ORIGINAL RESEARCH ARTICLE

Preparation and Properties of Silver Nanoparticles

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ABSTRACT

With the progress of science and technology, the research and development of silver nanoparticles has also developed. This paper attempts to prepare a silver nanoparticle by electrolyzing AgNO3 solution with electrochemical reduction method and citric acid as a complexing agent in a certain current and time. The crystal morphology and sample purity of silver nanoparticles were analyzed by X-ray diffractometer. The crystal structure of the nanoparticles was analyzed by scanning electron microscopy (SEM). The crystal structure of the nanoparticles was analyzed by X-ray diffraction. The particle size distribution of the particles was in the range of 125-199 nm, and the carbon paste electrode was modified with the prepared silver nanoparticles. The electrocatalytic activity of the carbon paste electrode was preliminarily explored.

KEYWORDS: silver nanoparticles; electrolysis; preparation; characterization

1. Introduction

1.1. Overview of nanoparticles

In the 21st century, the rapid development of nanotechnology has become a new industry. It has a huge market potential in many areas with a wide range of applications, closely related to people's life and production and it has unexpected potential. The particle size of the nanoparticles is usually between 1 and 100 nm, also called ultrafine particles. Studies of nanoparticles have shown that nanoparticles should have some novel physical and chemical properties. There are differences in the structure of the nanoparticles and the macroscopic objects, which are manifested in the large surface area, while the outer atoms are neither short nor longer. It is presumed that the atoms in the nanoparticles are arranged in an orderly manner, and that the surface atoms are more biased towards the gas state. Even so, as the external curvature, small particle size, the internal Gibbs pressure is high, the internal structure of a deformation. Its unique micro-structure to make it with excellent performance. Nanoparticles have the characteristics of small size effect, surface effect, macroscopic quantum tunneling effect and quantum size effect. They have the characteristics of macroscopic materials such as conductive properties, photoelectric properties and photocatalytic ability. They are widely used in various luminescence and display Device [1].

1.2. Application of Nanoparticles

Nano-particles external activation of the core, is the preparation of excellent catalyst for raw materials. At present, you can directly use nano-particles in the polymer oxidation, reduction and synthesis reactions in the catalyst such as platinum black, silver and so on. The use of nano-particles as a catalyst to react, can significantly increase the reaction efficiency, such as rocket fuel combustion reaction using nano-nickel powder as a catalyst, can increase the combustion efficiency of 100 times [2]. The catalytic reaction of the nanoparticles is also selective. For example, when the propionaldehyde is oxidized with nano-nickel as the catalyst, the particle size of the nickel particles has a great influence on the reaction. When the reaction is less than 5 nm, the reaction direction is more biased towards the formation of alcohol, and the reaction of the decomposition aldehyde [3].

Some materials are usually sintered at high temperatures, such as silicon carbide, tungsten carbide, high alloy, etc., but in the nanometer state at a lower temperature can be sintered to get high-density sintered body, which benefited from the volume effect of nanoparticles. On the other hand, if the active agent is used in the sintering process, the sintering process can be accelerated, the sintering temperature is lowered, the sintering time is shortened, and the nanoparticles can be used as an active agent. For example, adding 0.1% nano-nickel powder in the tungsten powder, the sintering

temperature can be reduced to 12000-1300 ℃, and under normal circumstances tungsten powder at 3000 ℃ high temperature sintering [4].

The sintering of the composite material is difficult for the sintering of the composite material because of its different melting point and different phase transition temperature. The surface effect and volume effect of the nanoparticles can be carried out at low temperature. The decomposition reaction reduces the melting point and the phase transition temperature, so that the composite material with good sintering performance can be obtained [5].

Pure nano-powder materials can be used to make fine ceramics. Ceramic made with these nanomaterials has the function of converting energy and transmitting information, and has the ability to wear, hard, high temperature and corrosion resistance. In addition, it can also be used as infrared absorption materials, such as Cr-based alloy nanoparticles to absorb infrared, the effect is good [6].

There are many applications of nanomaterials in medicine and bioengineering. 'Biological missile' drug technology has been successfully developed [7], which is a nano-magnetic materials for drug carrier targeted drugs. That is, carrying the drug on the surface of the protein, and then coated with magnetic iron oxide nanoparticles, injected into the human blood vessels, and then the role of the magnetic field under the action of drug-oriented movement can be direct lesions, reducing the drug on the human organs produced side effects, improve the treatment effect. Nano-sized nano-sensors can be used to obtain a variety of human body electrochemical information and biochemical reactions of information. You can also use nanotechnology developed into a robot, into the blood of the body, spread to the body, you can conduct a thorough examination of the human body, but also to clear the human brain thrombosis, remove the patient's arterial fat deposits, and even remove the virus, kill cancer Cells, etc., the study of nanotechnology on human medical technology is of great significance. It is predicted that the development of nanotechnology and the development of functional materials will develop rapidly in the future. More and more new nanometer materials will be widely used in many high-tech fields.

1.3. Overview of silver nanoparticles

Silver nanoparticles refers to the fine particles of silver atoms consisting of 1 to 100 nm. Studies have shown that the physical and chemical properties of materials are greatly altered when nanosized silver is mosaic on different materials. At present, people interested in silver nanoparticles research interest, promising.

Silver nanoparticles have very excellent performance, mainly in the information technology, physical components, chemical products, environmental protection and many other aspects, is a high-tech materials. Now widely used in ceramic building materials, medical care, environmental protection and coatings, researchers have paid great attention to the research and preparation of silver nanoparticles [8]. Chen water and so on [9] people found that the load of silver particles of activated carbon fiber has a strong bactericidal ability, so the load of silver prepared into silver nanoparticles, the reactivity of the material that is a substantial increase in bactericidal activity, because this do a substantial increase in surface area, increase the proportion of surface atoms. It has always been that the main components of ethylene epoxidation catalysts are silver and the effective way to improve the efficiency of the catalyst is to reduce the particle size. Therefore, it is very important to study the preparation of silver nanoparticles with smaller particle size to improve the performance of the catalyst. Nano-silver can also improve the methane selective reduction of NOx catalyst activity. The addition of an appropriate amount of silver nanoparticles to insulators and semiconductors gives them good optical properties and is suitable for the manufacture of optoelectronic devices [10].

The conductivity, catalytic efficiency and bactericidal ability of silver nanoparticles are affected by their particle size, so it is necessary to precisely control the particle size of silver nanoparticles. In addition, in recent years, the structure and properties of self-assembled and ordered assembled films of silver nanoparticles have become the focus of people's research. Among the most interesting ones are silver nanoparticles or semiconducting silver with uniform size distribution and smaller particle size Nanoparticles are assembled into ordered superlattices and investigated for their photoelectric properties [11]. The photoelectric properties of the solid composite film are affected by the size of the nanoparticles and the distance between the particles, so the research on this is of great significance.

1.4. Preparation of silver nanoparticles

Over the past decade, nanotechnology has flourished, and various physical methods and chemical methods for preparing nano silver particles have been endless. The focus of this field is to develop silver nanoparticle preparation methods that are less costly, more efficient, and more efficient and have large-scale industrial production prospects. One of the physical methods [12] have high-energy mechanical ball mill method, light method, evaporation condensation method. The simplicity of the principle is the advantage of the physical method, but its production cost is high, the requirements of the instrument device is also very high, in the silver nanoparticle size and shape of the requirements of the industrial preparation is not applicable. In the high performance requirements of nano-particles in general chemical synthesis of silver nanoparticles, such as optical, electrical and biomedical. It is the key technology to prepare the silver

nanoparticles by controlling the particle size of the particles, the smaller particle size distribution and the fabrication of specific, single and uniform crystal structures. Chemical preparation methods are photochemical reduction method, liquid chemical reduction method, electrochemical reduction method, microemulsion method, chemical precipitation method, sol-gel method and alcoholysis method. In recent years, the development of a new method of electrochemical synthesis of nanoparticles, Shen Mingming et al [13] by controlling the current density using electrochemical method to prepare a dumbbell, spherical and rod-shaped silver nanoparticles, B raun et al [14 The silver nanoparticles were synthesized by electrochemical synthesis of silver nanowires, and the silver nanoparticles were synthesized by electrodeposition. The effects of potential on the morphology of the particles were studied. Zhu et al. [16] synthesized the semiconductors by ultrasonic electrochemistry PbSe nanoparticles. The current electrochemical method is an effective means to synthesize nanomaterials because of its advantages of simple, rapid, non-polluting and high efficiency [17].

1.5. Research status quo

Gengtao et al. [9], 'Preparation and characterization of silver nanoparticles,' states that they prepared silver nanoparticles with ethylene glycol reduction under solvothermal conditions and characterized by X-ray diffraction (XRD) and transmission Electron microscopy (TEM). It was concluded that the nano-silver particles prepared by the hydrothermal reduction method of ethylene glycol had a face-centered cubic phase polycrystal structure with an average particle size of about 50 nm, mainly in the form of spherical granules. The agent played a very good dispersion.

Yang Biwen et al [18] with tyrosine as a reducing agent and stabilizer, in 60 ℃ constant temperature water bath under alkaline conditions to restore silver nitrate, reaction 20 min, successfully prepared silver nanoparticles. In the process of the formation of silver nanoparticles, the color of the mixed solution changed from pale yellow to brown, this phenomenon shows the formation of fine particles of silver. The UV-Vis and UV-Vis absorption spectra (TEM) were used to characterize the obtained product. The results showed that the UV absorption peak of the particles was near 412 nm, and the diameter of the silver nanoparticles At 15 - 25 nm, the shape is approximately spherical.

Sun et al. [19] used silver borohydride to reduce silver nitrate to prepare smaller silver nanoparticles. The silver nanoparticles were characterized by UV-Vis absorption spectroscopy (Uv-Vis), scanning electron microscopy (SEM) and cyclic voltammetry (CV). The results show that the size of silver nanoparticles is about 10 nm and can be assembled on the surface of conductive glass in the form of sub-monolayer. CV graph shows that silver nanoparticles have a pair of asymmetric redox peaks, and the concentration of nanoparticles can affect redox Potential.

Gu Daming [20] and so on in the pH = 1 - 2, temperature 40 - 42 ℃ under the conditions of sodium hypophosphite to reduce the silver nitrate solution to get purple silver paste, dried powder products. The results show that the size of the prepared silver nanoparticles is between 10 and 30 nm by TEM and XRD. The preparation cycle of this method is about 5 h, the yield can reach 70% - 80%.

Wang et al. [21] et al. Used EDTA as a ligand to prepare spherical nano-silver particles with different particle sizes by electrochemical method. They were characterized by XRD, TEM and UV-visible spectroscopy. The results show that the concentration of AgN03 solution is different, and the shape and size of the prepared silver nanoparticles are different.

Liao Xuehong [17] and so on with N'-hydroxyethylethylenediamine-N, N, N'-triacetic acid as the complexing agent to prepare dendritic nano silver by X-ray, and XRD and TEM were carried out on the nanoparticles. Characterization and discovery of the presence of ligands is the key to the formation of nanoparticles, and electrochemical method is an excellent method for the preparation of silver nanoparticles, simple and efficient pollution. At the same time, they were prepared by using EDTA as complexing agent and AgNO3 solution as raw materials. The silver nanoparticles with different spherical and dendritic shapes were successfully prepared by ultrasonic electrochemical method and characterized by XRD and TEM. Zhang Yunhong [22] and so on in 8 - 14 layer of silver stearate silver L-B film, the electrochemical method of preparation of nano-scale ultra-fine silver particles, spherical nano-silver particles detected diameter between 2-3 nm.

1.6. Research content

This method of preparation of silver nanoparticles is based on the solution of silver ions in a certain electrochemical window, can occur redox reaction, and was reduced to silver atoms. Under the specific potential, select the appropriate reaction conditions, the price of silver ions can be reduced to zero valence silver. In the electrochemical reaction carried out at the same time, the electrolyte exists in the stabilizer (the experiment is citric acid), the resulting protection of silver atoms isolated, can form dispersed silver nanoparticles. The basic principle is:

Anode 4OH - 4e - O2 ↑ + 2H2O

Cathode 4Ag ++ 4e- + Stabilizer 4Ag / Stabilizer

Total reaction 4AgNO3 + 2H2O 4Ag / stabilizer + O2 ↑ + 4HNO3

After the silver nanoparticles were synthesized, they were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and nano-particle size analyzer (C-V).

2. The experimental part

2.1. Experimental drugs

Drug	manufacturers
Silver nitrate (AgNO3, A. R. grade)	Sinopharm Group Chemical Reagent Co., Ltd.
Citric acid (C6H8O7 · H2O, A. R. grade)	Sinopharm Group Chemical Reagent Co., Ltd.
Potassium nitrate (KNO3, A. R. grade)	Sinopharm Group Chemical Reagent Co., Ltd.
Anhydrous ethanol (C2H5OH, A. R. grade)	Sinopharm Group Chemical Reagent Co., Ltd.
Ultra - pure water	Shanghai and Thailand Instrument Co., Ltd
High - purity graphite rods	China's new materials in Jiangsu Science and Technology Co., Ltd

2.2. Experimental apparatus

Equipment	manufacturers
Desktop High Speed Centrifuge (H1850)	Xiangyi Centrifuge Instrument Co., Ltd
Electronic Balance (AL204)	METTLER TOLEDO Instruments
Electrochemical workstation (CHI660C)	Shanghai Chen Hua Instrument Company
Electric thermostatic blast oven (DHG-9140A)	Shanghai Jinghong Experimental Equipment Co., Ltd.
Ultrasonic cleaning device (SK1200E)	Shanghai Branch guided ultrasound Instrument Co., Ltd
Nano-laser particle size analyzer (BT-90)	Dandong City Baxter Instrument Co., Ltd.
X-ray diffractometer (XRD-7000)	Shimadzu Corporation
Scanning Electron Microscope (S-3000N)	Hitachi (HITACHI)

2.3. Experimental steps

2.3.1 Preparation of silver nanoparticles

Use a balance to accurately weigh 4g of citric acid (as a complexing agent) and 0.2g of AgNO3 in a beaker. Measure 100ml of ultrapure water with a graduated cylinder until fully dissolved (adding a small amount of KNO3 to enhance the conductivity of the solution) to form an electrolyte. It is important to note that the beaker used in the experiment needs to be repeatedly washed with ultrapure water so as not to adhere to Cl - and Ag + to form AgCl. The electrode system is a graphite rod-graphite rod (8mm) double electrode system, the two-electrode system line connection for the working electrode caught in a graphite rod, the electrode and the reference electrode together with another graphite rod on the beaker for the electrolytic cell. Can be added in a beaker cup lid, on the one hand to catch the electrode to avoid shaking the impact of silver nanoparticles precipitation, on the other hand to prevent impurities fall into the pollution of the electrolyte.

The electrochemical workstation was used as the power source and was operated at a constant current of 25 mA for 40 min using the chronopotentiometry mode of operation. After the start of electrolysis need to pay close attention to the phenomenon of the electrolytic cell, can be observed on the working electrode black fluffy material generation, with the passage of time gradually grow up, the electrode has a small bubble evenly (for oxygen). After the electrolysis is completed, turn off the electrochemical workstation, carefully remove the graphite rod, placed in a clean beaker with ultra-pure water rinse the finished silver completely off, the beaker placed in the ultrasonic cleaner in the shock so that the silver particles fully dispersed to facilitate the washing. Take the silver suspension in a centrifuge tube and centrifuge for 15 min using a high-speed centrifuge (6500 r / min). Remove the supernatant and separate the precipitate again with water and ethanol. After the precipitate was washed and washed, it was placed on a surface dish and dried in an oven (40 ° C) for 12 h to obtain a product and the product was collected and stored.

2.3.2 Characterization of silver nanoparticles

Characterization of the product: X-ray diffraction (XRD), scanning electron microscopy (SEM) and nano-laser particle size analyzer were used to characterize it.

XRD: The silver nanoparticle powder was uniformly filled on a glass slide, covered and the surface was flattened, and the sample was placed in the instrument. The instrument parameters are set to:

Target = Cu

Voltage = 40kV

Current = 30mA

Scan rate of 2 ° / min, scan range of 10 ° - 80 °, set up after the start scan

SEM characterization: the first silver nanoparticle powder shocks fully dispersed, and then use a toothpick to pick a small number of samples applied to the stage, so that the powder paved evenly. After the preparation of the sample is placed in the instrument, respectively, in the magnification of 10,000 times and 20000 times the case to observe the camera.

Nano-laser particle size analyzer: Since the sample is powder, it must be pretreated to be tested. Weigh 0.05 g of silver nanoparticles in a 5 ml plastic capsule, and then add 5 ml of ultra-pure water to the capsule, close the capsule and place it in an ultrasonic cleaner for 30 min after ultrasonic shock until the silver particles are fully diffused to obtain silver Nano particle suspension. The resulting suspension was transferred to a quartz cuvette and tested.

2.3.3 Electrocatalytic activity of silver nanoparticles

1). Take 0.02 g of graphite powder in the surface dish, add 5 μL of liquid paraffin mixed evenly, grinding to paste, filled into the PTFE tube, filling to ensure that the mixture is empty and smooth contact surface, at the other end A copper paste was inserted as a conductor to obtain a carbon paste electrode.

2) Add 1 ml of liquid paraffin to 0.002 g silver nanoparticle powder, encapsulate the mixture in a closed container, and place it in an ultrasonic cleaner for 30 min to prepare a uniform silver nanoparticle suspension. Take 0.02 g of graphite powder in the surface dish, with a pipette to take 5μL suspension in the graphite powder mixed evenly, grinding to paste, filled into the PTFE tube, the same filling to ensure that the mixture is empty The contact surface is smooth and the copper wire is inserted into the copper wire as the conductor at the other end to prepare the carbon paste electrode modified by silver nanoparticles.

3). The electrode was immersed in potassium ferrocyanide solution and the starting potential of the two electrode reactions and the current through the electrode were measured by cyclic voltammetry respectively. The data were recorded and plotted.

The C-V parameter is set to: starting voltage -0.2V, maximum voltage 1V, minimum voltage -0.2V, scanning rate 0.05V / s, sensitivity 10-4A / V.

3. The experimental results and discussion

The prepared silver nanoparticles are gray-black solid powders

3.1. Characterization of X - ray Diffractors

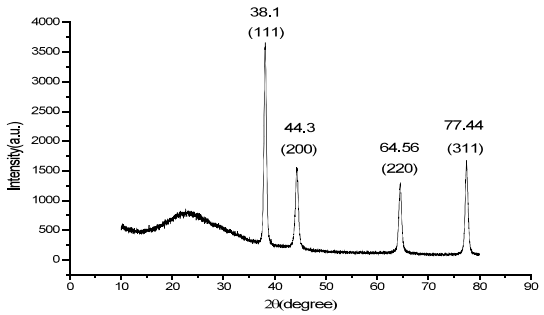


Fig 1 XRD diffraction of the Silver Nanoparticles

X-ray diffraction measurements can be used to confirm the presence of silver nanoparticles and to estimate their grain size [23]. It can be seen from Fig. 1 that the four diffraction peaks on the curve appear at 2θ = 38.1 °, 44.3 °, 64.56 ° and 77.44 °, respectively. (111), (200), (220) and (311) planes, respectively, which can be judged to be silver crystals, and the peaks are clear and pure Other peaks, indicating that the prepared samples were cubic-structured single-phase silver nanoparticles without any other impurities. The average grain size of the silver nanoparticles can be calculated using the X-ray diffraction peak Scherrer formula:

$D = k\lambda / b\cos 2\theta$

D is the average thickness of the crystal grains perpendicular to the crystal plane (nm)

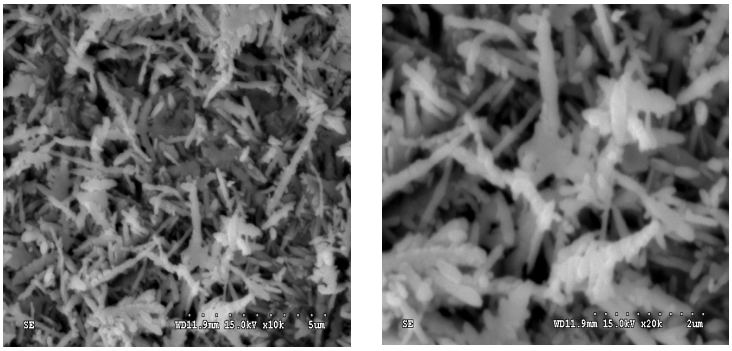
K is the Scherrer constant, if b is the half width of the diffraction peak, k = 0.89; if b is the integral height of the diffraction peak, then k = 1

Λ is an X-ray wavelength (Cu target) of 0.154056 nm

B is the diffraction peak of the measured sample

Θ is the diffraction angle, reflected at 2θ = 38.1 °, 44.3 °, 64.56 ° and 77.44 °. The average particle size of the grains is 21.2 nm, which is 18.5 nm, 17.2 nm, 25.4 nm and 23.7 nm, respectively.

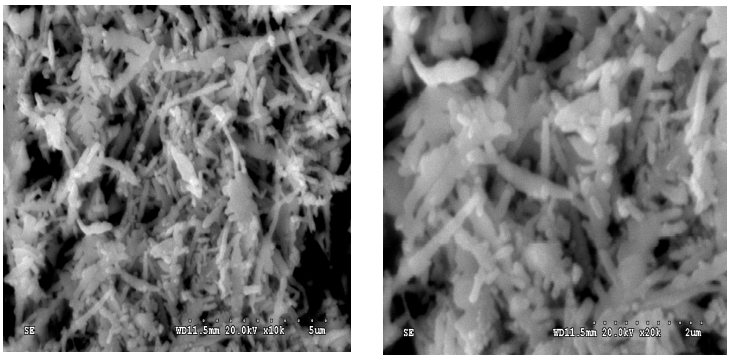
3.2. Scanning electron microscopy (SEM) characterization



A B

Figure 2 silver nanoparticle SEM image of the first group (Figure A for the amplification of 10k times, Figure B for the amplification of 20k times)

Fig. 2 The first group of the SEM image of the silver nanoparticles (Figure A is Magnified 10K times, and the B is Magnified 20K times).



C D

Figure 3 SEM chart of silver nanoparticles, the second group (Figure C for the amplification of 10k times, D Figure for amplification 20k times)

Fig 2 The second group of the SEM image of the silver nanoparticles (Figure C is Magnified 10K times, and the D is Magnified 20K times).

Figure 2 is a freshly prepared silver nanoparticle SEM image, Figure 3 for the preparation is completed, placed a week after the shot of the SEM.

It can be seen that the prepared silver nanoparticles are pineal crystal, the single pattern of the crystal form is symmetrical and the size is uniform and the dispersity is good. Also, compared the four pictures, you can clearly find the newly prepared and placed a week of silver nanoparticles SEM diagram reflects the morphology of the nature of the two are almost identical, there is no significant difference can explain the preparation of this experiment has a good reproducibility and the preparation of the product of stable nature, a certain period of time basically no change. It is estimated that the particle size of the pineal silver nanoparticles is 50-100 nm by comparison with the SEM scale. The coordination agent used in this experiment is citric acid, its existence is very important, is the key to the formation of nanoparticles. Previous studies have shown that silver nanoparticles cannot be produced if an appropriate amount of complexing agent is not added to the AgNO3 solution when the silver nanoparticles are synthesized by electrochemical reduction [17]. I also confirmed this by the control test, in the same experimental environment electrolysis did not join the citric acid AgNO3 solution, can be clearly observed in the graphite rod on the precipitation of silver-white large-size silver element particles, and Not gray black fluffy material. This is mainly due to the addition of complexing agent, the solution there is the following coordination dissociation balance:

Ag + - complexing agent Ag + + complexing agent

Under the action of the complexing agent, the free Ag + in the solution is surrounded by and bound with the complex, and the concentration of Ag + is controlled so that the rate of Ag + reduction on the electrode is indirectly controlled to achieve the control of the generation of silver atoms Rate, the preparation of nano-level silver particles. Since citric acid contains an atom having a strong coordination ability such as a carboxyl group, it can function with Ag +, so that the above object can be achieved.

The shape of the synthesized silver nanoparticles is also related to the structure of the ligands used. Citric acid contains carboxyl, only Ag + electrolysis before the role of coordination, in order to control a certain degree of electrolytic reduction. Dendritic crystals may be the cause of the fractal growth of particles, that is, within a certain range of particles through the diffusion, adsorption process, continuous growth, and finally grow into pine-like crystals. The results show that the crystal types of silver nanoparticles prepared by the use of different complexing agents are very different in crystal form size. Liao Xuehong et al. Use cysteine as the complexing agent, and the silver nanoparticles are spherical.] It can be seen that the use of different types and different structures of the complex agent is to achieve the manual control of silver nanoparticles shape and size of the effective means.

3.3. Nano laser particle size analyzer test

Particle size and distribution area

Particle size nm	125-140	140-158	158-177	177-199
Interval%	16.88	34.56	33.5	15.06
Accumulated%	16.88	51.44	84.94	100

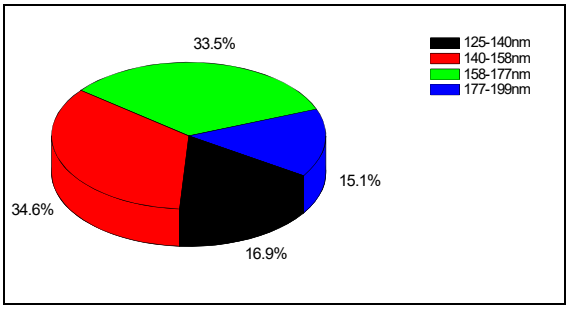


Fig. 4 Size and percentage distribution of Silver Nanoparticles

As a result of the nano-laser particle size analyzer, the average particle size of the prepared silver nanoparticles was 157 nm and the particle size range was 125 nm to 199 nm. The results show that the size of silver nanoparticles is very consistent, the particle size is evenly distributed in a small range, a single crystal, successfully control the particle size and crystal structure, and the product is excellent.

3.4. Electronactivity test results of silver nanoparticles

Cyclic voltammetry was used to scan the two electrodes for cyclic voltammetry, as shown in the following figure

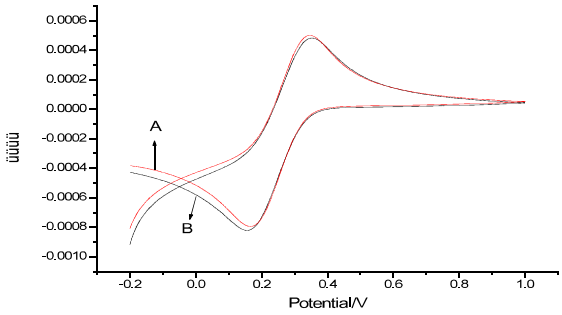


Fig. 5 Comparison of cyclic voltammetry on carbon paste electrode blankcarbon pasteelectrode with silver nanoparticles modified

A is the cyclic voltammetry of the carbon paste electrode modified with silver nanoparticles B is the cyclic voltammogram of the blank carbon paste electrode.

It can be seen from the above figures that the starting potential of the CV curve of the carbon paste electrode modified with silver nanoparticles is lower than that of the blank carbon paste electrode, indicating that the reaction is easier to proceed and the electrode current is larger, indicating that the reaction proceeds the more intense. From this we can speculate that silver nanoparticles have an effect on the electrochemical activity of the carbon paste electrode, which can make it have better electrochemical performance.

But the observation curve shows that before and after the modification of the difference is relatively weak, given the interpretation of persuasive is not very adequate. The electrochemical knowledge and electrochemical experiments are limited. The experiment is only a preliminary exploration of the electrocatalytic activity of silver nanoparticles. Further understanding is still to be studied and studied.

4. Conclusion

In this paper, high purity silver nanoparticles with uniform size, crystal symmetry and particle size of about 100 nm were prepared by electrochemical reduction method and citric acid as agent. AgNO3 solution was successfully prepared by cyclic voltammetry. It is concluded that silver nanoparticles have an effect on the electrode properties of carbon paste electrodes but need to be explored. It is very useful and promising method to prepare silver nanoparticles by electrochemical synthesis method. This method can be extended to the preparation of other metal nanoparticles, which is worthy of attention and sustainable development.

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