Effectiveness of Green Synthesized Silver Nanoparticles (Ag-Nps) of Delonix Regia Pods in Water Disinfection

Shilpa B S, Shruthi G

Abstract: ***Particles of sizes between 1 and 100 nanometers are known as nanoparticles. The natural materials like plant leaf extract, bacteria and fungi for the synthesis of silver nanoparticles has numerous benefits. In this scenario, the main objective of this work is to utilize Delonix Regia pod extract for the synthesis of silver nanoparticles (NPs) and check its feasibility as disinfectant. Pod extract prepared was subjected to EDS analysis. Transformed silver NPs are coated onto GAC(1.5mm) and subjected to SEM analyses. Column study was carried to check the disinfecting action of silver NPs for varied contact time by monitoring MPN value. From this research work, it can be concluded as, for pod extract, peak absorbance was found at 360nm. Delonix Regia pod extract transforms into silver nanoparticles moderately to about 4.63-8.85%. Silver nanoparticles are coated onto Activated Carbon, dried and stored for extended period without loss of nanoparticles. At 30min contact time efficiency was found to be 63% and 71 for NP2.5 and NP5 respectively. Green synthesized silver nanoparticles are proved to be moderately effective in disinfecting contaminated water.***

Index Terms: Delonix Regia, Disinfection, Green synthesis and Silver NPs.

I. INTRODUCTION

Particles of size varying between 1 and 100 nanometres are referred as nanoparticles. Researches on nanoparticle is presently one of the potential area which has applications in biomedical, optical and electronic fields. There is a great need to identify environmentally and economically friendly processes for nanoparticle synthesis. This made the researchers to use natural/ virgin materials for the synthesis of nanoparticles. The use of materials in nanoparticle synthesis ranges from bacterial cells to fungi and plants extract. The use of plant leaf extract, bacteria and fungi for the synthesis of silver nanoparticles leads to eco-friendly synthesis.

Delonix regia is a species of flowering plant in the family: Leguminous, commonly known as Gulmohar. Current research of natural nanomaterials has opened a new era in pharmaceutical industries. The metal which has antibacterial property ie., Silver is of atmost concern. Silver nanoparticles because of its unique property of oligo-dynamic effect, that silver ions has on the microbes, where silver ions bind to reactive groups in bacterial cells, resulting in their precipitation and inactivation.

Water is the universal solvent that supports survival all organism and also even many pathogens. The presence of bacteria is the main indication of water contamination. Water is disinfected to kill any pathogens which pass through the filters and to provide a residual dose of disinfectant to kill or inactivate potentially harmful micro-organisms in the storage and distribution systems. Disinfection can be attained by means of physical or chemical disinfectants. Nanoparticles are expected to play an important role in water purification technique. The use of silver nanoparticle as water disinfectant is one of the new disinfection process carried out in recent days. In this scenario, the main objective of this work is to utilize Delonix regia pod extract for the synthesis of silver nanoparticles. The specific objectives are (i) To synthesis the nanoparticles from Delonix regia pod extract at varied conditons (ii) To find out the percentage of transformation of Ag-NPs synthesized using EDS and (iii) To check the efficiency of green synthesized Ag-NPs in disinfection.

II. MATERIALS

A. Delonix regia pods

Delonix regia pods shown in Figure 1, required for the synthesis of Silver Nanoparticles was collected from in and around Vidyavardhaka College of Engineering, Gokulam III stage, Mysore.

B. Granular Activated Carbon (GAC)

Granular activated carbon of 1.5mm is used

Figure 1 Parts of Delonix Regia Tree

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C. Lakewater

To assess the effectiveness of coated GAC, water samples were collected from Kukkarahalli lake, Mysore . Figure 2 shows the location of Kukkarahalli lake.

Figure 2: Location of Kukkarahalli lake

III. METHODOLOGY

Figure 3 is the flow chart depicting the activities involved in the research work

i. Preparation of Pod Extract

Pods were thoroughly washed with tap water followed by distilled water and dried in hot air oven at 103^0C for 24 hours and powdered. 13g of dry powder extract of pods were added to 250 mL distilled water (1: 20) and boiled for about 15 minutes and filtered through Whatman No.1 filter paper.

ii. Preparation of 1mM Silver nitrate

Weight of silver nitrate required is calculated using the formula

W= (0.001 MW * Volume)/100 …… (1)*

MW= Molecular weight= 169.87 g

V=Volume= 1000 mL

*W= (0.001*169.87*100)/1000 …… (2)*

W= 0.0169 g

0.0169 g of silver nitrate is dissolved in 100 mL of distilled water

iii. Synthesis of Silver Nanoparticles

2.5mL of pod extract is added to 22.5 mL of silver nitrate and observed for color change. Color changes from colorless to brown, designated as **NP2.5**. The solution is checked for absorbance in UV-Vis spectrophotometer at 290- 370 nm and then it is kept for 24 hrs in the dark room. Again 5mL pod extract is added with 20 mL of silver nitrate and color changed is observed, designated as **NP5**.

iv. Centrifugation

The silver nanoparticle suspension is seperated after 24 hrs using centrifuge (Remi Motors) by repeated centrifugation for 20 minutes at 1200 rpm.

Figure 3 Methodology Followed in this Research Work

v. Energy Dispersive Spectroscopy Analysis (EDS)

After centrifugation the solution is subjected to EDS to assess the percent of silver ion present in the Silver Nanoparticle solution. EDS analysis was carried out in Vignana Bhavan, University of Mysore. EDS analysis was carried out on a thin evaporated layer on top of a nickel / iron alloy. The layer contained a mixture of barium, strontium and oxygen, with a small amount of magnesium. The thickness of the layer was calculated to be ~ 38 nm. The layer contained three shells namely K, L and M shell.

vi. Coating of Silver ions on GAC

Granular activated carbon are washed and dried in hot air oven for 2hrs, soaked in Silver nanoparticles suspension overnight for the adsorption of Silver ions. Dried GAC of 30g kept for suspension with 120mL of Ag-NPs(**NP2.5**) and GAC of 15g with 135mL Ag-NPs(**NP5**).

vii. Scanning Electron Microscopy (SEM) of coated GAC

The coated GAC is then subjected to SEM analysis at Vijnana Bhavan, UoM, Mysore using Scanning Electron Microscopy (Hitachi).

viii. Effective Contact Time of GAC for Disinfection

Feasibility studies on the efficiency of coated GAC in disinfection process was analysed by batch process. The water sample is filled and placed in jar test apparatus for uniform mixing of GAC within the sample and it was maintained at 30 rpm. 5g of coated GAC is added to each beakers. At every 5min interval samples are drawn from each beakers and Most Probable Number of coliforms present in the sample was determined using Standard procedure.

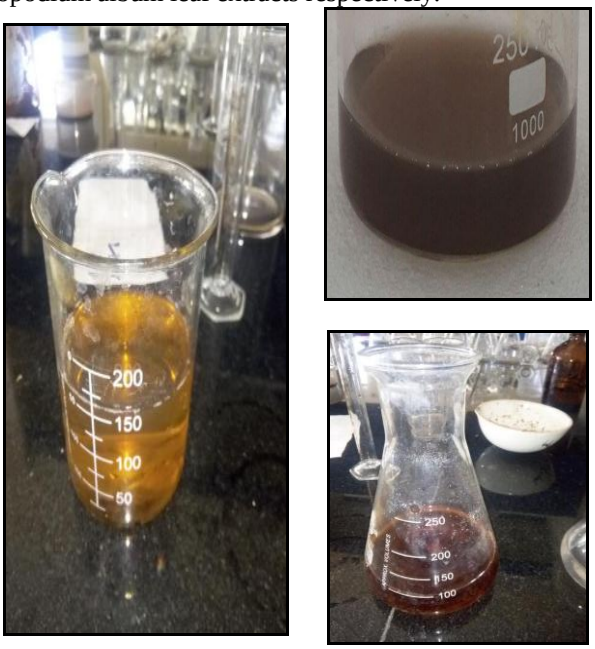
ix. Most Probable Number (MPN)

Standard procedure was followed to determine the MPN of the sample.

IV. RESULTS AND DISCUSSION

A. Synthesis of Silver Nanoparticles

The synthesis of silver nitrate using Delonix Regia pod extract was determined by the change of color in the solutions as shown in Figure 4. The progress of the reaction between metal ions and the pod extract was monitored by UV-Visible spectra of silver nanoparticles in aqueous solution with different wavelength in nanometers varying from 290 to 370 nm (Table1 & 2). The reduction of silver ions and formation of silver nanoparticles occurred within an hour of reaction. The maximum absorbance peak was found at 360 nm. Reduction of silver ions present in the aqueous solution of silver complex during the reaction with the ingredients present in the pod extracts observed by the UV-VIS spectroscopy revealed that silver nanoparticles in the solution may be correlated with the UV-VIS spectra. Shameli et al., (2013) obtained maximum absorbance at 423 nm and Gopal et al., (2010) obtained maximum absorbance at 460 nm for synthesis for Ag-NPs using Vitex negundo L and Chenopodium album leaf extracts respectively.



Mixture of pod extract+ 1 mM silver nitrate solution

Transformation of dry powder pod extraction as colloidal silver ions

Figure 4: Transformation of Delonix Regia Extract to Ag -NPs

Table 1: UV-VIS absorbance of silver ions for different time intervals at varied wavelengths (NP2.5)

Absorbance, nm	5 min	10 min	15 min	20 min
290	**0.026**	**0.026**	**0.027**	**0.027**
300	0.167	0.168	0.168	0.168
310	0.314	0.314	0.315	0.315
320	0.527	0.527	0.528	0.528
330	0.669	0.670	0.671	0.672
340	0.696	0.697	0.698	0.698
350	0.816	0.819	0.817	0.818
360	**0.869**	**0.869**	**0.867**	**0.868**
370	0.853	0.853	0.854	0.855

Table 2: UV-VIS absorbance of silver ions for different time intervals at varied wavelengths (NP5)

Absorbance, nm	5 min	10 min	15 min	20 min
290	0.010	0.011	0.011	0.012
300	0.092	0.092	0.093	0.094
310	0.327	0.328	0.328	0.329
320	0.576	0.576	0.577	0.577
330	0.775	0.776	0.777	0.777
340	0.730	0.731	0.732	0.732
350	0.823	0.823	0.824	0.825
360	**0.859**	**0.860**	**0.861**	**0.861**
370	0.855	0.856	0.856	0.857

EDS was carried out to check the percentage of silver ions present in Ag-NPs solution. The silver nanoparticles formed are necessarily subjected to EDS to know the percent of silver present. Ag-NPs solution is divided into K, L, M shell where silver ions were present about 89.80 % in L shell as shown in Figure 5a & b, which further confirmed the reduction of Delonix regia as silver ions. Most studies focused on the size of nanoparticles formed during synthesis, not many studies were carried out for assessing the percent of silver ions present.

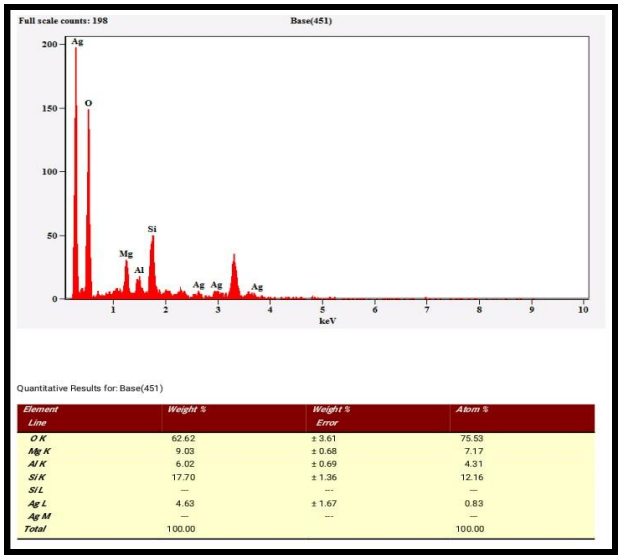
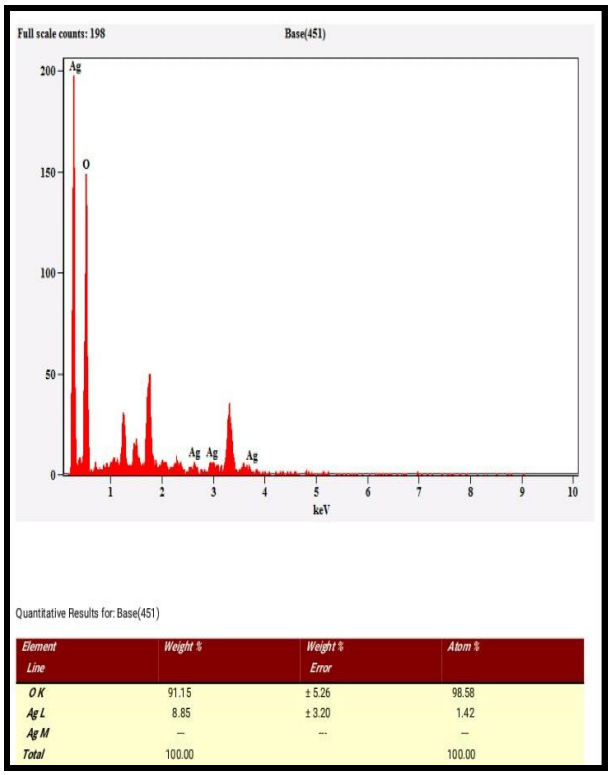


Figure 5 a: EDS spectrum of transformed silver ions of Delonix regia extract



Element Line	Weight %	Weight % Error	Atom %
O K	91.15	± 5.26	98.58
Ag L	8.85	± 3.20	1.42
Ag M	--	---	--
Total	100.00		100.00

Figure 5 b: EDS spectrum of transformed Ag-NPS NP2.5 and NP5 respectively

B. SEM analysis

SEM analysis is carried out know about the adsorption of Ag-NPs on GAC. Focused beam of high energy electrons generates a variety of signals at the surface of solid specimens. It is also capable of performing analysis of selected point locations on the sample. The Figure 6 shows the SEM images of uncoated GAC, while Figure 7 and Figure 8 shows the SEM images of coated GAC with NP2.5 & NP5 respectively. Figure 9 shows the comparison SEM images at 20mm magnification of uncoated and coated GAC. These images shows that adsorption of Ag-NPs is higher in NP5 when compared to uncoated and NP2.5.

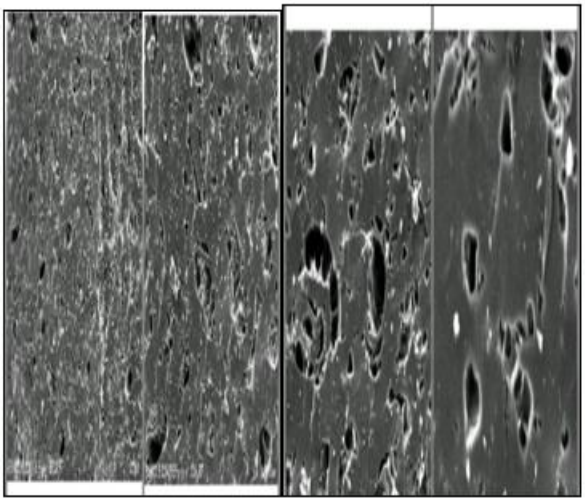


Figure 6: SEM images of uncoated GAC

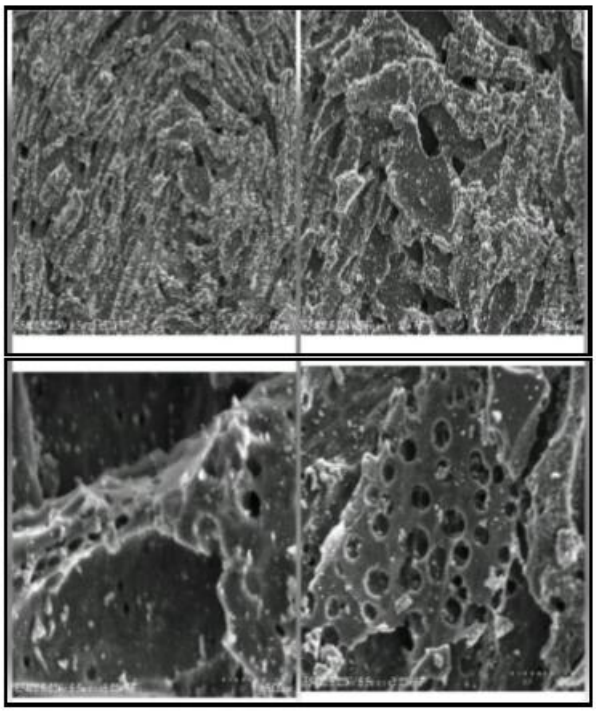


Figure 7: SEM images of GAC coated with Ag-NPs of NP2.5

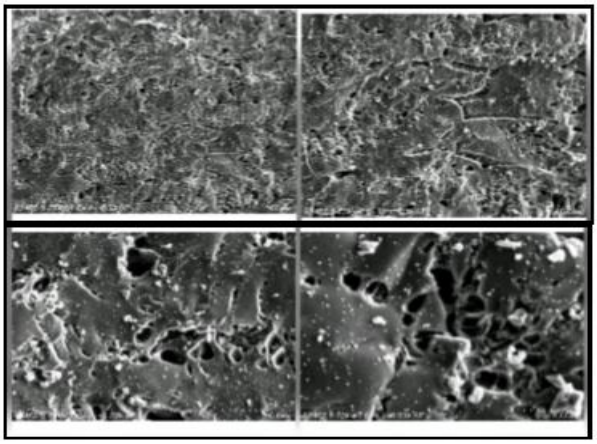


Figure 8: SEM images of GAC coated with Ag-NPs of NP5

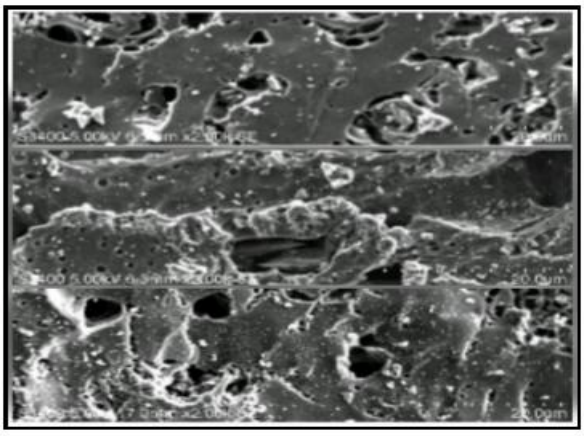


Figure 9: Comparison of Uncoated, Coated with NP2.5 & NP5 SEM images at 20 µm Magnification

C. Effective Contact Time of Disinfection

Most probable number test was carried out to assess the effectiveness of coated Silver ions on GAC. The effective contact time in disinfection is summarized in the Table 3. This shows decreasing value of MPN after treatment with coated GAC. The initial MPN value of NP2.5 84 is reduced to 31 at 30 mins contact time with GAC, and the initial MPN value of NP5 83 is reduced to 24 at 30 mins contact time with GAC.

Petri plates of complete test of MPN shows the decrease in the formation of colonies of coliform bacteria shown in Figure 11.

Table 3 : MPN Values of Collected Samples

GAC coated with	Initial MPN	Time in min	MPN Value
$NP_{2.5}$	84	5	73
		10	70
		15	55
		20	42
		25	36
		30	31
NP_5	83	5	69
		10	53
		15	47
		20	38
		25	30
		30	24

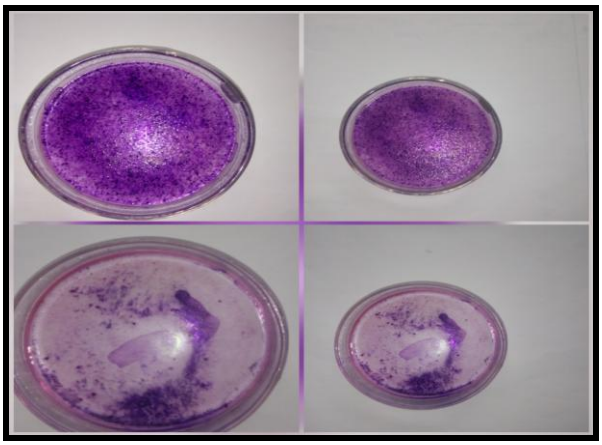


Figure 11: Petriplates of Complete Test of MPN

V. CONCLUSIONS

From this research work, the conclusions can be drawn as,

- For pod extract, peak absorbance was found at 360nm in UV-VIS spectrophotometer. Delonix Regia pod extract transforms silver nanoparticles moderately to about 4.63-8.85
- Silver nanoparticles can be coated onto Activated Carbon, dried and stored for extended period without loss of nanoparticles
- At 30min contact time, efficiency was found to be 63% and 71 for NP2.5 and NP5 respectively
- Green synthesized silver nanoparticles are proved to be moderately effective in disinfecting contaminated water.

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