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Green-Synthesized Nanoparticles from Coconut Husk Ash Extract with Potent Antioxidant Activity

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Abstract

The valorization of agricultural waste into functional nanomaterials represents a sustainable approach to nanotechnology. In this study, coconut husk ash extract was utilized as a green precursor for the synthesis of mesoporous silica nanoparticles (MSNs) via a sol-gel method using cetyltrimethylammonium bromide (CTAB) as a structure-directing agent. The nanoparticles were characterized for particle size, polydispersity index (PDI), zeta potential, surface morphology, and FTIR spectra. Results indicated particle sizes ranging from 247 to 722 nm with PDIs between 0.118 and 0.168, confirming acceptable homogeneity. Zeta potential values ranged from -18.9 to -22.2 mV, consistent with silanol group deprotonation. SEM analysis revealed spherical morphology with smooth surfaces, validating successful condensation and template removal. Antioxidant activity was evaluated using the DPPH radical scavenging assay, with the optimized formulation (SP4) showing dose-dependent inhibition and an IC_{50} value of 33.18 µg/mL. These findings highlight the potential of coconut husk ash-derived MSNs as eco-friendly nanocarriers with significant antioxidant activity, offering promise for biomedical and nutraceutical applications.

Keywords: Coconut husk ash; Green synthesis; Mesoporous silica nanoparticles; Antioxidant activity; DPPH assay

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Introduction

The increasing demand for sustainable nanotechnology has driven interest in agricultural waste valorization as a source of functional biomaterials. Coconut husk, a lignocellulosic by-product of *Cocos nucifera*, is abundantly available and rich in silica, lignin, and polyphenolic compounds, making it a promising precursor for green nanomaterial synthesis[1]. Traditional silica sources such as tetraethyl orthosilicate (TEOS) are expensive and environmentally taxing, whereas bioderived silica from coconut husk ash provides a cost-effective and eco-friendly alternative aligned with green chemistry principles[2].

Mesoporous silica nanoparticles (MSNs) synthesized via sol-gel methods using surfactants like cetyltrimethylammonium bromide (CTAB) exhibit high surface area, tunable pore size, and excellent biocompatibility[3]. Incorporating coconut husk ash extract as a silica precursor not only reduces synthesis cost but also integrates phytoconstituents that may impart antioxidant activity[4]. Antioxidants are crucial in mitigating oxidative stress, which is implicated in the pathogenesis of chronic diseases such as cancer, diabetes, cardiovascular disorders, and neurodegeneration[5].

Green-synthesized nanoparticles derived from plant extracts have demonstrated significant radical scavenging activity, cytotoxicity against cancer cells, and antimicrobial potential[6]. Coconut husk ash extract, in particular, contains phenolic and flavonoid compounds that contribute to free radical neutralization, enhancing the biomedical relevance of the resulting nanoparticles[7]. The DPPH assay, a widely accepted method for evaluating antioxidant capacity, measures the hydrogen-donating ability of nanoparticles to neutralize free radicals, thereby providing a quantitative assessment of their antioxidant potential[8].

Thus, the formulation and characterization of MSNs using coconut husk ash extract, followed by antioxidant evaluation, represent a novel approach to developing biogenic nanocarrier systems with therapeutic applications. This study aims to synthesize MSNs from coconut husk ash extract via sol-gel method, characterize their physicochemical properties, and assess their antioxidant activity using the DPPH radical scavenging assay.

Material and Methods

Materials

Coconut husk was procured from local coconut vendors. The chemicals and reagents like sodium hydroxide, petroleum ether, hydrochloric acid were procured from CDH, Rankem, Finar Chemicals and Loba Chemie and were of analytical grade.

Preparation of coconut husk for ash extraction

Coconut husk was collected, dried and cleaned before further processing. The dried coconut husk was reduced to smaller particles and then powdered using a slow speed blender. The coarse powdered material was then sieved to obtain a uniform material for ash extraction (Figure 1).

Figure 1. Coconut husk and its powder

Preparation of coconut husk ash

The powdered coconut husk was accurately weighed and placed in a tared fused silica crucible. The crucible was placed in muffle furnace and the temperature was gradually increased to 500 °C to obtain ash of the husk powder. The ash was then cooled to room temperature and treated with 50 mL of 0.1N HCl. The mixture was then heated to 50°C to ensure complete removal of acid soluble impurities. The ash-HCl mixture was then filtered and the residue was treated with 20 mL of 1N NaOH. The solution was heated at 80°C for 1h to dissolve the silica in the husk extract and filtered. The yellowish-filtrate was stored as coconut husk ash alkaline extract for further study[9].

Formulation of Mesoporous Silica Nanoparticles

Mesoporous silica nanoparticles were prepared by sol-gel method. Briefly, varying molar concentrations of CTAB and coconut husk ash alkaline extract were used while molar ratio of ethanol, water, and ammonium hydroxide was kept constant[10]. The composition is shown in Table 1. Initially ethanol, water and ammonia solution (NH_4OH) were mixed together to form silica sol. Then CTAB was added with constant stirring for 15 min. Finally, coconut husk ash alkaline extract was added drop by drop under continuous stirring for 2 h at room temperature. The start of the reaction was confirmed by immediate formation of opaque solution. The white powder precipitated was filtered and washed with deionized water. The particles were calcined at 550°C for 3 h to remove the surfactant[11].

Table 1. Composition of MSNs

Code	Coconut husk extract (mL)	CTAB (g)	Ammonium hydroxide (mL)	Ethanol (mL)	Water (mL)
SP1	1.5	0.1	1	80	20
SP2	1.5	0.2	1	80	20
SP3	1	0.1	1	80	20
SP4	1	0.2	1	80	20
SP5	0.5	0.1	1	80	20
SP6	0.5	0.2	1	80	20

Evaluation of MSNs

Determination of particle size and zeta potential

The particle size and zeta potential of each formulation batch was determined using a laser diffraction particle size analyzer[12].

Surface morphology

The surface morphology of the prepared nanoparticles was studied using scanning electron microscopy by gold coating of the lyophilized nanoparticle[13].

FTIR Spectra of MSNs

The FTIR Spectra of the prepared MSNs was obtained using FTIR spectrophotometer and the spectra was observed for the presence of various stretching and bending vibrations[14].

Antioxidant evaluation

The antioxidant action of the prepared MSNs (best formulation) was determined using 2,2-Diphenyl-1-picrylhydrazyl (DPPH) radical scavenging assay[15,16]. The free radical scavenging activity of the MSNs was measured in terms of hydrogen donating or radical scavenging ability using the stable radical DPPH. The test samples (100 μL, 100-500 µg/mL) were prepared in DMSO and were mixed with 1.0 mL of DPPH solution and filled up with methanol to a final volume of 4 mL. Absorbance of the resulting solution was measured at 517 nm in a visible spectrophotometer. Ascorbic acid was used as the reference compound. Lower absorbance of the reaction mixture indicated higher free radical scavenging activity. Radical scavenging activity was expressed as the inhibition percentage of free radical by the sample and was calculated using the following formula:

$$\% \, inhibition = \frac{(Ao - At)}{Ao} \, X \, 100$$

where Ao is the absorbance of the control (blank, without sample) and At is the absorbance in the presence of the test samples. All tests were performed in triplicate and the results were expressed as mean values ± standard deviations.

Results and Discussion

Preparation of husk ash

The determination of ash content from coconut husk powder provides valuable insight into its inorganic composition and potential applications. At 500 °C, the cellulose, hemicellulose, and lignin components of the husk undergo oxidative degradation, leaving behind a stable mineral residue. This ash typically contains elements such as potassium, calcium, magnesium, sodium, iron, and silica, which are characteristic of lignocellulosic biomass[17-19]. The ash was collected for various batches and yield was calculated. The yield ash was found to be 8.83 ± 0.392 %w/w (0.1007 ± 0.005 g). The ashes were combined and processed further to extract silica from the ash. In a previous study, similar ash percent was recovered from dried coconut husk[20].

FTIR Spectral study of ash

The coconut husk ash was studied by infrared spectrophotometry to identify the major functional groups present in the chemical composition of the ash. The ash revealed peaks due to O-H stretching vibrations of surface silanol (Si-OH) groups and adsorbed water molecules (3544-3394 cm^{-1}), C-H stretching vibrations from organic fragments (2921 and 2852 cm^{-1}), asymmetric stretching vibrations of Si-O-Si bonds (1099-1020 cm^{-1}) and symmetric Si-O-Si stretching and bending vibrations (868, 760, and 713 cm^{-1}) (Figure 2).

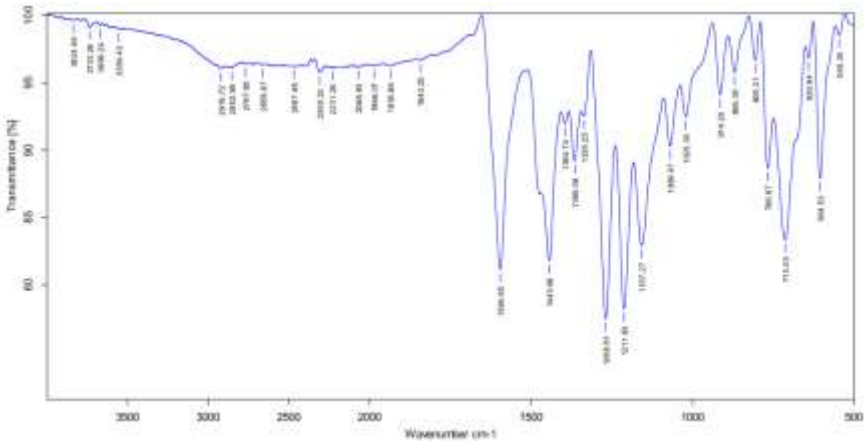

Figure 2. FTIR Spectrum of coconut husk ash

Extraction of silica from husk ash

The preparation of coconut husk ash alkaline extract through sequential acid-alkali treatment represents a classical approach for isolating silica and other alkali-soluble constituents from lignocellulosic biomass. Following incineration, the ash was cooled and treated with dilute hydrochloric acid (0.1 N HCl), to remove acid-soluble impurities such as carbonates, oxides, and trace metallic salts. Heating at 50 °C enhanced solubilization kinetics, ensuring complete removal of these contaminants.

Subsequent alkaline treatment with sodium hydroxide (1 N NaOH) at 80 °C for 1 h facilitated dissolution of silica. Under alkaline conditions, silica reacts to form soluble sodium silicate. The yellowish coloration of the filtrate may be attributed to trace transition metal ions or residual organic chromophores carried over from the husk matrix[21]. The filtrate, designated as the coconut husk ash alkaline extract, thus represents a silica-rich solution suitable for downstream applications.

Preparation of MSNs

Traditionally, TEOS has been the dominant silica source in sol-gel synthesis; however, integrating bio-derived silica not only reduces cost but also aligns with green chemistry principles. Coconut husk ash is rich in silica, and its alkaline extract (sodium silicate solution) provides a reactive precursor that undergoes hydrolysis and condensation under alkaline conditions. CTAB (cetyltrimethylammonium bromide) acts as a structure-directing agent, forming micelles that serve as templates for mesopore formation. The ethanol-water-ammonia mixture established the alkaline environment necessary for hydrolysis and condensation. Dropwise addition of the coconut husk ash alkaline extract ensured controlled nucleation, while constant stirring facilitated uniform particle growth. The immediate formation of an opaque solution confirmed sol-gel initiation. Calcination at 550 °C for 3 h successfully removed CTAB, leaving behind ordered mesoporous silica[22,23]. Variation in CTAB concentration and precursor (coconut husk ash alkaline solution) volume provides a means to fine-tune particle morphology and mesostructure.

FTIR spectral study of MSNs

The FTIR profile confirms the successful synthesis of mesoporous silica nanoparticles using coconut husk ash extract as the silica source. The dominant Si-O-Si and Si-OH bands validate the formation of a silica network, while the weak organic peaks indicate efficient removal of CTAB during calcination. The absence of strong C-H or N-H bands suggests high purity of the final product (Figure 3).

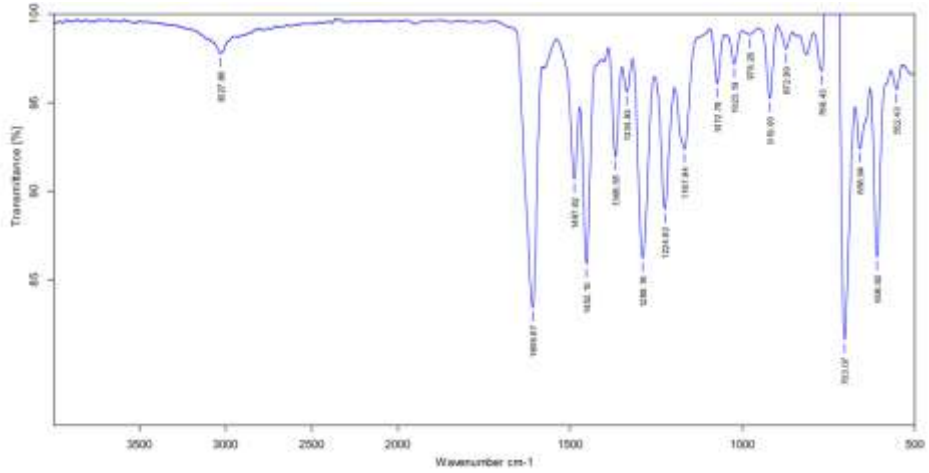

Figure 3. FTIR Spectrum of MSN

Particle size and PDI

The particle size and polydispersity index (PDI) data for formulations SP1-SP6 provide critical insights into how CTAB concentration and coconut husk ash extract volume influence the morphology of mesoporous silica nanoparticles (MSNs) synthesized via the sol-gel method. The particle size of the MSNs ranged from 722 to 247 nm with a PDI ranging from 0.168 to 0.118 (Table 2).

Table 2. Particle size and PDI of MSNs

Code	Particle Size (nm)	PDI
SP1	529	0.151
SP2	393	0.118
SP3	358	0.143
SP4	247	0.127
SP5	722	0.168
SP6	456	0.149

Effect of CTAB Concentration

CTAB acts as a surfactant and structure-directing agent, forming micelles that template mesopores. A lower CTAB (0.1 g; SP1, SP3, SP5) led to formation of larger particle sizes (529–722 nm) with higher PDIs (0.151–0.168), indicating less uniformity whereas a higher CTAB (0.2 g; SP2, SP4, SP6) resulted in smaller particle sizes (247–456 nm) with lower PDIs (0.118–0.149), reflecting improved homogeneity. This trend is consistent with previous reports where increased surfactant concentration enhances micelle density, providing more nucleation sites and restricting particle growth, thereby yielding smaller, more uniform nanoparticles.

Effect of Coconut Husk Extract Volume

The extract serves as the silica precursor. It was found that High extract (1.5 mL; SP1–SP2) resulted in larger particles (393–529 nm), likely due to increased silica availability leading to faster condensation. On the other hand, intermediate extract (1 mL; SP3–SP4) led to a balanced

precursor–surfactant ratio, producing smaller particles (247–358 nm) with good uniformity whereas low extract (0.5 mL; SP5–SP6) resulted in very large particles (456–722 nm), possibly due to insufficient silica for stable micelle templating, resulting in irregular growth. SP4 (1 mL extract, 0.2 g CTAB) yielded the smallest, most uniform nanoparticles (247 nm, PDI 0.127), suggesting this composition provides optimal balance between precursor concentration and surfactant templating.

Polydispersity Index (PDI)

All formulations exhibited PDIs between 0.118-0.168, which are within acceptable ranges for nanoparticle systems. SP2 (0.118) and SP4 (0.127) were highly monodisperse, indicating stable and uniform particle populations. SP5 (0.168) was found to be the most polydisperse, reflecting heterogeneous particle growth under low precursor and surfactant conditions. Values below 0.2 confirm that the synthesized MSNs are sufficiently homogeneous for biomedical and catalytic applications.

Zeta potential

The zeta potential values of formulations SP1-SP6 provide important information about the surface charge, colloidal stability, and interaction potential of the synthesized mesoporous silica nanoparticles prepared using coconut husk ash extract. The zeta potential of the produced MSNs was found to be in the range of -18.9 to -22.2 mV for the various formulations (Table 3).

Table 3. Zeta potential of MSNs

Code	Zeta Potential (mV)
SP1	-18.9
SP2	-22.1
SP3	-19.7
SP4	-20.4
SP5	-19.9
SP6	-20.1

All formulations exhibit negative zeta potential values**,** consistent with the presence of silanol (Si–OH) groups on the silica surface. The negative charge arises from deprotonation of silanol groups in alkaline medium, a typical feature of silica nanoparticles. Increasing CTAB concentration slightly enhances the negative zeta potential. This may be due to improved micelle templating and more uniform silica condensation, leading to higher surface exposure of silanol groups. Variations in extract volume did not drastically alter zeta potential, with values remaining within a narrow range. This indicates that precursor concentration influences particle size more strongly than surface charge.

Surface Morphology

The scanning electron microscope (SEM) image provides a detailed visualization of the surface morphology and particle size distribution of mesoporous silica nanoparticles (MSNs) synthesized using coconut husk ash extract. The magnification (×8,000) and accelerating voltage (15 kV) reveal spherical particles with smooth surfaces and varying diameters in the nanometer range (Figure 4). The particles appear predominantly spherical to slightly oval, indicating uniform nucleation during

the sol-gel process. The smooth surface texture suggests successful condensation of silica and removal of organic templates (CTAB) after calcination. The absence of visible agglomerates implies good dispersion and moderate colloidal stability, consistent with the zeta potential values. Measured diameters range from approximately 300 nm to 825 nm, with most particles falling between 325 nm and 600 nm. The observed morphology is consistent with literature reports for CTAB-templated silica nanoparticles synthesized via sol-gel methods. The use of coconut husk ash extract as a silica source yields comparable particle uniformity to conventional TEOS-based MSNs, validating its potential as a sustainable precursor.

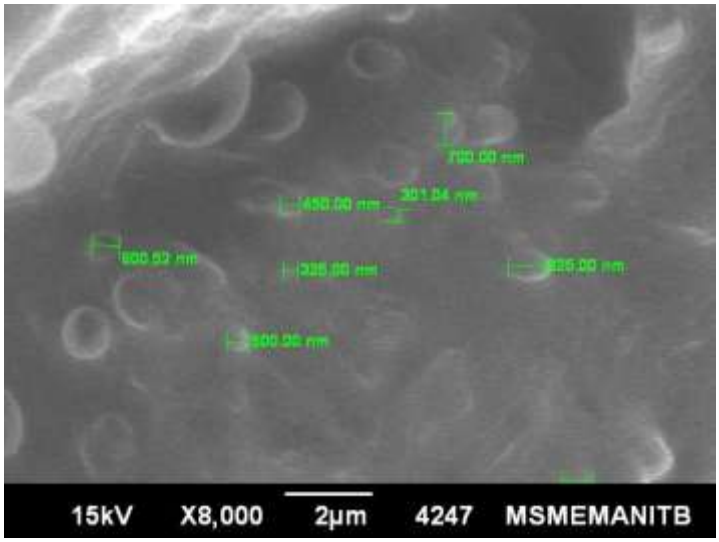

Figure 4. SEM pictograph of SP4

Antioxidant action of MSN

The DPPH assay results provide a quantitative measure of the antioxidant potential of the MSN (SP4), expressed as the percentage inhibition of the DPPH radical at varying concentrations (Table 4).

Table 4. Antioxidant activity data of SP4

Concentration (µg/mL)	DPPH inhibition (%)*
10	35.22 ± 0.5029
20	41.57 ± 0.8557
30	47.15 ± 0.3759
40	53.81 ± 0.1356
50	61.83 ± 0.2387
Ascorbic Acid (10 µg/mL)	85.43 ± 0.3536

*mean ± SD, n=3

The MSN produced using coconut husk ash extract exhibits a clear dose-dependent increase in DPPH radical inhibition. Ascorbic acid, a well-known antioxidant, showed 85.43% inhibition at 10 µg/mL, significantly higher than the extract. The DPPH radical scavenging mechanism involves electron transfer from antioxidant molecules to the DPPH radical, converting it from a deep violet

color to pale yellow. The gradual increase in inhibition with concentration reflects the cumulative effect of phenolic and flavonoid compounds derived from coconut husk ash extract. The alkaline extraction process may have preserved certain polyphenolic structures responsible for radical neutralization. The IC_{50} value (concentration required for 50% inhibition) can be calculated to be 33.18 μg/mL, indicating reasonable antioxidant strength for a bio-derived MSN.

Conclusion

This work demonstrates the successful green synthesis of mesoporous silica nanoparticles using coconut husk ash extract as a sustainable silica source. The sol-gel method, optimized with varying CTAB concentrations and precursor volumes, produced nanoparticles with favorable physicochemical properties, including nanoscale size, acceptable polydispersity, and stable negative zeta potential. The optimized formulation (SP4) exhibited strong antioxidant activity in a dose-dependent manner, with an IC_{50} value of 33.18 μg/mL, confirming its radical scavenging potential. The integration of bioderived silica not only reduces reliance on conventional precursors but also aligns with green chemistry principles, enhancing the biomedical relevance of the nanoparticles. Overall, coconut husk ash-based MSNs represent a cost-effective, eco-friendly, and functional nanocarrier system with potential applications in antioxidant therapy, drug delivery, and nutraceutical formulations. Future studies should focus on in vivo validation, biocompatibility assessment, and targeted delivery strategies to translate these findings into clinical and industrial applications.

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