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SYNTHESIS AND CHARACTERIZATION OF BUTYL GALLATE LOADED SILVER NANOPARTICLES BY PROBE SONICATION

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ABSTRACT

In recent scientific advancements, Nanotechnology has emerged as a prominent and dynamic field of study. It revolves around the manipulation and utilization of nanoparticles, which typically possess dimensions ranging from 1 to 100 nanometers in one dimension. This field has gained substantial significance across various disciplines, including medical chemistry, atomic physics, and numerous other scientific domains. Nanoparticles have garnered widespread attention due to their minuscule size, unique properties, and their demonstrated ability to influence the performance of materials they interact with. These tiny particles can be conveniently synthesized through various methods, including chemical, physical, and biological approaches. Among these methods, the biological approach stands out as the most promising, as it is not only simpler but also environmentally friendly and less time-consuming. The synthesis process involved the utilization of probe sonication technique on an aqueous solution containing Butyl Gallate and AgNO3. Silver nanoparticles were particularly intriguing for this study due to their remarkable physical and chemical characteristics. A fixed ratio of plant extract to metal ions was meticulously prepared, and a notable color change was observed, providing strong evidence of nanoparticle formation. To characterize these nanoparticles, a comprehensive array of analytical techniques was employed, including UV-vis Spectrophotometry, FTIR (Fourier-transform infrared spectroscopy), DLS (Dynamic Light Scattering), Zeta Analysis, XRD (X-ray diffraction), and SEM (Scanning Electron Microscopy). These analyses collectively revealed that the nanoparticles exhibited sizeswithin the range of 160 to 180 nanometers.

KEYWORDS: Nanotechnology, Nanoparticles, Butyl Gallate.

INTRODUCTION

Silver nanoparticles are of interest because of the unique properties (*e.g.*, size and shape depending optical, electrical, and magnetic properties) which can be incorporated into antimicrobial applications, biosensor materials, composite fibers, cryogenic superconducting materials, cosmetic products, and electronic components.[1-3] Several physical and chemical methods have been used for synthesizing and stabilizing silver nanoparticles. The most popular chemical approaches, including chemical reduction using a variety of organic and inorganic reducing agents, electrochemical techniques, physicochemical reduction, and radiolysis are widely used for the synthesis of silver nanoparticles.[4-7] Recently, nanoparticle synthesis is among the most interesting scientific areas of inquiry, and there is growing attention to produce nanoparticles using environmentally friendly methods (green chemistry).[8-10] Green synthesis approaches include mixed-valence polyoxometalates, polysaccharides, Tollens, biological, and irradiation method which have advantages over conventional methods involving chemical agents associated with environmental toxicity.[11,12] This chapter presents an overview of silver nanoparticle preparation by physical, chemical, and green synthesis approaches. The aim of this chapter is, therefore, to reflect on the current state and future prospects, especially the potentials and limitations of the above mentioned techniques for industries. Moreover, we discuss the applications of silvernanoparticles and their incorporation into other materials, the mechanistic aspects of the antimicrobial effects of silver nanoparticles.[13-15]

MATERIALS AND METHODS

Synthesis of Silver Nanoparticles

Four concentration ratios of plant and metal ions were prepared (30:1, 60:1,120:1 & 240:1) by increasing the concentration of plant extract concentration in the solution. 0.17% of 1mM AgNO3 metal ion was added in the prepared plant extract. 30 mL of Butyl Gallate solution was mixed with 1 mL aqueous 1MoL NaOH adjusted solution of silver nitrate (0.1 Mol) and the solution pH 9. The solution was then emulsified by subjection to high-power ultrasonic vibration in probe sonicator for different time intervals. Then the solution slowly changed colourless to deep brown colour in ionic liquid phase that indicates the formation of AgNPs. After the complete reduction, this solution was centrifuged at 10,000 rpm for 30 min at 4^{o} C. The supernatant was discarded, and the pellet was re-dispersed in dd H2O. Next, the

pellet(NPs) was dried and stored at vial for further use. Then the bio reduced nanoparticles was used for characterization.

CHARACTERIZATION OF SILVER NANOPARTICLES

FTIR analysis

The chemical composition of the synthesized silver nanoparticles was studied by using FTIR spectrometer **(**perkin-Elmer LS-55- Luminescence spectrometer**)**. The solutions were dried at 75^{o} C and the dried powders were characterized in the range 4000–400 cm^{-1} using KBr pellet method.

SEM Analysis

The morphological features of synthesized silver nanoparticles from neem plant extract were studied by Scanning Electron Microscope (JSM-6480 LV). After 24Hrs. of the addition of $AgNO_3$ the SEM slides were prepared by making a smear of the solutions on slides. A thin layer of platinum was coated to make the samples conductive. Then the samples were characterized in the SEM at an accelerating voltage of 20 KV.

DLS & Zeta-Potential Analysis

Dynamic light scattering (DLS) which is based on the laser diffraction method with multiple scattering techniques was employed to study the average particle size of silver nanoparticles. The prepared sample was dispersed in deionized water followed by ultra-sonication. Then solution was filtered and centrifuged for 15 min.at 25^{o}C with 5000 rpm and the supernatant was collected. The supernatant was diluted for 4 to 5 times and then the particle distribution in liquid was studied in a computer- c o n t r o l l e d particle size analyzer (ZETA sizer Nanoseries, Malvern instrument Nano Zs).

RESULTS AND DISCUSSION

SEM ANALYSIS

SEM provided further insight into the morphology and size details of the silver nanoparticles. Comparison of experimental results showed that the diameters of prepared nanoparticles in the solution have sizes of several µm in case of 30:1, 120:1 & 240:1 ratio where as in 60:1 ratio the size is of several nm. **(Figure: 1, Figure: 2, Figure: 3 & Figure: 4)**. The size of the prepared nanoparticles was more than the size of nanoparticle which should be; i.e.; between 1-100 nm. The size was more than the desired size as a result of the proteins which were bound in the surface of the nanoparticles.

The result showed that the particles were of spherical shape in case of 30:1, 60:1, and 120:1 ratio but sheet shape in case of 240:1 ratio. The shape varies dueto the concentration increased in 240:1 ratio.

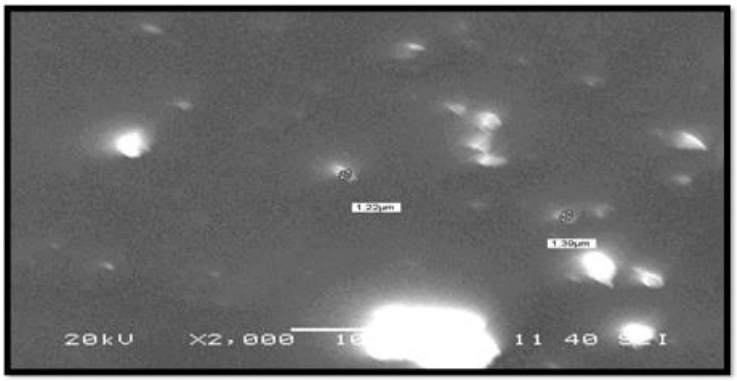

Fig. 1: SEM image for 30:1 ratio silver nanoparticles.

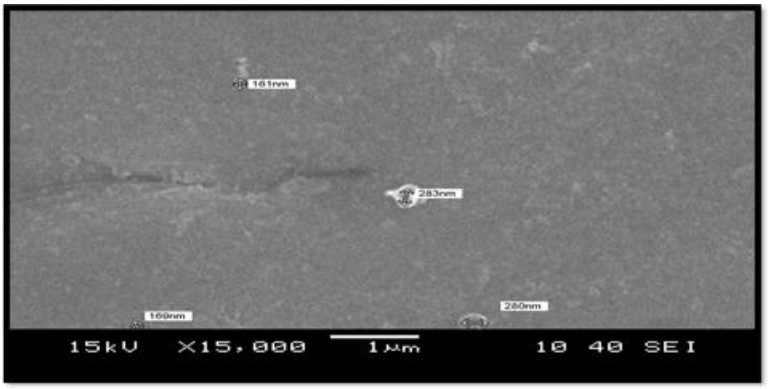

Fig. 2: SEM image for 60:1 ratio silver nanoparticles.

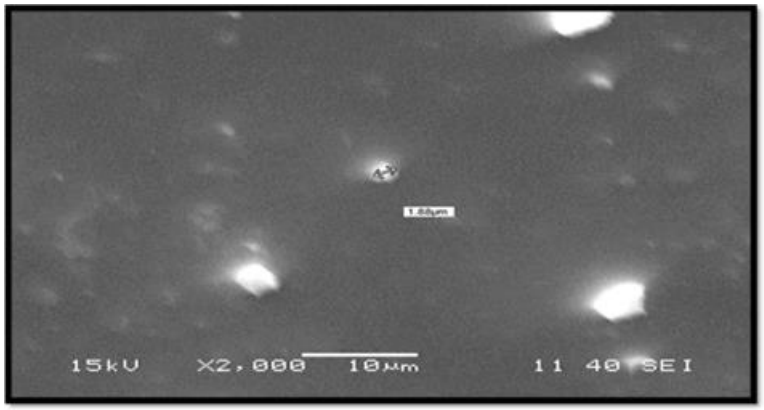

Fig. 3: SEM image for 120:1 ratio silver nanoparticle.

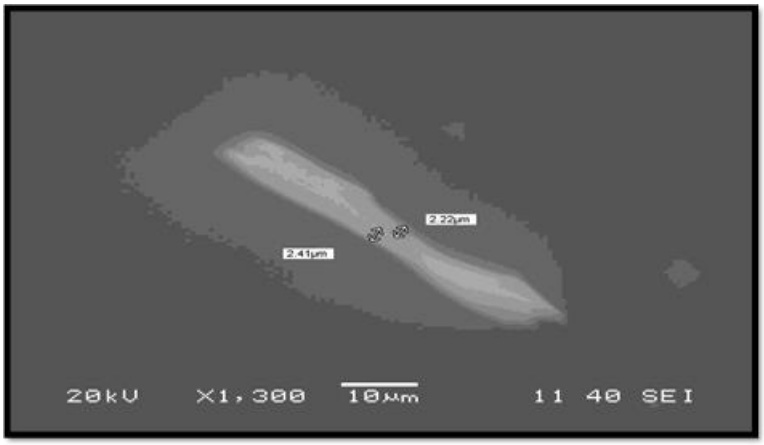

Fig. 4: SEM image for 240:1 ratio silver nanoparticle.

DLS ANALYSIS

The particle size distribution (PSD) of synthesized silver nanopaericles of different ratios like 30:1, 60:1, 120:1, and 240:1 is shown in the figures. **(Figure: 5, Figure: 6, Figure: 7 & Figure: 8)**. According to the **figure: 5** the colloidal solution of silver nanoparticles of ratio 30:1 contains particles of different sizes some were with average sizes ranging from 5 nm to 180 nm. But in case of 60:1, the solution contains particles of uniform sizes ranging from 68 nm to 396 nm. The average size of nanoparticles is 160 nm. The particle size in case of 120:1 ratio range from 78 nm to 255 nm with mean particle size of 169 nm. Similarly, the sizes of nanoparticles in case of 240:1 ratio range from 91 nm to 220 nm with average size of 164 nm. If we compare the above four results, we can conclude that the ratios like 60:1, 120:1, 240:1 give uniform distribution of particles but 30:1 ratio does not obey this principle. Among them 60:1 ratio is very appropriate since it gives lowest average size of nanoparticles.

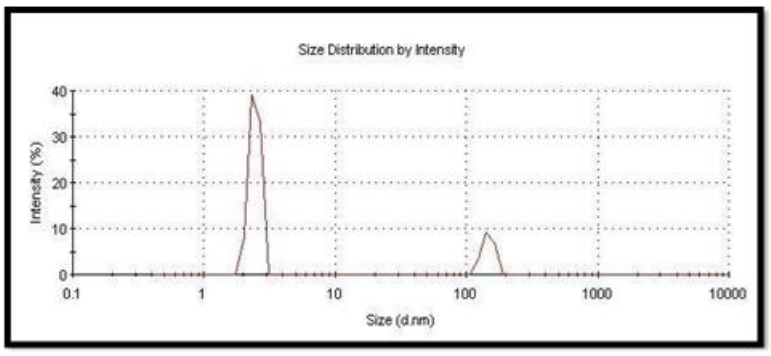

Fig. 5: DLS result for 30:1 ratio silver nanoparticle.

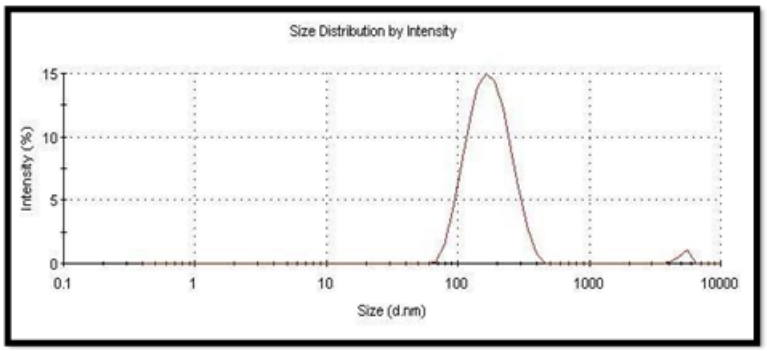

Fig. 6: DLS result for 60:1 ratio silver nanoparticles.

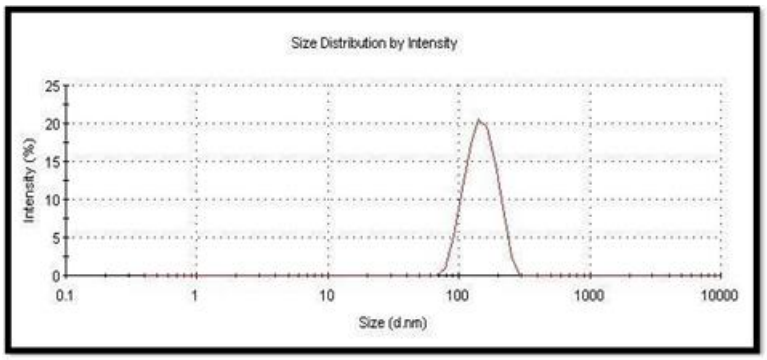

Fig. 7: DLS result for 120:1 ratio silver nanoparticles.

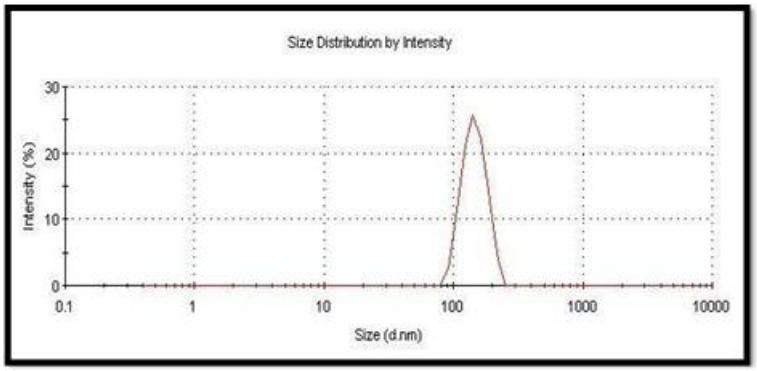

Fig. 8: DLS result for 240:1 ratio silver nanoparticle.

ZETA POTENTIAL ANALYSIS

The Zeta potential measurements of silver nanoparticles synthesized with different ratios like 30:1, 60:1, 120:1, and 240:1 is 15.5 mV, 1.92 mV, 6. 12 mV, and 2.45 mV respectively. (**Figure: 9, Figure: 10, Figure: 11 & Figure: 12)**. From the analysis the order of stability of nanoparticles synthesized from different ratios is 30:1> 120:1 > 240:1 > 60:1. Nanoparticles are very small in size for which they are energetically very unstable. Therefore, the particles undergo agglomeration/aggregation to stabilize themselves. So, there were some potential charges on the surface of the nanoparticles which makes them

stable. These charge potential we got from this analysis.

Zeta potential (Surface potential) has direct relation with the stability of a form/structure as mentioned below (**Table: 1)**

Table 1: A table showing the stability of the NPs according to the potential charge.

Zeta potential [mV]	Stability behavior of the colloid
from 0 to ±5	Rapid coagulation or flocculation
from ±10 to ±30	Incipient instability
from ±30 to ±40	Moderate stability
from ±40 to ±60	Good stability
more than ±61	Excellent stability

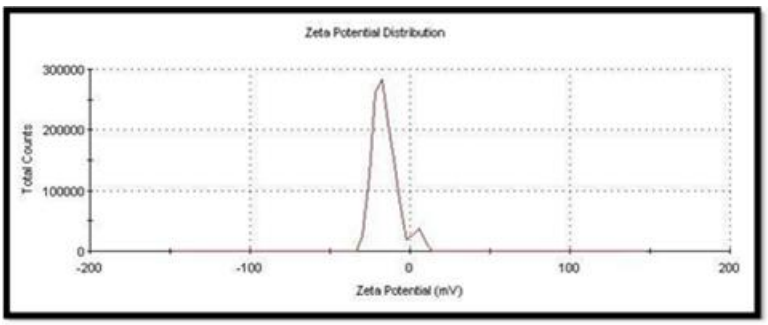

Fig.9: Zeta Analysis result for 30:1 ratio silver nanoparticle.

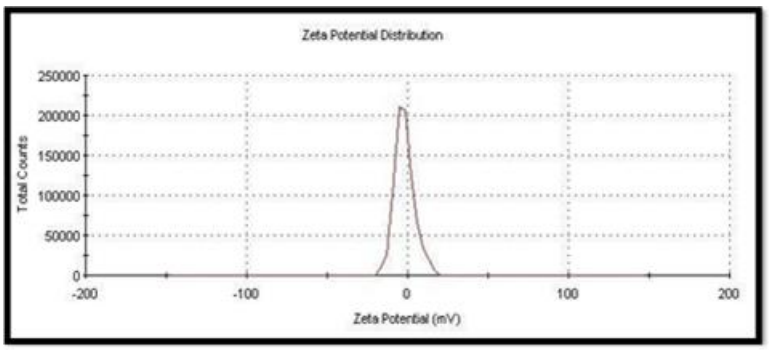

Fig.10: Zeta Analysis result for 60:1 ratio silver nanoparticle.

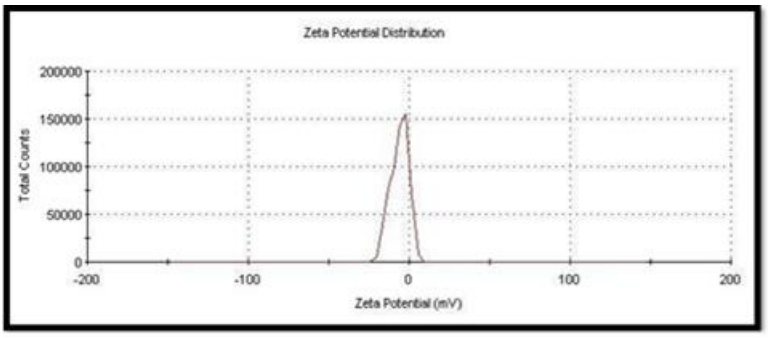

Fig. 11: Zeta Analysis result for 120:1 ratio silver nanoparticle.

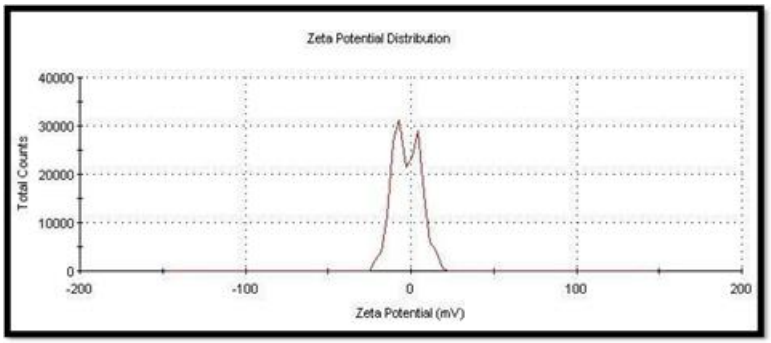

Fig. 12: Zeta Analysis result for 240:1 ratio silver nanoparticle.

FTIR ANALYSIS

FTIR measurements were carried out to identify the biomolecules for capping and efficient stabilization of the metal nanoparticles synthesized. The FTIR spectrum of silver nanoparticles **(Figure: 13 & Figure: 14)** in case both of 60:1 and 120:1 ratioshowed the band between 3490-3500 cm^{-1} corresponds to O-H stretching H-bonded alcohols and phenols. The peak found around 1500-1550 cm^{-1} showed a stretch for C-H bond, peak around 1450-1500 cm^{-1} showed the bond stretch for N-H. Wheres the stretch forAg- NPs were found around 500-550 cm^{-1}. Therefore, the synthesized nanoparticles were surrounded by proteins and metabolites such as terpenoids having functional groups. From the analysis of FTIR studies we confirmed that the carbonyl groups from the amino acid residues and proteins has the stronger ability to bind metal indicating that the proteins could possibly from the metal nanoparticles (i.e.; capping of silver nanoparticles) to prevent agglomeration and thereby stabilize the medium. This suggests that the biological molecules could possibly perform dual functions of formation and stabilization of silver nanoparticles in the aqueous medium. Carbonyl groups proved that flavanones or terpenoids absorbed on the surface of metal nanoparticles. Flavanones or terpenoids could be adsorbed on the surface of metal nanoparticles, possibly by interaction through carbonyl groups or π-electrons in the absence of other strong ligating agents in sufficient concentration. The presence of reducing sugars in the solution could be responsible for the reduction of metal ions and formation of the corresponding metal nanoparticles. It is also possible that the terpenoids play a role in reduction of metal ions by oxidation of aldehydic groups in the molecules to carboxylic acids. These issues can be addressed once the various fractions of the neem leaf extract are separated, identified and individually assayed for reduction of the metal ions. This rather elaborate study is currently underway.

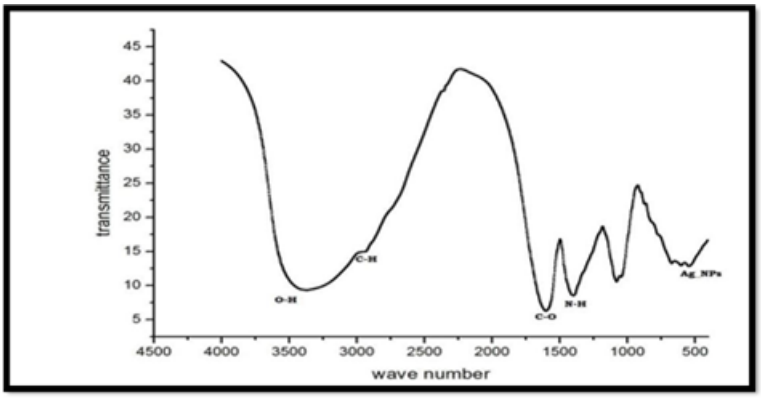

Fig. 13: FTIR result for 60:1 ratio silver nanoparticle.

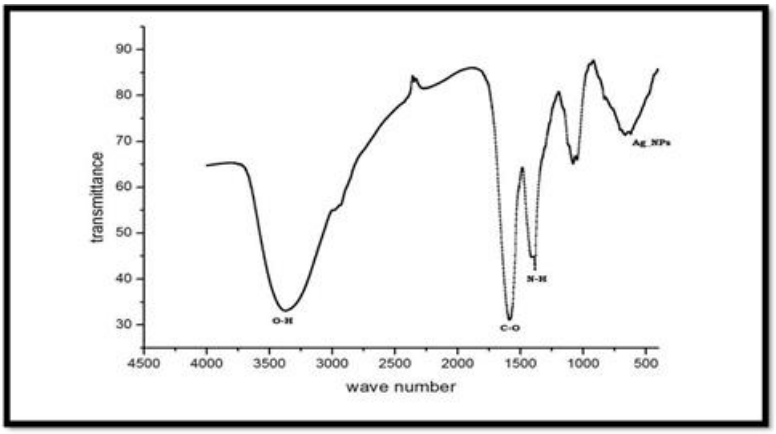

Fig. 14: FTIR result for 120:1 ratio silver nanoparticles.

CONCLUSION

The rapid synthesis of silver nanoparticles using Butyl gallate provides environmentally friendly, simple and efficient route for synthesis of benign nanoparticles. The synthesized nanoparticles were of spherical and sheet shaped and the estimated sizes were 160-180 nm. The size was bigger as the nanoparticles were surrounded by a thin layer of proteins and metabolites having functional groups of amines, alcohols, ketones, aldehydes, etc., which were found from the characterization using SEM, DLS, Zeta Analyzer, and FTIR techniques. All these techniques it was proved that the concentration of Butyl Gallate to metal ion ratio plays an important role in the shape determination of the nanoparticles. The higher concentrated nanoparticles had sheet shaped appearance whereas the lower concentrations showed spherical shaped. The sizes of the nanoparticles in different concentration were also different which depend on the reductionof metal ions. From the data of DLS it was found that the 30:1 ratio solution had sharp nanoparticles of around 5 nm and some has around 180 nm and the had the potential of around 15.5 mV. From the technological point of view these obtained silver nanoparticles have potential applications in the biomedical field and this

simple procedure has several advantages such as Higher Bioavailability compatibility for medical and pharmaceutical applications as well as large scale commercial production.

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