Phyllantus Emblica Mediated Silica Nanomaterials: Biosynthesis, Structural and Stability Analysis

M Sankareswaran
Muthayammal College of Arts and Science

M Vanitha
Karpagam Academy of Higher Education

P. Rajiv (rajivsmart15@gmail.com)
Karpagam Academy of Higher Education

A. Anbukumaran
Urumu Dhahalakshmi College

Short Report

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Abstract

The current investigation reports on a green route, simple and eco-friendly method for synthesis of silica nanoparticles from *Phyllantus emblica*. Appropriate characterization techniques were employed to assess the crystalline nature, microstructure, size, purity, elemental composition and stability of as-biosynthesized silica nanoparticles. The XRD analysis showed a wide-ranging peak at 22° of 2θ value and proved that the nanoparticles were crystalline nature with 32 nm average size of particles. FT-IR studies confirmed the occurrence of metal oxide group and presence of phyto-molecules namely hydroxyl, amide, and carboxyl functional groups, which were responsible for formation and stabilization of silica nanomaterials. TGA and Zeta potential analysis determined that silica nanoparticles are highly thermostable. EDX analysis revealed the purity of nanomaterials and spectra confirmed that formation of silica nanomaterials (72.97 weight percentage of SiO₂ content) with low impurities. SEM analysis shows that the particles are spherical in shape with low agglomeration. This research work concluded that the *P. emblica* was an excellent and reliable green resource for production of highly stable and potential silica nanoparticles.

1. Introduction

Nanotechnology refers to technology that is apply at the nanoscale and has applications in the here and now. Unique physical and chemical properties of nanomaterials can make use for applications that benefit for society (Bhushan, 2017). Nanomaterials are vital role in manufacturing new device with a vast range of applications in the field of nanoelectronics, nanomedicine, biomaterials energy production, nano sensor and consumer products (Pawliszak et al., 2019). Green synthesis is process for production of nanomaterials through the biological substrates/extracts to explore their numerous applications. Green synthesis methods are producing nanomaterials with biocompatibility and highly stable. (Namvar et al., 2021). It is a simple, environmentally friendly and single step. Bio molecules such as enzymes, proteins, amines, phenolic compounds, pigments and alkaloids are involved in fabrication of nanomaterials by reduction method (Nadaroglu et al., 2017). Green synthesis approach is offering safety, reliability, scalability, controllable particle size distribution, simplicity, and inexpensive technology during the production of nanomaterials (Shafiei et al., 2021). Nanoparticle have the ability to improve a wide range of agricultural, environmental, and forestry problems.

Silica nanomaterials are immensely stable and less toxic (Jeelani et al., 2020). Silica nanomaterials have unique characters such as high surface area, stability, biocompatibility and surface reactivity. Hence, silica nanoparticles have been used in various applications in drug delivery, optical imaging agents, sensor field, medical field and agriculture (Karande et al., 2021). Several researchers are focused on production/fabrication of silica nanomaterials by green synthetic protocols that utilize the medicinal plants (Babu et al., 2018), agriculture waste (Vaseashta and Kavaz, 2020), industrial waste (Yadav and Fulekar, 2019), etc. The green synthesis of silica nanomaterials is vital area of research having significant potential for further forthcoming developments. Silica is not important for the survival of plants, but it required to adapt the plants under different biotic and abiotic stress conditions (Rajiv et al., 2020). Silica
nanomaterials can be employed as transport agents for agrochemicals, proteins and nucleotides. The silica nanoparticles are enhancing the soil water retention and soil monitoring in agriculture sector (Rastogi et al., 2019).

*P. emblica* L. (Indian gooseberry) has various pharmacological properties, that is widely employed in traditional medicine in different countries. Several research reports determined that fruit, leaves and barks of *P. emblica* contain the huge amounts of phenolic compounds (Arun et al., 2018). Hence, *P. emblica* shows the better anti-microbial, anti-cancer, hypolipidemic, anti-inflammatory, and hypoglycemic activity. It has excellent reactive oxygen species (ROS) scavenging activities.

Hereby, this present research work aimed to synthesis of silica nanoparticles by a facile and green method. It is expected that this technology will minimize the toxicity to environment. Additionally, to assess the chemical composition, microstructure, chemical bonding, crystalline nature, stability and zeta potential for as-synthesized silica nanomaterials by suitable characterization methods. Moreover, the toxicity of synthesized silica nanomaterials was determined using the seed germination assay.

2. Materials And Methods

2.1. COLLECTION OF *P. EMBLICA* AND EXTRACT PREPARATION

Healthy and fresh leaves of *P. emblica* were collected from Karpagam Academy of Higher Education Campus, Karpagam Academy of Higher Education, Coimbatore, Tamilnadu. 10 g of fresh *P. emblica* leaves were washed thoroughly with tap water and distilled to remove the debris. The extract was prepared according to protocol of Rajiv et al., (2013). The filtered extract was stored at 4°C in order to be used for synthesis of silica nanomaterials.

2.2. SYNTHESIS OF SILICA NANOPARTICLE

Synthesis of silica nanoparticles by green chemistry approach. The silica nanoparticles were produced from the precursor of 12.5 ml Tetra Ethyl Ortho silicate. A 5 ml of ethanol and 17.5 ml of *P. emblica* leaf extract were added into precursor solution. Then, it allowed to stirred continuously for 15 minutes. At mean time 0.1 N HCl was added to the mixture. Finally, the jelly like precipitation was formed. The precipitation was dried in hot air oven for 12 h at 200 ºC. At the end, the white color powder was obtained and stored in sterile air tight container for further experimental analysis.

2.3. CHARACTERIZATION OF SYNTHESISED SILICA NANOPARTICLE

The chemical composition, crystalline structure, chemical bonding, microstructure, elemental analyses, particle size distribution, and purity of the extracted silica were determined by, X-ray powder diffraction,
Fourier-transform infrared spectroscopy, Scanning electron microscope, Energy Dispersive Spectroscopy, Zeta Potential Analysis and Thermo-Gravimetric Analysis.

3. Result And Discussion

3.1. CHARACTERIZATION OF SILICA NAOMATERIALS

The crystallinity of green synthesized silica nanomaterials was assessed by X-Ray Diffraction. The major peak at 2theta values of 22.01 refers to the crystalline nature of nano silica and shown in Figure 1. Debye-Scherrer's formula was employed to determine the average size of silica nanomaterials. The average size of *P. emblica* leaf extract assisted silica nanomaterials is 32 nm. Babu et al., (2018) reported the green synthesis of bio silica nanoparticles with average size of 60 nm using the extract of *Cynodon dactylon*.

Figure 2a and b depicts the FT-IR spectra of *P. emblica* leaf extract and silica nanoparticles. The peaks such as 3379, 1635, 1056, 964, 601 and 555 cm\(^{-1}\) are in Figure 2a and corresponds to hydroxyl, carboxyl and metal oxide groups. FT-IR of plant extract (Figure 2b) shows few important peaks (3356, 1635 and 678 cm\(^{-1}\)) and which confirms that presence of hydroxyl, amide, and carboxyl functional groups. FT-IR studies of silica nanomaterials confirms that biomolecules are present in surface of green synthesized nanomaterials. Yadav and Fulekar, (2019) observed the three major characteristic peaks of the silica nanoparticles in FT-IR spectra at the region of 1200-400 cm\(^{-1}\).

The shape, purity and elemental composition were observed by the SEM with EDX. Figure 3a shows the EDX spectrum for *P. emblica* mediated silica nanoparticles. The atomic percentages of carbon (27.03%), oxygen (53.43%), and silica (19.54%) were occurred in biogenic silica nanoparticles. SEM images revealed that the distribution and spherical shape of the silica nanoparticles Figure 3b. Sadek et al., (2013) were synthesized nano silica from rice husk and their EDX spectra shows the Si and O peaks. Adebisi et al., (2020) described the synthesis of silica nanoparticles using maize stalk and determined its morphology using the SEM analysis.

Analysis of thermal stability for *P. emblica* mediated silica nanoparticles was carried out to determine the weight loss of nanoparticles at various temperatures (the range between 50 to 1000°C) (Figure 4). The 35% of weight reduction was obtained due to degradation of volatile compounds and removal of water molecules on surface of as-synthesized silica nanoparticles. Peralta et al., (2019) determined that thermal decomposition of the organic polymer matter in silica nanoparticles at 700°C which was confirmed by TGA analysis. Zeta potential analysis is a traditional technique to find the stability of nanomaterials. Green synthesized silica nanoparticles have high value of zeta potential suspension due to the increased force of electrostatic repulsion between the particles. Wang et al., (2010) concluded that nanoparticles have low zeta potential due to aggregation of nanomaterials.

4. Conclusion
The aqueous extract of *P. emblica* leaf has been used as stabilizing and reducing agent for biosynthesis of silica nanoparticles. Biogenic silica nanomaterials were characterized by various techniques. Spherical-shaped biogenic silica nanoparticles were produced with an average size of 32 nm. High stable silica nanoparticles were produced by simple, rapid and green chemistry approach. The green synthesized silica nanoparticles may be used in medicinal and agriculture areas.

**Declarations**

**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
* Consent for publication – Not applicable
* Availability of data and materials – Not applicable
* Competing interests

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* Authors' contributions

**SN:** Conceptualization

**VM:** Investigation

**PR:** Supervision, Funding acquisition and Project administration

**AA:** Data Curation and Writing- Original draft preparation

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**Figures**

**Figure 1**

<p><strong>XRD spectrum of Silica nanoparticles</strong></p>

**Figure 2**

<p><strong>FT-IR spectra of silica nanoparticles (a) and <em>P. emblica leaf </em>extract (b)</strong></p>

**Figure 3**

<p><strong>EDX spectra of silica nanoparticles (a) and SEM image of silica nanoparticles (b)</strong></p>

**Figure 4**

<p><strong>TGA spectra of silica nanoparticles</strong></p>