Materials Letters 188 (2017) 197–200

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

Phytofabrication and encapsulated of silver nanoparticles from *Gloriosa superba*

Muthukrishnan Saradha Devi[a,*], Kaliyaperumal Ashokkumar[b], Subramanian Annapoorani[c]

[a] *Department of Biotechnology, Bharathiar University, Coimbatore, Tamil Nadu, India*
[b] *Department of Plant Biotechnology, TNAU, Coimbatore, Tamil Nadu, India*
[c] *Department of Biochemistry Avinashilingam University, Coimbatore, Tamil Nadu, India*

ARTICLE INFO

Keywords:
Nanoparticles
Bioactive capping
Electron microscope
X ray diffraction
Gloriosa superba

ABSTRACT

The present study has been investigated for the formation of silver nanoparticles (AgNPs) toxicity was reduced by a biological route employing methanolic extract of *Gloriosa superba* seed (MGsS) extract. AgNPs protected with -CH2- group, –NH2– groups and –CO– groups of colchicines and colchicine derivatives of *Gloriosa superba* seed extract are not cytotoxic, while, by contrast bare AgNPs have been found to be rather toxic. Capping and efficient stabilization of silver ions by MGsS confirmed by FTIR analysis. TEM analysis also revealed that the edges of the particles were lighter than the centers, confirmed that the bioorganic compounds such as alkaloids (colchicines) in MGsS capped the silver NPs contributing to reduction of Ag^{+} ions to Ag^{o}. Formation of AgNPs was confirmed by UV–visible absorbance peaks at 360 and 440 nm clearly indicated the interaction between AgNPs and biomolecules present in the MGsS. The XRD study confirmed the crystalline face-centered cubic lattice of AgNPs of methanolic extract of *Gloriosa superba* seed (AgMGsS). The average size of the nanoparticles was determined to be about 35–42 nm.

1. Introduction

Various researchers found that silver nanoparticles had a toxic effect on cells, suppressing cellular growth, multiplication and causing cell death depending on concentrations and duration of exposure. To reduce the toxicity of AgNPs is vital to select stabilizing agents and pathways that are environmentally friendly, non toxic and easy to implement. Plants are nature's “chemical factories”. Organic layer protected AgNPs-Tiopronin AgNPs [1], BSA capped Ag- Pt alloy NPs [2] and AgNPs protected with Na+-poly γ -glutamic acid [3] are not cytotoxic, while, by contrast bare AgNPs have been found to be rather toxic [4]. Furthermore, green synthesis of nanoparticles has been offered as a cost effective environmental friendly alternative to chemical and physical methods [5]. Currently, plant extracts act as biological-shell, reducing and capping agents for the synthesis of nanoparticles, has received special attention among others, due to maintaining an aseptic environment during the process [6–10]. Therefore, medicinal plants with therapeutic importance are being widely used for the size and shape controlled synthesis of silver nanoparticles [11–14]. Additionally, AgNPs synthesized using herbals have been reported to have good antioxidant, anti-fungal and anti-bacterial properties [15,16].

Gloriosa superba (L.) is often referred to as Malabar glory and it is a perennial creeper within the Liliaceae family, native to Africa continent and South-East Asia. *G. superba* are used in skin disease, wound, gout antidote to snake bite and for inflammatory disease [17–19]. In this perspective, we report first time the synthesis of bioactive silver capped with methanolic seed extract of *G. superba*.

2. Materials and methods

2.1. Reagents

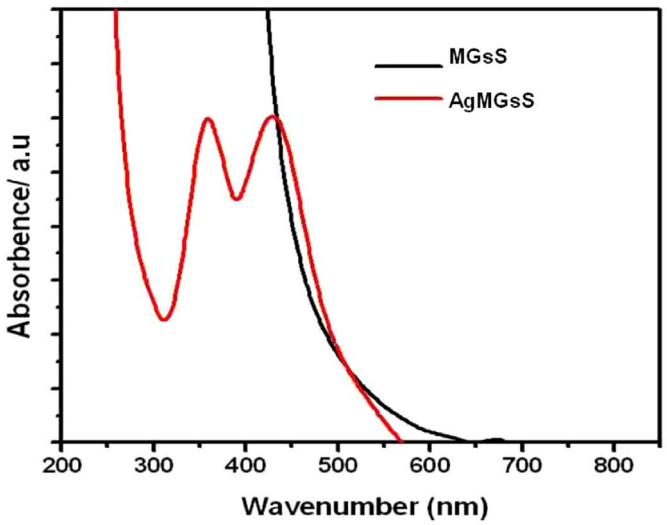
In this experimental study Silver nitrate, chemicals and enzymes were purchased from Sigma-Aldrich, (Bangalore, India) and SISCO Research Laboratories (Maharashtra, India). All chemicals used were of analytical grade.

2.2. Preparation of the extract

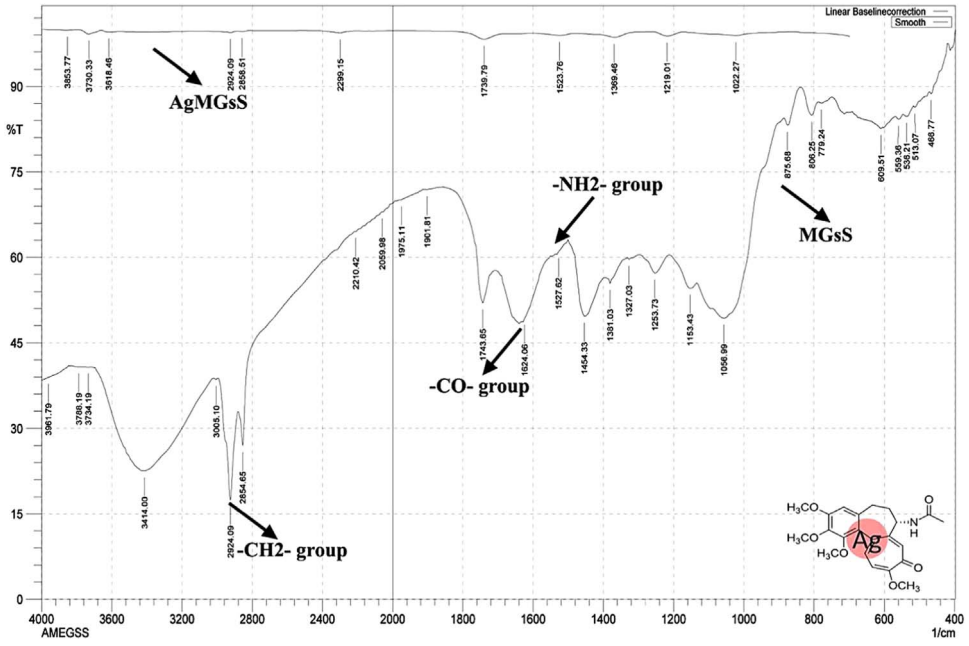
Fresh seeds of *G. superba* were collected from outskirts of Thanjavur district India. The plant was identified and authenticated from Department of Botany, Avinashilingam University, Tamil Nadu (India). The collected seeds were washed, shade dried and powdered. Ten grams of seed powder

* Corresponding author.
E-mail address: sribio770@gmail.com (M. Saradha Devi).

http://dx.doi.org/10.1016/j.matlet.2016.11.016
Received 6 January 2016; Received in revised form 2 September 2016; Accepted 5 November 2016
Available online 12 November 2016



A. UV-vis spectra of MGsS and AgMGsS



B. FT-IR spectrum of AgMGsS and MGsS

Fig. 1. A. UV–vis spectra of MGsS and AgMGsS. B. FT-IR spectrum of AgMGsS and MGsS.

of *G. superba* were extracted with 150 ml of methanol.. The distillated and condensed extract was used for the synthesis of silver nanoparticles. Besides, the extract was analysed for its major phytochemical constituents.

2.3. Synthesis of silver nanoparticles

Aqueous solution (1 mM) of silver nitrate was prepared by using deionized water used for the synthesis of AgNPs. 10 ml of methanol seed extract was mixed with 90 ml of 1 mM silver nitrate solution and incubated for a period of 15 h at room temperature. The color changed from yellowish brown to dark brown color indicates the formation of silver nanoparticles.

2.4. Characterization

The bioreduction of Ag^{+} ion in solution was monitored using UV–visible spectrometer (Perkin-Elmer). Further characterization was done using Perkin-Elmer spectrometer FT-IR Spectrum ONE in the range of 4000–400 cm^{-1} at a resolution of 4 cm^{-1}. The sample was mixed with KCl procured from Sigma. Thin sample disc was prepared by pressing with the disc preparing machine and subjected to FT-IR analysis [20]. Morphological characterization of the samples was carried out using LV-SEM (JEOL JSM-6480). A pinch of dried sample was coated on a carbon tape and platinum in an auto fine coater and subject to analysis. Samples for transmission electron microscopy (TEM) were prepared by drop-coating the Ag nanoparticle solutions onto carbon-coated copper grids and measured using a TECHNAI10-Philphs instrument. The average particle size and zeta potential of nanoparticles were performed on a Malvern Zetasizer Ver. 6.32 (Malvern Instruments, Malvern, UK). X-ray diffraction (XRD) analysis, carried out on a Phillips X′pert 1(Phillips, USA) XRD instrument

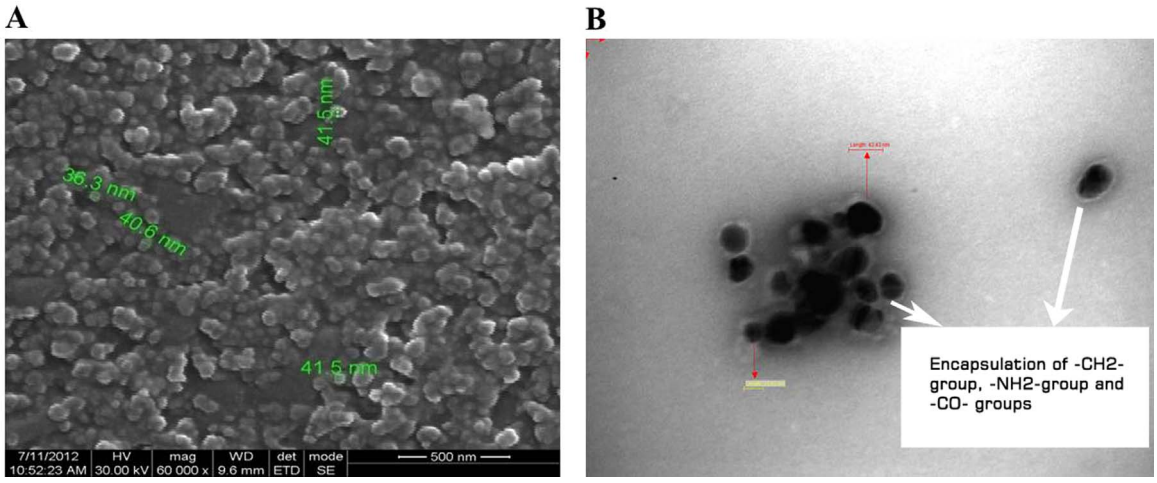


Fig. 2. (A) SEM image of AgMGsS at different nanometric scales. (B) TEM image of AgMGsS.

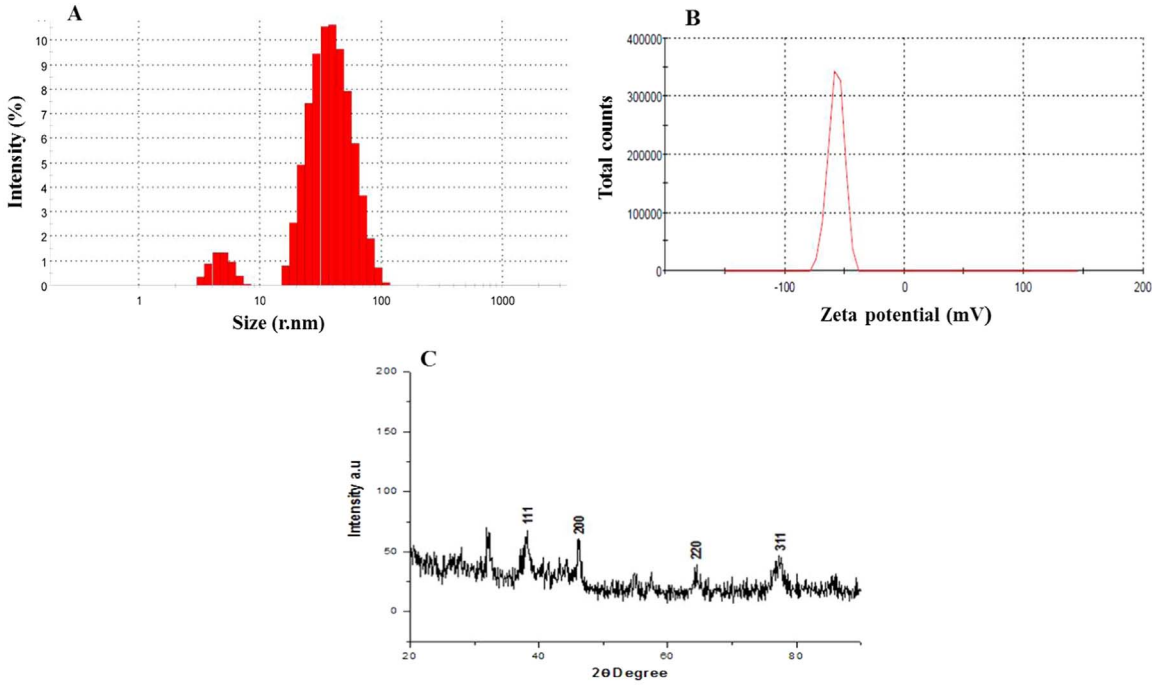


Fig. 3. Characterization of AgMGsS. (A), Particle size distribution histogram; (B) Zeta potential distribution; (C), XRD pattern.

operating at 45 kV and a current of 40 mA with CuKα radiation, was performed to determine the nature of nanoparticles.

3. Results and discussion

Phytochemical analysis of the extract revealed the presence of carbohydrates, alkaloids, glycosides, flavonoids, tannins, steroids, terpenoids and phenolics they serve as effective reducing and capping agents for converting silver nitrate into nanoparticles. The reduction of pure of pure silver ions was confirmed by UV-vis spectra where the maximum absorbance was seen at 300 nm and 440 nm (Fig. 1A). Accordingly remarkable broadening of peak indicated that the particles are polydispersed.

FTIR measurement was used to predict the role of reducing and stabilizing capacity of methanolic extract *of G. superba* seed. The FTIR spectrum shows 28 peaks and the prominent peaks at 3788.19, 3734.19, 2854, 1743.65, 1527.62, 1381.03, 1056.99 cm^{-1} of MGsS changed to 3730.33, 3618.46, 2924.09, 2858.51, 1739.79, 1523.76, 1369.46, 1022.27 cm^{-1} after synthesis confirming the reduction of silver ion to silverNPs (AgMGsS), (Fig. 1B). Compared to the standard colchicine in FTIR spectrum, MGsS and AgMGsS showed peaks at 2924 (both) corresponding to -CH2- group, peak at 1527 (MGsS) reduced to 1523 (AgMGsS) corresponding to –NH2– groups; peak found at 1647 (MGsS) reduced to 1639 (AgMGsS) attributed to –CO– groups of colchicine and colchicine derivatives were found but other phytoconstituents also responsible for capping. It shows that, these functional groups colchicine present in the extract responsible for capping, stabilization and reduction of silver ions and formation of the NPs. The SEM analysis was confirmed that the metal particles presence in nano-size. Synthesized nanoparticles were found to be highly scattered due to its spherical nature and the diameter of the particle was below 100 nm in the range of 35–42 nm (Fig. 2A) and it was consistent with earlier reports [21,22]. TEM analysis showed the morphology of AgNPs particles are being predominantly spherical, polydispersed and ranged in size from 20 to 69 nm (Fig. 2B). However, the separation between the silver nanoparticles seen in the TEM image could be due to capping effect of plant extract [23]. The reason for these large-sized particles is due to the aggregation of two or more NPs together due to the presence

of excess amounts of reducing moieties and the interactions between stabilizing molecules bound to the surface of particles and secondary reduction process on the surface of the preformed nuclei. It was noticeable that the edges of the particles were lighter than the centers, due to bioorganic alkaloids (colchicines) in MGsS, capped the silver NPs contributing to reduction of Ag^{+} ions to Ag^{o}.

The graphical representation of particle size distribution was ranged from of 3–110 nm (Fig. 3A). The average size of particles to be found 34 nm and this smaller diameter may increase the targeting efficiencies to specific sites to prolong the duration of the drug activity. The zeta potential of the synthesized AgNPs was determined in water as dispersant. Zeta-potential was found to be −57.0 mV (Fig. 3B). The high negative value confirms the repulsion among the particles and thereby increases in stability of the formulation [24].

The crystalline nature of AgNPs was confirmed by the analysis of XRD pattern as shown (Fig. 3C). The XRD spectrum of AgMGsS showed four distinct diffraction peaks at 2θ values at 32.10°, 38.15°, 46.28°, and 77.34° corresponding lattice plane value was indexed at (111), (200), (220) and (311) planes of face centered cubic (fcc) structure of silver were in good agreement with reference of fcc structure from joint committee of powder diffraction standard. Furthermore, these results clearly indicate that the nanoparticles were composed of highly crystalline Ag. The mean size of AgNPs was calculated by using the Debye–Scherrer's equation ($D=k\lambda/\beta \cos\theta$) by determining the width of the Bragg's reflection [25] and average size of the nanoparticles was thus determined to be about 3.05–5.17 nm. The XRD patterns obtained are consistent with earlier reports [26,27].

These green synthesized AgMGsS were found to exhibit excellent antitumor activity against DLA tumor both *in vitro* (MTT, tryphan blue and flow cytometric analysis)and *in vivo* (Swiss albino mice) studies (data not shown). Therefore, the substantial outcome of this study would help to formulate the herbal based value added nanomaterials in nanotechnology and biomedical industries.

4. Conclusions

The procedure described for the silver nanoparticles using the *G. superba* seed to reduce the toxicity of silver NPs it is vital to select encapsulating agents and environmentally friendly, non toxic and easy to implement extract.

References

[1] P.M. Castillo, J.L. Herrera, R. Fernandez-Montesinos, C. Caro, A.P. Zaderenko, J.A. Mejias, D. Pozo, Nanomedicine 3 (2008) 627–635.
[2] L. Singh, J.Y. Song, H.K. Jang, B.S. Kim, Process Biochem. 44 (2009) 1133–1138.
[3] D.G. Yu, Colloids Surf. B Biointerfaces 59 (2007) 171–178.
[4] S. Kim, J.E. Choi, J. Choi, K.H. Chung, K. Park, J. Yi, D.Y. Ryu, Toxicol. Invitro 23 (2009) 1076–1084.
[5] S. Ashokkumar, S. Ravi, S. Velmurugan, Spectrochim. Acta A 115 (2013) 388–392.
[6] S. Basavaraja, S.D. Balaji, A. Lagashetty, A.H. Rajasab, A. Venkataraman, Mater. Res. Bull. 43 (2008) 1164–1170.
[7] T.C. Prathna, N.A. Chandrasekaran, M. Raichur, A. Mukherjee, Colloids Surf. A 377 (2011) 212–216.
[8] Y. Liu, Y.A. Zhang, M. Zhang, Mater. Lett. 64 (2010) 1779–1781.
[9] M.D. Ali, N. Thajuddin, K. Jeganathan, M. Gunasekaran, Colloids Surf. B 85 (2011) 360–365.
[10] V.K. Vidhu, S.A. Aromal, D. Philip, Spectrochim. Acta A 83 (2011) 392–397.
[11] S.L. Smitha, D. Philip, K.G. Gopchandran, Spectrochim. Acta A 74 (2009) 735–739.
[12] S.P. Dubey, M. Lahtinen, M. Sillanpaa, Process Biochem. 45 (2010) 1065–1071.
[13] G. Zhan, J. Huang, M. Du, I. Abdul-Rauf, Y. Ma, Q. Li, Mater. Lett. 65 (2011) 2989–2991.
[14] V.G. Kumar, S.D. Gokavarapu, A. Rajeswari, T.S. Dhas, V. Karthick, Z. Kapadia, T. Shrestha, I.A. Barathy, Colloids Surf. B 87 (2011) 159–163.
[15] J. Banerjee, R.T. Narendira Kannan, J. Dig, Nanomater. Biostruct. 6 (2011) 961–968.
[16] E.K. Elumalai, Prasad TNVKV, V. Kambala, P.C. Nagajyothi, E. David, Sch. Res. Libr. 6 (2010) 76.
[17] S. Jana, G.S. Shekhawat, Fitoterapia 82 (2011) 293–301.
[18] J.C. John, J. Fernandes, T. Nandgude, S.R. Niphade, A. Savla, P.T. Deshmukh, J. Int, Green. Pharm. 3 (2009) 215–219.
[19] S. Suryavanshi, G. Rai, S.N. Malviya, Adv. Res. Pharm. Biol. 2 (2012) 45–52.
[20] S. Prati, E. Joseph, G. Sciutto, R. Mazzeo, Acc. Chem. Res. 43 (2010) 792–801.
[21] M.B. Prakash, S. Paul, Int. J. Appl. Biol. Pharm. Technol. 3 (2012) 105–111.
[22] S. Ashokkumar, S. Ravi, V. Kathiravan, S. Velmurugan, Spectrochim. Acta A 121 (2014) 388–392.
[23] K.B. Narayanan, N. Sakthivel, Mater. Lett. 62 (2008) 4588–4590.
[24] Y. Subba Rao, V.S. Kotakadi, T.N. Prasad, A.V. Reddy, D.V. Sai Gopal, Spectrochim. Acta A 103 (2013) 156–159.
[25] K.C. Bhainsa, S.F. D'Souza, Colloids Surf. B 47 (2006) 160–164.
[26] M. Safaepour, A.R. Shahverdi, H.R. Shahverdi, M.R. Khorramizadeh, A.R. Gohari, Avicenna J. Med. Biotechnol. 1 (2009) 111–115.
[27] J. Gandhiappan, R. Rengasamy, Der Pharm. Lett. 4 (2012) 875–880.