Rare Metal Materials and Engineering
Volume 48, Issue 11, November 2019
Available online at www.rmme.ac.cn

Science Press

Cite this article as: Rare Metal Materials and Engineering, 2019, 48(11): 3470-3475.

ARTICLE

Green Synthesis and Characterization of Monodisperse Gold Nanoparticles Using Aloe Vera Leaf Extract

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Abstract: By using Aloe vera leaf extract as both reducing and stabilizing agents, spherical and small-size gold nanoparticles were successfully synthesized. The method is a simple, straightforward and eco-friendly approach because of absence of toxic reagent and the neutral pH conditions. The synthesized gold nanoparticles were examined by UV-visible absorption spectroscopy (UV-vis), dynamic light scattering (DLS), scanning electron microscope (SEM), transmission electron microscopy (TEM), Fourier transform infrared (FT-IR) and powder X-ray diffraction (XRD). The change of color and the absorbance peak in UV-vis spectroscopy indicate the formation of gold nanoparticles. The XRD results show that the particles are highly crystallined in nature. The TEM and SEM results illustrate the particles are spherical in shape with a narrow distribution from 20 nm to 60 nm. The FT-IR results prove that the gold nanoparticles are capped with extracts, which keeps them from agglomeration and oxidation. The effect of reaction temperature, amount of chloroauric acid solution and extracts was also studied. The results display that these parameters play important roles in the generation of gold nanoparticles.

Key words: gold nanoparticles; green method; powder technology; nanocrystalline materials

In modern nanoscience, nanomaterials have been known as one of the most promising and popular fields of scientific research. Numbers of nanomaterials, such as nanodisk, nanowire, nanoparticles, have been produced by various methods[1-12]. Among nanomaterials, metallic nanoparticles have attracted intensive interest due to their unique optical and electronic properties, which may lead to a huge amount of potential applications, such as sensing, catalyst, diagnostics, photonics and therapeutics. Hence, the generation of metallic nanoparticles have important theme in nanotechnology[13-18]. Generally, the properties as well as the application of metallic nanoparticles are influenced by the size and morphology[19-24]. It is virtue to control the size and shape of metallic nanoparticles. Various methods, including irradiation reduction, laser ablation, ion implanation, electrolysis methods, thermal decomposition and chemical reduction, have been explored to produce of metallic nanoparticles[25-27]. In these procedures, the chemical reduction method own the merit of being able to prepare metallic nanoparticles with desired size by simple device and equipment[28-30]. Thus, the chemical reduction process is the most prevalent method to prepare metallic nanoparticles. However, some expensive and toxic reagent, such as sodium borohydride, hydrazine hydrate and ethylene glycol were employed in the common methods, which may limit the application of metallic nanoparticles.

In consideration of the environment sustainability, the green synthesis of metallic nanopartilces has received much attention in nanotechnology as it manufactures relatively less or non toxic metallic nanoparticles[27, 31-36]. The preparation of metallic nanoparticles using various plants extracts, fungi and microbes are considered as an environmental way, which avoid the use of the toxic chemicals. Compared with the microbial process, plant mediated route has gained more attention due to its cost effectiveness, rapid synthesis and improved stability. Numbers

Received date: November 19, 2018
Foundation item: National Natural Science Foundation of China (51405146); The Hubei Natural Science Foundation(2018CFB718); Hubei Polytechnic University Scientific Research Project(15xjz04R)
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of plants extract, such as Murraya Koenigii leaf, Mangifera indica leaf, Sesbania grandiflora leaf, have been used to synthesize of metallic nanoparticles[37-41].

In recent years, the synthesis of silver and gold nanoparticles using plant extracts has attracted remarkable attention due to their unique properties, which may lead to potential commercial application in many fields[9, 42-45]. Recently, we have developed an original approach using wolfberry fruit (lycium barbarum), osmanthus fragrans extract and ginkgo biloba leaf extract as capping agents as well as reducing agent for preparation of small silver and gold nanoparticles[6, 13, 46, 47]. In present research, Aloe vera leaf extract was used as reducing and capping agents for the bio-synthesis of gold nanoparticles. Aloe vera, belonging to Liliaceal family, is a perennial succulent which is grown in hot, dry climates. This plant is widely planted in China, Korea, Japan, Europe and USA[48, 49]. It is well known that Aloe vera plant extract can be used to curevarious diseases with their biologically important molecules. The Aloe vera was reported to own anti-inflammatory, UV protective, antibacterial and wound healing properties. The Aloe vera leaf consists of 75 bioactive components, including vitamins, enzymes, sugars, salicylic acids, lignins, saponins, amino acids, which may be used for medicinal purposes in several culture for millennia[50, 51]. These potentially active constituents also can be used as reducing agents and capping agents to prepare metallic nanoparticles. The metallic nanoparticles capped with Aloe vera leaf extracts can used as pharmaceutical drugs and antibacterial agents. Hence, we use the Aloe vera leaf extract to prepare gold nanoparticles.

1 Experiment

Chloroauric acid used in the experiment was analytical grade, which was obtained from Sinopharm Chemical Reagent Co. Ltd., and acted as the precursor for the synthesis of gold nanoparticles. Fresh Aloe vera leaf was gathered from Hubei Polytechnic University, Hubei, China. All other chemicals and solvents were used as received without further purification. Doubly distilled water was used in all experiments.

To obtain 0.2% chloroauric acid solution (w/v), 1.0 g of chloroauric acid was dissolve in 500 mL distilled water. The solution was kept in dark for further use.

For the preparation of plant extract, Sixty grams of Aloe vera leaf were washed with tap water to remove foreign material, and then rinsed with distilled water. The cleaned Aloe vera leaf were dried in shade for 24 h. The obtained dry Aloe vera leaf were mixed with 200 mL distilled water in a 500 mL flask, and boiled at 100 °C for 30 min. Then the gained plant extracts were cooled and subjected to repeated filtration through Whatman No.1 filter paper. The volume of plant extracts were adjusted to 200 mL by adding distilled water and stored in refrigerator at 4 °C for further studies in 3 d.

The preparation of gold nanoparticles was simply obtained by the mix of chloroauric acid and plant extracts. For a typical synthesis, 6 mL Aloe vera leaf extract and 3 mL chloroauric acid solution were mixed at room temperature. The total volume of reaction mixture was adjusted to 10 mL by adding appropriate amount of distilled water. The solution were stirred at slow speed for 2 h. The color of the reaction solution was gradually transformed from pale yellow to red, and wine red, which indicates the generation of gold nanoparticles. The gold colloids were separated and purified from the solution by repeated centrifugation for 15 min at 10 000 r/min. Finally, the purified metallic nanoparticles were further dried in vacuum at 40 °C for 12 h.

The synthesis of gold nanoparticles was easily achieved by reduction of chloroauric acid using Aloe vera leaf extract as reducing agent and capping agent. The biotransformation of gold nanoparticles can be easily confirmed by UV-vis spectroscopy. The UV-vis spectroscopy of different amount of chloroauric acid solution and leaf extract were monitored in a Specord S 600 UV-vis spectrophotometer operated at a resolution of 1 nm in 300~900 nm to prove the production of gold nanoparticles.

The morphology of gold nanoparticles was observed by a JEM2100 transmission electron microscope (TEM). The TEM observations were operated at an accelerating voltage of 200 kV. The TEM samples were obtained by transferring small amount of gold colloids suspension onto carbon coated copper grids and evaporating the water at room temperature. The sizes of gold nanoparticles were determined statistically by image analysis.

XRD technique was employed to check the composition and microstructure of synthesized gold nanoparticles. The measurement was conducted on an X'Pert PPO X-ray diffractometer, operated at a voltage of 40 kV, with Cu Kα radiation of wavelength of λ=0.15406 nm in the 2θ range of 10° to 85° at room temperature. The samples were prepared as uniform thin film by dropping the metallic nanoparticles on the slides.

The hydrodynamic diameter and distribution of the metallic nanoparticles were determined by dynamic light scattering (DLS) on a Malvern Zetasizer Nano ZS.

2 Results and Discussion

2.1 Characteristics of the synthesized gold nanoparticles

Recently, much research has focused on the preparation of nano metallic particles using environment friendly and renewable begins. In these forming methods, lots of plant extracts were used to manufacture metallic nanoparticles. In the present study, a straightforward and facile method was exploited to synthesize gold nanoparticles by mixing Aloe vera leaf extract and chloroauric acid solution together. The Aloe vera leaf extract was used as capping agent as well as reducing agent. To verify the formation of gold nanoparticles, UV-vis absorption spectrum was used. The UV-vis technique has been confirmed to be a useful tool to detect of noble metallic nanoparticles. The typical UV-vis absorption spectrum of gold nanoparticles is illustrated in Fig.1. A characteristic absorption band due to the surface plasmon resonance (SPR) of gold nanoparticles was observed at around 564 nm. Usually, the appearance and position of absorption peak are strongly affected by the size and shape of metallic nanoparticles. For nano gold particles, the SPR peak located at 564 nm was confirmed during the preparation of spherical gold nanoparticles. Further more, the shape of the plasmon band was symmetric, which indicates the narrow size distribution of gold nanoparticles.

To confirm the UV-vis analyses, the sample of gold nanoparticles was purified by ultra centrifugation and checked by XRD. The typical XRD pattern of Aloe vera leaf extract stable gold nanoparticles is illustrated in Fig.2, showing the crystalline nature of prepared gold nanoparticles. From the XRD pattern, five distinct diffraction peaks at 38.12°, 44.26°, 64.68°, 77.42° and 81.40° are indexed as (111), (200), (220), (311) and (222) planes, respectively, confirming that the samples were metallic gold nanoparticles with a face-centered cubic crystal structure. Strong and broadening peaks pertaining to gold nanoparticles was due to the crystalline nature and small particles of the prepared gold nanoparticles. Among the five Bragg reflections, the peak located at 38.12° pertaining to the (111) reflection is intenser than other peaks. These results demonstrate that the as-obtained gold nanoparticles may be likely to be enriched with (111) facets. Besides these five peaks, a broad peak is also observed which is may be from the Aloe vera leaf extract absorbed on the surface of gold nanoparticles. Because of the adsorption of leaf extract, the gold nanoparticles keep good oxidation resistance.

The particle size distribution was also proved by DLS analysis. The size particle distribution of noble metallic nanoparticles prepared in typical process is illustrated in Fig.3. According to the result, the average particle size of the prepared gold nanoparticles is about 100 nm with a narrow distribution.

The size and shape of gold nanoparticles was further confirmed by SEM and TEM. Fig.4 shows the typical SEM images of gold nanoparticles synthesized in a typical process. It is clearly seen that the morphology of the obtained gold nanoparticles is mostly spherical and the particles size ranges from 20 nm to 50 nm. The typical TEM image of gold nanoparticles is illustrated in Fig.5. As can be seen, the synthesized gold nanoparticles distribute uniformly, and exhibit a spherical shape with a particle size distribution from 20 nm to 60 nm. Further structural information of gold nanoparticles was obtained from HRTEM image shown in Fig.6. The *d*-spacing of the lattice can be clearly seen, suggesting the prepared gold nanoparticles are highly crystallined.

In order to identify the possible bio-molecules in the surface of gold nanoparticles, Fourier transform infrared (FT-IR) measurements were executed. Usually, the FT-IR spectroscopy is a special tool to reveal the structural information. The FTIR spectra of the obtained gold nanoparticles are illustrated in Fig.7. The broad absorption peak at 3409 cm^{-1} is attributed to the stretching vibrations of the hydroxyl group (-OH). The peak in 1631 cm^{-1} is due to the propyl stretching vibration. The peak located at 1077 cm^{-1} may be associated with the stretching vibration of the C-OH bond. The small peak located at 660 cm^{-1} can be allocated to C-H stretching. The FTIR analysis reveals the existence of organic bio-molecules on the surface of gold nanoparticles.

2.2 Effect of leaf extract amount

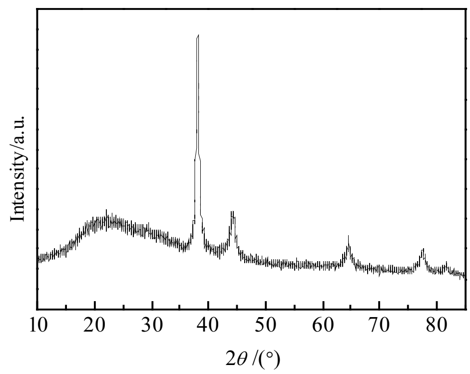

Fig.2 XRD pattern of gold nanoparticles

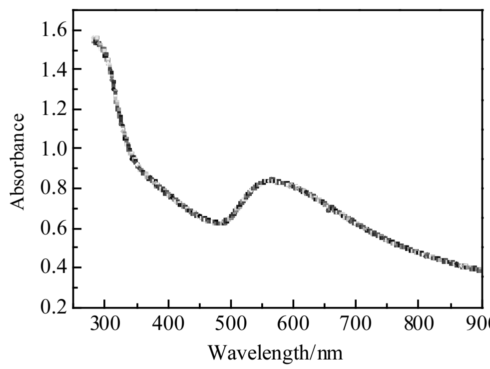

Fig.1 Typical UV-visible spectrum of the nano gold colloids

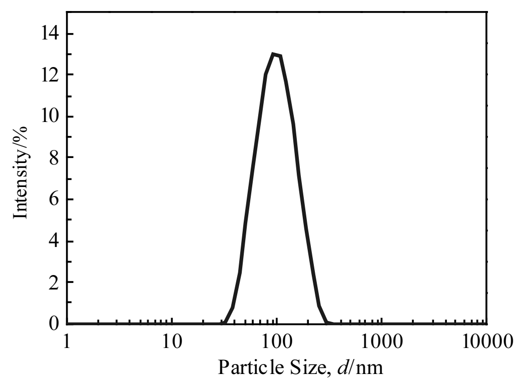

Fig.3 Particle size distribution of gold nanoparticles by intensity

In previous research, many leaf and fruit extracts were used as reducing agent and capping agent to prepare noble metallic nanoparticles. In the present study, Aloe vera leaf extract was used as reducing agent and stabilizing agent due to its good bio-compatibility and bio-degradability. Metallic gold nanoparticles were prepared in aqueous solution by reduction of chloroauric acid using Aloe vera leaf extract. Since the mixing of leaf extract and chloroauric acid solution, the color of mixture was changed from light yellow to red or to amaranth. It is well known that noble gold nanoparticles exhibit reddish color in colloids, which is attributed to the excitation of surface plasmon vibrations in gold nanoparticles. The change of color displays the generation of nano level gold particles, which is reduced from chloroauric acid by Aloe vera leaf extract. In this study, Aloe vera leaf extracts were used as reducing and capping agent, which play an important role in the preparation of gold nanoparticles. In order to illustrate the affect of leaf extract on the gold nanoparticles formation, 2, 4, 6 and 7 mL of Aloe vera leaf extract were used to prepare gold nanoparticles. The corresponding UV-vis absorption spectroscopy is shown in Fig.8. As the amount of extract is increased from 2 mL to 7 mL, the SPR peak shows a slight blue shift from 565 nm to 564 nm. The variation in SPR peak reveals the transformation of size in gold nanoparticles. The blue shift indicates the increase of particles size in the gold colloids.

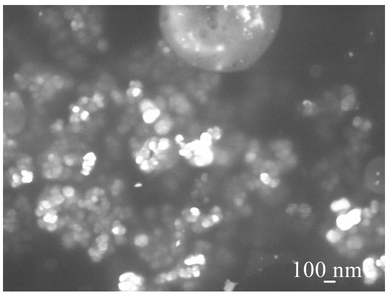

Fig.4 Typical SEM image of gold nanoparticles

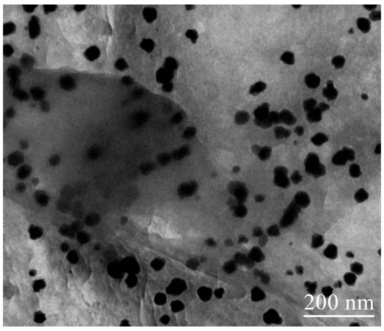

Fig.5 Typical TEM image of gold nanoparticles

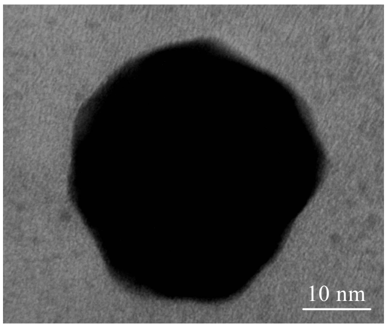

Fig.6 HRTEM image of gold nanoparticles

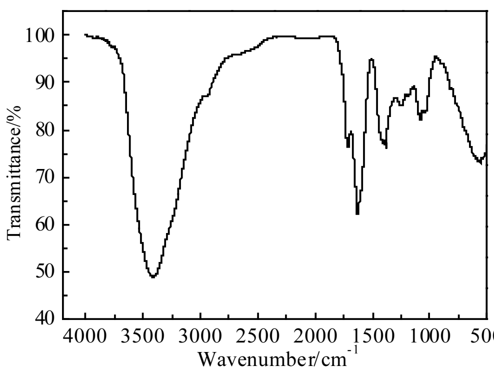

Fig.7 FT-IR spectrum of the prepared gold nanoparticles

2.3 Effect of amount of chloroauric acid solution

It is clear that the size, dispersibility, distribution and shape of the prepared metallic nanoparticles are influenced by the concentration of metal precursor. In this study, chloroauric acid was used as chloroauric acid to produce gold nanoparticles. To investigate the effect of chloroauric acid on gold nanoparticles generation, a multifarious amount of chloroauric acid solution (1, 2, 3 and 4 mL) were utilized to create gold nanoparticles. The UV-vis absorbance spectroscopy of gold colloids generated with different amounts of chloroauric acid solution is illustrated in Fig.9. The gold nanoparticles obtained at the lowest dosage of chloroauric acid solution show a SPR peak at 567 nm. As the amount is gradually increased to 2 and 3 mL, there is a blue shift from 567 nm to 565 nm and 564 nm, which demonstrates the decrease of particles size. As the amount increased to 4 mL, the SPR peak does not change.

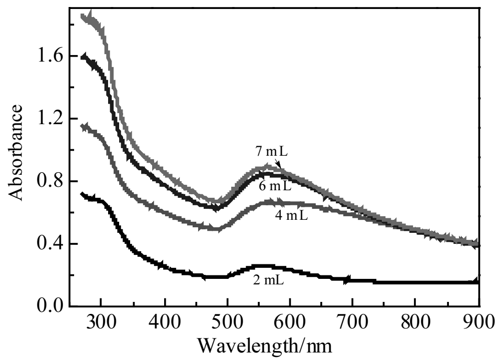

Fig.8 UV-vis spectra of gold colloids synthesized at different amount of leaf extract

2.4 Effect of reaction temperature

It is obvious that the size distribution and shape, and gold nanoparticles are influenced by reaction temperature. To investigate the effect of reaction temperature on the gold colloids, three comparative experiment at 25, 50 and 80 °C under the same reactant composition were carried out. The corresponding UV-vis absorbance spectroscopy is demonstrated in Fig.10. When the reaction is performed at 25 °C, the SPR peak is located at 564 nm. Gradually increasing reaction temperature, from 50 °C to 80 °C, the SPR peak disappears. Some black big sedimentation was obtained in the bottom of the reaction bottle. The room temperature seems the best reaction temperature.

2.5 Possible mechanism

The above results indicate that small size gold nanoparticles can be prepared by using Aloe vera leaf extract as reducing and capping agent. Generally, Aloe vera leaf contains some bioactive compounds such as vitamins, flavanoids, ascorbic acid and amino acids[50, 51]. These biochemicals can reduce chloroauric acid to gold nanoparticles and can be absorbed on the surface of gold nanoparticles, preventing them from aggregation.

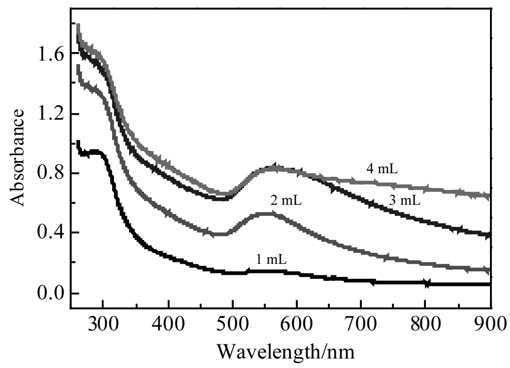

Fig.9 UV-vis spectra of gold colloids synthesized with different amounts of chloroauric acid amount solution

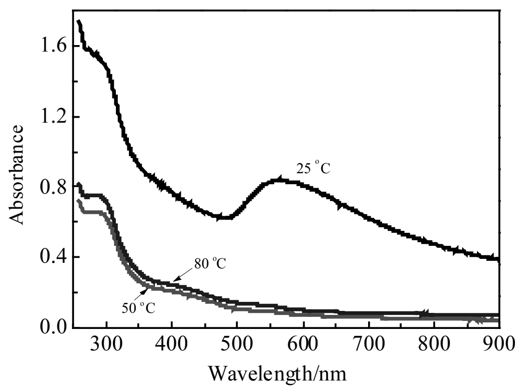

Fig.10 UV-vis spectra of gold colloids synthesized at different temperatures

3 Conclusions

1) In this work, we prepared gold nanoparticles by reducing chloroauric acid using Aloe vera leaf extract as reducing and stabilizing agents. The developed methodology is transcendent to other chemical reduction methods mentioned in the literature because of absence of toxic chemical reagent. In the method, gold nanoparticles were synthesized under ambient and neutral pH conditions.

2) The gold nanoparticles are protected by the extract from oxidation and aggregation.

3) The obtained gold nanoparticles are highly crystallined in nature with a fcc structure. The gold nanoparticles are mostly spherical in morphology, and polydispersity ranges from 20 nm to 60 nm.

4) The amount of extract, reaction temperature, and the amount of chloroauric acid solution play important roles in the synthesis of small-size gold nanoparticles. In this method, gold nanoparticles are obtained from chloroauric acid and Aloe vera leaf extract without using any other toxic reagent, which can be extended to prepare other nano metallic particles. The products have potential applications in pharmaceutical drugs and antibacterial agents.

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芦荟叶提取物绿色制备单分散纳米金及其性能

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摘　要：以芦荟叶提取液为还原剂和稳定剂，成功地制备了小粒径、球状的金纳米粒子。在这种方法中，简单的混合芦荟叶提取液和金源，没有使用有毒试剂，因此该方法是一种生态友好的合成纳米金的方法。混合溶液的颜色从浅黄色变到紫色，表明生成了纳米金粒子。采用紫外-可见吸收光谱（UV-vis）、激光粒径分析仪 (DLS)、扫描电子显微镜（SEM）、透射电子显微镜（TEM）、傅里叶变换红外光谱（FT-IR）、X 射线衍射（XRD）等方法对合成的纳米金粒子进行了表征和性能测试。紫外-可见光谱的吸收峰再次表明, 金纳米颗粒的形成。XRD 分析表明, 所生成的纳米金具有高度结晶性。TEM 和 SEM 表明, 纳米金颗粒呈球形，粒径分布在 20~60 nm 之间。FT-IR 证实了金纳米粒子被提取物所保护，使其不发生团聚和氧化。研究了反应温度、氯金酸溶液和提取物的用量对纳米金粒径的影响。结果表明，这些参数在金纳米粒子的合成中起着重要的作用。

关键词：纳米金；绿色方法；粉末技术；纳米晶材料

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