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BIOINSPIRED SYNTHESIS AND CHARACTERISATION OF SILVER NANOPARTICLES USING AQUEOUS SOLUTION OF *ALLIUM FISTULOSUM* LEAF EXTRACT

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ABSTRACT

In the present study Silver nanoparticles were synthesised from aqueous Silver nitrate through a simple and eco-friendly route using leaf extract of *Allium fistulosum* as reductant and stabilizer. Effect of *Allium fistulosum* leaf extract, Silver nitrate concentration, reaction time and temperature on reaction rate were investigated. The results recorded from UV-Visible Spectrometry, TEM and FTIR support the biosynthesis and characterisation of Silver nanoparticles. An intense Surface Plasmon Resonance band at 450 nm in the UV-Visible spectrum clearly reveals the reduction of Silver ions (Ag^+) to Silver (Ag^0) which indicates the formation of Silver nanoparticles. Water soluble organics present in the leaf extract are responsible for the reduction of Silver ions. This green method provides faster synthesis comparable to chemical methods and can be used in areas such as cosmetics, food and medicinal applications.

KEYWORDS: *Allium fistulosum,* Silver nanoparticles, Surface Plasmon Resonance, XRD.

INTRODUCTION

The field of nanotechnology is one of the most interesting areas of research in modern material science. Nanoparticles exhibit improved properties based specific characteristics such as size, distribution and morphology. Nanotechnology is mainly concerned with the synthesis of nanoparticles of variable sizes, shapes, chemical compositions and their potential use for human benefit.[1] In the recent years, noble metal nanoparticles have been the one of the focused research due to their unique optical, electronic, mechanical, magnetic, and chemical properties that are significantly different from those of bulk materials.[2] Various approaches using plant extract have been used for the synthesis of metal nanoparticles. These approaches have numerous advantages over chemical, physical methods as it is cost effective and environment friendly.[3-6] Many research papers reported the synthesis of silver nanoparticles using plant extracts such as Murraya koenigii (curry) leaf[7], Ocimum sanctum (Tulsi) leaf[8], Garcinia mangostana (mangosteen) leaf[9], Nicotiana tobaccum leaf[10], Citrus sinesis leaves[11], Arbutus unedo leaf[12], Ficus benghalensis leaf[13], mulberry leaves[14] and Olea europaea leaves.[15] Considering the vast potentiality of plants as sources this work aims to apply a biological green technique for the synthesis of silver nanoparticles as an alternative to conventional methods. In this regard, leaf extract of Allium *fistulosum* (commonly known as spring onion/bunching onion) a species of family Amaryllidaceae was used for bioconversion of silver ions to nanoparticles. Japanese bunching onion is a widely known pe-rennial plant in cultivation and usage in Europe, and especially in the Far East countries – Japan, Korea, and China.[16] In Poland it does not have a great significance, because the dominating species is the common onion, which is grown not only for the bulb, but also for its green leaves.An advantage of the Japanese bunching onion is its rapid growth, creating a large bunch of leaves of delicate consistency, which contain a great amount of vitamin C, flavonoids and other ingredients important for health.[17] Silver nanoparticles can be produced at low concentration of leaf extract without using any additional harmful chemical/physical methods. The effect of concentration of metal ions and concentration of leaf extract were also evaluated to optimize route to synthesise silver nanoparticle. The method applied here is simple, cost effective, easy to perform and sustainable.

MATERIALS AND METHOD

Materials: *Allium fistulosum* used for the preparation of extract was procured from a local supermarket Fig.1. The silver nitrate was supplied by Sigma-Aldrich Chemicals. All chemicals and reagents used in the study were of Analytical grade.

Fig.1: *Allium fistulosum.*

Preparation of the Extract

Allium fistulosum was used to make the aqueous extract. Fresh *Allium fistulosum* leaves weighing 25g were accurately weighed, thoroughly washed under running tap water followed by washing it with distilled water to remove surface impurities. They were cut into fine pieces, crushed into 100ml distilled water and heated over water bath maintained at 75^0 C for 15 minutes. The extract obtained was filtered through Whatmann No.1 filter paper and used for the synthesis of silver nanoparticles.

Phytochemical Screening

Preliminary phytochemical screening was carried out for the identification of flavanoids, phenols and alkaloids.[18]

Synthesis of Silver Nanoparticles

Aqueous solution of 1 mM Silver nitrate was prepared and used for the synthesis of silver nanoparticles. 5 ml of aqueous *Allium fistulosum* extract is mixed with 45 ml of silver nitrate for the synthesis of silver nanoparticles. The formation of silver nanoparticles is confirmed by colour change from pale yellow to brown and by UV-Visible spectroscopy.

Characterisation of Silver Nanoparticles

Visual Confirmation - The bioreduction of silver nitrate using aqueous *Allium fistulosum* extract was monitored and the appearance of brown colour indicates the formation of silver nanoparticles.

UV-Vis Spectroscopy - The reduction of silver nitrate to silver using aqueous *Allium fistulosum* extract was monitored by recording UV - Vis spectrum of the reaction mixture. The measurements are recorded on Shimadzu UV 2450 spectrophotometer.

FT-IR Analysis – FT-IR Spectroscopic analysis was carried out for silver nanoparticles to identify the possible bioactive molecules responsible for the reduction of the silver ions. The FT-IR spectrum of silver nanoparticles was obtained using Brucker model within the mid IR region of 400-4000 cm^{-1}. The dried experimental sample was mixed with KBr crystals and the spectrum was recorded in transmittance mode.

Transmission Electron Microscopy (TEM) - TEM technique was employed to visualise the shape of silver nanoparticles. TEM grid was prepared by placing a drop of the particle solution and drying under an IR lamp.

Fixation of different parameters – The reaction was monitored at different time intervals. The reaction was monitored using different concentration of silver nitrate (2mM, 4mM, 6mM and 1mM) and also by varying leaf extract solution (1-5ml) and their absorbance was measured.

RESULTS AND DISCUSSION

Phytochemical evaluation – The results of the phytochemical evaluation of the *Allium fistulosum* extract indicates the presence of metabolites such as flavanoids, phenols and alkaloids. The presence of phenolic compounds constitutes a major group of compounds that act as primary antioxidants which are responsible for the reducing property of the *Allium fistulosum* extract is shown in Fig.2. Phenolic compounds are very important plant constituents because of the scavenging ability of their –OH groups.

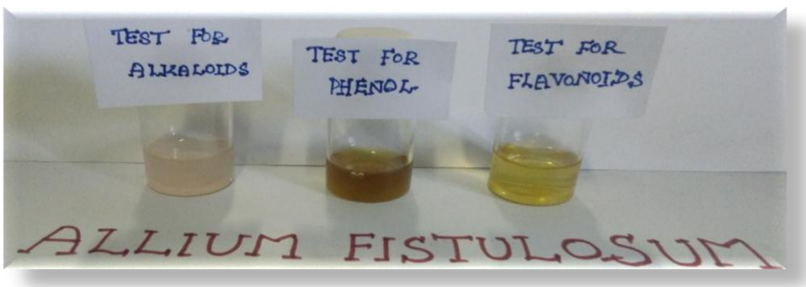

Fig 2: Phytochemical evaluation.

Visual observation and UV-Vis spectroscopy– As the *Allium fistulosum* extract was mixed with aqueous solution of 1mM silver nitrate it started to change colour from pale yellow to brown within reaction duration due to excitation of surface Plasmon vibrations in silver nanoparticles Fig.3.[19]

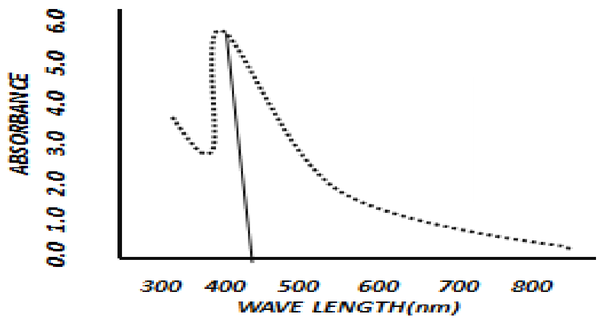

Fig 3: UV-Vis absorption spectrum of leaf extract of *Allium fistulosum*.

On addition of different concentration (1-5 ml) of leaf extracts to aqueous silver nitrate solution keeping its concentration 10 ml (1mM) constant, the colour of the solution changed from pale yellow to brown indicating formation of silver nanoparticles Fig. 4. Different parameters were optimized including concentration of silver nitrate and *Allium fistulosum* leaf extract, and time which had been identified as factors affecting the yields of silver nanoparticles. Silver nanoparticles were synthesised at different concentrations of leaf extract such as 1-5 ml using 1mM of silver nitrate were analysed by UV spetra of Plasmon resonance band observed at 430 – 450 nm similar to those reported in literature.[20] If we increase the leaf extract concentration to 4ml, the absorption spectrum shifted towards red (from 410 – 450 nm), indicating an increase in the size of silver nanoparticles[21] Fig 5.

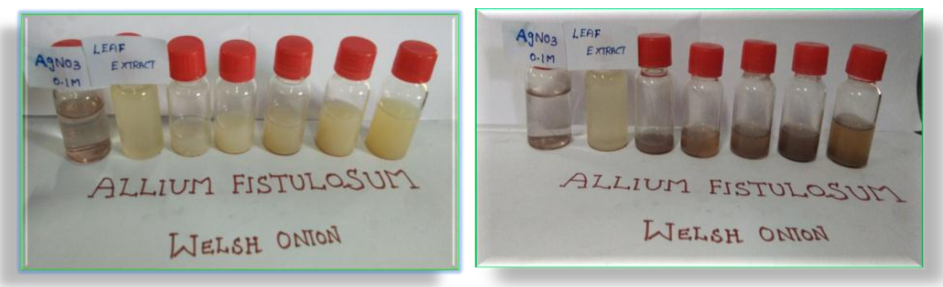

Fig.4 Digital optical image of synthesised AgNps with different conc(1-5ml) of leaf extract.

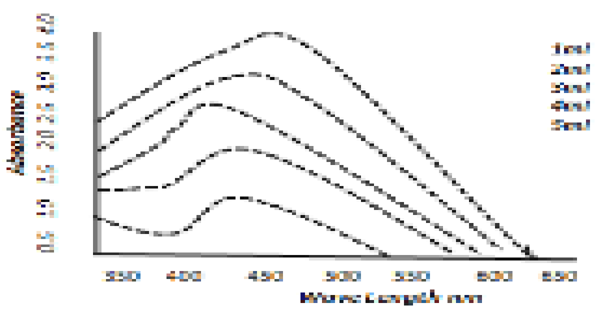

Fig 5: Absorption spectra of AgNps with various concentration (1-5ml) of leaf extract.

Parallel changes in colour have been observed when different concentrations (0.02mM – 0.08mM) of silver nitrate was used by keeping plant extract (1ml) constant Fig.6. The appearance of the brown colour was due to the excitation of the Surface Plasmon Resonance (SPR), typical of silver nanoparticles having adsorbance values which were reported earlier in the visible range of 440 – 450 nm[22] Fig.7.

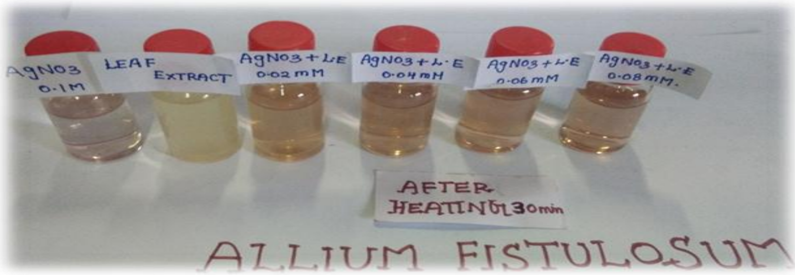

Fig.6 Digital optical image of synthesised AgNps with different conc (0.02-0.08mM) of $AgNO_3$.

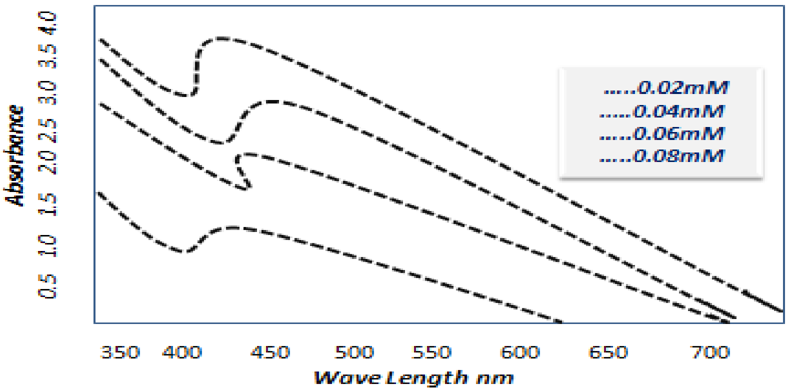

Fig.7 Absorption spectra of AgNps with various concentration (0.02-0.08mM) of $AgNO_3$.

Effect of Temperature on the formation of AgNPs

Temperature is an important factor that affects the synthesis of nanoparticles significantly.

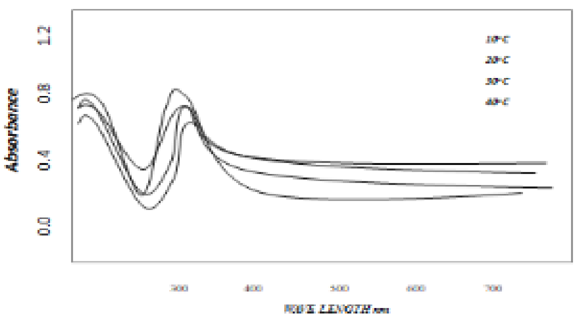

Fig. 8: Absorption spectra of AgNps at different reaction temperature (10 – 40^0C).

Fig. 8 shows the absorbance spectra of AgNps at different temperatures in the range of 10 – 40^{0}C. With the increase in temperature, the reduction of silver is enhanced as indicated by rapid change in the colour of the solution. The peak absorption wavelength shifted toward blue from 450 to 410 nm, as temperature varies from 10–40^{0}C. The shift in the band maximum is due to the localization of surface Plasmon resonance of the AgNPs. This indicates that the size of the nanoparticles decreases with increasing temperature, which may be due to the faster reaction rate at higher temperature. At high temperature, the kinetic energy of the molecules increases and silver ions gets consumed faster, thus leaving less possibility for particle size growth. Thus, smaller particles of uniform size distribution are formed at higher temperature. The present work is in complete correlation with the work report based on the banana peel extract.[23]

FTIR analysis

FT-IR spectrum was recorded to identify the possible phytochemical in *Allium fistulosum* leaf extract responsible for capping lead to efficient stabilisation of silver nanoparticles. (Fig.9) The absorption at 3377, 2926 cm^{-1} correspond to the O-H stretching vibrations of phenols. Peaks correspond to the wave numbers 1602, 1384, 1117, 1087 cm^{-1} show the presence of O-H stretching of alcohol/ phenol. The absorption peak between 917cm^{-1} & 617 cm^{-1} is responsible for C-H out of plane bending of aromatic hydrocarbons. These results confirms the presence of polyphenolic groups responsible of capping & stabilization of silver nanoparticles.

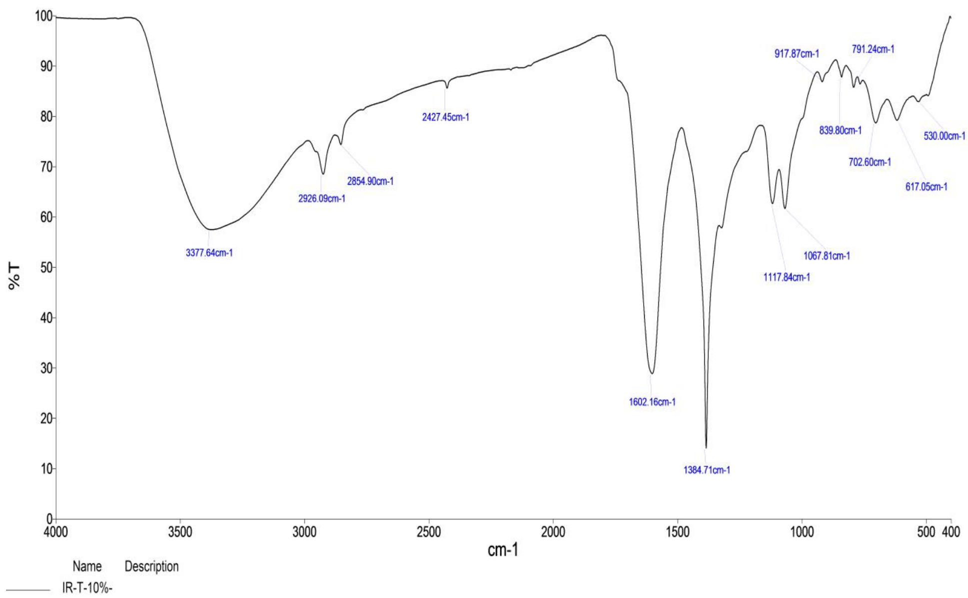

Fig. 9: FTIR spectra of AgNps synthesised from *Allium fistulosum*.

TEM analysis

Transmission Electron Microscopy (TEM) has been used to identify the size, shape and morphology of nanoparticles. It reveals that the silver nanoparticles are well dispersed and predominantly spherical in shape. TEM showed the nanoparticle formed are with diameter range 10-20 nm Fig 10.

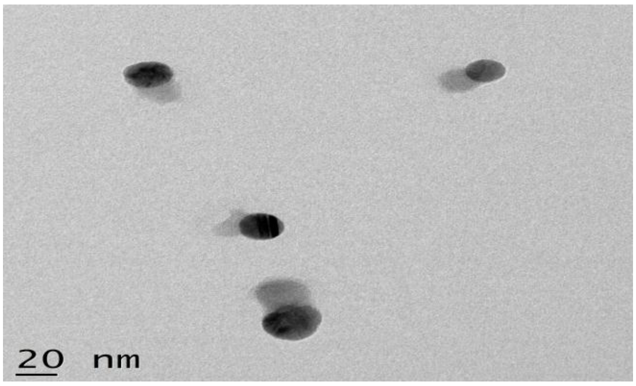

Fig 10: TEM image of silver nanoparticles synthesized from *Allium fistulosum*.

CONCLUSION

In conclusion, the present method of synthesis of silver nanoparticles is a rapid, green and economic. Reduction is accomplished probably due to phytochemicals such as phenols, flavanoids and alkaloids. Colour change from pale yellow to brown confirmed the formation of silver nanoparticles. Nanoparticles formed are spherical and the particle size of 20 nm are obtained. Colour change occur due to the surface plasmon resonance during the reaction with the ingredients present in the *Allium fistulosum* leaf extract resulting in the formation of silver nanoparticles, which is confirmed by UV-Vis Spectroscopy, FT-IR and TEM. By changing these environmental parameters, the size and shape of the synthesised nanoparticles can be altered.

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