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Structural, Optical and Morphological Properties of Silver Nanoparticles using Green Synthesis

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Abstract: The field of nanotechnology is one of the most active research areas in modern materials science. Nanoparticles exhibit new or improved properties based on specific characteristics such as size, distribution and morphology. There have been impressive developments in the field of nanotechnology in the recent past years, with numerous methodologies developed to synthesize nanoparticles of particular shape and size depending on specific requirements. New applications of nanoparticles and nanomaterials are increasing rapidly. In the present investigation, silver nanoparticles were prepared by green synthesis process from silver nitrate solution through the extract of *Citrus sinensis* (sweet lime). The prepared silver nanoparticles were characterized for phase composition, using X-ray diffractometry. Optical properties of silver nanoparticles have been carried out using UV-Visible absorption spectrum. The particle size and morphology were studied using the scanning electron microscope (SEM) and transmission electron microscopy (TEM). Dynamic light scattering (DLS) is used to monitor the size of the precipitating particles and to provide information about their concentration.
Key words: Nanotechnology, Silver, XRD, SEM, UV analysis and TEM.

1. Introduction

In recent years nanotechnology was promising as a rapidly growing field with numerous applications in science and technology for the purpose of manufacturing new materials. This technology is defined as the design, characterization and application of structures, devices and systems by controlling shape and size at nanometre scale level and has already found practical applications in health and daily life [1,2] such as better drug delivery methods[3,4] chemical deposition for environmental pollution cleanup [5,6] medical imaging [7,8] as well as military purposes [9,10]. Out of all kinds of nanoparticles, the metallic nanoparticles, including gold, silver, iron, zinc and metal oxide nanoparticles, have shown great promise in terms of biomedical applications, not only due to their large surface to volume ratio [11,12] but also because they exhibit different biomedical activities [13]. Silver nanoparticles are one of the promising products in the nanotechnology industry. The development of consistent processes for the synthesis of silver nanomaterials is an important aspect of current nanotechnology research. One of such promising process is green synthesis. Silver nanoparticles can be synthesized by several physical, chemical and biological methods. However for the past few years, various rapid chemical methods have been replaced by green synthesis because of avoiding toxicity of the process and increased quality. New technology advances in reducing silver compound chemically to nanoscale sized particles have enabled the integration of this valuable antimicrobial into a larger number of materials including plastics, coatings, and foams as well as natural and synthetic fibers. Nano-sized silver have already provides a more durable antimicrobial protection, often for the life of the product. Current research in

inorganic nanomaterials having good antimicrobial properties has opened a new era in pharmaceutical and medical industries. Silver is the metal of choice as they hold the promise to kill microbes effectively. Silver nanoparticles have been recently known to be a promising antimicrobial agent that acts on a broad range of target sites both extra cellularly as well as intracellarly. Silver nanoparticles shows very strong bactericidal activity against gram positive as well as gram negative bacteria including multi resistant strains [14] and also it was found to be in few studies [15,16]. This paper reveals the synthesis and characterization of silver nanoparticles were prepared by green synthesis process. The as prepared silver nanoparticles are characterized by X- ray diffraction, UV-Visible absorption spectrum, scanning electron microscopy (SEM), transmission electron microscopy (TEM), and dynamic light scattering (DLS) analysis.

2. Experimental Procedure

Citrus Sinensis has been thoroughly washed in distilled water, dried, cut into fine pieces and was smashed into 100 ml sterile distilled water and filtered through Whatman filter paper. The extract has been stored at 40°C. The aqueous solution of silver nitrate has been used for the synthesis of silver nanoparticles. 10 ml of *Citrus Sinensis* extract has been added into 90 ml of aqueous solution of silver nitrate and kept for incubation period of 12 h at room temperature. Silver nanoparticles which exhibit yellowish brown color in aqueous solution were obtained. To check phase formation and purity, XRD pattern was recorded using an X-ray diffractometer using CuKα radiation. The optical absorption spectrum of the silver nanoparticles has been taken by using the VARIAN CARY MODEL 5000 spectrophotometer in the wavelength range of 300 to 1000 nm. Scanning electron microscopy (SEM) was performed with a focusing on nanoparticles to study the morphology. The particle size was studied using the transmission electron microscopy (TEM). The particle size of the silver nanoparticles was analyzed, using the dynamic light scattering (DLS) experiment.

3. Results and Discussion

3.1 XRD analysis

The structural analysis of sample was done by the powder X-ray diffraction. The *Citrus sinensis* extract-mediated synthesized silver nanostructure was confirmed by the characteristic peaks observed in the XRD image which was shown in Fig.1. The X-ray diffraction patterns of silver nanoparticles are shown in Fig.1. The strong diffraction peaks in the XRD spectrum of the (1 1 1), (2 00), (2 2 0) and (331) planes can be indexed to a cubic and hexagonal pattern of silver nanoparticles. The broadened peak shows the nanometer-sized crystallites. The average nano-crystalline size (D) was calculated using the Debye-Scherrer formula,

$$D = \frac{0.9\lambda}{\beta \cos\theta} \qquad (1)$$

where λ is the X-ray wavelength (CuKα radiation and equals to 0.154 nm), θ is the Bragg diffraction angle, and β is the FWHM of the XRD peak appearing at the diffraction angle θ. The average crystalline size is calculated from X-ray line broadening peak and Debye-Scherrer equation to be about 48 nm.

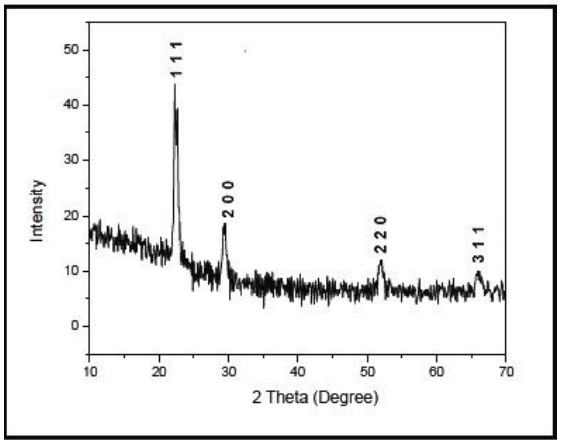

Fig.1 XRD patterns of Ag nanoparticles

3.2 UV–Vis spectral analysis

Study of materials by means of optical absorption provides a simple method for explaining some features concerning the band structure of materials. The transmittance spectrum of silver nanoparticles in the wavelength range 300 - 1000 nm are shown in the Fig 2.The absorbance near infrared domain (300 nm) is very low whereas transmittance is high at the same region. The absorption edge has been obtained at a shorter wavelength. The broadening of the absorption spectrum could be due to the quantum confinement of the nanoparticles. The as-prepared silver nanoparticles have good crystalline and show strong blue emission. A remarkable broadening of peak at around 350 nm to 480 nm indicates that the particles are polydispersed. It was observed that the peak was blue shifted in the absorption spectrum from 350nm to 480 nm with increasing reaction time. Silver nanoparticles have free electrons, which give surface Plasmon resonance (SPR) absorption band, due to the combined vibration of electrons of silver nanoparticles in resonance with light wave. A broad absorption peak was observed at 440 nm, which is a characteristic band for the silver nanoparticles. No other peak was observed in the spectrum which confirms that the synthesized products are silver nanoparticles only.

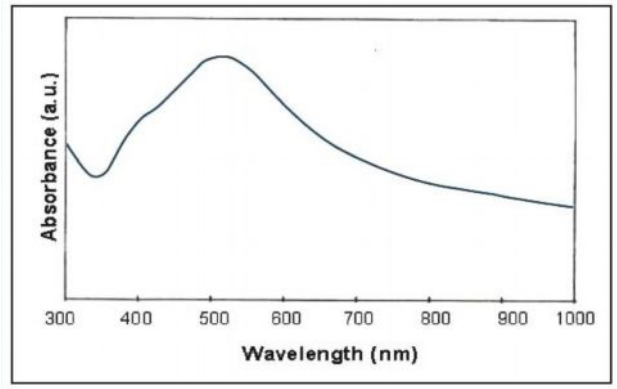

Fig.2 UV-Vis spectrum of silver nanoparticles

3.3 SEM analysis

External morphology, chemical composition, crystalline structure and orientation of materials making up the sample are revealed by SEM. Data are collected over a selected area of the surface of the sample, and a two-dimensional image is generated. In the present research work, the scanning electron microscope (SEM) was used for the morphological study of silver nanoparticles. Fig.3 shows the SEM images o f the as-prepared silver nanoparticles. The spherical shaped particles with clumped distributions are visible from the SEM analysis.

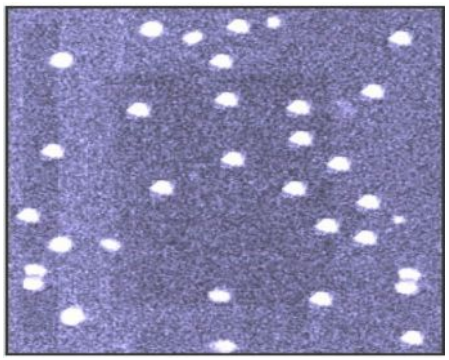

Fig.3 SEM Image of silver nanoparticles

3.4 TEM Analysis

TEM is commonly used for imaging and analytical characterization of the nanoparticles to assess the shape, size, and morphology. The structure and morphology of the samples were further confirmed by the TEM and TEM images of the prepared silver nanoparticles, as shown in Fig.4. The transmission electron microscopic analysis confirms the presence of the spherical shape morphology of the prepared silver nanoparticles with the particle size of around 20 to 50 nm (Fig.4). The average size of nanoparticles was found to be 48 nm.

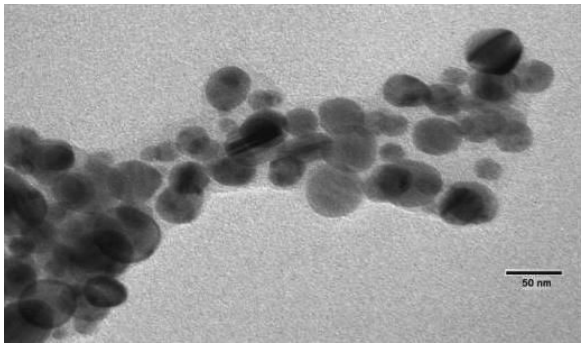

Fig.4. TEM image of the silver nanoparticles

3.5 DLS Studies

Dynamic light scattering (DLS) is an important tool for characterizing the size of nanoparticles in a solution. The DLS measures the light scattered from a laser that passes through a colloidal solution and by analyzing the modulation of the scattered light intensity as a function of time, the hydrodynamic size of the particles and particle agglomerates can be determined. Larger particles will diffuse slower than smaller particles, and the DLS instrument measures the time dependence of the scattered light, to generate a correlation function that can be mathematically linked to the particle size. The DLS is a valuable tool for determining and measuring the agglomeration state of the nanoparticles as a function of time or suspending solution. When the DLS sizing data is compared to the transmission electron microscopy images, the aggregation state of the particles can be determined. In an unagglomerated suspension, the DLS measured diameter will be similar or slightly larger than the TEM size. If the particles are agglomerated, the DLS measurement is often much larger than the TEM size, and can have a high polydispersity index (large variability in the particle size). Fig.5 shows the graphical representation of average particle size distribution of silver nanoparticles. They were in a range of 20-100 nm. However, beyond 100 nm range the percentage of nanoparticles present is very less. The highest fraction of silver nanoparticles present in the solution was of 50 nm. From the plot it was evident that the solution was consist of nanoparticles having various sizes which are indeed in agreement of the result obtained by TEM analysis.

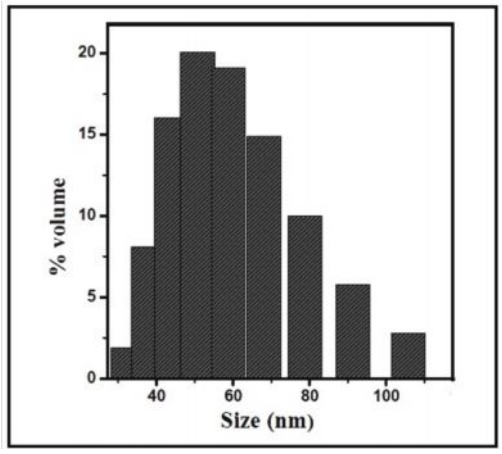

Fig. 5. Particle size distribution of silver nanoparticles

4. Conclusion

Silver nanoparticles have been successfully synthesized using the green synthesis process. The formation of silver nanoparticles was confirmed by X-ray diffraction (XRD). The optical properties were studied by the UV-Vis absorption spectrum. The size and morphology of the samples were characterized using scanning and transmission electron microscopy (SEM and TEM). The spherical shaped particles were confirmed through the SEM analysis. The transmission electron microscopic analysis confirms the prepared silver nanoparticles with the particle size of around 48 nm. The particle size of the silver nanoparticles range of 40 to 50 nm was determined using the dynamic light scattering (DLS) experiment which **is** in good agreement with the TEM analysis.

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