Preparation of Silver Nanoparticles and Their Characterization

Abstract

The preparation of stable, uniform silver nanoparticles by reduction of silver ions by ethanol is reported in the present paper. It is a simple process of recent interest for obtaining silver nanoparticles. The samples have been characterized by X-Ray diffraction (XRD) and Transmission electron microscopy (TEM), which reveal of the nano nature of the particles. These studies infer that the particles are mostly spherical in shape and have an average size of 16 nm. The UV/Vis spectra show that an absorption peak, occurring due to Surface Plasmon Resonance (SPR), exists at 410 nm.

Keywords

Nanoparticle, Surface Plasmon Resonance (SPR), silver nanoparticles

Introduction

Nanoparticle synthesis and the study of their size and properties is of fundamental importance in the advancement of recent research [1,2,3]. It is found that the optical, electronic, magnetic, and catalytic properties of metal nano particles depend on their size, shape and chemical surroundings[2,3].

In nanoparticle synthesis it is very important to control not only the particle size but also the particle shape and morphology as well. In the present investigation the synthesis of silver nanoparticles by chemical route [4,5] is discussed, which is an easy, simple and convenient route for preparing metal particles in nanometer range. The prepared silver nano particles have been dispersed in chloroform and then examined using X-ray diffraction (XRD), Transmission Electron Microscope (TEM) and UV/Vis absorption spectroscopy. These studies reveal that the prepared nanoparticles are of an average size of 16 nm, which indicates the importance of the present work.

Perhaps the most important factor in this process is that the silver nano particles prepared by this process are stable for months.

Synthesis of Silver Nanoparticles

Uniform silver nano particles can be obtained through the reduction of silver ions by ethanol at a temperature of 80°C to 100°C under atmospheric conditions [4]. In this synthesis process, 20 ml of aqueous solution containing silver nitrate (0.5g of AgNO₃), 1.5 g sodium linoleate (C₁₈H₃₂ONa), 8 ml ethanol and 2 ml linoleic acid (C₁₈H₃₂O₂) are added in a capped tube under agitation. The system is sealed and treated at the temperatures between 80°C to 100°C for 6 hours.

In the aqueous solution of silver ions, sodium linoleate and the mixture of linoleic acid and ethanol are added in order. A solid phase of sodium linoleate, a liquid
phase of ethanol and linoleic acid, and water ethanol solution phase containing silver ions formed in the system. Ethanol in the liquid and solution phases reduced the silver ions into silver nanoparticles.

Along with the reduction process, linoleic acid is absorbed on the surface of the silver nanoparticles with the alkyl chains on the outside which the produced silver nanoparticles of near circular shape.

The product which collected at the bottom of vessel after cooling to room temperature, was dispersed in chloroform to form a homogenous colloidal solution of silver nanoparticles. The colour of the sample (colloidal solution of silver nanoparticle) becomes reddish brown. On changing the concentration of electrolyte, it is found that the colour become reddish brown on adding linoleic acid at the same proportions. This reddish brown colour of prepared nanoparticles indicates nearly 100 % conversions of silver ions into nanoparticles. The preparation of silver nanoparticles with different electrolyte concentrations has been tried, but neither the samples with concentration other than the present one is found to be stable over 2 weeks nor of smaller size (more than 60 nm). Hence, we have recorded the data of the particle of optimum size and of comparatively better stability (over 4 months).

**Experimental Result and Discussion**

**XRD Analysis**

The structure of prepared silver nanoparticles has been investigated by X-ray diffraction (XRD) analysis. Typical XRD patterns of the sample, prepared by the present chemical method are shown in the Fig.1.

![Figure 1. X-ray diffraction pattern of Ag nanoparticles.](image)

The XRD study indicates the formation of silver (Ag) nanoparticles. Table 1 shows the experimentally obtained X-ray diffraction angle and the standard diffraction angle [4] of Ag specimen.

**Table1.** Experimental and standard diffraction angles of Ag specimen

<table>
<thead>
<tr>
<th>Experimental diffraction angle [2θ in degrees]</th>
<th>Standard diffraction angle [2θ in degrees]</th>
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<tr>
<td>45</td>
<td>44.3</td>
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From this study, considering the peak at 45 degrees, average particle size has been estimated by using Debye-Scherrer formula [6,7].
Where 'λ' is wave length of X-Ray (0.1541 nm), 'W' is FWHM (full width at half maximum), 'θ' is the diffraction angle and 'D' is particle diameter (size). The average particle size is calculated to be around 14 nm. Table 2 gives the diffraction planes, d spacing, and average size.

### Table 2. Size, diffraction plane, d spacing of Ag sample

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<tbody>
<tr>
<td>45</td>
<td>0.011</td>
<td>0.1788</td>
<td>200</td>
<td>14</td>
</tr>
</tbody>
</table>

**TEM Analysis**

A TEM image of the prepared silver nano particles is shown in the fig.2. The Ag nano particles are spherical in shape with a smooth surface morphology. The diameter of the nano particles is found to be approximately 16 nm. TEM image also shows that the produced nano particles are more or less uniform in size and shape.

**Figure 2.** TEM image of Ag nano particles

**UV/ Vis Spectroscopy Analysis**

In metal nano particles such as in silver, the conduction band and valence band lie very close to each other in which electrons move freely. These free electrons give rise to a surface plasmon resonance (SPR) absorption band [8,9,10,11], occurring due to the collective oscillation of electrons of silver nano particles in resonance with the light wave [6]. Classically, the electric field of an incoming wave induces a polarization of the electrons with respect to much heavier ionic core of silver nanoparticles. As a result a net charge difference occurs which in turn acts as a restoring force. This creates a dipolar oscillation of all the electrons with the same phase.

When the frequency of the electromagnetic field becomes resonant with the coherent electron motion, a strong absorption takes place, which is the origin of the observed colour. Here the colour of the prepared silver nanoparticles is dark reddish brown. This absorption strongly depends on the particle size, dielectric medium and chemical surroundings [9,10]. Small spherical nano particles (< 20nm) exhibit a single surface plasmon band [5]. The UV/Vis absorption spectra of the silver nano particles dispersed in chloroform is shown in the fig. 3.

The absorption peak (SPR) is obtained in the visible range at 410 nm. With the above mentioned concentration. The stability of silver nanoparticles is observed
for 4 months and it shows a SPR peak at the same wavelength.

**Figure 3.** The UV/Vis absorption spectra of Ag nano particles.

**Electroluminescence**

Fig. 4 displays the room temperature electroluminescence spectra of silver nanoparticles when the silver nanoparticles (assembly of nanoparticle) are biased with ac supply voltage. This experiment reveals that unlike fluorescence (FL), silver nanoparticles also exhibit electroluminescence (EL).

Radiative recombination of electron hole pairs between d-band and sp-conduction above the Fermi level produces FL emission [12], which occurs practically at 480 nm when biased with ac voltages. Also, the absorbed linoleic acid during the formation of silver nanoparticles further enhances the intensity of emission [13, 14].

We believe that the reasons behind both the types of luminescence are same as EL and FL peaks occur nearly in the same position which is around 480 nm. Fig 5 shows the variation of luminescence intensity as a function of bias (applied voltage). It is observed that EL intensity varies with bias almost in a linear fashion. This study indicates that silver nanoparticles (assembly of silver nanoparticle) can act as "nano laser" when stimulated (excited) with electrical energy (bias voltage).

**Figure 4.** Electroluminescence spectra of Silver nanoparticle

**Figure 5.** Variation of EL intensity with bias (AC Voltage)

**Conclusion**

Silver nanoparticles have been prepared through the reduction of silver ions by ethanol, which is dispersed in chloroform.
This is one of the simplest and cheapest processes for obtaining silver nanoparticles. UV/Vis spectroscopy reveals the surface plasmon property, while XRD analysis and TEM images reveal the nano nature of the prepared samples. Average size estimated from above studies is 16 nm. Electroluminescence (with peak at around 480 nm) shows the possibility of silver nanoparticles being used as a "nano laser".

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References