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Microwave Synthesis of Au Nanoparticles with the System of $AuCl_4$-CH_3CH_2OH

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Abstract: The Au nanoparticles has been prepared by microwave high-pressure procedure with alcohol as the reducing agent. The color of colloidal Au nanoparticles is blue-violet. The maximum absorption spectrum of colloidal Au is at 580 nm, and the resonance scattering peak is at 580 nm. Using this method, the colloidal Au of long-time stability can be prepared simply and quickly.

Keywords: Microwave high-pressure, alcohol, Au nanoparticles, resonance scattering spectroscopy(RSS).

Au nanoparticles attract keen research efforts due to their unique optical electronic properties[1,2]. The preparation of nanoparticles has important significance in nanochemistry, material science, physics and life science[3]. The technique of microwave has been paid attention by the researchers of chemistry, physics, and materials[4-6]. But the liquid preparation of Au nanoparticles with both microwave high-pressure and reducing agent of alcohol has not been reported to date. As the $HAuCl_4$-CH_3CH_2OH-H_2O systems in the microwave reaction device, the polar molecules in the systems absorb the energy of microwave radiation, and begin to rotate at high-speed. So the temperature of the systems grows up evenly, and the pressure of reaction device increases fast too (maximum to 12 atm). The process helps the oxide-reduce reaction between $HAuCl_4$ and CH_3CH_2OH engaged quickly and evenly. The results indicated that the liquidly Au nanoparticles were in good dispersion and stability. The RSS and absorption spectrum were used to research the $HAuCl_4$-CH_3CH_2OH system.

Experimental

All reagents were of analytical grade used without further purification. All of the solutions prepared with deionized water. The colloidal Au nanoparticles was prepared as follows: 0-2.0 mL 228 μg/mL $HAuCl_4$ solution and 3.0 mL CH_3CH_2OH was put into a 10 mL tested tubes, and diluted to the mark with water. After shaken, it was transferred into a microwave high-pressure reaction device, which was made with polytetrafluoroethylene. Then the reaction device is put into the microcomputer-microwave-oven(Galanz 800W). After irradiating 3 min (480 W), the Au nanoparticles was prepared and the reactor device was taken out, cooled with water. The absorption spectra and the RSS of the Au nanoparticles were obtained by Hitach UV-3400 spectrophotmeter (Japan) and Kyoto RF-540 spectrfluorophotometer (Japan), respectively. The size and the shape of Au nanoparticles (**Figure 1)** were observed by H-600 transmission electron microscopy (Japan).

Figure 1 The TEM for Au nanoparticle

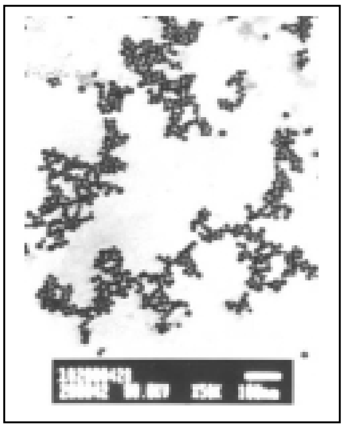
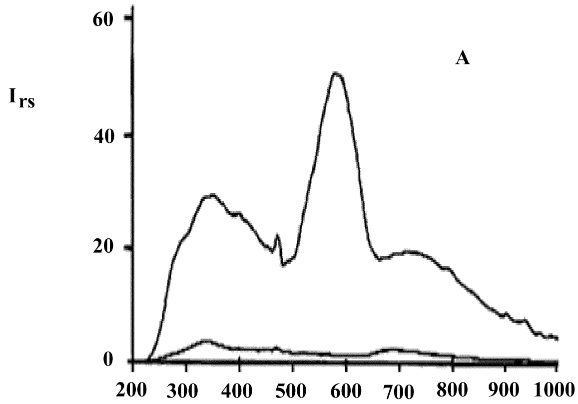


Figure 2 The RLS of colloidal Au



λ / nm
A: 2.32 × 10^{-4}mol/L Au; B: blank

Results and discussion

The results showed that the maximum absorption peak (λ_{max}) of the Au nanoparticles was at 580 nm. Although the Au^{3+} concentrations are different, its λ_{max} do not significantly vary. The test of TEM also showed that the sizes (11 nm) of particles are very close. So, the Au nanoparticles of the same size can be prepared at a wide concentration range of Au^{3+}.

Resonance scattering spectra

The RRS is a new analytical technique and has been applied to biochemical research and trace inorganic analysis[7-14]. Recently, it has been utilized to study the nanoparticle supramolecules, cell, and so on[15-20]. The maximum resonance scattering peak of the colloidal Au is about 580nm (**Figure 2)**. As the concentration of Au^{3+} increase, the amount of Au nanoparticles will increase too, and the purple of colloidal Au nanoparticles turn to deep. The results indicated a good between A_{580} (the absorbance at λ = 580 nm) the concentration of Au^{3+}in the range from $1.16 \times 10^{-5} \sim 2.32 \times 10^{-4}$

mol/L can be obtained. The linear regression equation is A_{580} = 4297C-0.0206, and the linear coherent coefficient is 0.998. The I_{580} increases with the Au concentration in the range of $1.16\times10^{-5} \sim 1.16\times10^{-4}$ mol/L. When the concentration is more than 1.16×10^{-4} mol/L the interesting phenomenon can be observed. The more Au particles are formed in colloid the lower intensity of RSS is obtained. Owing to the wavelength of maximum absorption(580nm) approximately is closed to that of RSS(580nm), the most of the excitation light is absorbed by the nanoparticles and transferred into heat energy . As a result the intensity of RSS is decreased[21].

The selection of the power and time

The results indicate that homogeneous Au nanoparticles are formed with 480-W power of microwave and 3 min irradiation. If the power is lower and the time of irradiation is shorter, there are many imperfections in the systems. But if the power is higher or the time of radiation is longer, Au particles will partialy be agglomerated.

The selection of the reducing agent

The Au nanoparticles can also be prepared with the reducing agent of methanonic acid, methanol, alcohol, acetone, acetaldehyde, ether and hydrogen peroxide *etc*. The alcohol is a no toxic, neutral , water soluble. So alcohol is an ideal reducing agent. The studies indicate that as the amount of alcohol is in the range of 1.5-6 mL, homogeneous Au nanoparticles will be obtained. If the amount of alcohol is lower or higher, the speed of the reaction will go slower, and the size Au nanoparticles will be bigger. The reason is that when the amount of alcohol is higher the polar of solution molecules is lower comparing with water, so the absorption of microwave is decreased too.

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