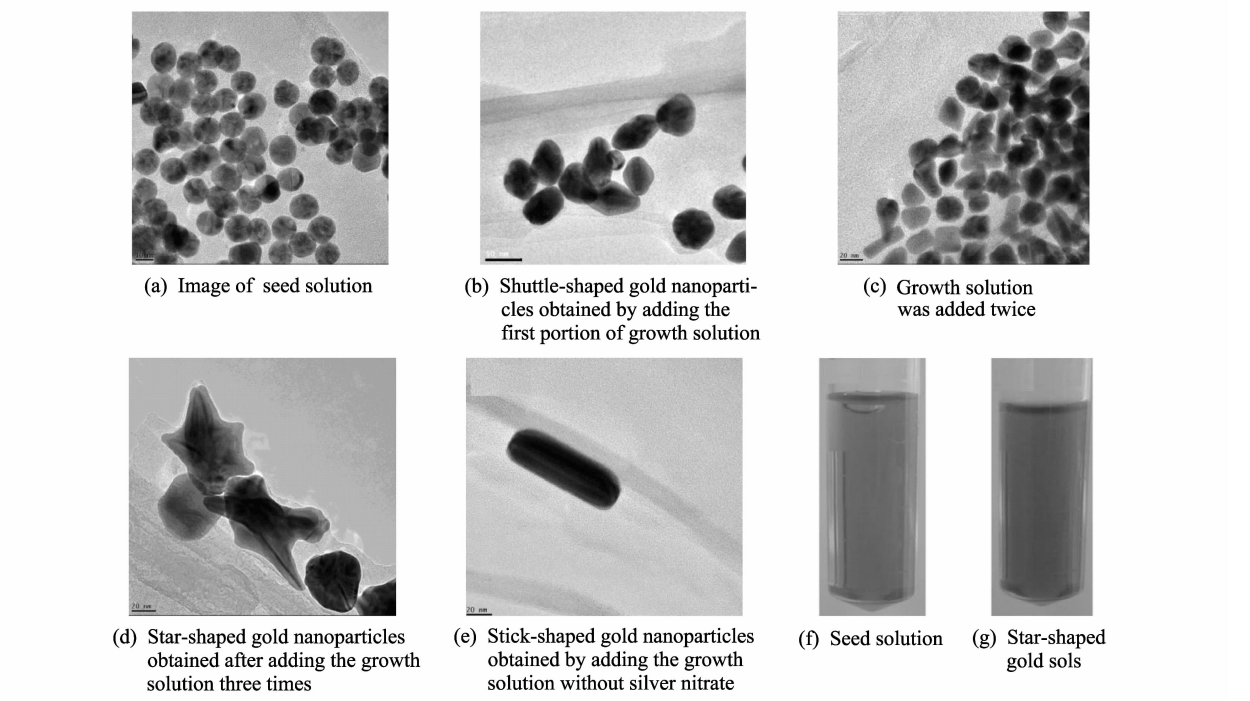


Fig. 2 TEM images of gold nanoparticles with different patterns

2 Results and discussion

2.1 Growth process of gold nanoparticles

In this study, spherical gold nanoparticles were obtained using the reduction method, where sodium citrate was used for reduction and protection, as shown in Fig. 3(a). Shuttle-shaped, star-shaped, and stick-shaped gold nanoparticles were fabricated using the seed-mediated solution growth method[17]. Fig. 3 illustrates the principle in synthesizing gold nanoparticles with different patterns, i.e., (a) spherical shape, (b) shuttle shape, (c) star shape,

and (d) stick shape.

To synthesize the seed solution, auric chloride was reduced using sodium borohydride. Sodium citrate was used as chelation agent to obtain a stable gold seed solution. The growth solution includes a weak reduction ascorbic acid, template hexadecyl trimethyl ammonium bromide, auric chloride, and silver nitrate. Nucleation control is important in the seed-mediated solution growth method[18]. To make sure that there was no new core formatting during the growth process, the following two operations were performed. Firstly, the weak reduction ascorbic acid was monitored, such that Au3+ was not reduced until the seed solution was added to the growth solution. Secondly, by adding three portions of the growth solution, the ratio of the gold seed to the growth solution was controlled to 1∶10.

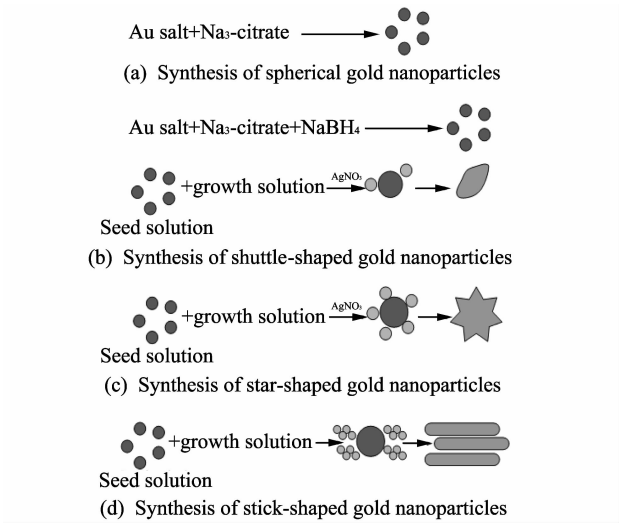


Fig. 3 Principles of the synthesis of spherical gold nanoparticles

Different components and amounts of the growth solution lead to different patterns of gold nanoparticles. Silver nitrate could not be reduced by a weak reduction ascorbic acid under a low PH value. Ag+ could replace the gold growth particles on the gold seed particle surface. The blocking phenomenon was the key in the synthesis of the star-shaped gold nanoparticles. In Fig. 3(b), shuttle-shaped gold nanoparticles were synthesized after the first portion of the growth solution was added. Fig. 3(c) shows that star-shaped gold nanoparticles were obtained after three portions of the growth solution were added. Stick-shaped gold nanoparticles were obtained by adding the growth solution without silver nitrate, as shown in Fig. 3(e).

2.2 Optical porperties of gold nanoparticles

The most important feature of the metal nanoparticle absorption spectra lies in their surface plasmon band. The absorption peaks of surface plasmon resonance (SPR) are excited by free electric concussion[19], and different sizes and morphologies of gold nanoparticles lead to different absorption peaks[20,21]. The absorption spectra were characterized by UV-Vis spectrometer (Cary-300).

Fig. 4 shows that the absorption of 10 nm spherical gold nanoparticles peaks at a wavelength of 520 nm (see curve (a)), and that the absorption of 50 nm spherical gold nanoparticles peaks at a wavelength of 530 nm (see curve (b)). These results agree with the electromagnetic theory, which states that the larger the nanoparticles are, the longer the wavelength is in which the absorption peak occurs. The width of the half wave depends on the size distribution of the gold nanoparticles. The wider the half-wave width is, the wider the size distribution of the gold nanoparticles is[14]. The results in Fig. 4 verify that the size distribution of 10 nm spherical gold nanoparticles is similar to that of 50 nm spherical gold nanoparticles.

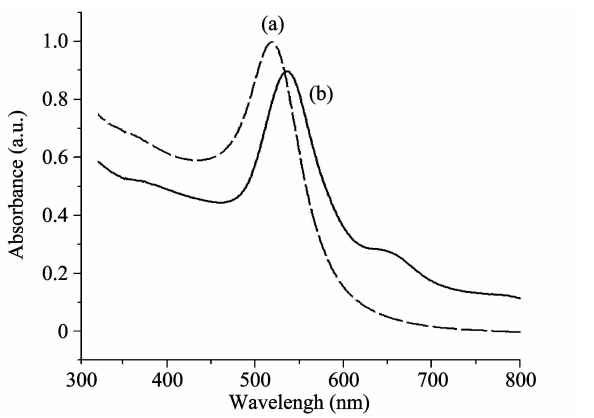
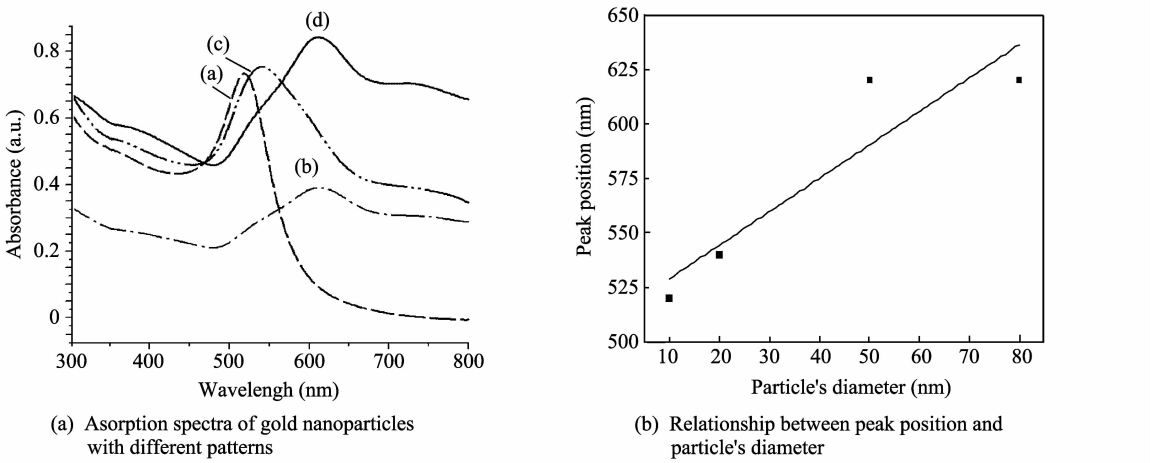


Fig. 4 Absorption spectra of gold nanoparticles with different sizes

Fig. 5(a) illustrates absorption spectra of the gold nanoparticles with different shapes, which were obtained by adding three portions of the growth solution into the seed solution. Curve (a) is the spectrum of the seed solution. Curve (b) is the spectrum of shuttle-shaped gold nanoparticles obtained by adding the first portion of the growth solution. Curve (c) is the spectrum of the gold nanoparticles obtained by adding the first portion of the growth solution. Curve (d) is the spectrum of the star-shaped gold nanoparticles obtained after adding the growth solution three times. Fig. 5(b) shows the relationship between peak position and particle's diameter, which demonstrates the red-shifting of the absorption spectrum as the particle diameter increases. The reason is that the nanoparticle's diameter is inversely proportional to energy level gap of the adjacent electronic states[20]. The bigger the diameter of the nanoparticle, the wider the level gap

between electrons on the highest and lowest orbitals. Less energy is needed for electron transition during the photochromic process[20]. Therefore, the red shift occurs when the nanoparticle's diameter increases.

In Fig. 5(a), curve (a) is the absorption spectrum of 10 nm gold nanoparticles in the seed solution, in which the absorption peak occurred at a wavelength of 520 nm. Curve (b) is the absorption spectrum of 50 nm shuttle-shaped gold nanoparticles, which were obtained by adding the first portion of the growth solution. The absorption peak occurred at a wavelength of 620 nm, which had 100 nm red shift with respect to the peak of curve (a), indicating that the size of the gold nanoparticles is bigger. The width of the half wave of curve (b) was wider, indicating that the size distribution of shuttle-shaped gold nanoparticles was wider. Curve (c) is the absorption spectrum of gold nanoparticles obtained after adding the second portion of the growth solution. Au3+ was reduced using ascorbic acid. In Fig. 2(c), the gold nanoparticles are smaller but not still star-shaped, as seen in curve (c). There was about 80 nm blue shift compared with curve (b), indicating that the size of the gold nanoparticles is smaller. Curve (d) is the absorption spectrum of gold nanoparticles obtained after adding the third portion of the growth solution. Compared with curve (c), curve (d) has 80 nm red shift, indicating that the size of the gold nanoparticles after adding the third portion of the growth solution was bigger. The above characterization results, which agree with Fig. 2, demonstrate that spherical gold nanoparticles turned to irregular double cones, and then to twig-like star-shaped gold nanoparticles. The results in both Fig. 5 and Fig. 2 also indicate that the absorption peaks are shifting as the morphologies change during the growth process. The different absorption spectra of the gold nanoparticles with different patterns make the gold nanoparticles be applied to the design of optical sensors with different wavelengths.



(a) Asorption spectra of gold nanoparticles with different patterns

(b) Relationship between peak position and particle's diameter

Fig. 5 Absorption spectra of gold nanoparticles and relatiorship between peak position and particle's size

To study the optical properties of gold nanoparticles with different shapes, both spherical and star-shaped gold nanoparticles were tested in SERS. Crystal violet was used as the probing molecule to detect SERS signal. The steps for preparing samples are described as follows.

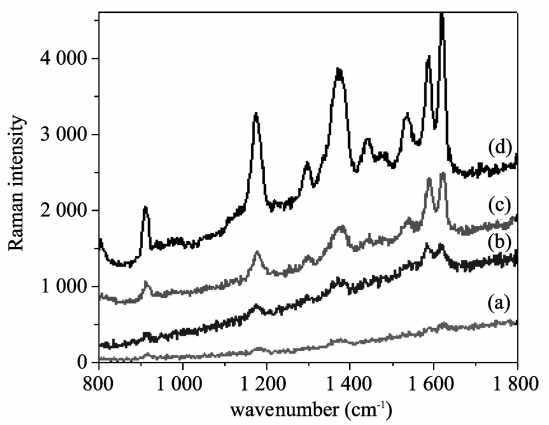
A total of 6 mL spherical and star-shaped gold sols were put into 2 mL centrifuge tubes, which were centrifuged at 14 000 r/min for 10 min by high-speed desktop centrifuge (TGL-16K). The upper solution without nanoparticles was removed, and then restored to its original volume in deionized water. This step was repeated three times to remove undissolved hexadecyl ammonium bromide as much as possible. The two kinds of gold sols in the bottom were pipetted into a new centrifuge tube. Deionized water was used to restore these two kinds of gold sols back to 2 mL. By conducting the above procedures, the concentration of the gold sols increased.

Then, 0.1 mL of 10^{-5} g/L crystal violet solution was added into the gold sols so that the solutions were dispersed ultrasonically. The solutions were moved into quartz capillaries whose two sides were obdurate. The capillaries with samples were maintained for 30 min. The parameters of Raman spectroscopy (made by Renishaw, UK) were as follows: excitation wavelength is 514.5 nm, laser power is 5 mW, and integration time is 10 s. Fig. 6(a) shows Raman spectra of the low-concentration crystal violet solution with different appearances of gold nanoparticles. Curve (a) is the Raman spectrum of low-concentration crystal violet solution. Curve (b) is the spectra of the solution with spherical gold nanoparticles. Curve (c) is the spectra of the solution with shuttle-shaped gold nanoparticles. Curve (d) is the spectra of the solution with star-shaped gold

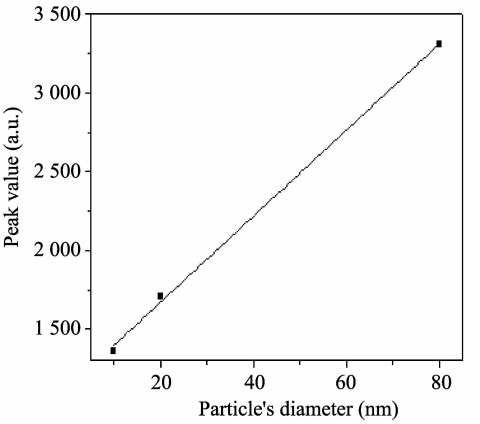
nanoparticles. Fig.6(b) illustrates the relationship between peak position and particle's diameter, indicating that the gold nanoparticles with larger diameter can better enhance Raman signal.

Gold nanoparticles can enhance Raman signal of the solution with very low concentration, which is affected by different patterns of the nanoparticles. Although mental molecules have no Raman peak, they can enhance Raman intensity of other molecules in very low concentration. The Raman peak cannot be found in curve (a), but is found clearly in curves (b), (c) and (d). Curves (b), (c) and (d) have Raman peaks at 785, 901, 1 025, 1 163, 1 279, 1 358, 1 464, 1 517, 1 569, 1 607 and 1 617 cm^{-1}, which are all the wavenumbers of crystal violet Raman peaks[15].

Fig.6 demonstrates that all spherical, shuttle-shaped and star-shaped gold nanoparticles can enhance Raman intensity. When crystal violet molecules are absorbed by gold nanoparticles, the LSPR of gold nanoparticles can increase Raman intensity of the crystal violet molecules. It can be seen from Fig.6 that the star-shaped gold nanoparticles can enhance Raman signal by 6.62 times at 1 617 cm^{-1}, while shuttle-shaped and spherical nanoparticles can enhance Raman signal by 3.42 and 2.73 times respectively at the same wavenumber. Due to different shapes, the charging storage ability of gold nanoparticles decreases in the order of star-shape, shuttle-shape and spherical shape, which also leads to reduction of LSPR of the nanoparticles. Fig.6 demonstrates that the gold nanoparticles with different patterns have different effects on Raman signal. Utilizing this property, star-shaped gold nanoparticles can be used to detect micro-amount molecules, and the gold nanoparticles with different shapes can be used to measure different micro-amount molecules.



(a) Reman spectra of low-concentration crystal violet solution with different appearances of gold nanoparticles



(b) Relationship between peak value and particle's diameter

Fig.6 Test results of Raman spectra

3 Conclusion

This paper presents an approach to synthesize gold nanoparticles with different shape patterns, such as spherical, star, shuttle, and stick shapes. Spherical gold nanoparticles with different sizes are obtained using different amounts of Na3-citrate as reducer. Star-shaped, shuttle-shaped, and stick-shaped gold nanoparticles are obtained using growth solutions with different amounts of components. The results of TEM characterization show that star-shaped, shuttle-shaped and stick-shaped gold nanoparticles can be obtained successfully. The absorption spectroscopy and SERS characterization results also demonstrate that the optical features of gold nanoparticles change during the growth process. The gold nanoparticles with different patterns appear to have different absorption spectra. This property can be utilized to design optical sensors with different wavelengths. The SERS characterization results further demonstrate that the gold nanoparticles with different patterns have different effects on Raman signal, which can be used for probing different molecules.

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不同形貌尺寸金纳米颗粒的制备及其光学性能研究

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摘　要: 本文制备了不同形貌的金纳米颗粒, 并对其形貌对光学性能的影响进行了研究。本文用还原法制备了不同粒径的金纳米颗粒, 采用晶种生长法成功地制备出了星形、梭形和棒状的金纳米颗粒。颗粒的形貌和大小并采用投射电子显微镜(TEM)进行了表征, 结果说明, 本文成功制备出了不同形貌大小的金纳米颗粒。UV-Vis 光谱和拉曼光谱仪对制备的颗粒的表征测试说明, 不同形貌大小对颗粒有着不同的光学性能。拉曼光谱的结果说明, 不同形貌大小的金纳米颗粒可以用作不同浓度分子的探针, 对物质进行检测。

关键词: 星形金纳米颗粒; 球星金纳米颗粒; 表面增强拉曼散射(SERS); 晶种生长法(晶种法)

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