Facile Synthesis Photochemical, Dye Degradation and Ecotoxicological Properties of Silver Nanoparticles Using *Sansevieria angolensis* Bojer Ex. Hook Plant Extract

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ABSTRACT

Background: It has been found in recent years that metallic nanoparticles can have significant impacts in various fields because of their low cost, ease of synthesis, and environmental friendliness. **Objectives:** This study focuses on eco-friendly silver nanoparticles synthesis using *Sansevieria angolensis* as catalyst. **Materials and Methods:** Synthesized silver nanoparticles were characterized by UV, FTIR, XRD, SEM and EDX studies. Three different organic dyes as crystal violet, coomassie blue and congo red the photocatalytic dye degrading capacity of synthesized nanomaterials was evaluated. Earthworm mortality rate with nanomaterial exposure was done to ensure that the SASNPs (*Sansevieria angolensis* Silver Nanoparticles) are non-hazardous to the environment. **Results:** The present study green synthesized NPs were characterized with absorption range above 250 nm, particle size ranging from 40-50 nm and the presence of 1.5 mg of silver per kg. This study focuses on the rapid effect of SASNPs in degrading crystal violet, congo red and coomassie blue dyes in the presence of light. The heavy metal accumulation was measured in SASNPs indicated a 1.5 ppm build-up and ecotoxicology of NPs by using earthworm mortality rate of earthworm is observed to be as 0.3333±0.333 in the lowest concentration of 25 mg/100 mL. **Conclusion:** Amidst of numerous metals used to synthesize nanoparticles, silver-based nanoparticles synthesis is prominent as silver is essential for all living organisms. This study reveals that the biogenic synthesis of NPs showed low ecotoxicity on earthworm *Eisenia fetida*.

Keywords: Eco-friendly synthesis, Non-hazardous, Photochemical dye degradation, *Sansevieria angolensis*, Silver nanoparticles.

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INTRODUCTION

Plant-mediated synthesis of nanoparticles is now very popular and very successful due to its eco-friendly and time-conservation properties. The synthesis of metal nanoparticles using plant extracts is one of the most easy, convenient, cost-effective, and ecologically benign approaches for reducing the use of harmful chemicals.[1] Silver nanoparticles can be synthesised from leaf extract at low concentrations without the use of any additional damaging chemical/physical processes. The influence of metal ion concentration and leaf extract concentration was also investigated in order to optimise the pathway for synthesis of silver nanoparticles. The method used here is straightforward, cost effective, easy to use, and long-lasting.[2]

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One of the main causes of water pollution is the discharge of dye effluents from the textile sector[3] causing cancer, toxicity, birth defects, and teratogenicity, as well as reducing light penetration through the water's surface.[4] Congo red (CR) dye is an organic contaminant that is harmful to the environment,[5] Coomassie Brilliant Blue is a known water contaminant,[6] Crystal violet is a poisonous, refractory organic dye that causes serious health concerns and damage in the environment.[7]

The need for the use of nanomaterials may result in the release of these anthropogenic materials into soil and water, which might harm the environment by changing water and soil properties.[8] Unused silver nanoparticles may enter the soil via bio-solids from wastewater treatment or effluent from industrialization processes, where they may be hazardous to organisms living in terrestrial environments. Furthermore, silver ions are among the most hazardous heavy metals produced from dissolved Silver Nitrate ($AgNO_3$) and AgNPs via dissolution or oxidation. The study looked at the effect of manufactured AgNPs and $AgNO_3$ on earthworms, which are a key bio indicator for assessing toxicity in soil environments.[9]

The present study focuses on the synthesis and characterization of nanoparticles by combining silver metal and *S. angolensis* plant extract as reducing agent. The present study evaluated on the ecotoxic effects of silver nanoparticles on earthworm, heavy metal accumulation and photocatalytic degradation of textile dyes.

MATERIALS AND METHODS

Collection of the plant material

The plant is recognized as *Sansevieria angolensis* and accumulated from (10.92900N, 77.03330S), Chettipalayam, Coimbatore, Tamil Nadu, India. Matured plant grown in wild condition used to be collected and utilised to obtain excellent results.

Facile synthesis of SNPs

S. angolensis was powdered after washing, chopping and shade drying. Water extract was prepared by the use of magnetic stirrer. The extract is filtered using Whatmann No. 1 filter paper and stored for further utilization. The extract was added to the 5 mL $AgNO_3$ solution in drop wise and incubated in UV light until colour change.

Characterization of SNPs

In order to track the reduction process and ascertain the kinetic behavior of silver nanoparticles in solution, a Perkin-Elmer UV-VIS Spectrometer Lambda-35 was utilized. To identify the substance that is responsible for their formation FTIR PERKIN ELMER instrument with a wavelength range of 4000 to 400 nm was used. The crystalline structure of SASNPs was examined using X-ray powder diffractometer. The presence of silver and other elementary particle compositions was determined using EOL-2100 Energy-dispersive X ray spectroscopy. The particle size is observed by QUANTO 250 scanning electron microscopy.

Heavy metal accumulation studies of silver

Estimation of silver in the sample by AAS *(*Atomic Absorption Spectrometry) method: The silver standards (4.1) (NIST traceable) have been prepared in a 100 mL flask at 1, 3, 5, 8, and 10 g/L for thousand ppm. Approximate weight of the sample is positioned in an RB flask, and 20 mL of HCl is delivered before boiling for 15 min. The flask is kept open in the course of the boiling process, then cooled and transferred to a standard 100 mL flask with H_2O. The work directions are set in accordance with the AAS guidelines. The absorbance of silver at 196 nm was once measured to calculate the absorbance of the blank, standards, and pattern solution.

Photocatalytic dye degradation

Three different organic colorings similar as crystal violet, coomassie blue and congo red were used for this trial. One milligram of each color was dissolved in 100 mL of double distil water and the result was kept on a stirrer for 30 twinkles to insure the result's equilibrium. Each color result entered 10 mg of *S. angolensis* nanoparticles and was exposed to sun. 3 mL suspensions were centrifuged at 10,000 rpm for 10 min at different time intervals. The absorbance of the sample was also measured using a UV-visible spectrophotometer.

Earthworm mortality rate analysis

Dissolving the samples in 100 mL of sterile distilled water yielded four distinctive concentrations of SASNPs solutions viz., 25 mg/100 mL, 50 mg/100 mL, 100 mg/100 mL, and 150 mg/100 mL were yielded by dissolving the samples in 100 mL of sterile distilled water. Red soil is gathered from agricultural fields and blended with cow dung before introducing the earthworms. For this experiment, 40-60 earthworms had been accumulated from the field. The soil mixture is treated with 4 extraordinary concentrations of synthesized nanoparticles. 10-15 earthworms have been used for every of the 4 beds and the mortality rate was observed after four weeks.

RESULTS

Synthesis of silver nanoparticles

The *S. angolensis* plant extract was added to the 5 µm silver nitrate, solution kept in magnetic stirrer in room temperature in 9:1 ratio. The solution was kept in UV light to induce the synthesis of silver nanoparticles. The colour change from dark brown to light brown indicates the primary characterization of synthesis (Figure 1).

Characterization of Silver nanoparticles

UV-visible spectrum analysis

UV analysis of SASNPs showed absorption of the nanoparticles above wavelength of 200 nm. Sharp peak lies between 250-310 nm is determined (Figure 2). This may presence of phytochemicals in the leaf extracts of this medicinal plant also serve as capping agents, stabilizing SNPs through their capping action.

FT-IR analysis

FT-IR is done to find the functional groups responsible for the formation of nanoparticles and the chemical components present in the sample. 400-4000 cm^{-1} is the typical IR absorption range for covalent bonds SASNPs showed 11 intense peaks such as 421.13, 415.26, 403.90, 551.76, 570.52, 598.14, 1077.37, 1408.50, 1631.69, 2280.24, 3253.51 cm^{-1} (Figure 3). The identified functional groups are alcohols, phenols, alkynes, 1° amines, aromatics, aliphatic amines, primary alcohol, alkane and alkyl halides respectively (Table 1). The results of FT-IR analysis showed a strong broad

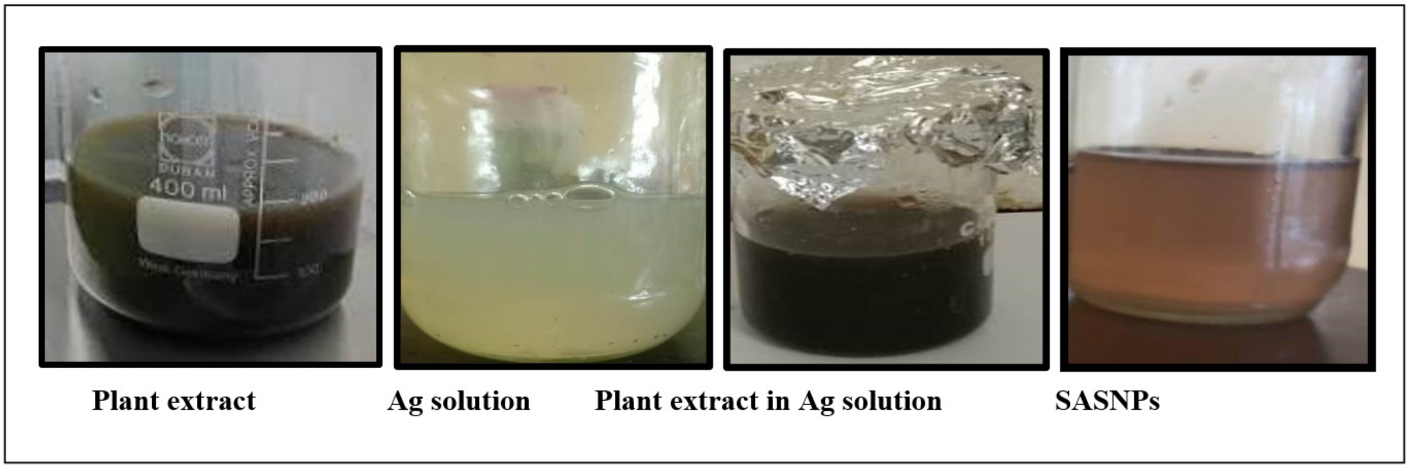

Figure 1: Synthesis of silver nanoparticles.

peak at 421.13 cm^{-1} that corresponds to O-H stretch alcohols and phenols suggesting a strong hydrogen bonding interaction between silver and the O-H groups.

XRD analysis

Using XRD diffract gram which shows the crystalline nature, size and alignment, the 2θ values of the peaks ranging from 0° to 80° in SASNPs were observed. X-Ray Diffraction studies showed 4 intense peaks viz**.,** 32.28°, 32.2°, 32.24°, 32.26° of which 32.28° is highest (Figure 4). The crystalline character of the SNPs produced by the reduction of Ag^+ ions using *S. angolensis* extract.

SEM analysis

Quantitative information on the chemical composition of the sample's contents can also be determined by Scanning electron microscopy. SEM microscopy was also used to evaluate morphology, including voids, fractures, porosity, agglomeration, grain distribution, and average grain size. The shape of SNPs is spherical. The silver nanoparticles range from 42.57-44.85 nm (Figure 5).

EDX analysis

Through Energy dispersive X-ray composition analysis the presence of Ag (Silver) screened, which confirms the sample contains the metal. The peak at 3 keV demonstrates the ability of silver to bind with plant phytochemical components (Figure 6). For the production of SNPs, the phytochemical group in *S. angolensis* serves as a capping and binding agent. This investigation demonstrates that NPs are composed of the purest Ag^+ (Figure 6).

Heavy metal accumulation study of SASNPs

The AAS technique was used to conduct a heavy metal accumulation investigation in order to control the minimal amount of reported harmful effects for applications, particularly in the food business. The present study in SASNPs extract indicated a 1.5 ppm build-up. The result was represented in (Table 2). These policies involve measuring metal concentrations in soils and establishing threshold values.

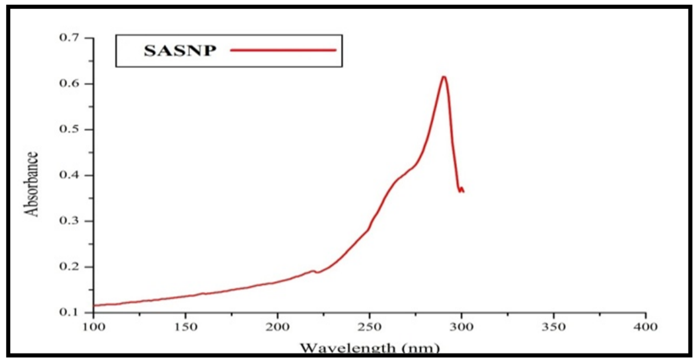

Figure 2: UV-vis spectrum of SASNPs.

Photocatalytic Dye Degradation

Optical analysis

Degradation of dyes such as crystal violet, congo red and coomassie blue were observed by exposing these dye solution containing SASNPs to sunlight. After subjecting the sample to sunlight for 30 min, reduction of pigments in the dyes were clearly visible with the increase in time (Figures 7-9). Congo red was reduced to transparent light red at 25 min (Figure 7), crystal violet was reduced to transparent at 30 min (Figure 8) and the intensity of coomassie blue has also reduced at 30 min (Figure 9).

UV-analysis

The use of the dye's crystal violet, coomassie blue, and congo red allowed to demonstrate the photocatalytic activity of silver nanoparticles on dye degradation. At various times in the visible region, dyes were degraded in the presence of silver nanoparticles. The lowered peaks for crystal violet, coomassie blue, and congo red were visible in the absorption spectra at various times. Initially, the absorption peaks were at 600 nm for crystal violet, 500 nm for congo red and 600 nm for coomassie blue dye. These peaks were decreased gradually with the increase of the time exposure

Table 1: FT-IR analysis of SASNPs.

Sl. No.	Peak value	Appearance	Frequency	Bond	Functional group name
1	421.13	Strong, broad	3500-3200	O-H stretch, H-bonded	Alcohols, phenols
2	415.26	Weak	2260-21000	-C≡C- stretch	Alkynes
3	403.90	Medium	1650-1580	N-H bend	1ºamines
4	551.76	Medium	690-515	C-Br stretch	Alkyl halides
5	570.52				
6	598.14				
7	1077.37	Strong	1085-1050	C-O stretch	Primary alcohol
8	1408.50	medium	1440-1390	O-H bend	Alkane, gem dimethyl
9	1631.69	Medium	1500-1400	C-C stretch	Aromatics
10	2280.24	Weak	2260-2222	C-N stretch	Aliphatic amines
11	3253.51	Strong, broad	3550-3200	O-H stretch	Alcohol

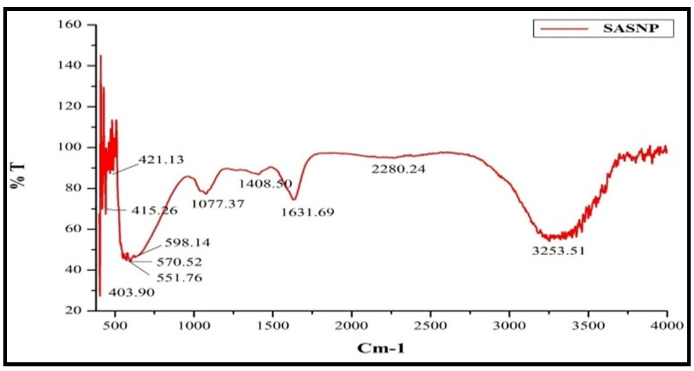

Figure 3: FT-IR peaks of SASNPs.

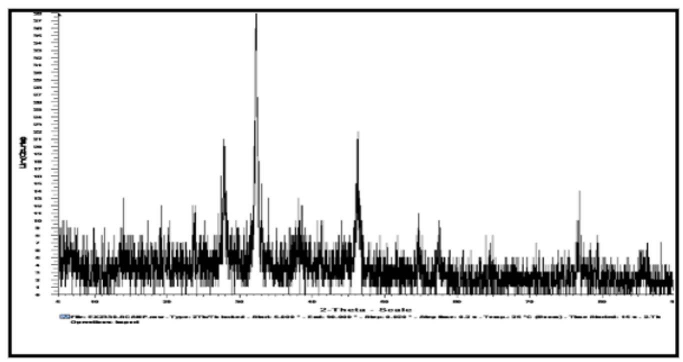

Figure 4: XRD analysis of SASNPs.

which indicates the photocatalytic degradation reaction of these dyes due to the reducing capability of SASNPs (Figure 10). Two peaks at 664 and 614 nm in the UV-vis absorption spectrum of methylene blue in an aqueous media were observed.

Ecotoxicology

The synthesised silver nanoparticles are diluted in sterile water at concentrations of 25, 50, 100, and 150 mg per 100 mL, respectively (Figure 11). Before introducing earthworms, the sample are sprayed into the soil for three days. *Eisenia fetida*, a type of earthworm that ISO, OECD, and USEPA advocate using as a test organism, is gathered from the fields and placed into the soil that has been treated with the sample solutions (Figure 12). After 4 weeks of introduction, the mortality rate of earthworm is observed to be as 0.3333±0.333 in the lowest concentration of 25 mg/100 mL followed by 1.33±0.33 in 50 mg/100 mL, whereas the mortality rate is 7.6±0.882 in 100 mg/100 mL and 9.66±0.882 in 150 mg/100 mL (Table 3).

Table 2: Heavy metal analysis in SASNPs.

Sl. No.	Name sample	Test method	ppm: mg/kg
1	SASNP	Association of Official Analytical Chemists	1.5

DISCUSSION

The dark brown color was a sign that the aqueous silver ions in the reaction mixture were reduced to silver nitrates[10] and was generated because the Surface Plasmon Resonance (SPR) excitation of the AgNPs.[11,12] The free electrons of AgNPs gave Rise to SPR absorbance as a result of the combination of electrons vibration of the MNPs in resonance with the light waves.[13]

A Rapid synthesis using UV also influenced the absorbance. According to Rama Krishna *et al.*,[14] the *Sansevieria roxburghiana* SPR band was first discovered at 429.95 nm. These may be impacted by some physical or chemical conditions since the nanomaterial should be between 200 and 800 nm in size. According to Prabu and Johnson,[15] the AgNPs' peaks were

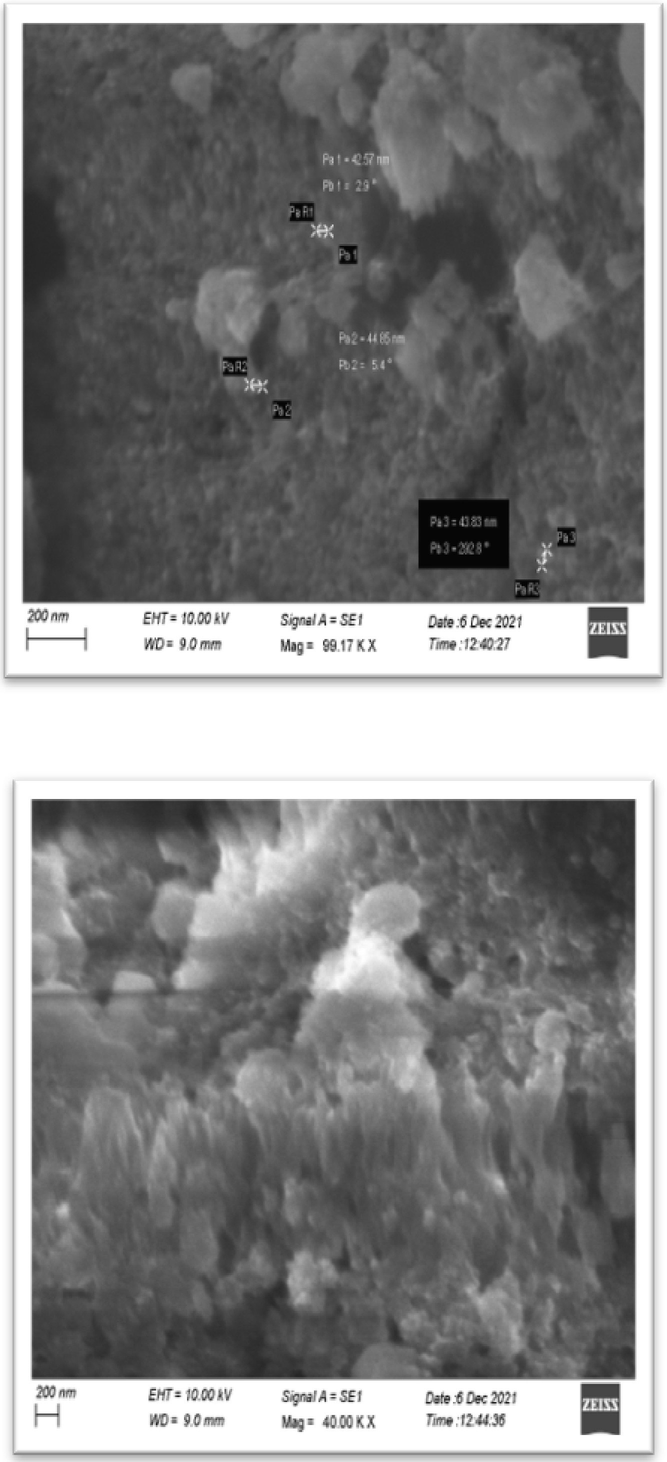

Figure 5: SEM analysis of SASNPs.

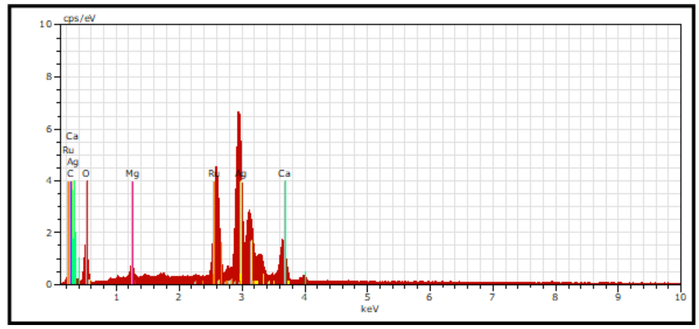

Figure 6: EDAX analysis of SASNPs.

in the 250-400 nm range and were connected to their SPR. As previously described,[16,17] these noble metal nanoparticles exhibit an absorbance characteristically between 400 and 430 nm. The absorption (SPR) peak of these biogenic AgNPs did not change over the course of six months. This may indicate that the phytochemicals in the leaf extracts of this medicinal plant also serve as capping agents, stabilizing SNPs through their capping action.

The findings and those in the literature are well-aligned.[18] The bioorganic molecules from the FT-IR studies[19] show that a significant capping agent was produced on the nanoparticles by "*S. roxburghiana*" extracts. The FTIR spectra revealed a number of functional groups in charge of the production of SNPs. Ag nanoparticles capping layers and synthesis were most likely accomplished by these functional groups. The result was that the synthesized SNPs were less dangerous and toxic compared by Arif *et al.*[20] SNPs.

Furthermore, the Debye-Scherrer equation revealed an average size of 27 nm, and the outcomes were evaluated in comparison to earlier investigations in the literature.[21,22] The crystalline character of the SNPs produced by the reduction of Ag^{+} ions using *S. angolensis* extract was clearly demonstrated by the well-resolved and strong XRD pattern.

SEM microscopy was also used to evaluate morphology, including voids, fractures, porosity, agglomeration, grain distribution, and average grain size. The silver nanoparticles range from 42.57-44.85 μm. According to earlier research,[23] coriander leaf extract was used in the production of gold nanoparticles.

The physical properties of nanoparticles as determined by TEM and EDX were found to be positively linked with results from earlier investigations.[24] For the production of SNPs, the phytochemical group in *S. angolensis* serves as a capping and binding agent. This investigation demonstrates that NPs are composed of the purest Ag^{+}.

The AAS technique was used to conduct a heavy metal accumulation investigation in order to control the minimal amount of reported harmful effects for applications, particularly in the food business. Due to the long history of human usage of metals including lead, gold, silver, copper, and tin, heavy metals are among the most common pollutants detected in soils.[25] In order to distinguish between soils that do not pose a risk and those that might or already do so for the purposes intended, governments have also put policies in place to protect both human health and the environment.[26] These policies involve measuring metal concentrations in soils and establishing threshold values.

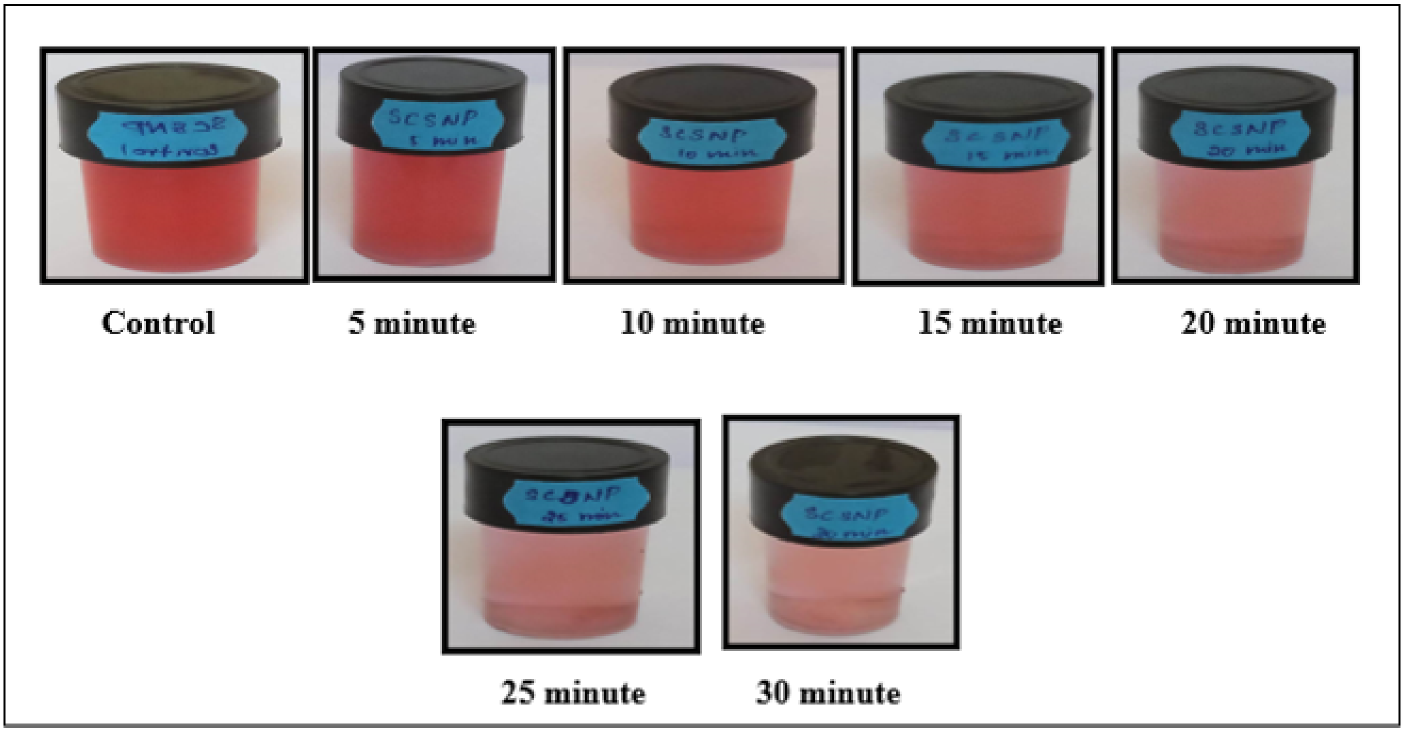

Figure 7: Reduction of congo red dye.

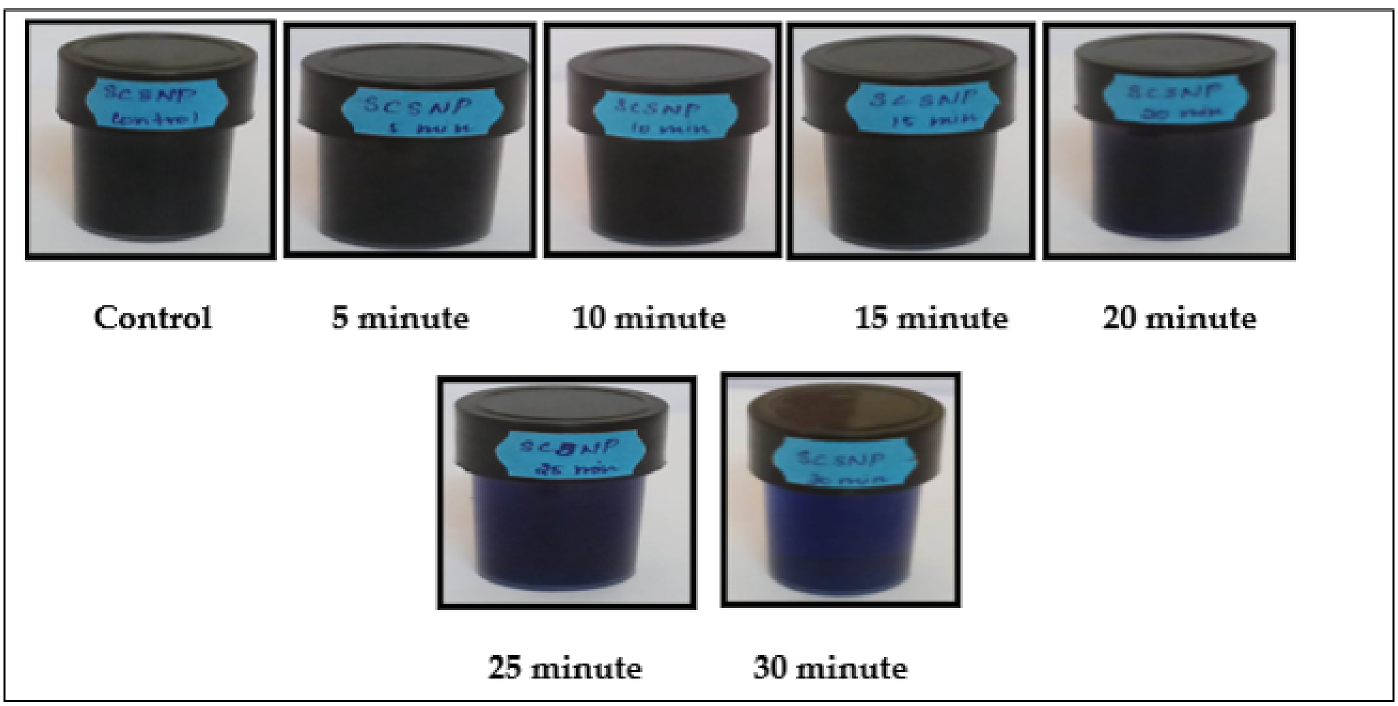

Figure 8: Reduction of coomassie blue dye.

In the presence of $NaBH_4$, the produced biogenic gold nanoparticles demonstrated strong catalytic capability for the oxidation of harmful dyes like 4-nitrophenol, bromothymol blue, acridine orange, phenol red, congo red, and methylene blue. The rate of dye degradation in the presence of gold nanoparticles was analyzed using the Langmuir and Hinshelwood model to determine its connection to time.[27] Anionic diazo dye congo red has been shown to have mutagenic and carcinogenic properties.[28] According to research by Ganapuram *et al.*[29] and Kang *et al.*,[30] methylene blue, which is released by the textile, paper, rubber, and plastic industries, poses a major threat to the environment.

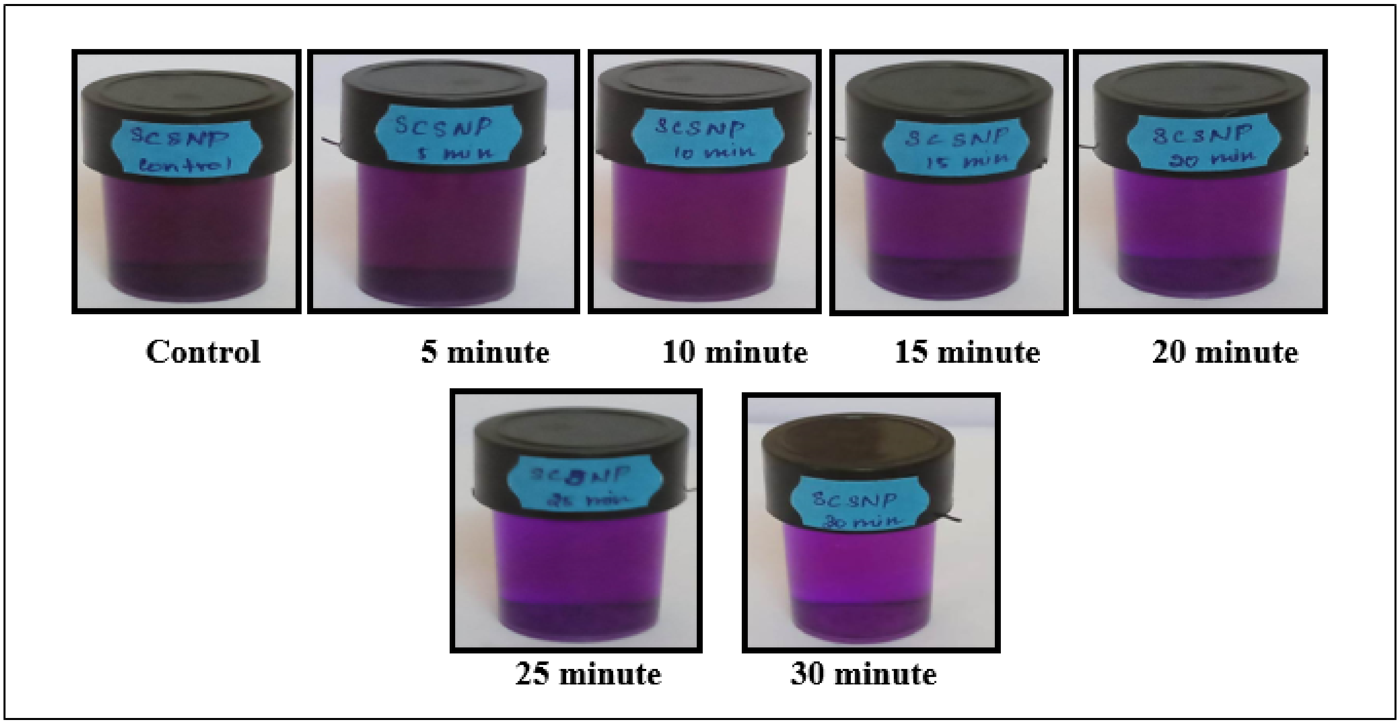

Figure 9: Reduction of Crystal violet dye.

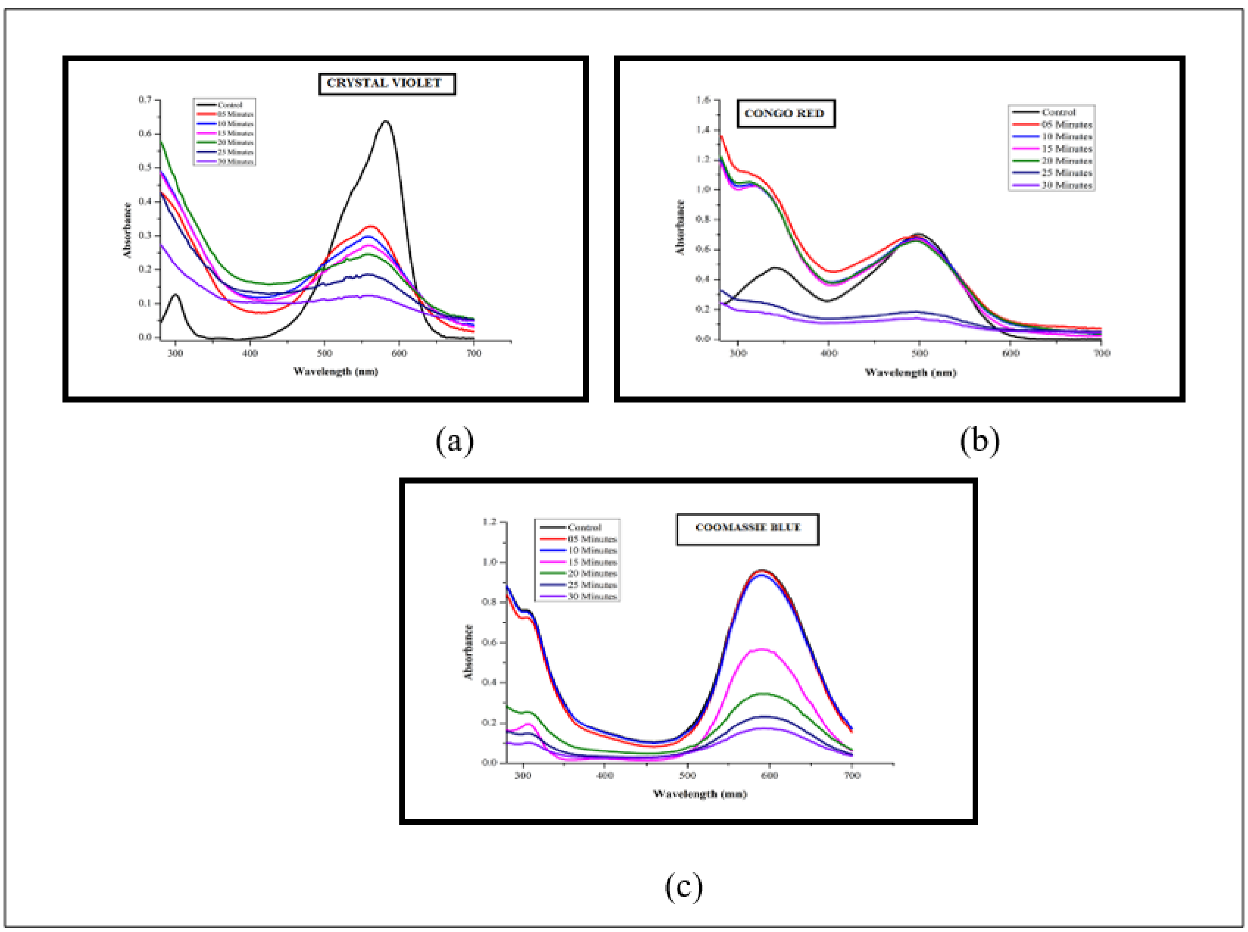

Figure 10: UV-analysis of crystal violet dye (a), congo red dye (b) and coomassie blue dye (c).

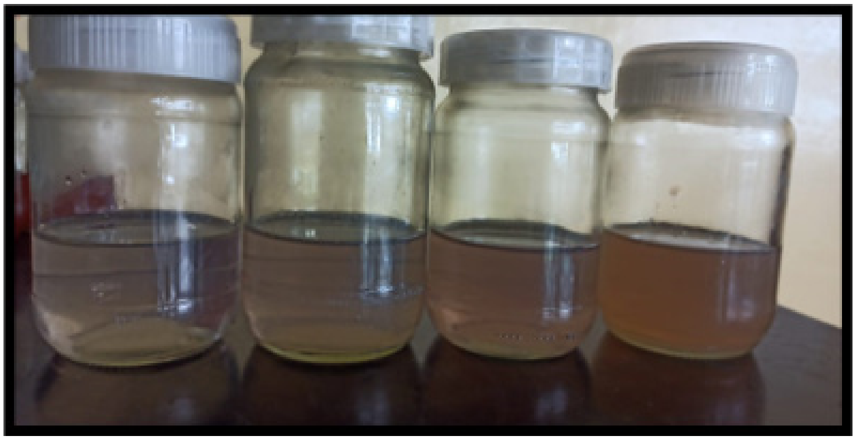

Figure 11: SASNPs solutions.

Figure 12: Earthworm beds (1-25 mg/100 mL, 2-50 mg/100 mL, 3-100 mg/100 mL, 4-150 mg/100 mL.

Table 3: **Ecotoxicity of SASNPs in earthworm**

Sl. No.	Concentration of SASNPs solution	No. of earthworms during introduction			No. of worms alive after 4 weeks			Rate of mortality
	Per 100 mL	Trial 1	Trial 2	Trial 3	Trial 1	Trial 2	Trial 3	
1	25 mg	10	10	10	10	10	9	0.3333±0.333
2	50 mg	11	11	11	10	10	9	1.33±0.33
3	100 mg	15	15	`15	7	9	8	7.6±0.882
4	150 mg	12	12	12	4	2	1	9.66±0.882

The use of the dye's crystal violet, coomassie blue, and congo red allowed to demonstrate the photocatalytic activity of silver nanoparticles on dye degradation. At various times in the visible region, dyes were degraded in the presence of silver nanoparticles. Two peaks at 664 and 614 nm in the UV-Vis absorption spectrum of methylene blue in an aqueous media were observed, correlating with the findings of Uddin *et al.*[31] These results revealed that the biogenic gold nanoparticles by *S. roxburghiana* leaf extract showed catalytic activity in the reduction of dye pollutants. Due to their large surface to volume ratios,[32] the synthetic biogenic gold nanoparticles made by *S. roxburghiana* work well as catalysts for the breakdown of hazardous organic contaminants.

The samples are sprayed into the soil for three days. After 4 weeks of introduction, the mortality rate of earthworm is observed to be as 0.3333±0.333 in the lowest concentration of 25 mg/100 mL followed by 1.33±0.33 in 50 mg/100 mL, whereas the mortality rate is 7.6±0.882 in 100 mg/100 mL and 9.66±0.882 in 150 mg/100 mL. According to a prior study, coated AgNPs were about 10 times less dangerous than uncoated AgNPs, with no appreciable impacts on growth, mortality, or reproduction, even if the results for $AgNO_3$ were identical. The average earthworm survival rate across all treatments was higher than 80% during the experiment, indicating that the experiment was acceptable.[33] Mwaanga demonstrated that weight loss in earthworms was dose-related.[34] Other researchers have noted that the survival of earthworms may be due to the agglomeration of nanoparticles at greater concentrations.[35]

CONCLUSION

Eco-friendly synthesis of nanoparticles is recognized by enthusiastic scientists for the sustainable future. Amidst of numerous metals used to synthesize nanoparticles, silver-based nanoparticles synthesis is prominent as silver is essential for all living organisms. For the production of SNPs, the phytochemical group in *S. angolensis* serves as a capping and binding agent. It is also used as the mix-ups in fertilizers to enhance the plant growth and inhibit pests. Bio-synthesized SNPs have very low metal accumulation compared to chemically synthesized NPs. This study reveals that the Biogenic synthesis of NPs showed low ecotoxicity on earthworm *Eisenia fetida*.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

AUTHORS' CONTRIBUTIONS

Manoharan Laxmi Kiruthika: developed the concept, did the experiments, generated and analyzed the data, provide literature review, data analysis, guarantor. Ammasaikutty Vanitha: experimental design, and literature, reviewed the concept, prepared the first draft of this manuscript, literature search, data collection, analysis and statistics, updating and editing, guarantor. Kandasamy Kalimuthu: organization and analysis, detailed review of the manuscript, supervised the day-to-day experiments of the project, guarantor. All authors contributed to the final version of the manuscript.

ABBREVIATIONS

SASNPs: *Sansevieria angolensis* Silver Nanoparticles; **FT-IR:** Fourier transform infrared spectroscopy; **XRD:** X-ray powder diffraction analysis; **SEM:** Scanning electron microscope; **EDX:** Energy dispersive X-ray analysis; **$AgNO_3$:** Silver nitrate; **AAS:** Atomic absorption spectrometry; **$NaBH_4$:** Sodium borohydride; **OECD:** Organization for Economic Cooperation and Development; **USEPA:** United States Environmental Protection Agency.

SUMMARY

The leaves of the plant *Sansevieria angolensis* belonging to the family Asparagaceae were used and investigated for synthesis of silver nanoparticles as well as their characterization through UV, FTIR, XRD, SEM, and EDX analysis, as well as their phytochemical screening, dye degradation, heavy metal accumulation and ecotoxicological properties in aqueous extract. Aqueous extract from SASNPs was found to be effective in all of these studies.

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