Rapid Green synthesis of Nano SiO₂ using Bryophyllum pinnatum leaf and evaluation of physicochemical quality attributes

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Abstract

The present study focused on determining the phytochemical analysis of the possible chemical groups present in leaves extract, green synthesis, its characterization, and applications. *Bryophyllum pinnatum* leaf extract was used to synthesized the Silica nanoparticles. Green synthesized nanoparticles were characterized by different techniques such as UV-Visible absorbance spectroscopy, Fourier Transform Infra-Red, X-Ray Diffraction, Scanning Electron Microscope, Energy Dispersive X-Ray, Zeta Potential Analysis and Thermo-Gravimetric Analysis. A UV-Visible spectrum of silica nanoparticles displayed an absorption peak at 280 nm, and FT-IR results highlighted the key bioactive compounds that could be responsible for capping and reduction of Silica nanoparticles. The XRD pattern analysis showed its crystalline nature and an average size is 24 nm. SEM analysis revealed that synthesized nanomaterials are spherical in shape. Silica nanoparticles showed excellent stability with negative zeta potential value (– 32 mV) and uniform dispersion in aqueous media. Moreover, the seed germination assay was carried out on *Vigna radiata* using the biogenic silica nanoparticles. The low concentration of silica nanoparticles was enhancing the seed germination. Meanwhile, the higher concentration of the silica nanoparticles decreased seed germination and shoot and root formation. Silica nanoparticles at optimum concentration could be used in the agriculture field as nano growth promoters.

1. Introduction

The field of nanoscience and nanotechnology has the potential for revolutionizing the methods to create the novel materials. It will give the significant impact on biological sciences and increase its values exponentially in the future (Kumar and Kumbhat, 2016; Hulla et al., 2015). These unique nanomaterials have wide-ranging physicochemical properties and are extensively used in food packaging industries, agricultural industries, biosensors, biomedical, textile and environmental applications (Hulla et al., 2015; Nguyen et al., 2018).

Among all the available nanoparticles, inorganic mesoporous silica nanoparticles are the newcomers to the field, contributing with their unique physical and chemical properties and excellent properties (small size, strong surface energy, high scattered performance, and thermal resistance) (Ma et al., 2013; Wang et al., 2015). Silica nanoparticles have numerous applications, including drug delivery, biosensing, catalysis, bioimaging, and energy storage (Nandanwar et al., 2013; Kordasht et al., 2020). Silicon plays a vital role in increasing plant tolerance against pests and diseases. Mesoporous silica nanoparticles were used as a solid adsorbent for the removal of lindane pesticides from aqueous solutions (El-Said et al., 2018). The synthesis, properties, and applications of silica nanoparticles have become a quickly expanding field of research (Nandanwar et al., 2013). The silica nanoparticles have been produced from agricultural wastes (Adebisi et al., 2019). Stober process is used for the preparation of monodispersed silica colloids 'white carbon black' by hydrolysis of alkyl silicates and silicic acid in alcoholic solutions using ammonia as catalyst (Ibrahim and Zikry et al., 2010).

Synthesis of metal/metal oxides nanomaterials, hybrid materials, and bioinspired materials using plants and microbes/other natural resources has received significant attention as a reliable, sustainable, cost-effective and an eco-friendly technology in nanoscience alternative to chemical and physical methods (Singh et al., 2018; Parveen et al., 2016).

Bryophyllum pinnatum (Figure 1) is commonly known as *Pattharcaṭṭa*. It belongs to Crassulaceae family. The herb contains a wide range of valuable chemicals that could be responsible for its various pharmacological effects (Latif et al., 2019). It is mainly used as a natural anti-inflammatory agent (Fernandes et al., 2019). It is used in ethnomedicinal practices to treat kidney stones, jaundice, skin diseases, hypertension, and urinary problems (Oufir et al., 2015; Yadav et al., 2016). The leaves of *B. pinnatum* possess both sedative and muscle relaxant properties (Von Manitius et al., 2019).

This study focused on the green synthesis of silica nanoparticles using an aqueous extract of *B. pinnatum* by sustainable approach, simple, cheap, and environmentally friendly manner. In addition, the current research aimed to investigate the effect of silica nanoparticles on seed germination of *Vigna radiata* and its development of root and shoot after the germination.

2. Materials And Methods

The analytical grade chemicals and reagents were used in the experimental studies. Tetra Ethyl Ortho Silicate (TEOS), Hydrochloric acid, Ethanol and Double distilled water (DDW) were obtained for this investigation. All the glasswares were soaked in acids and washed with distilled water.

2.1. PREPARATION OF PLANT EXTRACT AND PHYTOCHEMICAL SCREENING

Fresh and healthy leaves were collected from villages of south Coimbatore (District), Tamilnadu, India, on January 2021. The leaves were cleaned with running tap water to remove debris and other organic contents. The aqueous extract was made by mixing and macerating 10 grams of fresh *B. pinnatum* leaves with 200 ml of double-distilled water. The mixture was boiled for 20 minutes at 50°C. The extract was cooled down and filtered by Whatman filter paper, and the extract was stored at 4°C for further analysis. The phytochemicals of *B. pinnatum* were screened by the methods of Harborne (1998).

2.2. SYNTHESIS OF SILICA NANOPARTICLES

Tetra Ethyl Ortho Silicate (TEOS) is a precursor for the production of silica nanoparticles. 20 mL of plant extract was mixed with 12 mL of precursor solution and allowed continuous stirring at 50 to 65°C for 10 min. Then, 1 M HCL was mixed on the above mixture. After 20 min, jelly-like precipitation was formed. Next, the precipitation was dried in a hot air oven. Finally, the white color power was obtained and stored in a sterile air tight container for further analysis.

2.3. CHARACTERIZATION OF SYNTHESIZED SILICA NANOPARTICLES

Various techniques were used to characterize the synthesized biogenic silica nanoparticles. The optical property of biogenic silica nanoparticles was determined by the UV-Visible Double beam spectrophotometer. The nature of biogenic silica nanoparticles was determined by the X-ray diffractometer. The atomic percentage of elements present in the biogenic silica nanomaterials was determined by EDX. The surface morphology of silica nanoparticles was assessed by the SEM analysis. The functional group of silica nanoparticles and aqueous plant extract were identified by the FT-IR spectrometer. The charge of silica nanoparticles was determined using a Malvern particle size analyzer. Thermal stability of synthesized silica nanoparticles was analyzed by Mettler-Toledo TGA.

2.3. SEED GERMINATION ANALYSIS

Seeds of *Vigna radiata* were used for this investigation. Four different concentrations (25, 50, 75, and 100 µg/mL) of silica nanoparticles were prepared using distilled water. The seeds were surface sterilized using HgCl₂ and distilled water. Meantime, the sterile Petri plates were taken, and cotton was placed on them. Then, the 15 seeds were placed on respective plates. A 15 mL of biogenic silica nanoparticles at various concentrations was poured on seeds in corresponding plates. Positive control (distilled water) was maintained. Next, the plates with seeds were incubated in the dark condition at 37°C for five days. After incubation, the germinated seeds were counted, and the root and shoot length were measured. Five replications were used for this analysis.

3. Results And Discussion

3.1. PHYTOCHEMICAL ANALYSIS

Table 1 determined the phytochemicals analysis of *B. pinnatum* (aqueous extract). The steroids, alkaloids, flavonoids, tannins, saponins, and reducing sugar were found in *B. pinnatum* leaf extract. Terpenoids and Glycosides were absent in the extract. Kavita et al., (2013) reported the medicinally active phytoconstituents like tannins, alkaloids, terpenoids, steroids, and saponins in the leaves of *Phyllanthus fraternus*.

Table 1
Screening of phytochemicals in *B. pinnatum* extract.

S.No	Qualitative test	Colour Obtained	Aqueous extract (<i>B. pinnatum</i>)
1.	Steroids	Blue green	Presence
2.	Alkaloids	Orange to Red	Presence
3.	Flavonoids	Yellow	Presence
4.	Terpenoids	Red, Pink or Violet	Absence
5.	Tannins	White precipitate	Presence
6.	Saponins	Foam formation	Presence
7.	Glycosides	Blue or Green	Absence
8.	Reducing sugar	Yellow to Orange	Presence

3.2. CHARACTERIZATION OF SILICA NANOPARTICLES

3.2.1. UV-VISIBLE SPECTROSCOPY

UV-Visible absorbance spectroscopy is broadly being utilized as a technique to determine the optical properties of nanosized particles. The result obtained from the analysis of UV-Visible spectroscopy of the sample is presented in Figure 2. The silica nanoparticles formation was confirmed by the peak occurrence in the range between 230 to 300 nm. to Winardi et al., (2020) were analysis the optical properties of silica-coated zinc oxide nanoparticles using the UV-Visible spectroscopy and obtained peaks between the wavelength range of 280 to 400 nm in UV spectra.

3.2.2. X-RAY DIFFRACTION (XRD)

The crystallinity of biogenic silica nanoparticles was evaluated from the X-Ray Diffraction pattern. The most substantial peaks at 2theta values of 22.01 correspond to the crystalline nature silica nanoparticles and are shown in Figure 3. The average size of the silica nanoparticles was calculated by Debye-Scherrer's formula. The size of the *Bryophyllum pinnatum* leaf extract mediated silica nanoparticles is 24 nm. Mohd et al., (2017) carried out the XRD analysis to find out the crystalline nature and size of the sugarcane bagasse mediated nanomaterials.

3.2.3. ENERGY DISPERSIVE X-RAY DIFFRACTION (EDAX)

The elemental composition and purity of *Bryophyllum pinnatum* leaf extract mediated silica nanoparticles were determined by the EDAX analysis. Figure 4 shows the EDAX spectrum for phyto-genic mediated silica nanoparticles. The atomic percentages of carbon (21.68%), oxygen (50.95%), and silica (27.37%) were present in green synthesized silica nanoparticles. The carbon was derived from plant

extract. The silica nanoparticles were synthesized by Dubey et al., (2021), and they assessed the level of its composition. They reported the presence of silica and oxygen with no impurities.

3.2.4. SCANNING ELECTRON MICROSCOPE (SEM)

The shape or morphology of the synthesized silica nanoparticles was determined by the SEM analysis. The microscopic images of *Bryophyllum pinnatum* leaf extract mediated silica nanoparticles are presented in Figure 5 and clearly show the distribution and spherical shape of the silica nanoparticles. Adebisi et al., (2020) reported the production of biogenic silica nanoparticles using maize stalk and determined its morphology using the SEM analysis.

3.2.5. FOURIER TRANSFORM INFRARED SPECTROSCOPY (FT-IR)

FT-IR spectra of plant extract and silica nanoparticles are shown in Figures 6a and b. The peaks such as 3348, 2970, 2885, 1921, 1658, 1442, 1087, 879, 671, and 555 cm⁻¹ are in Figure 4a and correspond to the hydroxyl, amide, and carboxyl functional groups. The spectrum of silica nanoparticles shows the peaks such as 2978, 2893, 1589, 1396, 1143, 1072, 956, 678, and 555 cm⁻¹. The nanomaterials spectrum has metal oxide functional groups and hydroxyl, amide, and carboxyl functional groups. FT-IR study was determined the functional groups of capping, reducing, and stabilizing agents from *Bryophyllum pinnatum* leaf extract to form the nanomaterials. Anuar et al., (2020) were found the functional groups or chemical groups, namely Si-O-Si, CH₂, -OH, and Si-OH from the FT-IR spectrum of coconut husk ash mediated silica nanomaterials.

3.2.6. ZETA POTENTIAL MEASUREMENT

Zeta potential analysis is a traditional method to determine the stability of the nanomaterials. The silica nanoparticles from *B. pinnatum* show negative charges and the zeta potential value of -32.4 mV (Figure 7). The high value of zeta potential refers to the stability of the suspension due to the increased force of electrostatic repulsion between the particles. The low zeta potential value indicates the aggregation of the nanomaterials (Wang et al., 2010). The silica nanoparticles with antibacterial properties were produced by Joni et al., (2020). In addition, they reported the zeta potential value of -24.69 mV for the silica nanoparticles.

3.2.7. Analysis of Thermal stability

Thermal stability of synthesized silica nanoparticles was performed to determine the weight loss of green synthesized silica nanoparticles at different temperatures (the range between 30 to 1000°C) (Figure 8). The 50% of weight loss was occurs at 150°C because to removal of water molecules and degradation of volatile compounds on surface of as-synthesized silica nanoparticles.

3.3. SEED GERMINATION

Different concentrations (5, 10, 15, and 20 µg/mL) of silica nanoparticles were used in the present investigation. The maximum level (100%) of seed germination was achieved on 5 µg/mL silica nanoparticles treated treatment, and meanwhile, the minimum level (40%) of seed germination was observed on 20 µg/mL of silica nanoparticles treated treatment. The increased shoot length of 4.3 cm was observed on the treatment of 5 µg/mL silica nanoparticles, and decreased shoot length of 1.8 cm was achieved with a concentration of 20 µg/mL of silica nanoparticles. The highest root length (1.0 cm) was observed on T2 treatment (5 µg/mL of Silica nanoparticles), and the lowest root length (0.2 cm) was recorded on T4 treatment (20 µg/mL of silica nanoparticles) (Table 2). Results showed that silica nanoparticles have a significant effect on seed germination, the length of the shoot, and the root. Roohizadeh et al., (2015) observed that silica nanoparticles improved the seed germination on *Vicia faba*.

Table 2

Analysis of seed germination, root and shoot length on Silica nanoparticles treated *Vigna radiata*.

S.No	Concentrations	% of seed germination	Shoot length (cm)	Root length (cm)
1	Control – Distilled water	100%	4.0 ± 0.1	1.0 ± 0.2
2	T1 – 5 µg/mL	100%	4.3 ± 0.1	1.5 ± 0.1
3	T2 - 10 µg/mL	70%	3.4 ± 0.2	0.8 ± 0.1
4	T3 - 15 µg/mL	50%	2.0 ± 0.3	0.5 ± 0.1
5	T4 - 20 µg/mL	40%	1.8 ± 0.1	0.2 ± 0.1

4. Conclusion

The aqueous extract of *Bryophyllum pinnatum* leaf has been used as capping and reducing agents for the green synthesis of silica nanoparticles. The phyto-bioactive like steroids, alkaloids, flavonoids, tannins, saponins, and glycosides were present in leaf extract, confirming the phytochemical analysis. Bio-synthesized silica nanomaterials were characterized by various techniques. Spherical-shaped biogenic silica nanoparticles were produced with an average size of 24 nm. The green synthesized silica nanoparticles enhanced seed germination, shoot, and root formation at low concentration levels. So, Silica nanoparticles could be used to improve seed germination and crop production in agriculture areas.

Declarations

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DECLARATION OF INTERESTS

☐ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

*Ethics approval – Not applicable

* Consent to participate – Not applicable

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* Availability of data and materials – Not applicable

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* Authors' contributions

SN: Conceptualization

SM: Investigation

AA: Methodology

PR: Supervision, Funding acquisition and Project administration

SD: Data Curation and Writing- Original draft preparation

MMA: Data Curation and Writing- Original draft preparation

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Figures

Figure 1

Bryophyllum pinnatum

Figure 2

UV spectra of a) plant extract and b) Silica nanoparticles

Figure 3

XRD spectrum of Silica nanoparticles

Figure 4

EDX spectrum of Silica nanoparticles

Figure 5

SEM images of Silica nanoparticles

Figure 6

FT-IR spectrum of a) Silica nanoparticles and b) plant extract

Figure 7

Zeta potential analysis of Silica nanoparticles

Figure 8

TGA analysis of Silica nanoparticles