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Synthesis and Characterization of PEG-Ag Nanoparticles: Investigation of their Antibacterial Properties.

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Abstract: *The polyethylene glycol capped silver nanoparticles have been synthesized through the aqueous preparation method by means of exploiting polyethylene glycol as reducing agent. The chain length of polyethylene glycol plays an important role in the formulation of silver nanoparticles. In the current study, $AgNO_3$ acts as a precursor, polyethylene glycol used as the reducing as well as a capping agent and the pH of the reaction mixture was maintained by using NaOH solution. X-ray diffraction, UV-Visible, Fourier transform infrared spectroscopy, Scanning electron microscope and Energy dispersive spectroscopy measurements were performed to confirm the formation of the polyethylene glycol capped silver nanoparticles. The size of the polyethylene glycol capped silver nanoparticles was found to be 17 nm by using the Debye-Scherer formula. This polyethylene glycol capped silver nanoparticles were tested for Antibacterial activity against four tested bacteria Bacillus subtillis, Klebsiella pneumonia, Pseudomonas putida, staphylococcus aureus which was proved to be a good effect.*

Keywords *: Silver nanoparticles, Polyethylene glycol, Aqueous solution method, X-ray diffraction analysis, Antibacterial activity.*

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I. Introduction

The nanoparticles stated to be small particles between 1 and 100 nanometers in size and with novel properties that differ its bulk materials [1-2]. The metal nanoparticles have got much attraction because of their unusual size dependent unique properties like optical, electrical, magnetic and catalytic properties. Now a day a large spectrum of research has centered on the dimensions manipulate like size, shape, distribution control of the metal particles which lead to vast changes in their physical, chemical and optical properties [3-6]. Among the metal nanoparticles, silver nanoparticles have potential applications in several fields such as oxidation catalysis, sensors, biomedicine, optics, electronics, fuel cell, photovoltaic cells, optical data storage systems and more [7-13]. The silver was widely recognized for antibacterial activity from ancient Greece's times. In fact, the silver nanoparticles are the most efficient antibacterial agents with a lower concentration than Ag+ ions [14]. Hence, the silver nanoparticles are highly commercialized due to their strong antibacterial activity. The nano-sized silver particles are utilized in medicine to lessen infections in burn treatments, to prevent bacterial colonization on dental materials, to extinguish microorganisms on textile fabrics and for water treatment, used in cosmetic products, antibacterial sprays, detergents and more [15-20]. A number of approaches are available for the synthesis of silver nanoparticles but the most and simplest is a bulky-solution synthetic method in which preparation of metal nanoparticles by using metal salts are chemically reduced [21]. We pronounced here to use the soluble metal salts, a reducing agent was polyethylene glycol (PEG) and it additionally acts as a stabilizing agent for synthesizing silver nanoparticles by way of the aqueous solution method. The synthesized polyethylene glycol capped silver nanoparticles (PEG-Ag) had been characterized by employing various chemophysical techniques such as UV-Visible absorption spectroscopy (UV-Vis), fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscope-energy dispersive spectroscopy (SEM-EDS) and studied their Antibacterial activity.

II. Experimental

Materials

All chemicals utilized in the experiment are analytical reagent grade. Silver nitrate (AgNO3) and Sodium hydroxide (NaOH) pellets were purchased from S.D Fine-Chem. Limited, Mumbai, India. Polyethylene glycol (PEG) was purchased from Lobha Chemie. The chemicals used for antibacterial activity were of Himedia grade, Mumbai, India. The bacterial test strains were procured from IMTECH, Chandigarh. Deionized water was used throughout the experiment.

Synthesis of PEG-Ag Nanoparticles

An aqueous solution of silver nitrate (0.2M) is prepared in a clean round bottom flask by completely covering it using aluminum foil to prevent photoreduction. Then PEG is added to the above aqueous solution and heated to 80^{o}C with constant stirring and 0.1M NaOH solution was added to above-heated solution till pH reaches between 9 to 10. The addition of NaOH solution was employed to maintain the pH basic in nature and it can act as an accelerant to the formulation of nanoparticles leads to completion of the reaction. The color of the solution turned to the characteristic dark brown to black color which suggested the formulation of colloidal silver nanoparticles. The precipitate centrifuged and collected later washed 3-4 times with deionized water. The prevailed precipitate was dried in a hot-air oven for 24 h. The silver nanoparticles in PEG are stable for more days without changing properties at room temperature and reveals that the PEG matrix is a good stabilizer for the nanoparticles. The obtained silver nanopowder is further used for the characterization and biological properties.

Experimental Methods

The absorption spectra of synthesized PEG-Ag nanoparticles were registered with UV-Vis spectrometer UV-3600 series, Shimadzu in the wavelength range from 200- 800 nm after small aliquot of sample dispersed in distilled water. The FTIR spectra of the PEG-Ag nanoparticles were registered with an IR Affinity-1 Shimadzu spectrometer in the range of 4000-500 cm^{-1} using a KBR pellets technique. The powder X-ray diffraction (XRD) of the sample was performed using Philips Holland, XRD system PW 1710 with nickel-filtered CuKα (λ = 1.5405 Å) radiation, with generator settings 40kV, 30mAh with the scanning rate 2°min^{-1} in θ=2θ configuration. The average crystallite size has been calculated from the line broadening using Debye-Scherer's relation. The SEM images of the synthesized PEG-Ag nanoparticles were carried out by using model ZEISS Special Edition 18. The energy dispersive spectroscopy microanalysis coupled to the SEM system which automatically distinguishes the elements corresponded to the peaks in the energy distribution.

Antibacterial studies

antibacterial studies of synthesized PEG-Ag nanoparticles were investigated by the well-diffusion method. Fresh cultures of antibacterial assays were prepared for four bacteria *Bacillus subtillis, Klebsiella pneumonia, Pseudomonas putida, staphylococcus aureus*, all sterilized Labware was used to perform this test. The sterilized nutrient agar medium was used to culture the bacteria. The sterilized media were poured into the petri plates at the laminar flow and spread with 50 µL specific bacteria in each of the plates. The wells are punched at different places in the petri plates and labeled to them respective samples were added and then petri plates were incubated at 37 °C for 12 hours to observe the zone of inhibition of the bacteria [22].

III. Results And Discussion

UV-visible measurements

UV-visible spectrum is very much useful in recognition of the nanoparticles [23]. The absorption spectra of synthesized PEG-Ag nanoparticles with capping agent PEG were shown in Fig.1. The synthesized PEG-Ag nanoparticles exhibit a band in UV region at 404 nm. The dark brown color solution indicating the formulation of colloidal silver nanoparticles, which can be reaffirmed by using UV-Vis spectroscopy. The color observed due to the surface plasmon resonance (SPR) characteristic phenomena observed by cause of the resonant oscillation of conduction electrons at the interface between negative and positive permittivity material stimulated by incident light [24-25].

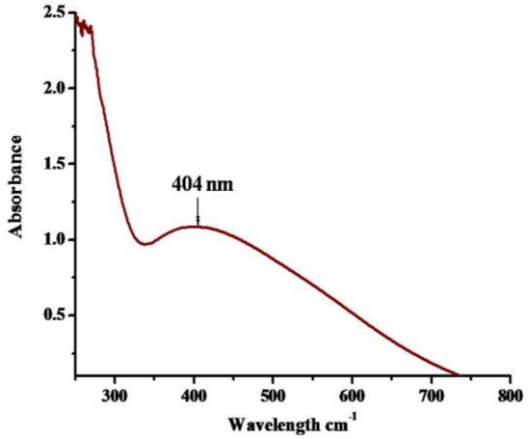

Fig.1. UV-Vis spectroscopy of PEG-Ag nanoparticles

Fourier transform Infrared measurements

The FTIR spectra of PEG-Ag nanoparticles were recorded in the range of 4000-400 cm^{-1} and are shown in Fig.2. In the spectra of PEG-Ag nanoparticles, a broadband recognized around 3390 $cm^{-1}$was assigned to O-H stretching vibrations of a hydroxyl group. The strong band at 1427 cm^{-1} was due to C-H scissor and bending vibrations of alkanes. The low band at 1133 cm^{-1} was attributed to stretching vibrations C-O of Alcohol or C-O-C of ethers from PEG indicating the formation of a coordinate bond between the oxyzen atom of the PEG and the Ag ions[26-27]. These peaks confirm the formation of Ag nanoparticles with PEG as capping as well as reducing agent indicating the involvement of PEG in the reduction reaction [28].

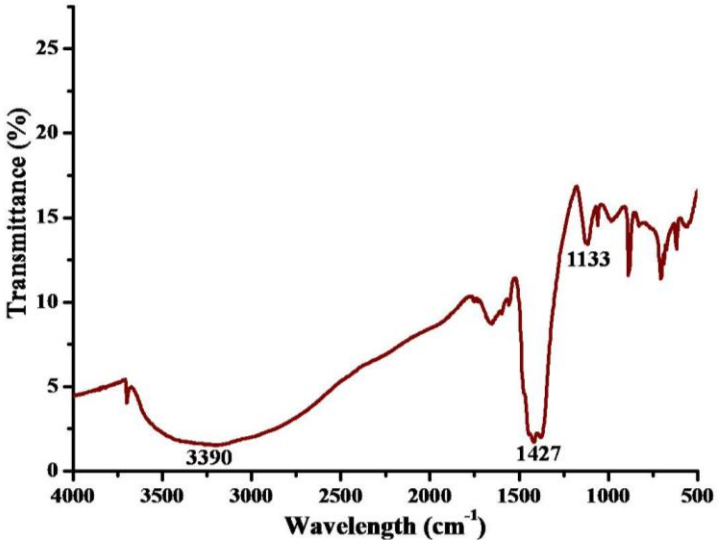

Fig.2. FTIR spectroscopy of PEG-Ag nanoparticles

X-diffraction measurements

The synthesized PEG-Ag nanopowder, dried at 60ºC and its XRD is illustrated in Fig.3. XRD pattern unveils that the intense and wide diffraction peaks at 2θ = 32.20º, 37.78º, 54.84° 65.78° and 76.98° were corresponding to (111) (200) (220) (311) sets of lattice planes of nanosilver particles of Ag respectively. The data of XRD of PEG-Ag nanoparticles designating face centric cubic (FCC) structure [29]. The size of nanoparticles has been calculated using Debye-Scherrer's formulae [30].

$$D = K\lambda / \beta\cos\theta \quad (1)$$

Where the constant K is taken to be 0.94, λ is the wavelength of X-ray (1.5406 Å), and β the full width at half maximum of the diffraction peak corresponding to 2θ. From the above equation (1), The average size of synthesized Ag nanoparticles was found in the range of 7-24 nm. The average particle size was determined to be 17 nm. It gives a single-phase with a monoclinic structure. The intensity and positions of the peaks are in good agreement with the reported values (JCPDS file No. 05-661). Impurities peaks are not found in XRD pattern and the peaks are broad due to the nano-size effect.

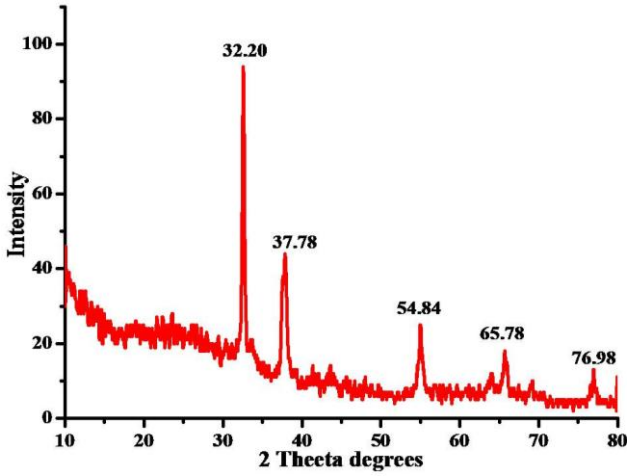

Fig.3. X-ray diffraction spectroscopy of PEG-Ag nanoparticles

Scanning electron microscope - Energy dispersive spectroscopy

The SEM image of synthesized PEG-Ag nanoparticles was expressed in Fig.4. at various magnifications. The SEM pictures show the size of synthesized Ag nanoparticles are very small. It shows that the particles are well crystallized and monoclinic in structure. The EDS spectra of Fig.5 shows the peaks corresponding to Ag, Cl, and Oxyzen molecules confirm the formation of Ag nanoparticles. The distinctive peak was noted at ~3 KeV within the spectra indication for the crystalline nature of synthesized nanosilver [31]. The Table.1 gives information about Ag atomic % was 41.67, weight % was 74.95%.

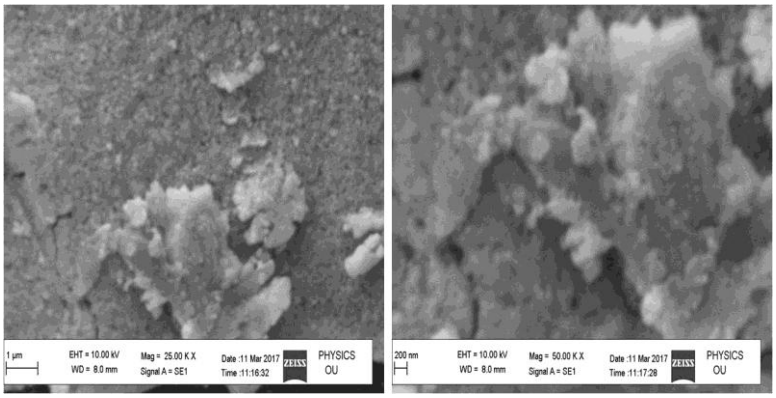

Fig.4. Scanning electron microscopy images of PEG-Ag nanoparticles

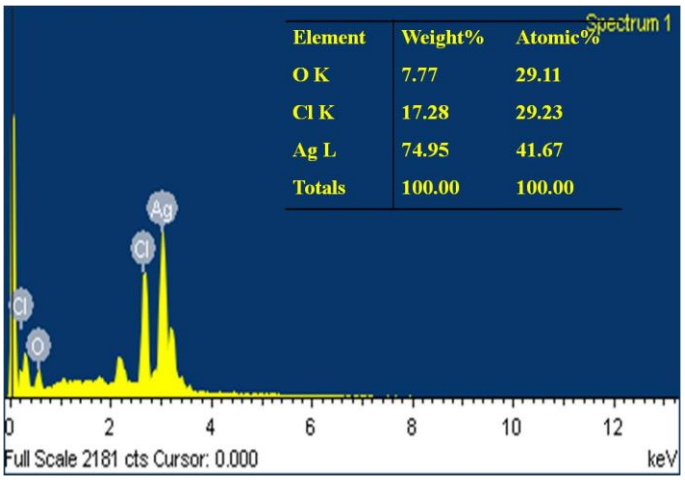

Fig.5. Elemental analysis by energy dispersive spectroscopy of PEG-Ag nanoparticles

Antibacterial activity

The Fig.6 of PEG-Ag Nanoparticles exhibited antibacterial activity against all the four bacterial strains used in the study. 50 µL concentrations of samples were added to the wells as ampicillin, PEG-Ag nanoparticles, NaOH solution, and only PEG sequentially. Ampicillin used as a control sample at the upper side of the plate which labeled as well 1. The Synthesized PEG-Ag nanoparticles were added in well 2. The clear zones of inhibition were seen, although smaller zone of inhibition was found when compared to the standard antibiotic ampicillin. The results of the zone of inhibition in mm for different bacteria in respective samples are measured in mm using a scale. The zone of inhibition for synthesizing PEG-Ag nanoparticles detected as 7 mm, 8 mm, 7 mm and 9 mm for four bacteria *Bacillus subtillis, Klebsiella pneumonia, Pseudomonas putida, staphylococcus aureus* respectively, whereas ampicillin activity shown as 16 mm, 9 mm, 12 mm, 14 mm for the same. The other samples PEG and NaOH did not show any activity at all. The proposed mechanism of antibacterial activity was the silver ions from PEG-Ag nanoparticles released and that they may attach to the negatively charged bacterial cell wall and damage it which leads to protein denaturation and induce cell death. On the other hand, the presence of capping agent PEG further enhances the cell death.

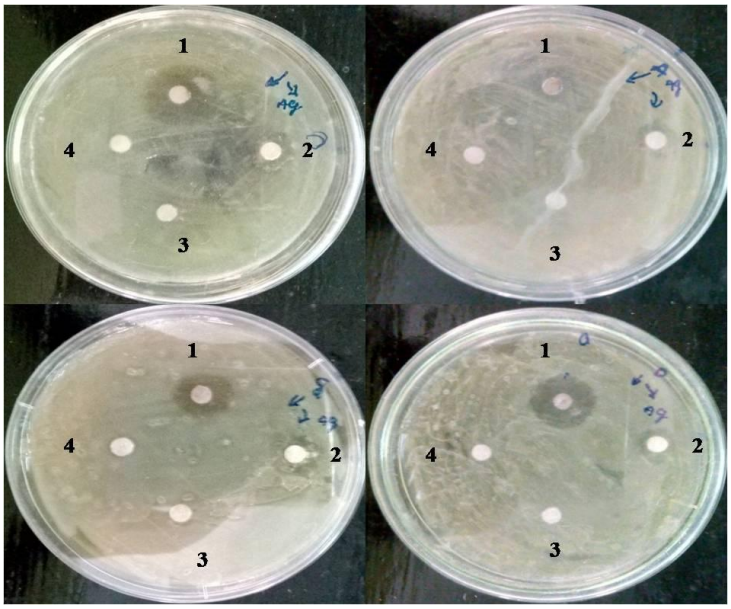

Fig.6. Antibacterial activity of PEG-Ag Nanoparticles for various bacteria 1. Ampicillin 2. PEG-Ag Nanoparticles 3. NaoH Solution 4. PEG

IV. Conclusions

In this article, we have successfully synthesized PEG-Ag nanoparticles through a simple chemical method by exploiting polyethylene glycol as reducing and capping agent. The UV-Vis spectroscopy analysis indicates the formation of colloidal PEG-Ag nanoparticles by noticing the distinct absorption peak at 404 nm. The FTIR analysis revealing the vibrations of functional groups from PEG confirmed the involvement of this PEG in the reaction where it acts as a capping agent. The crystalline nature and face centric cubic structure were confirmed by the XRD data and average particle size was found to be 17 nm. The nanosized particles and elemental analysis with atomic and weight percentage were determined by SEM-EDS. The PEG-Ag nanoparticles were stabilized in polyethylene glycol and their antibacterial ability was has given profound effect against Bacillus subtillis, Klebsiella pneumonia, Pseudomonas putida, staphylococcus aureus which may be used as a commendable antibacterial drug in the medical field.

Acknowledgments

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