

Green Synthesis of Nano-silver Particles Using Plant Active Substance from Lemongrass Extract

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Biosynthesis of nanoparticles by plant extracts is currently under exploitation. Plant extracts are very cost effective and eco-friendly and thus can be an economic and efficient alternative. This study investigates the mechanism of preparation of nano-silver using a plant active substance from lemongrass extract. The lemongrass ethanol extract was prepared using an ultrasonic cell crusher. Spherical silver nanoparticles were prepared with lemongrass extract and silver nitrate. The reaction mechanism of the preparation of nano-silver by plant extracts was analyzed by infrared and X-ray photoelectron spectrometer (XPS) measurements. The uniform and stable spherical silver nanoparticles with an average particle size of 22.99 nm were synthesized. Amide compounds in plant extracts may act as reductants and protective agents. The biomass of plants produces their nanomaterials through a process called biomineralisation. Thus, plant active substances can be used widely, and biological methods are completely feasible and worth studying for the chemical procedures, which are environmentally friendly and convenient.

Keywords: Lemongrass extract; Silver nanoparticles; X-ray photoelectron spectrometer

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INTRODUCTION

Nano-silver particles show many unique physical and chemical properties due to their surface effect, quantum size effect, and macroscopic quantum tunneling effect (Ball and Garwin 1992). In recent years, nano-silver particles have attracted much attention because of their excellent conductivity, oxidation resistance, and low temperature sintering performance (Wang *et al.* 2007; Park *et al.* 2008). They are widely used in thick film printed circuits, internal electrodes, and sensors in multi-layer ceramic capacitors (Lin and Wang 1996; Zhang *et al.* 1996). In addition, nano-silver particles are also used as antibacterial materials (Lee *et al.* 2003), biosensor materials (Shaik *et al.* 2017), and low-temperature superconducting materials (Hirano *et al.* 2003). Therefore, it is of great significance to study the preparation and application of nano-silver particles.

Nano-silver particles can be prepared through chemical reduction (Chou and Ren 2000; Pastoriza-Santos and Liz-Marzán 2002; Sun and Xia 2002; Khanna *et al.* 2005b; Ryu *et al.* 2005), electrochemical method (Rodríguez-Sánchez *et al.* 2000; Yin *et al.* 2003), electron beam evaporation deposition method (Korchagina *et al.* 2005), photocatalytic reduction method (Khanna *et al.* 2005a), magnetron sputtering (Xionget *al.* 2000), electrochemistry (Zhou *et al.* 2006), supercritical fluids (Chang *et al.* 2003), γ -ray method (Biswal *et al.* 2009), green synthesis method (Naik *et al.* 2002), seed media (Tian *et al.* 2007), microemulsion (Xie *et al.* 2006; Zhang *et al.* 2006), and laser melting method (Tsuji *et al.* 2003). Each method has advantages and disadvantages. In these methods, green

synthesis is not only a good way to produce favorable nanostructured materials, but also to reduce the production of harmful substances in human health and the natural environment (Chandran *et al.* 2006).

Silver nanoparticles can be synthesized using plant extracts. Geetha *et al.* (2012) prepared silver nanoparticles of 40 nm by heating a mixture of native Indian grasses and silver nitrate solution at 70 °C. Silver nanoparticles with different particle sizes under different reaction conditions have been prepared by the reaction of chestnut leaves extract and silver nitrate (Dan 2009). Manish *et al.* (2009) synthesized 50 to 150 nm silver nanoparticles by a eucalyptus methanol solution and silver nitrate. Chandran *et al.* (2006) prepared triangular gold nanoparticles and spherical nanosized silver particles using aloe extract. Song and Kim (2009) prepared silver nanoparticles of about 32 nm using magnolia, Platanus, pine, and *Ginkgo biloba* leaves.

Lemongrass is widely produced in the southern China subtropical region. It has a high survival rate, low price, fresh lemon flavor, and antibacterial properties. Some researchers earlier had successfully prepared the nano-silver particles by using *Cymbopogon citratus* (lemongrass) (Shalaka *et al.* 2011), but they had used very different experimental conditions in their method. It has been shown that using starch as a protective agent can make the prepared nano-silver particle size smaller, more stable, and more uniform. Compared with previous reports, this method is time-saving and effective.

This paper focuses on using lemongrass active extract to prepare nano-silver particles (Ajayi and Afolayan 2017). The effects of the amount of active substance and the soluble starch were examined. The synthesized nanoparticles were measured by ultraviolet-visible (UV-vis) spectroscopy, transmission electron microscopy, Malvern nano-size and potential analyzer, and X-ray photoelectron spectrometry.

EXPERIMENTAL

Materials and Equipment

Analytical grade sodium hydroxide (NaOH) (Tianjin Futong Chemical Reagents Factory, Tianjin, China), analytical grade silver nitrate (AgNO₃) (Sinopharm Chemical Reagent Co., Ltd., Beijing, China), analytical grade ammonia water (Zhejiang Sanying Chemical Reagent Co., Ltd., Lanxi, China), analytical grade anhydrous ethanol (Sinopharm Chemical Reagent Co., Ltd., Beijing, China), and analytical grade soluble starch (Shanghai Aladdin Biochemical Technology Co., Ltd., Shanghai, China) were used. Lemongrass was procured from crop ground of Xishuangbanna in Yunnan province.

The following laboratory equipment was used for this study: FZ102-type plant crusher (Beijing Zhongxing Albert Instrument Co., Ltd., Beijing, China), AL204-type electronic balance (Mettler-Toledo International Trading (Shanghai) Co., Ltd., Shanghai, China), DELTA320-type pH meter (Mettler-Toledo), TG16-W-type centrifuge (Shanghai Huyeming Technology Instrument Co., Ltd., Shanghai, China), ZEN3690-type Malvern nano-size and potential analyzer (Malvern Instruments, Ltd., Malvern, UK), DHG-type hot air drying oven (Shanghai Yiheng Technology Instrument Co., Ltd., Shanghai, China), and XH100B-type microwave catalytic synthesis/extract instrument (Beijing Cheung Swan Technology Development Co., Ltd., Beijing, China).

Preparation of Lemongrass Extract

Three grams of washed and dried lemongrass was crushed into a powder and mixed with 70 mL 70% ethanol water solvent. The mixture was sonicated at 40 °C for 20 min, using a reaction power of 350 W. Then it was subjected to suction filtration to obtain a crude extract, and condensed into a sticky state by rotary evaporator, and freeze-dried to prepare the lemongrass extract powder.

Preparation of Silver Nanoparticles

Lemongrass extract powder (0.4 g) was dissolved in 20 mL of aqueous NaOH solution prepared with 0.8 mg of solid sodium hydroxide (NaOH), to provide an alkaline environment, and the pH was adjusted to 11.0. The prepared extract solution was placed in a microwave reaction apparatus and heated at 75 °C; the power of the device was 700 W; 1.2 mmol silver nitrate and 25% dilute aqueous ammonia were placed in a silver ammonia solution. Then a mixture of silver ammonia solution and 10 mL of 1% starch solution was prepared (the soluble starch is amylose and can act as a protective agent in the process of synthesizing silver nanoparticles). The mixture was injected into the lemongrass extract solution when the temperature of the extract solution reached the set temperature. The reaction was carried out for 3 min. The color of the solution changed from yellow to pale brown. The nano-silver solution was prepared.

Analytical Methods

UV-VIS characterization of silver nanoparticles

Deionized water was measured in a UV-2100-type UV-VIS spectrophotometer (Unike (Shanghai) Instrument Co., Ltd., Shanghai, China) as a control. A sample of diluted nano-silver aqueous solution was placed in the sample cell for measurements.

X-ray diffractometry (XRD)

After centrifugation of the nano-silver solution at 10000 rpm, the obtained black powder was dried. The powder was tested in a D8 ADVANCE X-type X-ray diffractometer (Bruker, Karlsruhe, Germany) under the following conditions: copper target, incident wavelength of 0.15418 nm, Ni filter, tube pressure of 40 KV, tube flow of 40 mA, scan step size of 0.02°, scanning speed of 19.2 s/step, slit DS of 1°, and RS of 8 mm (corresponding to LynxExe array detector).

Transmission electron microscopy (TEM)

The dispersed nano-silver solution was dripped onto a copper mesh. The samples were dried for 1 min before observation under a Philips Tecnai10 transmission electron microscope (Amsterdam, Holland).

Malvern nano-size and potential analyzer

The Malvern nano-size and potential analyzer can determine the amount of different diameter particles in the solution by measuring the amount of light scattered at different angles in the solution in the case of incident light, whereby the particle size of the prepared nano-silver particles size has a rough judgment. After setting the test conditions in the software, the nano-silver solution was diluted several times and placed in the Malvern particle size tester for testing.

Fourier transform infrared spectroscopy (FTIR)

The nanometer silver powder and the extract powder were mixed with potassium bromide powder. The mixture was ground and pressed into a sheet. The sample was placed in a Bruker TENSOR27 apparatus (Karlsruhe, Germany) for testing.

X-ray photoelectron spectrometry (XPS)

An AXIS Ultra DLD X-ray photoelectron spectrometer (Kratos Co., Manchester, Britain) was used. The chamber was operated at a vacuum of $\sim 5 \times 10^{-9}$ torr. The X-ray source was a monochromatic Al K α source (Mono AlK α) with an energy of 1487.7 eV and 5 mA \times 15 kV, with a beam spot size of 700 μ m \times 300 μ m. The scanning mode was CAE, with a full-spectrum scan of 170 eV and narrow-band scan of 40 eV. The number of scans was 1.

RESULTS AND DISCUSSION

Characterization of Nano-silver Samples

UV-VIS analysis

According to Mie scattering theory, the peak in Fig. 1 appeared at 416 nm, which belongs to the band of the characteristic peak of ultraviolet spectroscopic spectrum of the spherical nano-silver particles (Ahmad *et al.* 2003). This symmetrical curve shows that the synthesized nano-silver particles had good dispersibility. After 30 days, there was no obvious agglomeration, indicating that the synthesized nano-silver particles were stable.

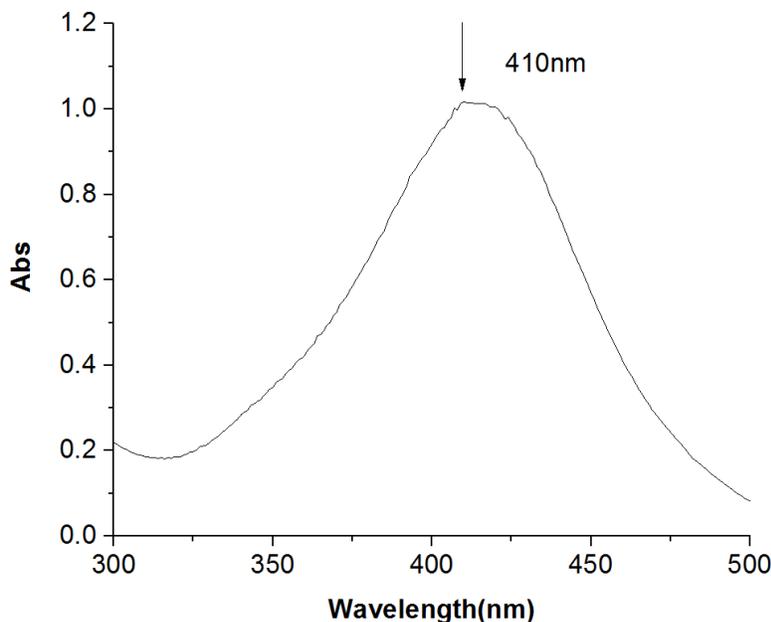


Fig. 1. Ultraviolet spectra of silver nanoparticles

XRD analysis

As shown in Fig. 2, the five principal peaks were 38.1°, 44.2°, 74.4°, 77.4°, and 81.5°, which correspond to the five peak positions of the Ag spherical particles coinciding with elemental silver XRD pattern (JCPDS, NO 04-0783). These characteristic peaks correspond to 111 au, 200 au, 220 au, 311 au, and 222 au, respectively, which shows that

the nanoparticles prepared in this paper had the same face-centered cubic crystal structure as previously reported (Janardhanan *et al.* 2009). The width of the peak is related to the size of the nanoparticle (Prasad *et al.* 2006). It is further illustrated that the product obtained by this environmentally friendly method was pure crystal silver.

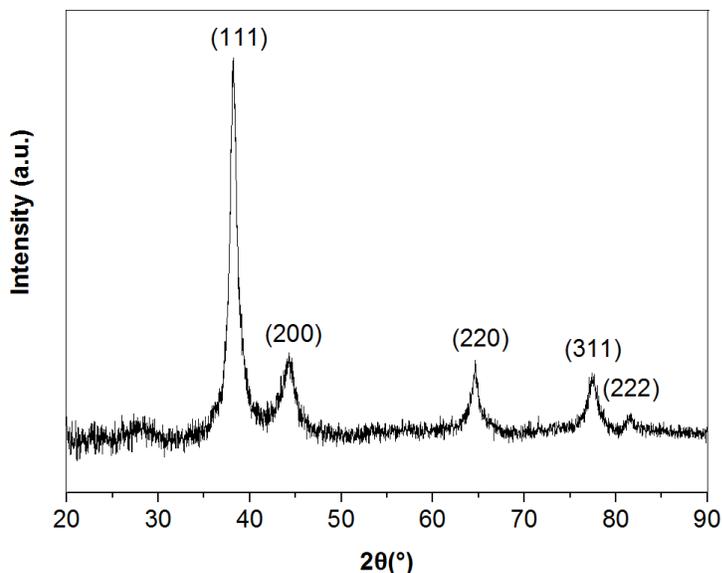


Fig. 2. X-ray diffraction pattern of silver nanoparticles

TEM analysis

Figure 3 shows the result of field emission transmission electron microscopy of the synthesized silver nanoparticles. The Fig. 3(a), (b), (c) and (d) shows the morphologies of the spherical nano-silver particles under 200 nm, 50 nm, 100 nm, and 20 nm scale, respectively.

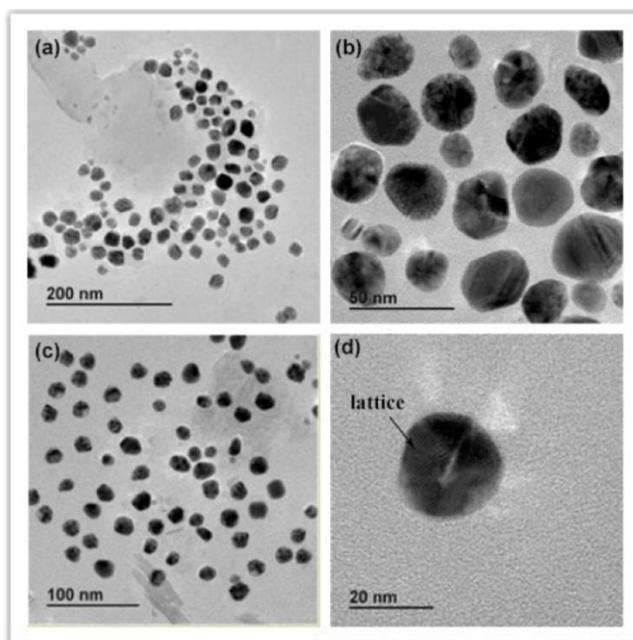


Fig. 3. Field emission transmission electron microscopy

The reaction product consisted of spherical particles with a high crystallinity, and the average particle diameter was 25 nm. The individual particles less than 10 nm had spherical shapes (Fig. 3), while the particles in the range from 20 nm to 30 nm were pentahedral or decahedral strictly, such that they could be called multiply twinned particles. The synthesized nano-silver particles were isotropic in shape and had a low aspect ratio. The results are consistent with the result of the symmetrical UV spectra obtained above, reflecting the monodispersity of the silver nanoparticles. Thus, the synthesized nano-silver particles had a high concentration and no agglomeration, indicating that the synthesized silver nanoparticles were stable and uniform in particle size distribution. Moreover, the particle size of the prepared silver nanoparticles was concentrated at 20 to 28 nm, according to results from the Malvern nano-size and potential analyzer; the average size of uniform and stable spherical nanoparticles was 22.99 nm.

Malvern particle size analysis

As shown in Fig. 4A, the prepared silver particles were concentrated in the distribution range of 20 to 28 nm. There were some very small particles, but not a lot. There were almost no particles in the range of 40 nm or more, showing that the prepared nano-silver particles were relatively stable, and there was no apparent reunion of the larger particles. The calculated average particle size was 22.99 ± 11.01 nm.

In order to observe the stability of the nano silver solution prepared by the lemon grass extract, the sample was stored at room temperature (25 °C) for 90 days to observe whether it had a significant agglomeration. Part B of Fig. 4 shows colorless, deionized water on the left. The right side is the silver nanoparticles-ethanol dispersion after 3 months, which was brown with no obvious precipitation. The silver nanoparticles prepared by the method described in this paper were stable and difficult to agglomerate.

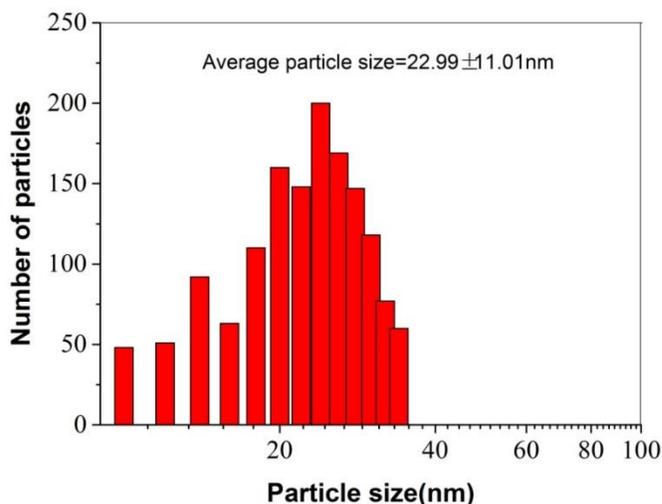
**A****B**

Fig. 4. A: Particle size distribution of spherical silver nanoparticles; **B:** The comparison deionized water and the silver nanoparticles-ethanol dispersion placed 3 months

FTIR analysis

The Fourier transform infrared spectra are shown in Fig. 5. Curve *a* is the infrared spectrum of lemongrass extract. Curve *b* is the spectrum of the mixed solution after the

reaction. There was an absorption peak near 3000 cm^{-1} both in *a* and *b*, which is the stretching vibration absorption of C-H bond belonging to $-\text{CH}_2-$ and $-\text{CH}_3-$. There was an absorption peak at 1577 cm^{-1} in curve *a*, which is attributable to the bending vibration of the N-H bond and it weakened in curve *b*. The absorption peak at 1395 cm^{-1} in curve *a* is ascribed to the C-N stretching vibration. The absorption peak was shifted to 1371 cm^{-1} in the infrared spectra *b*, indicating that the amino group played an important role in the whole reduction process. The C-O bond was obtained at 1156 cm^{-1} and the C=O bond was 1648 cm^{-1} in *b*, indicating that carboxylate ions were present in the reaction product. Carboxylate ions and silver ions may also occur in Ag-O chemical bonding reactions. There was a strong absorption peak near 1028 cm^{-1} in curve *a*, which belongs to the vibration absorption of -OH in the primary alcohol, which indicates that the soluble starch macromolecules may have been coated on the surface of nano-silver particles and were acting as a protective agent.

Through the infrared analysis of the lemongrass extract, it can be concluded that most of the lemongrass extract is amide compounds, which may be derived from the macromolecules such as proteins and peptides in plant cells. The amino group were hydrolyzed in alkaline environment under heating conditions: $\text{RCONH}_2 + \text{OH} \rightarrow (\text{heating}) \text{-RCOO-} + \text{NH}_3\uparrow$.

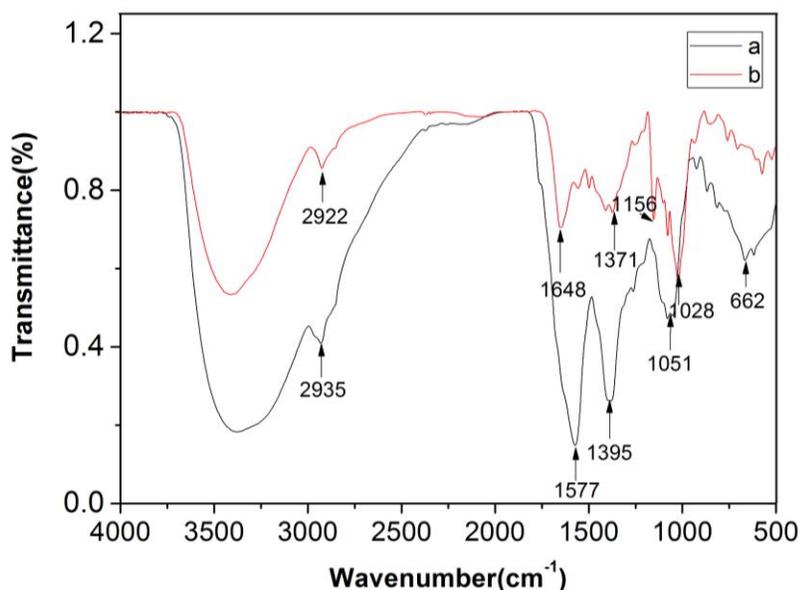


Fig. 5. Infrared spectra of lemongrass extract and reaction products

XPS analysis

As shown in Fig 6, the reduction reaction products were mainly constituted by the three elements carbon, oxygen, and silver. The XPS peak software was employed to divide the resulting spectrum into the peaks. Figure 7 shows the binding energies of two obvious peak positions of silver nanoparticles are 374.27 eV and 368.20 eV , corresponding to $3d_{3/2}$ and $3d_{5/2}$ binding energy. These positions are as same as the positions of the standard Ag, indicating that the prepared nano-silver existed in the form of crystals. The binding energy of peak 1 was 368.60 eV , which belonged to the $3d_{5/2}$ binding energy of the silver element.

This result indicates that this part of the silver element came from the silver-containing conjugates, confirming that some silver nanoparticles were bound with a protective agent and an active substance from the reaction system. Peak 2 corresponds to the silver element $3d_{5/2}$ binding energy of 368.04 eV, indicating that part of the silver element was constituted as AgO. There may be a small part of the silver by air oxidation in the high-temperature reaction process. Peak 3 corresponds to the $3d_{3/2}$ binding energy of the silver element, which indicates that most of the synthesized material was elemental silver.

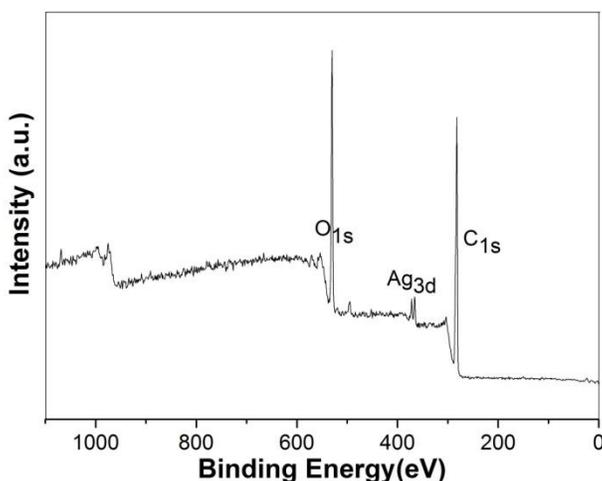


Fig. 6. X-ray photoelectron spectroscopy of silver nanoparticles

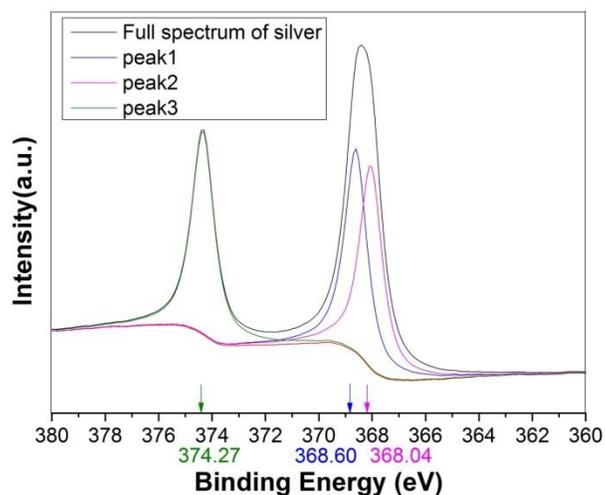


Fig. 7. XPS silver spectrum of reaction product

CONCLUSIONS

1. The spherical nano-silver particles were prepared using ethanol extracts of lemongrass and silver nitrate by microwave heating. This approach is time-saving, low in energy and resource costs, and environmentally friendly.

2. Through the analysis of XPS and infrared spectra, it was presumed that the amide compounds in the plant extracts played a role as reductant during the course of the reaction.
3. The particle size of the prepared silver nanoparticles was concentrated at 20 to 28 nm, and the average size of uniform and stable spherical nanoparticles was 22.99 nm. It is worthwhile to study whether preparation of nano-silver by lemongrass extract can be put into industrial production in large quantities.

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