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A Review on Green Synthesis, Characterization, Multifunctional Applications of Biogenic Silicon Nanoparticles

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Abstract: This review paper provides a comprehensive evaluation of the green synthesis of silicon nanoparticles (SiNPs) derived from sustainable, biogenic fruit peel biomass and analyses their structural transitions and multifunctional industrial applications. Convectional chemical and physical synthesis routes face critical bottlenecks due to extremely high-energy demands and toxic alkoxide precursors, which leave hazardous chemical residues that compromise material biocompatibility. Upscaling biogenic resources addresses these constraints but introduces structural variability due to complex native organic networks. This paper establishes an optimized, low-cost laboratory-processing framework: raw biomass undergoes targeted acid pre-treatment with 2.0 M HCl for 2 hours to strip alkali metallic impurities, followed by controlled low-temperature calcination at 550°C for 4 hours to prevent structural sintering and retain a purely reactive amorphous silica ash backbone. High-shear alkaline dissolution under reflux using 2.0 M NaOH at 95°C for 3 hours successfully cleaves solid siloxane bonds to generate a highly viscous, transparent sodium trisilicate stock solution. Controlled sol-gel titration via dropwise acid addition (1.0 Ml/min) under rapid agitation (750 RPM) precipitates uniform nanosilica networks when terminated strictly at a neutral pH endpoint of 7.2. To bypass expensive inert gas setups, a localized molten salt (NaCl) thermal blanket method executed inside a porcelain crucible at 650°C safely drives the final methallothermic reduction to isolate high-purity, crystalline elemental SiNPs. Sequential structural characterization via FTIR and XRD tracks the precise chemical purity and amorphous-to-crystalline phase transition. Finally, mechanistic evaluation validates the high efficiency of these biogenic frameworks across three advanced domains: absorbing volume expansion (300%) as high-capacity lithium-ion battery anodes; serving as high-capacity adsorbents and radical photocatalysts in environmental wastewater remediation; and functioning as tortuous physical barriers and stimuli-responsive smart nanocarriers in anti-corrosion protective coatings. Ultimately, this review bridges the gap between low-infrastructural bench biosynthesis and predictable, high-performance industrial engineering. A bibliometric analysis was performed on “Silicon nanoparticle” using Scopus and VOSviewer.

Keywords: Silicon nanoparticle; Green synthesis; Sol-gel processing; Metallothermic reduction; Bibliometric analysis

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1.0 Introduction

Recently, nanotechnology has evolved from a fascinating area of science to a crucial force behind industry innovation. Silicon nanoparticles (SiNPs) are highly appealing options among the vast array of manufactured nanomaterials for numerous scientific domains. Silicon's abundance, intrinsic semiconducting nature, large theoretical capacity, and tuneable surface chemistry are the main reasons for this considerable interest in academia and industry (Nayl *et al.*, 2022). Silicon has distinct optoelectronic characteristics, quantum confinement effects, and high surface area to volume ratios at the nanoscale that are not present at all in the bulk form. As a result, SiNPs have been instrumental in the development of high-performance protective coatings, precise electrochemical sensors, targeted environmental clean-up systems, and next-generation energy storage devices (Seroka *et al.*, 2025).

The environmental and financial consequences of conventional synthesis techniques significantly impede the mainstream commercialization of SiNPs, despite their enormous technological promise. Extreme temperatures, vacuum systems, and dangerous chemical precursors are key components of both bottom-up and convectional top-down processes, such as chemical vapour deposition and sol-gel synthesis, and high-energy ball milling and laser ablation. Toxic alkoxides such as tetraethyl orthosilicate (TEOS), which produce volatile organic molecules, chemically contaminated nanoparticle surfaces, and hazardous secondary wastes, are commonly used in chemical routes. These hazardous wastes not only restrict compatibility of the nanoparticles but also conflict with the global mandate for sustainable manufacturing (Zulfiqar *et al.*, 2023).

To address these environmental and financial constraints, the paradigm of “green synthesis” or biosynthesis has emerged as a disruptive alternative. This sustainable approach replaces synthetic chemical precursors with biogenic resources, prominently leveraging agricultural waste (such as rice husk or sugarcane bagasse), microorganisms, and diverse plant extracts. In a typical green synthesis pathway, naturally occurring phytochemicals – including flavonoids, polyphenols, terpenoids, and organic acids – serve a dual function. They act simultaneously as eco-friendly reducing agents to convert silicon precursors and as natural capping matrices that stabilize the resulting nanoparticles against agglomeration. Furthermore, by utilizing agricultural by-products, green synthesis upcycles hazardous waste into high-value functional materials, exemplifying the principles of circular economy (Al- Gheethi *et al.*, 2023)

However, the transition from chemically controlled synthesis to biogenic fabrication introduces complex materials science challenges. Because biogenic extracts possess inherently variable and complex molecular compositions, controlling the precise structural evolution of green SiNPs remains remarkably

difficult. Small alterations in extract temperature s, pH or biological source profile can lead to drastic variations in nanoparticle crystallinity (amorphous silica vs crystalline silica frameworks), particles size distributions, surface functional groups, and pore configurations. In sensitive interfacial applications, these structural features dictate performance (Rahman *et al.*, 2022).

Searching for "Silicon nanoparticle" yields more than 62,000 documents from 1970 to 2026 and only 2287 articles when "green" is associated in the period (1996-now). The Scopus analysis, as well as VOSviewer, provides more information on the top 10 authors and countries that contributed to this area and maps showing potential collaborations between researchers and institutions (Eck and Waltman, 2007; Hammouti *et al.*, 2025; Igwe *et al.*, 2025; Kachbou *et al.*, 2025). Green synthesis is widely studied in recent decades, as shown in **Figure 1**. Interest in this theme spans several areas, with materials science, chemistry, engineering, and physics playing a prominent role (**Figure 2**). Scopus analysis yields the number of keywords from the recorded articles (22,261), resulting in 1,230 keywords that appear in at least 10 articles (**Figure 3**).

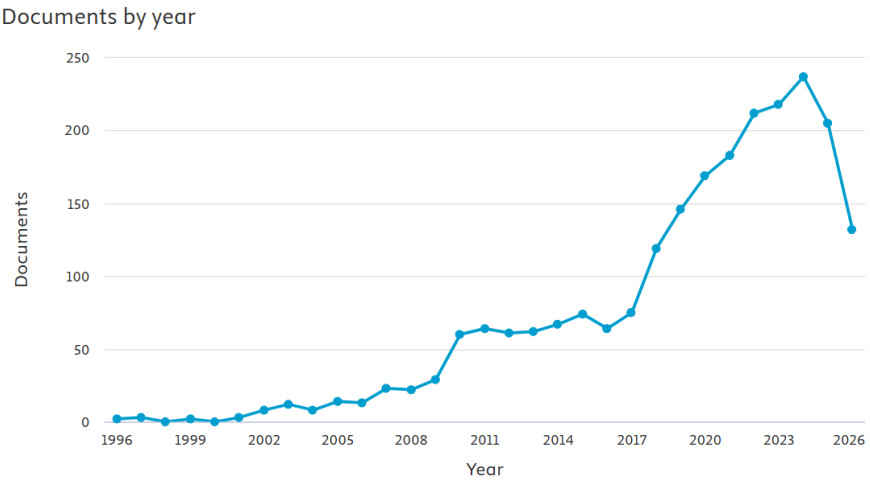


Figure 1. Evolution of the number of articles over the years.

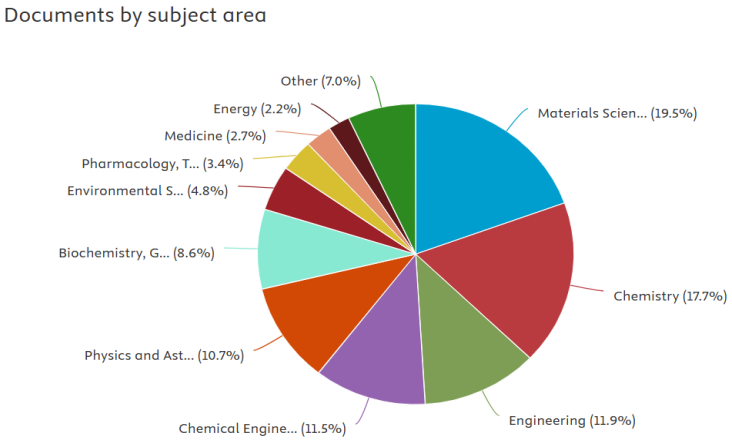


Figure 2. Distribution of documents by area during (1996-2026) period.

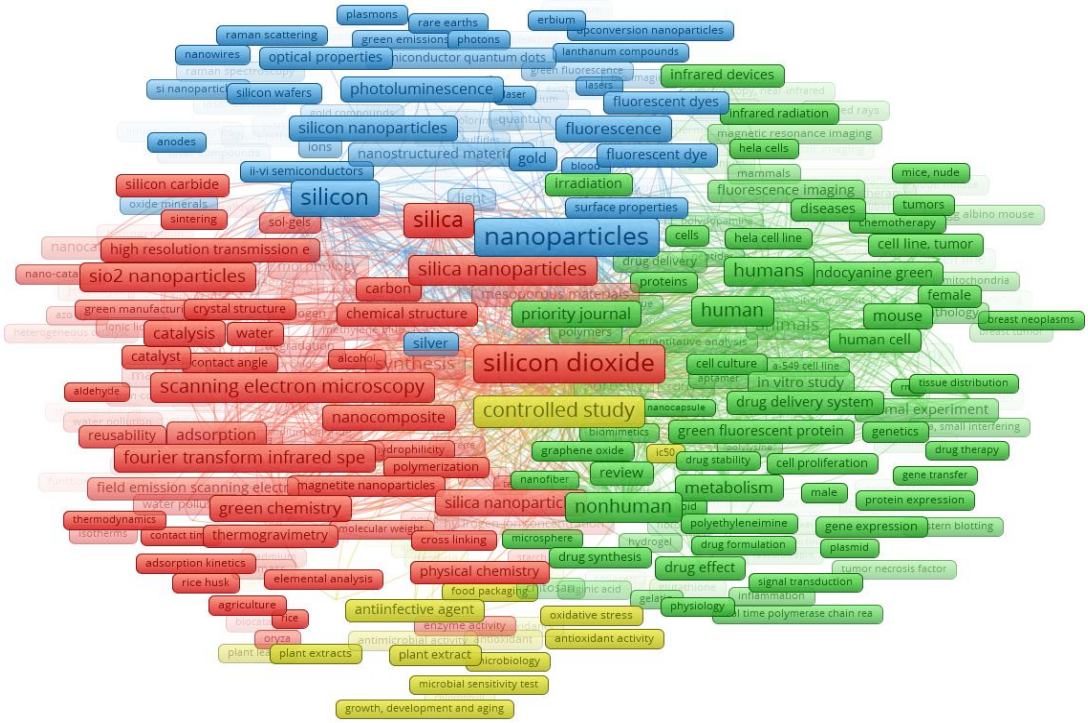
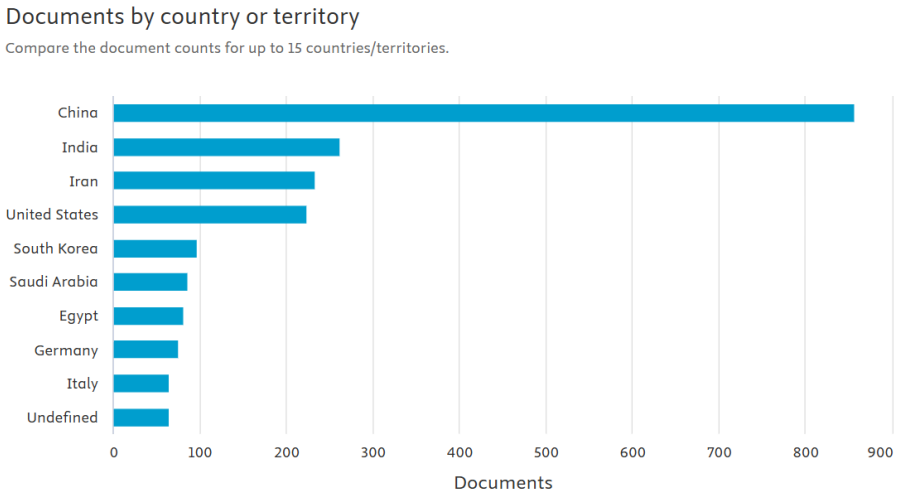


Figure 3. Ten top countries during (1996-2026) period.

As in many fields, China is increasingly becoming the leader—particularly in this area, where it has published over 850 articles—followed by India, Iran, the US, South Korea, and Saudi Arabia (**Figure 4**). This result is clearly visualized through the VOSviewer map, which indicates the contribution of countries using colored nodes as shown in **Figure 5**. The largest circle, the one in red, is reserved for China. The second circle is blue for India, the third is green for Iran, and the red one is for the US.



China	India	Iran	US	South Korea	Saudi Arabia
851	261	232	223	96	85

Figure 4. Ten top countries during (1996-2026) period.

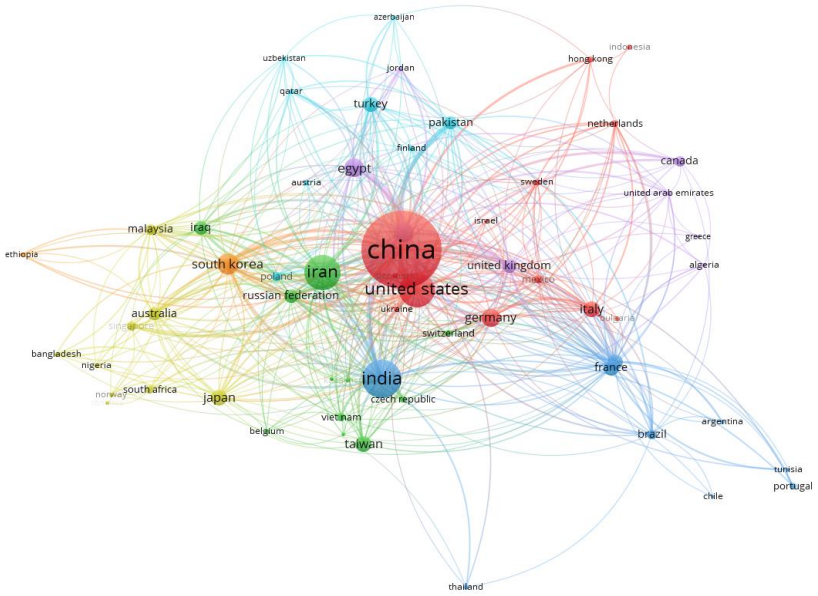


Figure 5. VOSviewer map of the best countries during (1996-2026) period

The direct causal linkages among green processing factors, the ensuing structural changes, and their related multi-domain performance are not well mapped in the literature, despite the fact that separate research has examined isolated aspects of biogenic nanomaterials. This important gap was filled in this review of the literature. This publication offers a comprehensive overview of green synthesis pathways for SiNPs by systematically gathering and critically evaluating recent research over the past 7 years. It offers a thorough, mechanistic assessment of their cutting-edge uses in electrochemical devices, environmental remediation, and anti-corrosion surface technologies and links their biogenic origins with specific structural and morphological traits. In conclusion, this analysis creates a standardized framework to bridge the gap between reliable, high-performance industrial deployment and bench-scale green synthesis (Ashish *et al*., 2012).

a. Biogenic Sources of Silicon Nanoparticles

In contrast to traditional top-down chemical methods that rely on expensive ore mining and hazardous synthesis, the green synthesis paradigm's sustainability is essentially based on the selection of biogenic precursor materials. It extracts silicon and silica frameworks directly from biological matrices, which can be broadly classified into agricultural wastes, plant tissues, and microbial sources (Debnath *et al*., 2012).

b. Rice Husk

Rice husks possess an exceptionally high ash content (approaching 20 wt. %), of which more than 85-95% consists of pure amorphous silica. This structural silica functions as a protective mechanical barrier during the plant's life cycle and serves as an ideal nanoscale precursor post-harvest (Garg *et al*., 2012).

c. Sugarcane Bagasse Ash

Industrial burning of bagasse for bioenergy yields an ash by-product that is dense with silicates. Literature highlights sugarcane bagasse as one of the cheapest raw matrices for manipulating engineered silica at the nano-level (Han *et al.*, 2026).

d. Alternative Agro-Wastes

Recent literature has expanded the biogenic pool to include wheat husk, cornhusk, coconut husk, banana peels, and groundnut shells. Each biomass contains varying baseline concentrations of minerals, requiring custom processing (Jeelani *et al.*, 2020).

e. Plant Extracts

Direct extraction, from living plant tissues (aloe Vera, bamboo leaves or Bermuda grass), skips the preliminary combustion phases needed for agricultural ash. Plant extracts are rich in water-soluble phytochemicals such as organic acids, flavonoids and polyphenols. They bind to the surfaces of the nanoparticles and serve as natural capping ligands to prevent uncontrolled particle growth and agglomeration (Hwang *et al.*, 2021).

f. Microorganisms

Certain bacterial strains and fungi (such as *Aspergillus Niger* and *Fusarium oxysporum*) have a bio-catalytic ability to breakdown mineral silicates through internal or extracellular enzymes. Fungal bioleaching by secretion of organic acids (e.g. oxalic acid or citric acid) can be carried out at room temperature, but over a longer time than biomass processing, and enables extraction and subsequent precipitation of nanoparticles (Kristof *et al.*, 2026).

2.0 Extraction and Isolation

A multi-phase chemical and thermal refinery process is required to convert raw biogenic materials into customized, high-purity silicon nanoparticles. The extraction processes are developed to eliminate metal contaminants and lignocelluloses matrices while maintaining nanoscale morphology:

Raw biomass → Acid leaching → Calcination → Alkaline dissolution → Controlled precipitation → pure silicon nanoparticle.

Step 1: Pre-treatment and Acid leaching

To get rid of dust, dried material, and volatile surfaces, raw biomass is carefully cleaned with deionized water. Usually, 1-2 M HCl, HNO_3, or environmentally benign citric acid are used for acid leaching. Structural carbohydrates and dissolved alkali metal pollutants (such Ca^{2+} and Mg^{2+}) are hydrolysed during this leaching stage. It is crucial to remove these metal cations because, if they remain in the

matrix, they would function as fluxing agents during heat treatment, fusing the silica into the crystalline, inactive quartz structure (Mahawar *et al.*,2023).

Step 2: Calcination (ashing)

The leached, organic –stripped biomass is place in a muffle furnace for controlled thermal degradation. The choice of temperature is a crucial parameter in determining the final material structure.

Amorphous threshold (600 ^{0} C - 700^{0} C): Calcination within this window burns off remaining lignocellulose, yielding highly reactive, high –surface area amorphous silica ash.

Crystalline Fusion (800^{0}C): Exceeding these temperatures prompts a phase transition into crystalline cristoballite or tridymite silica, which exhibits poor chemical reactivity and is difficult to process downstream.

Step 3: Alkaline dissolution (sol-Gel Extraction)

To isolate the nanoparticles, the amorphous ash is reacted with a strong base – predominantly sodium hydroxide (NaOH) or potassium hydroxide (KOH) – at temperatures 80 ^{0} C and 100 ^{0} C under continuous stirring. This process dissolves the solid silica network to form a water –soluble sodium silicate ($NaSiO_3$) precursor solution:

$$SiO_2\,(S) + 2NaOH\,(aq) \longrightarrow Na_2SiO_3\,(aq) + H_2O\,(L)$$

Comparative studies indicates that NaOH generally achieves higher extraction yields and superior structural uniformity than KOH.

Step 4: Controlled Neutralization and Precipitation

After cooling the sodium trisilicate solution, a mineral or organic acid is added dropwise while stirring quickly to bring the pH down to a neutral or slightly acidic range (pH 7-8). An interconnected network of siloxane (Si-O-Si) is created by this titration, which sets off a regulated condensation process that result in the precipitation of pure nanosilica gel. Researchers can modify the average pore radius and surface area by varying factors such as aging time (6 hours is commonly regarded as optimal), stirring velocity, and surfactant additives.

Step 5: Post-processing and Reduction to Elemental Silicon

The resulting gel is dried after being carefully cleaned with deionized water to get rid of any remaining salts, such as NaCl. Advanced electrochemical and electrical applications need elemental silicon nanoparticles (Si NPs), however many green synthesis pipelines end at biogenic silica nanoparticles (SiO2 NPs). This is accomplished via a final metallothermic (magnesiothermic) reduction of the biogenic SiO2 in an inert atmosphere at lower temperature thresholds (6000 C):

$$SiO_2 + 2Mg \longrightarrow Si + 2MgO$$

The magnesium oxide by-product is subsequently dissolved via mild acid washing, leaving behind

highly porous, crystalline elemental SiNPs that preserve the biogenic architecture.

2.2 Characterization

Because biogenic extraction methods introduce variable phytochemical capping matrices, which depend on a suite of advanced analytical techniques to verify phase purity, morphology, surface chemistry, and the porous architecture, rigorous structural validation is necessary for the successful deployment of green-synthesized silicon nanoparticles in electrochemical, environmental remediation, and as an anticorrosion (Bai *et al*., 2023).

2.2.1 Crystallinity and Phase Purity:

a. X-Ray Diffraction (XRD)

X-ray Diffraction is the primary non- destructive technique used to evaluate the physical state- amorphous or crystalline – of biogenic nanoparticles.

Amorphous Validation: Silicon nanoparticles extracted directly from agricultural ash below 700 ^{0} C typically manifest as biogenic silica (SiO_2). Their XRD pattern s exhibit a characteristic, broad diffraction halo cantered between $2\Theta = 20^0$ and 24^0. This lacks sharp Bragg peaks, confirming the disordered, and short –range atomic arrangement typical of amorphous frameworks.

Crystalline Validation: Following successful magnesiothermic or aluminothermic reduction of biogenic silica into elemental silicon (Si), the amorphous profile disappears. It is replaced by highly distinct, sharp Bragg reflection at 2Θ = 28.4, 47.3, 56.1, 69.1, and 76.4. These correspond precisely to the (111), (220), (311), (400), and (331) lattice planes of face- centered cubic (FCC) crystalline silicon (JCPDS card no.27-1402).

Crystalline Sizing: The average crystalline size (D) is calculated using the Scherer Equation:

$$\mathbf{D} = \frac{K\lambda}{\beta \cos \theta}$$

Where K = is the shape factor (typically 0.9), λ is the x-ray wavelength (cu-Kα radiation, = 0.15406 nm), β is the full width at half maximum (FWHM) of the dominant peak, and Θ is the Bragg angle.

2.2.2 Morphology and Size Distribution: Electron Microscopy (TEM, SEM, HR-TEM)

Electron microscopy translates diffraction data into tangible physical dimensions, mapping particle clusters and interfacial boundaries

a. Scanning Electron Microscopy (SEM)

SEM imaging evaluates the overall surface topography and agglomeration states of the dry powders. Green –synthesized SiNPs often display a micro-clustered, cauliflower –like morphology due to the bridging effects of natural organic residues. Coupling SEM with (EDS)

b. Energy –Dispersive X-ray Spectroscopy (EDS)

EDS maps elemental purity, verifying high weight percentages of Si and O while checking for residual extraction contaminants (e.g. Na, Cl, K)

c. Transmission Electron Microscopy (TEM)

TEM captures the true internal boundaries and individual core diameters of isolated nanoparticles. While traditional chemical methods to produce uniform spheres, green synthesis yields highly diverse geometries –ranging from spherical and irregular polygon nodes to highly porous interconnected clusters, with diameters spinning 10 nm to 80 nm (Nsikak *et al*.,2023).

d. High Resolution TEM (HR-TEM)

HR-TEM resolves individual atomic planes. In elemental crystalline SiNPs, it measures a distinct d-spacing of 0.31 nm, which matches the distance between (111) lattice planes. It also visualizes thin, amorphous, biogenic organic shells (1-3 nm thick) surrounding the core particles. This proves the successful in- stu capping of plant- derived biomolecules (Owusu *et al*., 2023).

2.2.3 Surface Chemistry and Functional Group:

Characterizing the outer boundary layer is vital because functionalities directly dictate target performance.

a. Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy screens the vibrational modes of chemical bonds to identify both the silicon infrastructure and remaining plant -derived biomolecules

Siloxane Framework: Predominant peaks appear around 1080-1100 cm^{-1} (asymmetric stretching of Si-O-Si bond), 800 cm^{-1} (symmetric stretching of Si-O-Si), and 460 cm^{-1} (bending vibrations of Si-O-Si). These peaks confirm the structural construction of the silica backbone.

Silinol Interfaces: A broad band between 3200-3600 cm^{-1} corresponds to –OH stretching from silanol groups (Si-OH) and physically adsorbed water molecules.

Biogenic Fingerprints: Weak but critical band at 1630 cm^{-1} (C=O, carbonyl stretching from protein/flavonoids) and 2920 cm^{-1} (C-H stretching from aliphatic chains) confirm that biological capping agents are chemically or physically anchored to the SiNP surfaces (Vetchinkina *et al*., 2019)

b. X-ray Photoelectron Spectroscopy (XPS)

XPS evaluates the precise elemental compositions and valence states of the near –surface region (top 1-10 nm). The Si 2p core-level spectrum serves as a vital diagnostic tool for phase composition:

Peaks located at binding energy of approximately 99.5 e V indicate elemental, non-oxidized zero-valent silicon (Si^0)

Peaks at shifting toward higher binding energies around 103.5 e V confirm fully oxidized silicon (Si^{4+} in a SiO_2 environment).

Intermediate peaks signal sub-oxide (SiO_X), which routinely form a protective passivation layer over green crystalline nanoparticles (Tang *et al*., 2012)

2.2.4 Porosity and Surface Area Analysis: BET and BJH

Because anticorrosive matrices and environmental adsorbents rely heavenly on high surface contact, nitrogen adsorption- desorption isotherms are essential.

a. Brunauer-Emmett-Teller (BET)

Surface area: Green-synthesized SiNPs regularly display type (IV) isotherm with an H3 –type hysteresis loop, which is indicative of mesoporous material containing slit-shaped pores. While bulk sand possesses a negligible surface area, biogenic nanoparticles routinely register remarkably high BET surface areas ranging from 150 m^2/g to over 600 m^2/g. This offers an abundant array of active target anchoring sites (Wang *et al*., 2020)

b. Barrett-Joyner-Halenda (BJH) Pores Distribution

BJH modelling extracts pore volume metrics from desorption data. Studies reveal that green processing parameters can be tuned to yield uniform pore diameters between 2 nm and 10 nm. This structural trait is ideal for loading active corrosion inhibitors or trapping bulky heavy metal ions during water filtration (Vivar *et al*., 2018)

3.0 Applications

3.1 Electrochemical Application

The integration of green-synthesized silicon nanoparticle (SiNPs) into electrochemical systems addressed a critical limitation of modern material science: the need for high performance, cost effective and ecologically benign electrode matrices. Nanoscale silicon exhibits an exceptionally high theoretical specific capacity, making it a disruptive material in energy storage and electrochemical sensing (Kim *et al*., 2024).

3.1.1 High Capacity Anode for Lithium –ion Battery (LIBs)

Due to its remarkable theoretical specific capacity of over 4200 mAh/g (by the production of the Li_2Si_4 alloy), which is 10 times higher than commercial graphite anodes (372 mAh/g), silicon is widely acknowledged as the leading next-generation anode material for lithium-ion batteries. Conventional silicon anodes have significant mechanical deterioration despite their potential. Silicon undergoes a tremendous volumetric expansion of more than 300% during lithiation (charging). This severe swelling

causes rapid capacity fading in dense, chemically produced silicon by inducing mechanical pulverization, electrical separation from the current collector, and constant rupture and reformation of the solid electrolyte interphase (SEI) layer (Liu *et al*.,2024). The solution to this problem is biogenic solution, literature demonstrates that the unique architecture of green synthesized SiNPs inherently mitigate this expansion mechanism;

a. Internal void Utilization: Mesoporous SiNPs derived from agricultural wastes possess an internal network of interconnected pores (2-10 nm) and hollow core geometries. These internal voids act as mechanical buffer zones, allowing the silicon to expand inward rather than outward, preserving electrode structural integrity.

b. Carbon –Silicon Nanocomposites

Many green synthesis pathways yield an in-stu carbonaceous matrix derived from residual biomass carbonization. This integrated carbon shell enhances the baseline electronic conductivity of the silicon core while serving as secondary mechanical constraint against outward particles expansion (Bagherzadeh *et al*., 2015).

c. Electrochemical Performance Metric's

Optimization of these biogenic frameworks yield anodes capable of maintaining a stable specific capacity of greater than 1200 mAh/g after 500ncharge –discharge cycles, showcasing superior capacity retention compared to bulk silicon synthetic alternative (Garg *et al*.,2012).

3.1.2 Supercapacitors:

The high surface area and structured mesoporous networks facilitate quick ion transport and efficient electrostatic charge separation at the electrode-electrolyte boundary, boosting overall power density.

3.1.3 **Electrochemical Sensors**

Surface –modified biogenic biosilica SiNPs accelerate electron transfer rate when layered on working electrodes. These properties make them sensitive platform for detecting trace biomolecules, heavy metals or nitroaromatic explosives.

3.2 Environmental Remediation

According to Siaw M., *et al.(*2024). Green-synthesized silicon nanoparticles (SiNPs) have emerged as a destructive, eco-friendly alternative for environmental remediation. Their high specific surface area,

tuneable porosity, and reactive surface functional groups allows them neutralized pollutants via two primary pathways: efficient adsorption and advanced photocatalytic degradation.

3.2.1 Heavy Metal Adsorption and Sequestration

Biogenic SiNPs function as high –capacity nano-adsorbents capable of trapping highly toxic heavy metal ions –such as lead (Pb^{2+}), cadmium (Cd^{2+}), mercury (Hg^{2+}), and hexavalent chromium (Cr^{6+}) from contaminated water systems (Badr *et al.*, 2019).

3.2.2 Mechanism of Adsorption

The binding efficiency of green SiNPs is fundamentally governed by their surface chemistry and structural porosity (Adach *et al.*, 2021).

3.2.3 Electrostatic attraction and complexation:

FTIR characterization reveals that biogenic SiNPs are densely populated with surface silanol (Si-OH) groups and residual plant-derived carboxyl (COOH) and hydroxyl (OH) functionalities. At the surrounding pH, these functional groups deprotonate, conferring a strong negative surface charge that electrostatically attracts and coordinates with positively charged heavy metal cations (Nayl *et al.*, 2022).

3.2.4 Ion Exchange:

Cation temporarily trapped within the biogenic silica matrix during synthesis can undergo localized ion exchange with heavy metal pollutants in the effluent (Nsikak *et al.*, 2023).

3.3. Anticorrosion

Metallic corrosion represents a severe industrial challenge, causing catastrophic structural degradation and imposing a massive global economic burden. Traditional protective strategies rely heavenly on chemical conversion coatings containing hexavalent chromium [Cr (v1)], or synthetic polymer primers filled with toxic zinc-based compounds. Because environmental mandates strictly restrict these hazardous substances, green –synthesis silicon nanoparticles have emerged as highly attractive, eco-friendly alternative. Biogenic silicon nanoparticles protect metallic substrates through two main strategies: acting as dense physical barrier fillers within organic coatings, and serving as smart Nano-carriers for self –healing anticorrosion system (Rahman *et al.*, 2022).

3.3.1 Core Protection Mechanisms

a- The Barrier Effect: Nanoparticles physically fill micro-void and cracks within polymer paints (like epoxy).

b- The Tortuous Path: They create a twisted maze that delays water, oxygen, and chloride ions from reaching the metal substrate.

c- Smart Self-Healing: Mesoporous SiNPs acts as tiny nanocarriers that safely encapsulate eco-friendly organic inhibitors

d- Stimuli Release: Localized corrosion changes the surrounding p H, triggering the nanoparticles to autonomously release inhibitors and heal scathes.

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