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Structural and Optical Properties of Gold Nanoparticles Formed by Wet-Chemical Method

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ABSTRACT

Our aim in this work is the synthesis and study optical and structural properties of gold nanoparticles.Gold (III) chloride trihydratewas taken as metal precursor, ascorbic acid at the presence of an appropriate amount of NaOH as reducing agent and polyvinylpyrrolidonek-30 (PVP K-30) as a stabilizing and capping agent. The reaction was performed in high-speed stirring rate at room temperature.Scanning electron microscopy (SEM with EDAX), X-ray diffraction (XRD) and DRS UV-Vis analysis have been used for characterization of the sample. SEM images shows formation of spherical shape nanoparticles. X-ray diffraction (XRD) measurements showed that the nanoparticles are crystalline and mainly composed of face-centered cubic (FCC). Using the Debye-Scherer formula average particle size of nanoparticles was calculated to be about 4.28 nm. UV-Vis absorption spectrum reveals the formation of gold nanoparticles by showing surface plasmon absorption maxima at 500 nm.

Keyword: Optical properties; Polyvinylpyrrolidone K-30; Gold nanoparticles; Wet-chemical; Ascorbic acid.

1. INTRODUCTION

Gold is known to be a noble metal which means that it is inert and shows small reactivity. The resistance of gold to corrosion has been known to the human for millennia and is reflected in the use of gold in jewelry and currency. Also it was found that very small gold particles can catalyze, i.e. accelerate, important chemical reactions [1]. Gold nanoparticles (Au NPs) generally exhibit plasmon absorption bands, depending on their size and shape [2-4], they have a wide variety of potential applications. For example, effective drug delivery to a cancerous tumor, biomedicals, a wide range of cosmetic and beauty applications, biosensors and catalysts [2]. Among synthesis methods, wet-chemical is the most preferred, because this method is simple and economical, and it can realize better size and size distribution control by optimizing the experimental

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Table 1: *Detailed experimental parameters for preparation of nanostructured gold-PVP nanocomposites.*

Morphology	$HAucl_4$ solution ($MolL^{-1}$)	Ascorbic acid solution ($MolL^{-1}$)	$HAucl_4$/PVP molar ratio	Surfactant(the type and the concentration)	Sediment color
Spherical Figure 2a,b	0.05	0.2	50.1	PVP- 0.001	Black

parameters. Au NPs with narrow size distribution are generally fabricated by a chemical reduction method, typically performed by reducing a HAuCl4 aqueous solution using a reducing agent. The use of stabilizers such as polyvinyl alcohol (PVA), polyethylene glycol (PEG), Polyvinylpyrrolidone (PVP), Hyperbranchedpolyethylenimines, Phosphine, thiol, polythiophene, Poly(3-thiophene acetic acid) (PTAA) and poly(acrylic acid) (PAA), or organic solvent to protect Au NPs against agglomeration is necessary [2, 5-12]. Thus, stability can be seen as one of the important issues for preparation of Au NPs. The stability issue that we are interested in is the stability of particle size over a period of time by monitoring particle size as a function of time. In this work, we synthesized gold NPs by wet-chemical method. Scanning electron microscopy (SEM with EDAX), X-ray diffraction (XRD) and Fourier UV-Vis spectra analysis have been used for characterization of the sample.

2. EXPERIMENTAL

2.1. Materials

In this study, all chemicals used were of analytical grade, Gold (III) chloride trihydrate ($HAucl_4.3H_2O$) was purchased from sigma Aldrich. Polyvinlpyrrolidone (PVP, average molecular weight of 30000) from TCI America was used as the capping agent. Ascorbic acid (99.7%, Merck), Sodium hydroxide NaOH (> 98%, Merck) was also used to adjust the pH; and de-ion water were used as source materials without further purification.

2.2. Method

The three-step preparation scheme for nanoparticles starts with dissolving PVP (0.001M), in deionized water. Next Gold (III) chloride trihydrate (0.05M) was dissolved in water and added to the aqueous solution containing the surfactant (PVP) while vigorously stirring. In the third step, with constant stirring and under N_2 atmosphere mixture ascorbic acid (0.2M) and sodium hydroxide (0.2M) added to the synthesis solution. Color change occurred in the aqueous phase to black. After separation from the mixed solution, the precipitation was washed 3-4 times by de-ion water and then 2-3 times by ethanol. Detailed experimental parameters concerning to the preparation of gold nanostructured composites given in Table 1. The powder X-ray diffraction (XRD) analysis were performed on a D 5000-siemens with Cu Kα radiation (λ= 1.541A°) using a 30 KV operation voltage and 40 mA current. Scanning electron microscopy (SEM) images were obtained using a LEO 1430VP microscope. DRS UV-Vis spectra of the synthesized materials were recorded in the scan range 200-1000 nm, using a UV-Visible spectrophotometer (S-4100, scinc Korea).

3. RESULTS AND DISCUSSION

3.1. XRD characterization

Figure 1represent powder XRD pattern of the nanoparticles sample. The peaks observed at 2θ = 38.58°, 44.8°, 64.9°, 77.9° corresponding to the (111), (200), (220) and (311) lattice planes can be well indexed to face-centered cubic (FCC) structure related to JCPDS card No.1-1172. The crystalline size has been estimated from the broadening of the first diffraction peak using Debye – Scherrer formula:

$$D = 0.9\lambda / \beta \cos\theta \qquad (1)$$

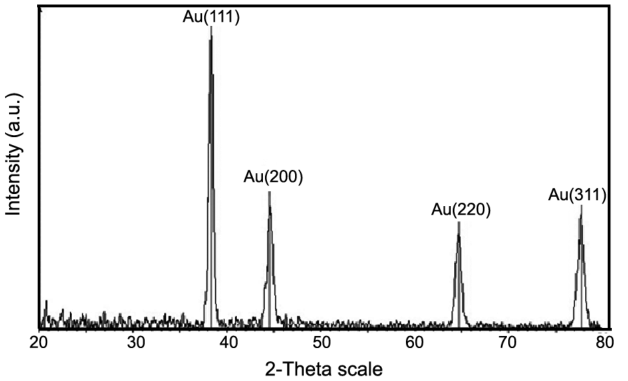


Figure 1: *XRD pattern of asprepared gold-PVP nanoparticles.*

The grain size of the sample was calculated from Eq. (1) using (111) reflection in XRD pattern.

$$\beta = (39.5-37.5)\times\frac{3.14}{180} = 0.0348 \ \text{radians}$$

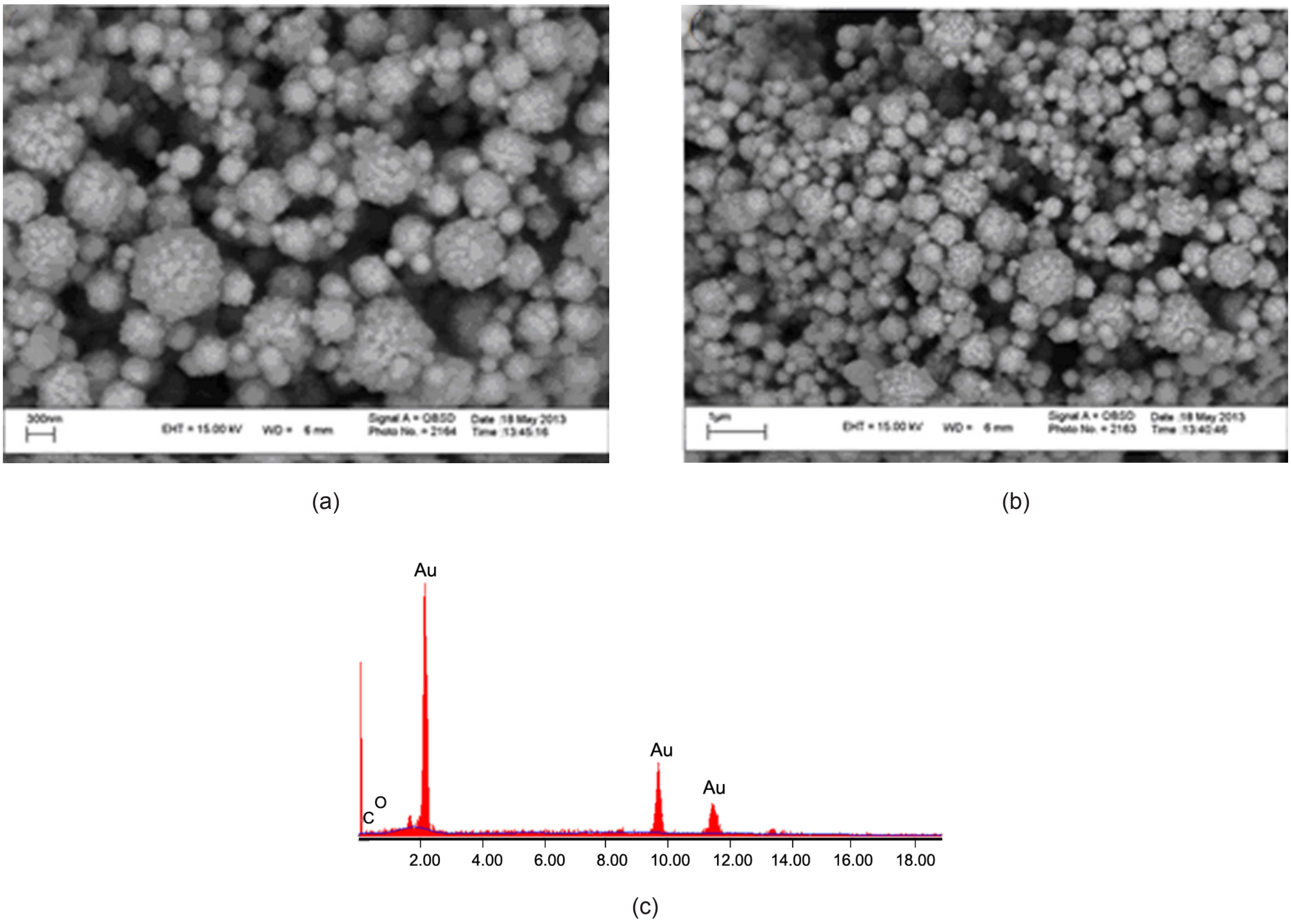
$$2\theta = 38.58 \rightarrow \theta = 19.29$$

$$D = \frac{0.9\lambda}{\beta\cos\theta} \rightarrow D = \frac{0.9\times0.154}{0.0348\times\cos 19.29} = 4.28 \ \text{nm}$$

The particles size of nanogold is 4.28 nm. It was observed that the intensity of the diffraction peak (111) is slightly stronger than other peaks, indicating that the gold sample is mainly dominated by the crystal form (111) an mentioned above, and thus, the crystal growth direction is favorably oriented parallel to the (111) direction.

3.2. SEM characterization

The morphology of the PVP-Au nanostructured was studied with SEM (Figure 2a,b). The spherical particles are seen from the SEM micrographs. Mainly, syn-



(a) (b)

(c)

Figure 2: *(a, b) Typical SEM images of PVP- gold nanostructures of as prepared nanoparticles, (c) EDAX spectrum of an as-synthesized PVP-Au.*

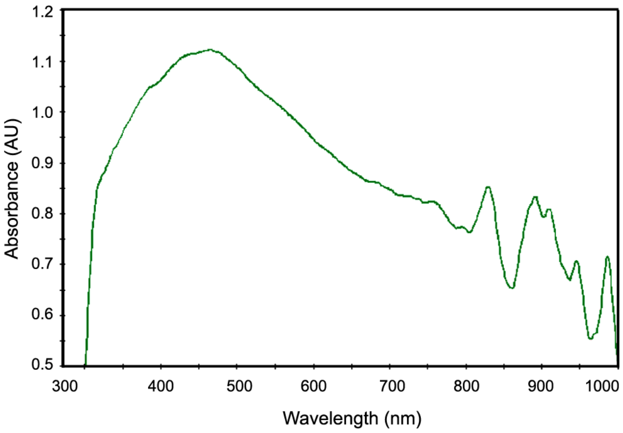


***Figure 3:** UV-Vis absorption spectrum of Au nanoparticles.*

thesis through bottom-up process is a process of reduction of gold cations into gold nanoparticles. Gold cations in the form of HAucl4 are reduced by ascorbic acid to generate Au atom that would soon join the others in the number, thousands or even millions. These atoms will soon interact with the strong metal bonding. Spherical nanoparticles is created, surrounded by PVP, finally larger spherical nanoparticles is crested. The EDAX spectrum of PVP-Au nanostructured indicates that Au is major element, with 94.80% Au, 4.77% C, and 0.43% O (Figure 2c). The results of EDAX instruments analysis provide information that the synthesis of nanogold in matrix of PVP.

3.3. UV-Visible spectra

For gold nanoparticles, the SPR wavelength is around 520 nm depending on the size the nanoparticles [13]. The SPR band of AuNPs with size smaller than 10 nm is largely damped due to the phase changes resulting from the increased rate of electron-surface collisions compared to larger particles [14].

Increasing particle size red shifts the SPR wavelength and also increases the intensity. For particles larger than 100 nm, the band broadening is evident due to the overcome contributions from higher order electron oscillations. Figure 3 represents the UV-Vis absorption spectra of Au nanoparticles; the maximum in the Au absorbance intensity around 500 nm. The optical properties such as absorption maxima and absorption intensity are particle size depend. An intense absorption peak at 500 nm is usually attributed to the surface plasmon excitation of small spherical gold particles.

4. CONCLUSIONS

A simple and easy method for synthesized of nanogold with diameter 4.28 nm is reported. PVP macromolecule in solution may take part in some form of association with the metal atoms, can increase the probability of nucleus formation. Au NPs show the SPR band around 520 nm in the visible region. The SPR band is affected by the particle size.

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