WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 8.453
Volume 13, Issue 16, 853-862. Research Article ISSN 2277–7105

ALGAL MEDIATED SYNTHESIS AND CHARACTERIZATION OF SILVER NANOPARTICLES USING *SARGASSUM SWARTZII* C.AG.

[1]Annalakshmi K., *[2]John Peter Paul J., [3]Sakthivel M. and [4]Velvizhi A.

[1]Reg.No. 20UBO110, B.Sc., Botany, Centre for Advanced Research in Plant Sciences (CARPS), Department of Botany, St. Xavier's College (Autonomous), Palayamkottai – 627 002, Tamil Nadu, India.
[2]*Assistant Professor of Botany and Director, Centre for Advanced Research in Plant Sciences (CARPS), Department of Botany, St. Xavier's College (Autonomous), Palayamkottai - 627 002, Tamil Nadu, India.
[3]Reg.No. 20UBO134, B.Sc., Botany, Centre for Advanced Research in Plant Sciences (CARPS), Department of Botany, St. Xavier's College (Autonomous), Palayamkottai – 627 002, Tamil Nadu, India.
[4]Reg.No. 20UBO135, B.Sc., Botany, Centre for Advanced Research in Plant Sciences (CARPS), Department of Botany, St. Xavier's College (Autonomous), Palayamkottai – 627 002, Tamil Nadu, India.

Article Received on
28 June 2024,
Revised on 18 July 2024,
Accepted on 08 August 2024
DOI: 10.20959/wjpr202416-33545

***Corresponding Author**
Dr. John Peter Paul J.
Assistant Professor of Botany and Director, Centre for Advanced Research in Plant Sciences (CARPS), Department of Botany, St. Xavier's College (Autonomous), Palayamkottai - 627 002, Tamil Nadu, India.

ABSTRACT

In this work, the synthesis of stable silver nanoparticles by the bioreduction method was investigated. Aqueous extract of *Sargassum swartzii* C.Ag. was used as reducing and stabilizing agent. Change of colour of the solution from light yellow to yellowish brown was used to monitor the quantitative formation of silver nanoparticles. The characteristics of the obtained silver nanoparticles were studied using UV-Visible Spectroscopy, FT-IR, X-ray diffraction analysis (XRD), energy-dispersive spectroscopy (EDX), and Scanning Electron Microscopy (SEM). The EDX spectrum of the solution containing silver nanoparticles confirmed the presence of an elemental silver signal without any peaks of impurities. The average diameter of the prepared nanoparticles in solution was about 27-63nm.

KEYWORDS: Silver nanoparticles, bioreduction, *Sargassum swartzii*, FT-IR, XRD.

1. INTRODUCTION

Nanotechnology is the fastest growing area of manufacturing in the

world today and there is an increasingly frantic search for new nanomaterials and methods to make them. It has been well known that living cells are the best examples of machines that operate at the nanolevel and perform a number of jobs ranging from generation of energy to extraction of targeted materials at very high efficiency.[1]

One of the fields in which nanotechnology finds extensive applications is nanomedicine, an emerging new field which is an outcome of fusion of nanotechnology and medicine. Medicine is no more physician job exclusively; the materials and devices designed at the level of nanoscale are for diagnosis, treatment, preventing diseases and traumatic injury, relieving pain and also in the overall preservation and improvement of health. Nanotechnology can improve our understanding of living cells and of molecular level interactions. A number of nanoparticles based therapeutics have been approved clinically for infections, vaccines, and renal diseases. Oligodynamic silver having antimicrobial efficacy extends well beyond its virotoxicity and it has lethal effects spanned across all microbial domains.[2]

Silver nanoparticles are widely used for its unique properties in catalysis, chemical sensing, biosensing, photonics, electronics and pharmaceuticals. Silver nanoparticles have a great potential for use in biological including antimicrobial activity. Antimicrobial capability of silver nanoparticles allows them to be suitably employed in numerous household products such as textiles, food storage containers, home appliances, and medical devices. Silver is an effective antimicrobial agent which exhibits low toxicity. The most important application of silver and silver nanoparticles is in medical industry such as tropical ointments to prevent infection against burn and open wounds.[3] Silver nanoparticles play a profound role in the field of biology and medicine due to their attractive physiochemical properties. Silver products have long been known to have strong inhibitory and bactericidal effects, as well as a broad spectrum of antimicrobial activities, which has been used for centuries to prevent and treat various diseases, most notably infections.[4] Hence the present study was concentrated on the synthesis and characterization of silver nanoparticles using the aqueous extract of *Sargassum swartzii* C.Ag.

2. MATERIALS AND METHODS

2.1. Collection of Plant Sample

The present study area is Manapad located in the south east coast of Tamil Nadu, India. The collection of *Sargassum swartzii* C.Ag. belonging to Phaeophyceae (Brown algae) was made

during the low tidal and subtidal regions (up to 1m depth) by hand picking.

2.2. Preparation of aqueous extract

2g dried algal powder was taken in a 250ml Erlenmeyer flask with 100ml of sterile distilled water and then boiled the mixture for 2 minutes. After boiling, the mixture was filtered in the Whatmann No.1 filter paper.

2.3. Preparation of silver nitrate solution and synthesis of silver nanoparticles

Silver nitrate ($AgNO_3$) used in the present investigation was of analytical grade purchased from Hi media laboratories Pvt. Mumbai, India. 3mM aqueous silver nitrate solution was prepared with double deionized water in sterile brown colour bottle and kept at room temperature for 24 h. For the reduction of silver ions 5mL of aqueous extract was mixed separately with 8 95mL of 3mM aqueous solution of $AgNO_3$ in a Erlenmeyer flask and incubated at room temperature in dark.[5]

2.4. Change in colour of the solution

Changes in colour of the aqueous silver nitrate solution from light yellow to yellowish brown appeared indicated the excitation of surface plasmon resonance due to reduction of silver nitrate.[6]

2.5. UV -Visible spectroscopy analysis

UV-visible spectroscopic analysis was carried out by using UV-Visible absorption Shimazdu spectrophotometer with a resolution of 20nm between 200 to 1100nm. The silver nanoparticles exhibit a unique peak in the range of 400 to 480nm.[7] The formation of reddish brown colour was observed and λ max at different time intervals were taken for at 15min, 30min, 1h, 2h, and 4h using a UV-Visible spectroscopy. After 48 hours, the reaction mixture was centrifuged and the supernatant was discarded. 1ml of distilled water was added to the pellet and washed by using centrifugation. The pellet was collected and dried in the watch glass and the silver nanoparticles were stored.

2.6. FTIR Analysis

Perkin-Elmer spectrometer in the range 4000 to 400cm^{-1} at a resolution of 4cm^{-1} was used. The sample was mixed with KCl procured from Sigma. Thin sample disc was prepared by pressing with the disc preparing machine and placed in Fourier Transform Infra Red (FTIR) for the analysis of the nanoparticles.[8]

2.7. XRD analysis

The dry powder of the silver nanoparticles was used for XRD analysis. The diffracted intensities were recorded from 20° to 80° at 2 theta angles. The phase variety and grain size of synthesized silver nanoparticles was determined by X-ray diffraction spectroscopy (Philips PAN analytical). The synthesized silver nanoparticles were studied with Cu Kα radiation at voltage of 40kV and current of 30 mA with scan rate of 0.030/s. Different phases present in the synthesized samples were determined by XPERT-PRO software with search and match facility. The particle size of the prepared samples were determined by using Scherrer′s equation as follows $D=0.9\ \lambda/\beta\cos\theta$. Where D is the crystal size, λ is the wavelength of X-ray, θ is the Bragg's angle in radians and β is the full width at half maximum of the peak in radians.

2.8. EDX and SEM Analysis

The structure, composition, and average size of the synthesized silver nanoparticles were analyzed by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray microanalysis spectroscopy (EDX). The morphological features of synthesized silver nanoparticles were studied by Scanning Electron Microscope (JSM-6480 LV). 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. The samples were characterized in the SEM at an accelerating voltage of 20 kV followed by EDX observations were also carried out.

3. RESULTS AND DISCUSSION

3.1. Synthesis of Silver Nanoparticles

Reduction of silver ion into silver particles during exposure to the seaweed extract could be followed by color change. Silver nanoparticles exhibit dark yellowish brown color in aqueous solution due to the surface Plasmon resonance phenomenon. The appearance of the yellowish brown color indicated the formation of silver nanoparticle synthesis in the reaction mixture, as it is well known that silver nanoparticle exhibited from light yellow to dark brown due to excitation of surface plasmon vibrations in the particles (Figure-1). It was reported that some amount of OH- groups tended to promote the reduction of silver ions in some chemical methods.

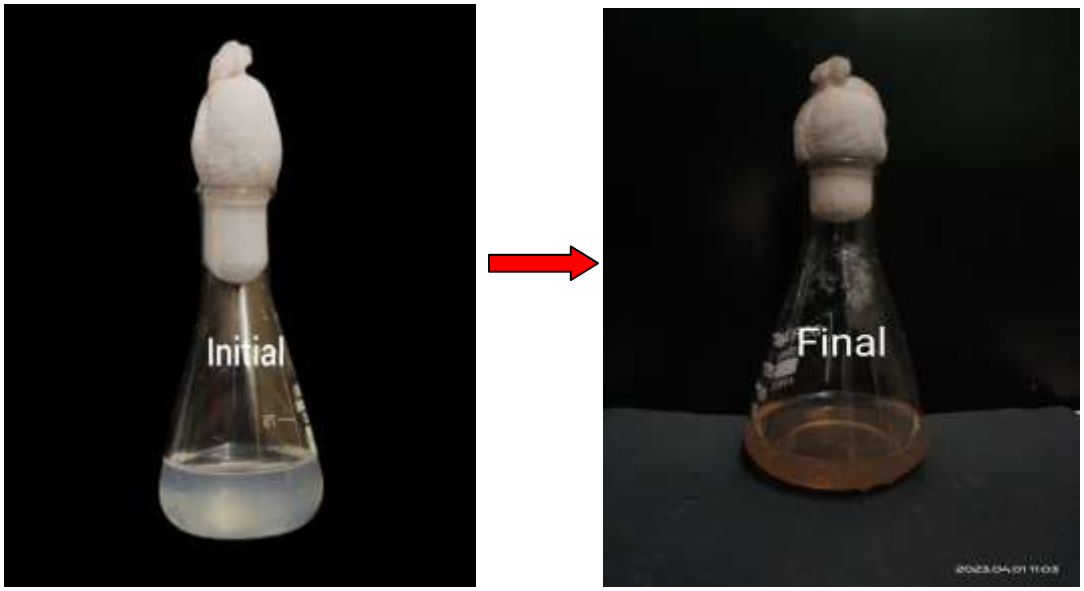

Figure 1: Plant extract and silver nitrate solution with initial and after the formation of silver nanoparticles using *Sargassum swartzii* C.Ag.

3.2. UV-Visible Spectrum

UV-Visible spectra of the reaction media were taken at different time intervals. That the Surface Plasmon Resonance (SPR) vibrations were found between 400 to 450nm with λmax at 420nm with absorption of 0.023 which was blue colour shifted at 15min the brown colour shifted at 417nm with absorption of 0.354 at 30min followed by yellow colour at 410nm with absorption of 0.713at 1h red colour at 417nm with absorption of 1.037 at 2h and green colour at 429nm with absorption of 1.118 at 4h was related to an increase the amount of silver nanoparticles (Figure-2). In the present study, there is only one peak at the centre indicating the formation of silver nanoparticles in spherical shape.

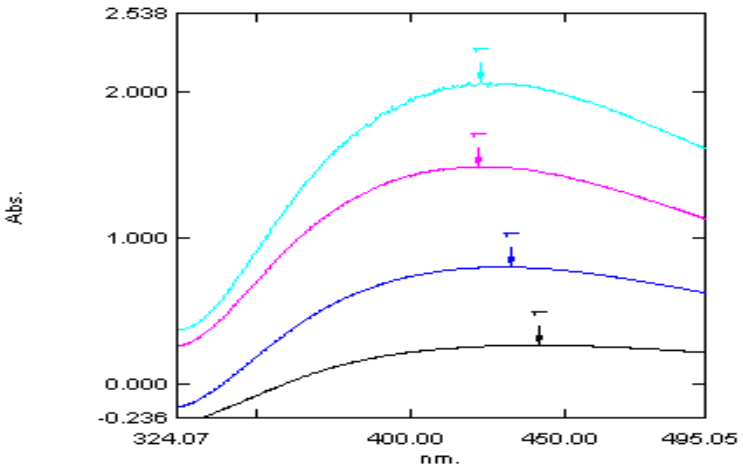

Figure 2: UV-Visible spectrum of silver nanoparticles using *Sargassum swartzii* C.Ag.

3.3. FT-IR Spectrum

FT-IR spectrum of silver nanoparticles is shown in Figure-3 and Table-1. This spectrum shows the presence of bands at 2360.71, 1637.45, 1304.79, 1201.57, 1139.85, 1095.49 and 632.61. The bands at 2360.71cm^{-1} corresponds to primary amine O-H band, 1637.45cm^{-1} corresponds to primary amine N-H band, the band at 1304.79cm^{-1} is assigned to methylene scissoring vibration from the protein in the solution and the band at 1095.49cm^{-1} were assigned to C-N stretching vibration of the proteins. The positions of these bands were close to that reported for native proteins.[9] This evidence suggests that the protein molecules could possibly perform the function of the formation and stabilization of silver nanoparticles in the aqueous medium.

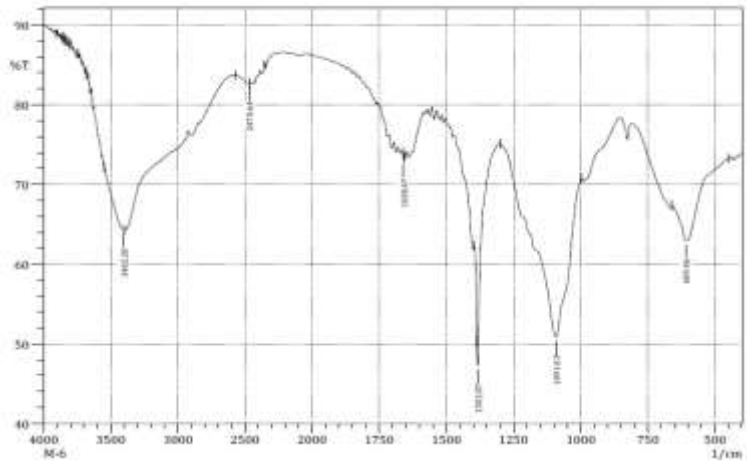

Figure 3: FTIR spectrum analysis of silver nanoparticles using *Sargassum swartzii* C.Ag.

Table 1: FTIR spectrum analysis of silver nanoparticles using *Sargassum swartzii* C.Ag.

Peak value	Functional group	Assignment
609.46	Ar-OH in phenols	OH
1091.63	C-NH_2 in 1^0 aliphatic amines	C-N
1382.87	SO_2 in sulfonyl chlorides	SO_2
1658.67	C=O in β-ketone esters	C=O
2470.64	Carboxylic acids	O-H
3402.20	NH_2 in aromatic	NH

3.4. X-Ray Diffraction studies

XRD pattern taken using powder X-ray diffractometer instrument (XRDML) in the angle range of 10˚C-80˚C of the silver nanoparticles at 2θ, scan axis: Gonio. A number of Bragg reflections corresponding to 32.37, 46.42, 27.47 and 54.65 sets of lattice planes are observed which can be indexed to face-centered cubic silver (Figure-4 and Table-2). The peaks matches with the Joint Committee on Powder Diffraction Standards (file No. 04-0783), which

further proves the formation of crystal silver nanoparticles.[10] The peaks were identified as silver nanoparticles according to XPERT-PRO software (PDF#030921). The XRD pattern thus clearly shows that the silver nanoparticles are crystalline in nature.[11]

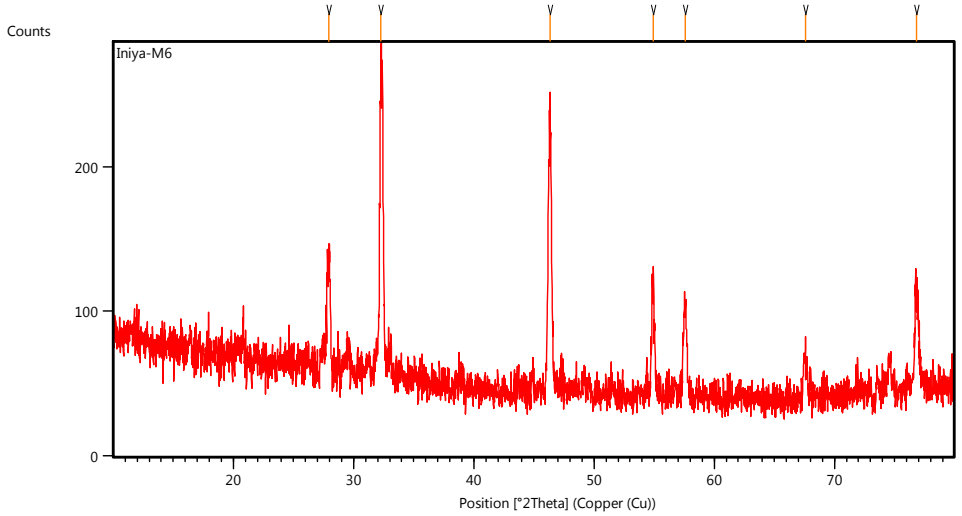

Figure 4: XRD analysis of silver nanoparticles using *Sargassum swartzii* C.Ag.

Table 2: XRD analysis of silver nanoparticles using *Sargassum swartzii* C.Ag.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
27.9319	79.29	0.2676	3.19433	40.39
32.2422	196.34	0.2676	2.77647	100.00
46.3730	181.64	0.3011	1.95806	92.52
54.9058	77.68	0.2676	1.67225	39.56
57.5711	62.46	0.3346	1.60101	31.81
67.5985	21.09	0.8029	1.38587	10.74
76.8066	78.94	0.2676	1.24106	40.21

The diffracted intensities were recorded from 10° to 80° at 2 theta angles. The diffraction pattern corresponds to pure silver metal powder. The XRD pattern indicates that the nanoparticles had a spherical structure. No peaks of the XRD pattern of Ag^2O and other substances appear and it can be stated that the obtained silver nanoparticles had a high purity. The observed peak broadening and noise were probably related to the effect of nano sized particles and the presence of various crystalline biological macromolecules in the plant extracts. The obtained results illustrate that silver ions had indeed been reduced to AgO by the extracts under reaction conditions.[12]

3.5. EDX and SEM Analysis

To gain further insight into the features of the silver nanoparticles, the element analysis of the

silver nanoparticles was performed using EDX on the SEM. The freeze dried silver nanoparticles were mounted on specimen stubs with double sided taps, coated with gold in a sputter coater and examined under a Philips XL-30 SEM at 12-16 kV with a tilt angle of 45°. Figure-5 shows the EDX spectrum of spherical nanoparticles prepared with this bioreduction method. The peaks around 3.40 keV correspond to the binding energies of AgL. Throughout the scanning range of binding energies, no peak belonging to impurity was detected. The results indicated that the reaction product was composed of high purity Ag nanoparticles. A similar EDX spectrum was obtained for each sample analyzed. Scanning electron microscopy provided further insight into the morphology and size details of the silver nanoparticles. Comparison of experimental results showed that the diameter of prepared nanoparticles in the solution was about 27-63nm. Figure-6 shows the scanning electron micrographs of silver nanoparticles obtained from the proposed bioreduction method.

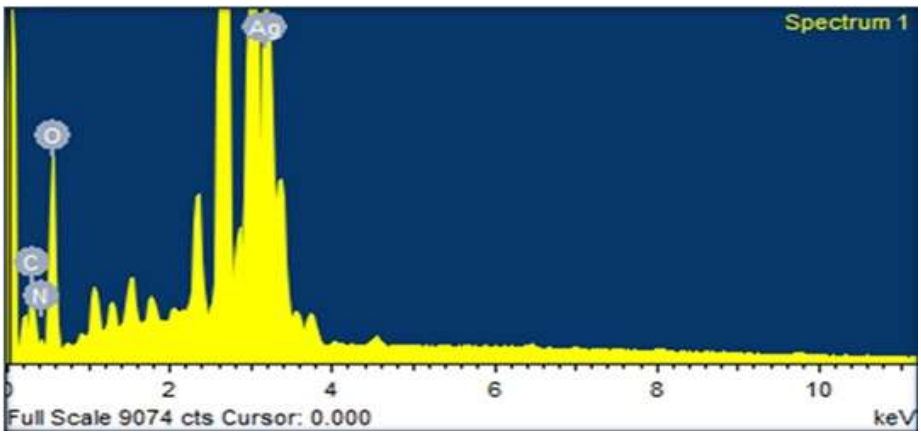

Figure 5: Energy Dispersive X-Ray (EDX) spectrum of silver nanoparticles using *Sargassum swartzii* C.Ag.

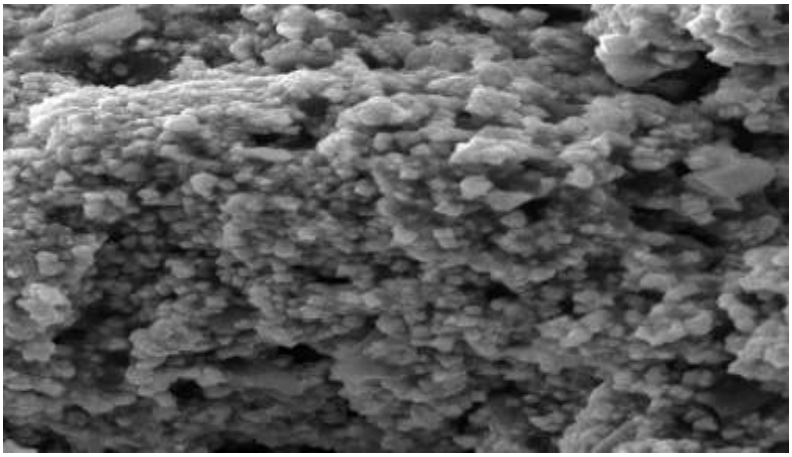

Figure 6: Scanning Electron Microscopic (SEM) image of silver nanoparticles using *Saragassum swartzii* C.Ag.

4. CONCLUSION

The aqueous extract of *Sargassum swartzii* C.Ag. was used to synthesis silver nanoparticles. The silver nanoparticles were characterized by UV-Vis Spectrophotometer, FT-IR, XRD, EDX and SEM. There is only one peak at the centre at different nanometer at indicating the formation of silver nanoparticles in spherical shape using UV-Visible spectrophotometer. FT-IR spectrum of silver nanoparticles shows the presence of bands at 632.61, 1095.49, 1139.85, 1201.57, 1304.79, 1637.45 and 2360.71cm^{-1}. XRD pattern of silver nanoparticles shows a number of Bragg reflections corresponding to ˚2 theta of 32.37, 46.42, 27.47 and 54.65 sets of lattice planes of silver. EDX and SEM analysis show the presence and the size of the synthesized silver nanoparticles about 27-63nm.

REFERENCES

1. Slawson R, Van Dyke MI, Lee H, Trevors JT. Germanium and silver resistance accumulation and toxicity 4. in microorganisms. Plasmid, 1992; 27: 72-79.
2. Bhakuni DS, Rawat DS. Bioactive Marine Natural Products. New Delhi, Anamaya Publishers, 2005.
3. Zhao Y, DeLancey GB. A predictive thermodynamic model for the bioreduction of acetophenone to phenethyl alcohol using resting cells of Saccharomyces cerevisiae. Biotechnology and Bioengineering, 1999; 64: 442-451.
4. Ankanna S, Prasad KV, Elumalai EK, Savithramma N. Production of biogenic silver nanoparticles using boswellia ovalifoliolata stem bark. Digest Journal of Nanomaterials and Biostructures, 2010; 5: 369-372.
5. Muthu Sheeba M, John Peter Paul J, Iniya Udhaya C. Green synthesis of silver nanoparticles using Enteromorpha linza (L.) J.Ag. (Green seaweed) from Hare Island, Thoothukudi, Tamil Nadu, India. European Journal of Molecular Biology and Biochemistry. 2014; 1(5): 201-206.
6. Jae YS, Beom SK. Rapid biological synthesis of silver nanoparticles using plant leaf extracts. Bioprocess Biosyst. Eng, 2009; 32: 79-84.
7. Vaidyanathan R, Kalishwaralal K, Gopalram S, Gurunathan S, Nanosilver-the burgeoning therapeutic molecule and its green synthesis. Biotechnolgy Advance, 2009; 27: 924-937.
8. Iniya Udhaya C, John Peter Paul J, Amster Regin Lawrence R. Seaweed mediated biosynthesis of silver nanoparticles using Gracilaria fergusonii J.Ag. collected from Hare Island, Thoothukudi in the south east coast of Tamil Nadu, India. International Journal of Plant Biotechnology, 2015; 1(2): 17-24.

9. Kapoor S. Preparation, characterization and surface modification of silver particles. Langmuir, 1998; 14: 1021-1025.
10. Kumar P, Senthamil Selvi S, Lakshmi Prabha A, Prem Kumar K, Ganeshkumar RS, Govindaraju M. Synthesis of silver nanoparticles from *Sargassum tenerrimum* and screening phytochemicals for its antibacterial activity. Nano Biomed. Eng, 2012; 4(1): 12-16.
11. Huang Z, Mills G, Hajek B. Spontaneous formation of silver particles in basic 2-propanol. J. Phys. Chem, 1993; 97: 11542-11550.
12. Dhanalakshmi PK, Riyazulla A, Rekha R, Poonkodi S, Thangaraju N. Synthesis of silver nanoparticles using green and brown seaweeds. Phykos, 2012; 42(2): 39-45.