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Synthesis of Silver Nanoparticles by Chemical Reduction Method

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ABSTRACT

Metal nanoparticles are gaining importance nowadays in nanoscience. The nanoparticles have better physical and chemical properties compared with solid particles due to their large surface area. Silver nanoparticles are employed mostly in medical and electrical applications having outstanding conductivity and antimicrobial activity. In the present investigation, a simple and reproducible method of synthesis of silver nanoparticles is developed. The silver nanoparticles are prepared by chemical reduction method using sodium citrate as a reducing agent. The average size, size distribution, morphology, and structure of particles were determined by UV/Visible absorption spectrophotometry, Particle size analysis, XRD, SEM, and zeta potential determination. We found that the silver nanoparticles with a size about of 103 nm and good distribution are prepared without using any external surfactant or stabilizer. The results of this work suggest that good properties of Ag NPs are synthesized by this facile method and are a cost-effective, safe, convenient, and beneficial technique for the large-scale production of nanoparticles.

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INTRODUCTION:

Nanotechnology is the study of very small structures. It deals with the study of smaller structures with a size range between 1 to 100 nm, in the production of innovative materials and devices which exhibit new and exclusive characteristics. These fabricated systems have interesting physical, electrical, and optical properties which make them appealing in a variety of fields, ranging from material science to biology.[1,2]

Nanotechnology is responsible for the production and study of metal nanoparticles (NPs). This nano-derived structure exhibits several applications that highlight anti-microbial activity and this property is a crucial tool in combating microorganisms resistant to conventional drugs. Recent advances in nanotechnology include the merging of metallic NPs into diverse industrial, medical, and household products.[3]

Nanoparticles can be synthesized using two primary approaches known as top-down and bottom-up. In the top-down approach, NPs are synthesized from bulk materials into smaller ones. This strategy mostly falls under the physical method for NP synthesis as it acquires big tube furnaces to small bulk material into smaller parts. While in the bottom-up technique, the NPs are synthesized to the required material from smaller molecules. The chemical reduction method is mostly used in this technique, sometimes combined with the Capping agent for NPs synthesized stabilizing purposes. The biological method of NP synthesis is also categorized under the bottom-up approach, in which biomolecules incorporate metallic substances for nanoscale material production.[4]

In recent years, many chemical methods for the synthesis of silver nanoparticles (AgNPs) have been reported including the polyol method and the liquid-liquid method. However, the chemical reduction is the most commonly used due to its simplicity. This technique also enables variation in the molar concentration of the reactant, dispersant, and feed rate of reactant to produce AgNPs with controlled particle sizes, shapes, and particle size distribution. In this method, the selection of an appropriate reducing agent is also an important factor, as the size, shape, and particle size distribution strongly depend on the nature of the reducing agent.[5] Owing to all these advantages chemical reduction method can be selected as it provides a highly pure, simple and reproducible technique for AgNPs synthesis.

Metallic Nanoparticles

Metallic nanoparticles display fascinating properties that are unique from those of individual atoms, surfaces, or bulk materials. They are a focus of interest for fundamental science and, because of their huge potential in nanotechnology, they are the subject of intense research efforts in a range of disciplines. Nanoparticles of noble metals, particularly silver, gold, gold-silver alloy, selenium, tellurium, platinum, silica, etc. have been studied extensively in past few years. The physical and chemical properties of the metallic NPs are different as they are dependent on their size as well as their structure, shape, and size distribution. These NPs are widely used in various domains which include medical, healthcare, food, consumer, and industrial purposes owing to their peculiar physical and chemical properties.[6]

Silver Nanoparticles

Nanoparticles of noble metals, especially NPs derived from silver have received great attention due to their unique properties and are found to be widely used in different fields of science. As a result of their unique properties, AgNPs have been used for several applications, including as anti-bacterial agents, in the industrial, household, and healthcare-related products, pharmaceutical industry, the food industry, diagnostics, orthopedics, drug delivery, etc. AgNPs have exhibited activity against bacteria, fungi, and viruses. AgNPs have seemed to be alternative anti-bacterial agents to anti-biotics and can overcome bacterial resistance against antibiotics. They are reported to have antibacterial activity against *Staphylococcus epidermidis, Pseudomonas aeruginosa, Staphylococcus aureus, and E. coli.* These NPs have exhibited enhanced and prolonged anti-fungal activity against strains of *Candida albicans*. [7] However, the synthesis of AgNP may lead to the generation of toxic waste during the reaction process.

Accordingly, new methods using non-toxic compounds have been researched. A variety of preparation techniques have been reported for the synthesis of AgNP, some of them including, laser ablation, chemical reduction, photochemical methods, microwave processing, biological synthetic methods, etc. In recent years, many chemical methods have been reported for the synthesis of AgNP which includes the polyol method and the liquid-liquid method. However, the chemical reduction method has its advantages due to its simplicity. This technique also enables variation in the molar concentration of the reactant, dispersant, and feed rate of reactant to produce AgNP with controlled particle sizes, shapes, and particle

size distribution. [5] In our study, trisodium citrate was used as both a reducing and a co-ordinating agent to synthesize AgNPs using chemical reduction method.

MATERIALS AND METHODS:

Silver nitrate was procured from Sigma Aldrich. Other reagents used were of laboratory grade. Triple distilled water was used in the study.

Synthesis of silver nanoparticles

Aqueous 0.01 M silver nitrate solution was heated to boiling and silver nanoparticles were synthesized by using 1% trisodium citrate as a reducing agent. The solution was mixed vigorously during this process and heated. Aqueous silver ions when exposed to 1% trisodium citrate were reduced and resulted in a color change from yellowish to pale brown indicating the formation of silver nanoparticles. The nanoparticle solution was stirred for 20 minutes on a magnetic stirrer at 90°C. The reaction mixture was centrifuged at 6000 rpm for 15 min. The pellet was collected, rinsed thrice with triple distilled water, and dried in a hot air oven at 80°C. The resultant silver nanoparticles were characterized by UV spectroscopy, Particle size analysis, zeta potential determination, X-Ray Diffraction, and Scanning Electron Microscopy.[8-10]

UV-vis spectroscopy

UV-vis spectroscopy is a very useful and reliable technique for the primary characterization of synthesized nanoparticles which uses electromagnetic radiations between 190 nm to 800 nm and is divided into the ultraviolet (UV, 190-400 nm) and visible (VIS, 400-800 nm) regions. It is helpful in the detection of functional groups, identification of unknown compounds, and determining the purity of compounds. AgNPs have unique optical properties which make them strongly interact with specific wavelengths of light. In AgNPs, 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, occurring due to the collective oscillation of electrons of silver nanoparticles in resonance with the light wave. Accordingly, the suspension was scanned in the range of 600 to 200 nm.[11-16]

Particle size analysis

Dynamic light scattering is a method that depends on the interaction of light with particles. This method can be used for the measurement of narrow particle size distributions, especially in the range of 2–500 nm. DLS is the most commonly used technique among all other techniques for the characterization of nanoparticles. DLS measures the light scattered from a laser that passes through a colloid and mostly relies on Rayleigh scattering from the suspended nanoparticles. Next, the modulation of the scattered light intensity as a function of time is analyzed, and the hydrodynamic size of particles can be determined. For size measurement, prepared nanoparticles were dispersed in triple distilled water (1:50 ratio) and sonicated for 30 minutes. The sample was then introduced into the cuvettes. The sizes of nanoparticles were recorded.[17-20]

Zeta potential of synthesized nanoparticles

Zeta potential can be used to gain further insights into the stability of the obtained colloidal AgNPs. The stability of nanoparticles is directly proportional to the zeta potential value. It also describes the degree of interaction between charged particles i.e. the degree of repulsion between the surface charge of nanoparticles dispersed in the solvent used. Higher zeta potential values indicate high stability of nanoparticle suspension. Zeta potential is measured using the technique of electrophoretic light scattering where particle motion is detected in an applied electric field. Synthesized silver nanoparticles were dispersed in water and zeta potential was measured.[21]

Scanning electron microscopy

SEM is a surface imaging method, fully capable of resolving different particle sizes, size distributions, nanomaterial shapes, and the surface morphology of the synthesized particles at the micro and nanoscales. In this technique, samples were thoroughly degassed and dried to eliminate any outgassing from organic contamination and water. Samples were then cleaned ultrasonically using water and blown dry using compressed gas. The powder sample was compressed into small disks for sample mounting. Samples were attached to the flat plates using double-sided carbon or copper tapes and observed under a scanning electron microscope. [22-24]

X-ray diffraction

X-ray diffraction (XRD) is a well-known analytical technique that has been used for the analysis of both molecular and crystal structures, qualitative identification of various compounds, quantitative resolution of chemical species, measuring the degree of crystallinity, isomorphous substitutions, particle sizes, etc. When X-ray light reflects on any crystal, it leads to the formation of many diffraction patterns, and the patterns reflect the physicochemical characteristics of the crystal structures. Here, silver nanoparticles were freeze-dried for 12 hr. and then ground to a fine powder. The sample was placed in a sample holder and scanned.[25,26]

RESULTS AND DISCUSSION:

Characterization of silver nanoparticles

Primary confirmation of synthesized silver nanoparticles can be observed visibly. Aqueous silver ions when exposed to 1% trisodium citrate were reduced and resulted in a color change from colorless (Figure 1) to pale brown (Figure 2) indicating the formation of silver nanoparticles.

Figure no 1: Aqueous 0.01 M silver nitrate solution

Figure no 2: Colour change that indicates synthesis of silver nanoparticles

UV-Vis Spectroscopy

The primary synthesis of silver nanoparticles was determined with color change and UV-Vis spectroscopy. The synthesis of the silver nanoparticles in an aqueous solution was confirmed by recording the absorption spectra at a wavelength range of 200-600 nm (Figure 3). Synthesized silver nanoparticles exhibited the Surface Plasmon Resonance phenomenon with absorption maxima at 432 nm. This is in line with the observations of several other researchers who have carried out studies on surface Plasmon resonance phenomena exhibited by silver nanoparticles.[27-29]

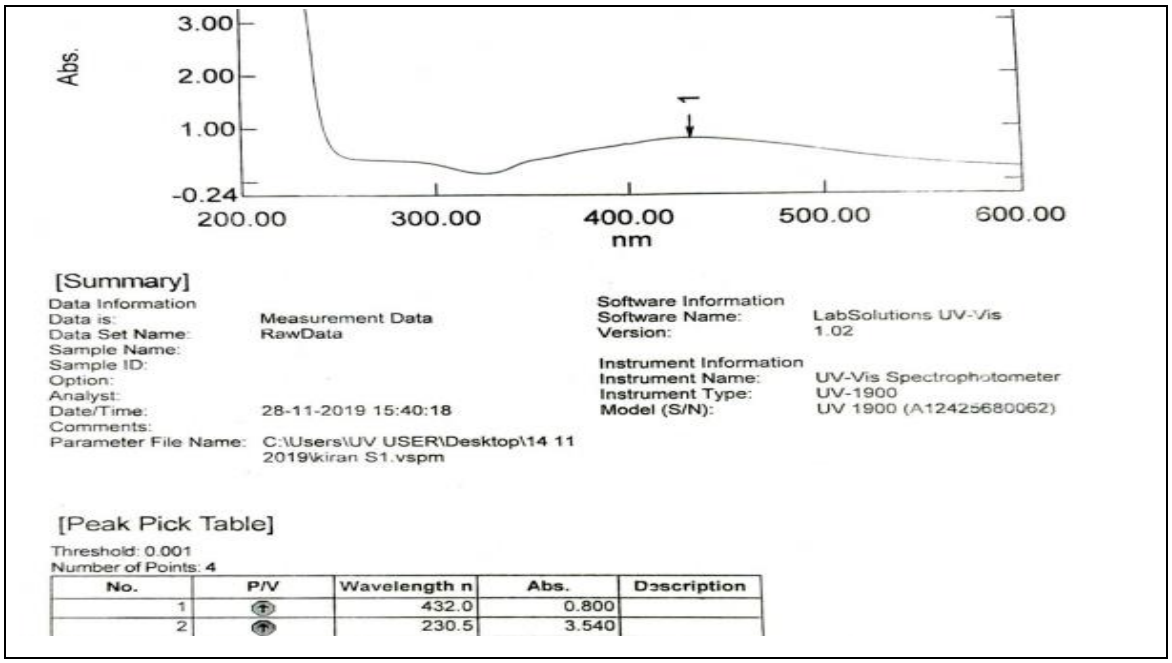

Figure no 3: Surface Plasmon Resonance exhibited by silver nanoparticles.

Particle Size Analysis

The average size of the particles and polydispersity index (PDI) of the synthesized silver nanoparticles were determined by a particle size analyzer and the results are shown in Figure 4. Data shows the average particle diameter is 103.2 nm and the Polydispersity index is 0.543 indicating that the particle size distribution is narrow.

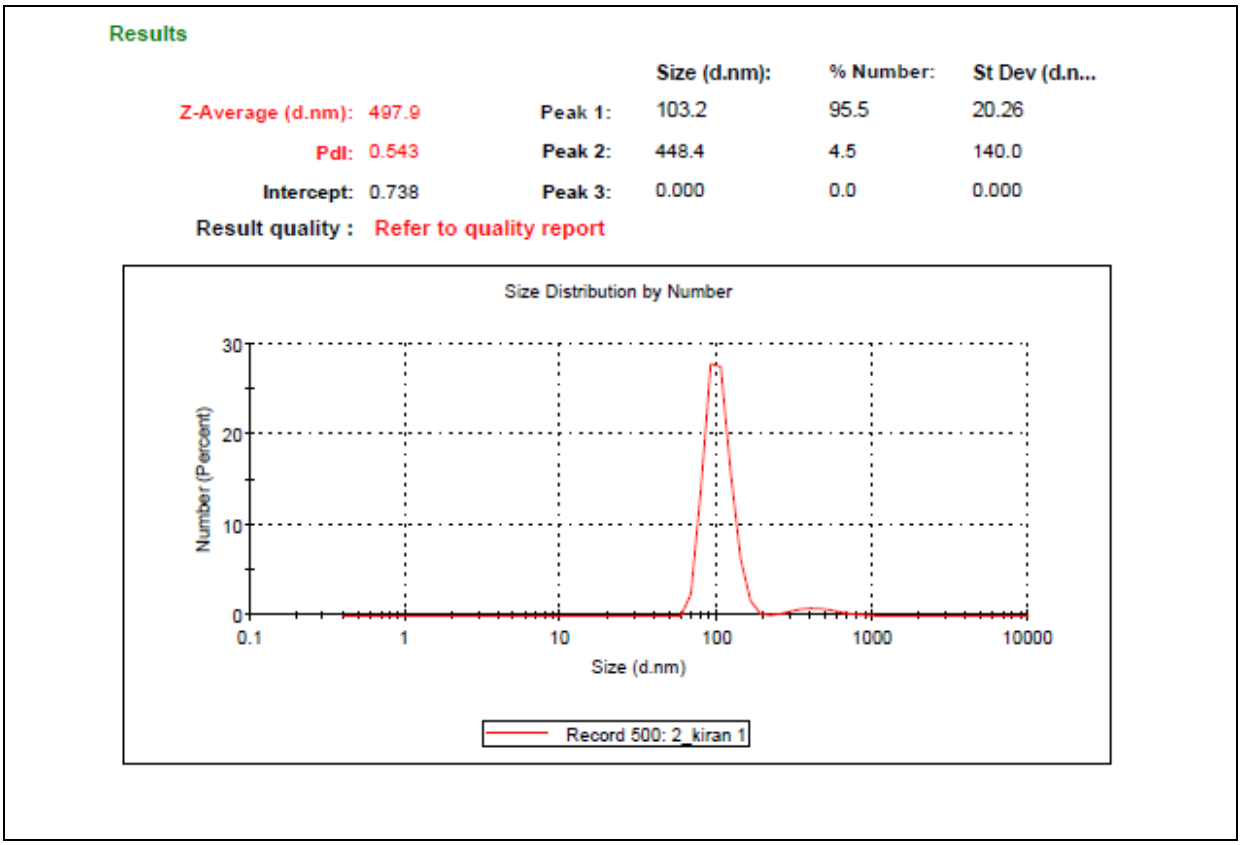

Figure no 4: Particle size analysis of silver nanoparticles.

Zeta potential of synthesized nanoparticles

Zeta potential indicates the surface charge on the silver particles which gives an idea of particle stability in the formulation. Results indicate that synthesized silver nanoparticles had a zeta potential value of +11.2mV (Figure 5). This indicates that the surface of synthesized silver nanoparticles is positively charged and proves that the particles will be stable in solution due to repulsive forces.

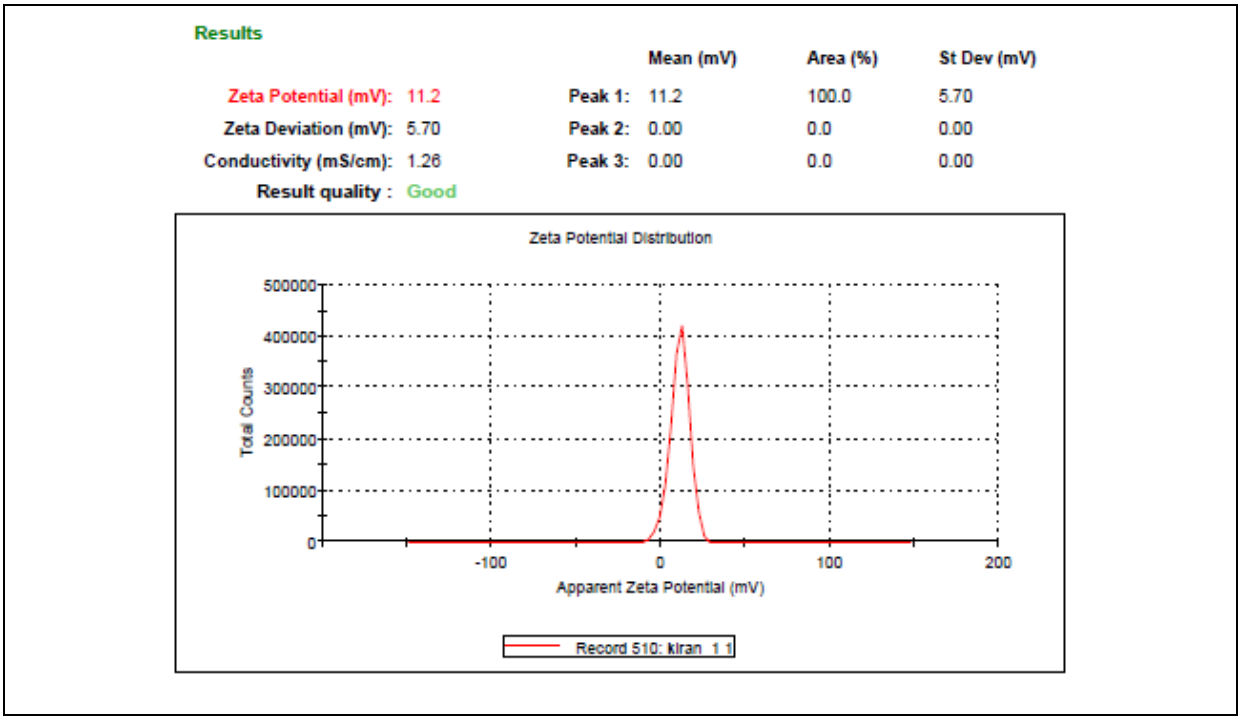

Figure no 5: Zeta Potential of silver nanoparticles

Scanning Electron Microscopy

As seen in this micrograph (Figure 6), Rod-shaped nanoparticles in the size range of 100 nm-300 nm were observed. Aggregation of particles was not seen indicating that silver nanoparticles have surface charge and were stabilized by trisodium citrate (TSC) as a capping agent.[30,31]

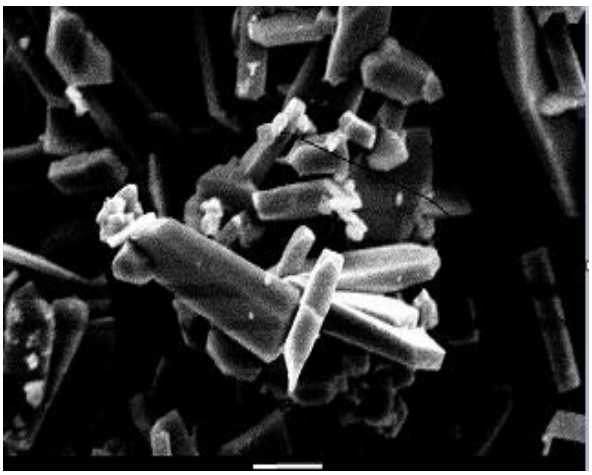

Figure no 6: SEM of Silver Nanoparticles

X-ray diffraction (XRD)

Figure 7 shows the XRD peaks of the silver nanoparticle. The peaks were observed at 2θ values of 38.02°, 44.65° and 64°corresponding to Ag (111), (200), and (220) planes respectively. High intensity at 38.02° reflection indicates that the crystallites are mainly orienting in this plane. The XRD pattern also showed a few additional intense peaks which are weaker than the silver peaks, these may be attributed to the presence of inorganic compounds on the surface of silver nanoparticles. Other studies prove ample proof of the presence of constituents on the surface of nanoparticles when they are synthesized by chemical route. [32,33]

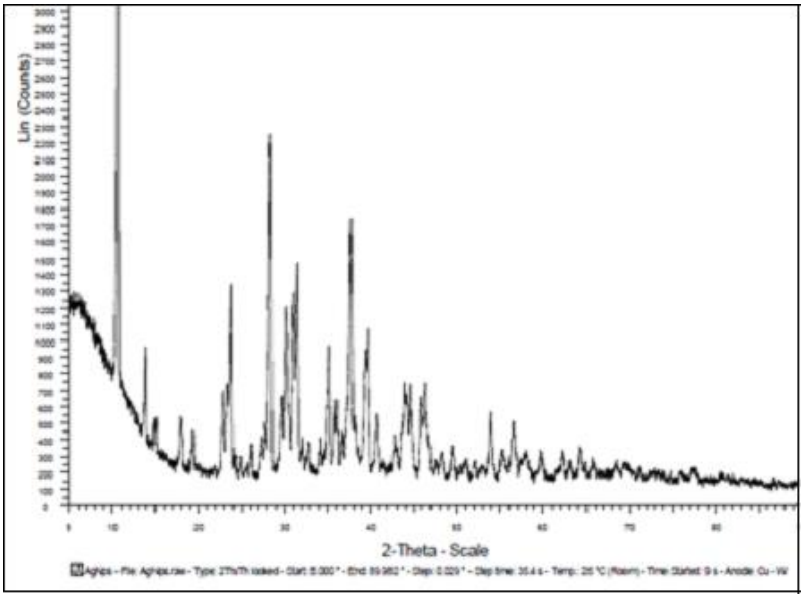

Figure No 7: XRD of silver nanoparticles

CONCLUSION:

There are considerable studies that have been carried out on silver NPs elucidating their role as promising antimicrobial, antibacterial, antioxidant, and anticancer agents. In the current study, a simple and facile method of synthesis of silver nanoparticles with small size was developed. The silver nanoparticles are prepared by chemical reduction method using sodium citrate as a reducing agent. We found that the silver nanoparticles with a size about of 103 nm and good distribution are prepared without using any surfactant or stabilizer. The primary indicator of the completion of the reduction of silver ions is a change in color from colorless to pale brown. In this study, the synthesized NPs were evaluated and confirmed to be in the nanometric size range, having acceptable surface charge. Minimal aggregation of particles

was confirmed by SEM analysis indicating that the procedure employed for synthesis was also contributing to capping and stabilization. Further, these particles were examined by SEM and found to have rod shape. The current study contributes to the body of knowledge regarding AgNPs and provides a simple reproducible technique of silver NP synthesis. Application of Ag NPs based on these findings may lead to valuable discoveries not only in the anti-bacterial field but also in various fields such as medical agents.[34-36]

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