

GREEN SYNTHESIS OF CTAB-TEMPLATED MESOPOROUS SILICA FROM SNAKE FRUIT PEEL FOR EFFICIENT REMOVAL OF Fe AND Zn FROM LANDFILL LEACHATE

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ABSTRACT

Snake fruit (*Salacca zalacca*) peel, an agro-industrial residue, was processed into mesoporous silica (MS) by sequential acid-alkali extraction followed by CTAB-assisted sol-gel synthesis. The product was tested as an adsorbent for Fe and Zn removal from actual landfill leachate. Acid-alkali extraction gave high-purity silica (~99.9 wt%) with a yield of ~60%. CTAB templating markedly changed the textural properties: the BET surface area increased from ~3.3 m²/g for non-templated silica to as high as ~896 m²/g for MSB, while the templated samples showed mean pore diameters of 3.9–8.8 nm and pore volumes up to ~0.96 cm³/g. XRD indicated an amorphous mesostructure, and FTIR showed the expected Si–O–Si band near 1095 cm⁻¹ and the Si–OH band at 3425–3441 cm⁻¹. In leachate treatment, MS removed 79% of Fe and 87.5% of Zn. The results show that snake fruit peel can be converted into a high-surface-area silica adsorbent for metal-bearing leachate, linking agricultural-waste valorization with SDG 6 (Clean Water and Sanitation).

Keywords: Heavy Metals, Landfill Leachate, Mesoporous Silica, Snake Fruit Peel, Water Treatment.

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INTRODUCTION

Heavy metals such as lead, cadmium, and zinc pose significant environmental and health risks due to their toxicity and bioaccumulation potential. Treatment methods that are effective but still affordable are therefore needed. Mesoporous silica synthesized from agricultural waste, including snake fruit (*Salacca zalacca*) peel (SFP), is one possible green-chemistry route. Through sol-gel templating, organic surfactants generate porous silica structures with enhanced adsorption properties.¹ The approach also gives practical value to a residue that is usually discarded.² Snake fruit peel contains silica precursors suitable for producing mesoporous silica, enabling waste-to-resource conversion and reducing environmental burdens from agricultural residues.^{3,4,5} Mesoporous silica is attractive for metal adsorption due to its high surface area, tunable pore size, and abundant silanol groups. Surfactant-templated approaches, particularly using CTAB, allow controlled pore formation through micellar structuring.⁶⁻⁹ Variations in synthesis parameters influence pore ordering, accessibility, and ion transport, directly affecting adsorption performance.^{9,10} Recent biomass- and biosorbent-based studies also report selective removal of heavy metals from aqueous, seawater, and wastewater systems, which supports the continued investigation of waste-derived adsorbents.¹¹⁻¹⁴ This type of adsorbent is relevant to landfill leachate, which often contains elevated levels of Cu, Zn, Fe, and Pb along with organic contaminants.^{15,16} In Indonesia, increasing waste generation highlights the need for affordable and effective adsorbents capable of functioning in complex wastewater matrices. Despite numerous studies on mesoporous silica synthesized from agricultural by-products, the utilization of snake fruit peel as a precursor for CTAB-templated silica and its application in the treatment of actual landfill leachate have received relatively little attention. The gap is also relevant to Sustainable Development Goal 6 (Clean Water and Sanitation), because landfill leachate remains a direct pressure on water quality in waste-impacted areas. To address this gap, we extracted silica from snake fruit peel via acid-alkali treatment and synthesized mesoporous silica through CTAB-templated sol-gel processing.¹⁷ Different CTAB concentrations were employed to adjust textural properties, which were then correlated with heavy-metal removal performance in real

landfill leachate. The work therefore examines both the preparation route and the performance of the resulting biomass-derived silica in a real wastewater matrix.

EXPERIMENTAL

Materials

Snake fruit peels were collected from a local agro-processing outlet, thoroughly washed with deionized water, and air-dried. The chemicals used in this study included hydrochloric acid (HCl, analytical grade), sodium hydroxide (NaOH, analytical grade), cetyltrimethylammonium bromide (CTAB, $\geq 99\%$), nitric acid (HNO₃, trace-metal grade), and silver nitrate (AgNO₃, analytical grade). Deionized water was employed throughout all experiments. Samples of real landfill leachate were collected from the Piyungan Landfill, Yogyakarta, Indonesia, using acid-cleaned sampling bottles and subsequently filtered through 0.45 μm membrane filters to obtain dissolved-metal fractions for further analysis.

Synthesis of Mesoporous Silica

The snake fruit peels were dried, cut, and calcined at 500 °C to obtain ash, which was then acid-activated using 6 M HCl and subsequently subjected to alkaline extraction with 6 M NaOH to produce sodium silicate. Acid titration of the silicate solution yielded a silica gel that was aged, thoroughly washed to remove residual ions, and dried to form a silica xerogel. Mesoporous silica was synthesized through CTAB-templated sol-gel processing using CTAB concentrations of 0.01, 0.05, and 0.10 M (MSA, MSB, and MSC, respectively). The silica sol was added to CTAB solutions at 40 °C, aged for 24 h, filtered, washed, and dried. Template removal was performed by calcination at 550 °C, after which the powders were cooled, ground, and stored for use. All steps involving corrosive chemicals adhered to institutional safety standards, and waste solutions containing metals or acids were managed according to hazardous-waste regulations.

Characterization of Mesoporous Silica

Comprehensive physicochemical characterization of the mesoporous silica samples was performed using multiple analytical techniques. XRF was used to determine elemental composition and SiO₂ purity. FTIR (4000–400 cm⁻¹) identified Si–O–Si and Si–OH functional groups and confirmed CTAB removal through the disappearance of C–H stretching bands. XRD with Cu K α radiation was conducted using small-angle scans (0.7–5° 2 θ) to assess mesostructural ordering and wide-angle scans (5–80° 2 θ) to evaluate amorphous characteristics. SEM provided particle morphology, while EDX offered qualitative elemental mapping before and after adsorption. TEM imaging confirmed pore-channel structures and mesopore arrangement. Nitrogen adsorption-desorption at 77 K (after degassing at 120 °C) was used to determine BET surface area, BJH pore-size distribution, and total pore volume.¹⁸ These parameters, along with XRD-derived crystallinity, were used to evaluate the structural and textural properties of the synthesized materials.

Adsorption of Heavy Metals in Landfill Leachate

Fe and Zn removal was evaluated by measuring metal concentrations in landfill leachate before and after contact with mesoporous silica (MS) using AAS. SEM-EDX mapping was then used to check whether the metals were retained on the MS surface. The acid activation, alkaline extraction, and gelation steps were selected to convert peel ash into soluble silicate with minimal residual impurities, while the CTAB concentration was varied to tune micelle formation and mesopore development. XRF, XRD, FTIR, SEM-EDX, TEM, and N₂ physisorption were used together to relate composition, mesostructure, and texture to adsorption behavior. BET surface area and BJH pore-size data were used to interpret Fe and Zn uptake, and SEM-EDX provided supporting evidence for metal adsorption on the silica surface.

RESULTS AND DISCUSSION

Mesoporous Silica

As shown in Fig.-1, FTIR analysis of ash-derived silica shows the characteristic vibrational bands of siloxane and silanol groups, including a strong asymmetric Si–O–Si stretching band at $\sim 1095\text{ cm}^{-1}$, a broad Si–OH stretching band at $3425\text{--}3441\text{ cm}^{-1}$, and an H–OH bending band near 1635 cm^{-1} associated with adsorbed water. In CTAB-templated mesoporous silica (MS), spectra prior to calcination exhibit an

additional absorption band at $\sim 1560\text{ cm}^{-1}$, assigned to residual CTAB-related alkyl-chain and ammonium-associated vibrations, which disappears after calcination, indicating that most of the template was removed. The low-frequency band around 470 cm^{-1} corresponds to Si–O–Si bending, while the $450\text{--}1300\text{ cm}^{-1}$ region represents the typical silica fingerprint dominated by Si–O–Si vibrations.¹⁹ The remaining silanol-related –OH bands and the retained Si–O–Si network after calcination support formation of the silica framework from snake fruit peel.^{20,21,22}

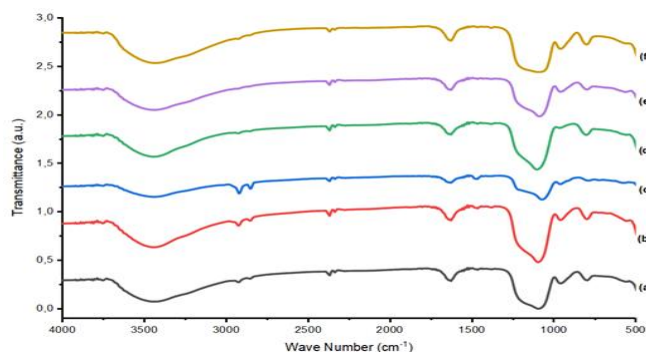


Fig.-1: FTIR Spectra of MS Synthesized from SFP: before Calcination (a) MSA, (b) MSB, (c) MSC; and after Calcination (d) MSA, (e) MSB, (f) MSC

Gas Sorption Isotherm and Pore Size

Gas sorption analysis shows a clear textural difference between non-templated silica and CTAB-templated mesoporous silica (MS), as summarized in Table-1. The non-templated silica exhibits a low specific surface area, small pore volume, and a comparatively large mean pore diameter, whereas all three CTAB-templated samples (MSA, MSB, and MSC) have higher surface areas and pore volumes, with smaller mean pore diameters, consistent with the formation of a mesoporous structure.^{23,24,25} Among the templated samples, the textural properties do not change monotonically with CTAB concentration: MSB exhibits the highest surface area and pore volume, followed by MSC and then MSA.

Table-1: Textural Properties of Non-Templated Silica and CTAB-Templated Mesoporous Silica (MSA, MSB, MSC)

Sample	BET Surface Area (m^2/g)	Pore Volume (cm^3/g)	Mean Pore Diameter (nm)
Non-templated silica	3.3	0.012	15
MSA	60.4	0.13	8.8
MSB	896	0.96	4.3
MSC	436	0.42	3.9

Nitrogen adsorption–desorption isotherms of MS (Fig.-2A) display a typical type IV profile with a pronounced hysteresis loop, indicating capillary condensation in mesopores, while the non-templated silica exhibits an isotherm closer to type I(b), reflecting limited pore development. Pore-size distribution analysis (Fig.-2B and 2C) further shows that MS possesses a broader distribution dominated by pores in the 1–10 nm range, with additional contributions at larger diameters, whereas non-templated silica presents a narrower distribution with lower surface area.

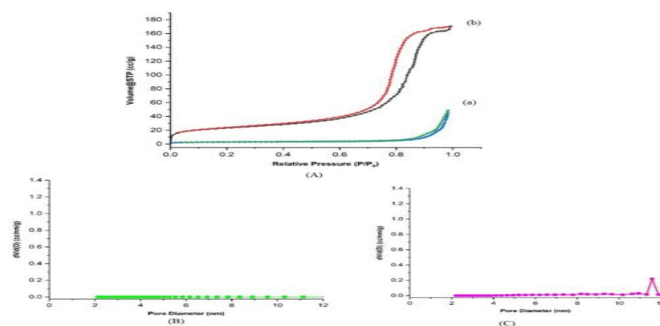


Fig.-2: (A) N_2 Adsorption–Desorption Isotherms of (a) Non-Templated Silica and (b) CTAB-Templated Mesoporous Silica (MS); (B, C) Corresponding Barrett–Joyner–Halenda (BJH) Pore-Size Distributions

These results, consistent with the textural parameters in Table-1, confirm that CTAB templating effectively promotes pore formation, connectivity, and enhanced textural properties suitable for adsorption applications.^{22,23}

Crystal Phase of MS

XRD analysis results (Fig.-3) indicate that both silica and mesoporous silica (MS) are predominantly amorphous, as evidenced by broad diffraction halos at wide angles and the absence of sharp Bragg reflections, reflecting a lack of long-range crystallinity typical of mesoporous silica materials. The diffraction profiles of the synthesized MS closely resemble those of commercial silica, which exhibits weak low-angle features around $2\theta \approx 9.1^\circ$ and 9.72° associated with amorphous or poorly ordered silica domains. Thus, the synthesized MS retains an amorphous mesoporous silica phase, with limited short-range order and without detectable crystallization.²¹

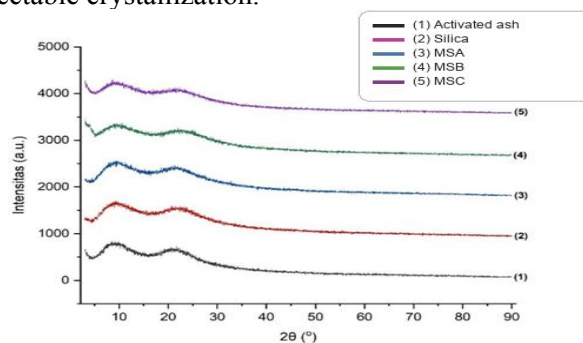


Fig.-3: XRD Diffractograms of Silica and Mesoporous Silica (MS) Samples, Showing Broad, Low-Intensity Halos Characteristic of an Amorphous Mesostructure

Morphology of Mesoporous Silica

TEM images (Fig.-4) show the morphological change from snake fruit peel (SFP) to peel ash, silica, and mesoporous silica (MS). Activated ash has more visible pores than raw SFP, and conversion to silica produces a more open framework. In the MS samples, the pores appear as irregular wormhole-like channels, consistent with CTAB acting as the structure-directing agent. At higher CTAB concentration, some pore blocking and local densification are observed, which may limit access to part of the mesopore network.

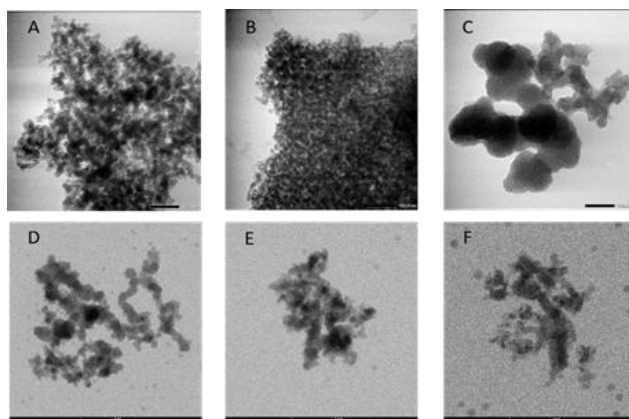


Fig.-4: TEM Images Showing Morphology of (A) SFP, (B) Activated Ash, (C) Silica, and (D–F) MSA, MSB, and MSC, respectively

Electron microscopy also shows the morphological change from biomass-derived precursors to silica and mesoporous silica (MS). SEM observations (Fig.-5) show that silica forms near-spherical aggregates composed of relatively uniform sub-particles, whereas MS exhibits coral-like aggregates with rougher surface textures. Increasing CTAB concentration from MSA to MSC enhances localized agglomeration, attributed to variations in micelle distribution, sol–gel interactions, and mixing hydrodynamics.⁴ TEM observations are consistent with the gradual development of porosity from raw biomass to MS, with

relatively uniform pore networks at low CTAB concentrations and more pronounced clustering and domain formation at higher template loadings. Similar template-driven aggregation has been reported for mesostructured silica materials.^{26,27}

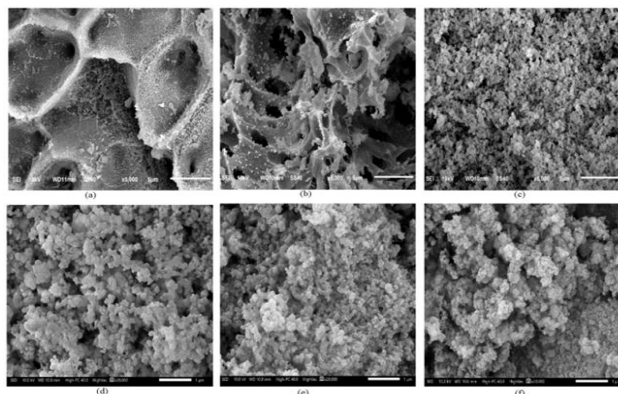


Fig.-5: SEM Images Showing Morphology of (a) SFP, (b) Activated Ash, (c) Silica, (d) MSA, (e) MSB, and (f) MSC

Removal of Fe and Zn from Landfill Leachate

Landfill leachate from the Piyungan site contained elevated concentrations of Fe (9.408 ppm) and Zn (0.258 ppm) at near-neutral pH, exceeding Indonesian regulatory limits. Visual observation indicated a reduction in color intensity after treatment with mesoporous silica (MS), suggesting partial removal of chromophoric species. Fig.-6 and 7 show that metal removal increases with CTAB concentration, with MSC exhibiting the highest adsorption performance, followed by MSB and MSA. Although MSB exhibits the highest BET surface area and pore volume among the three CTAB-templated samples (Table-1), MSC—which has the smallest and most uniform mean pore diameter (3.9 nm) — achieves the highest Fe and Zn removal, suggesting that, in this aqueous system, mean pore diameter is more closely related to uptake than total N₂-accessible surface area: part of MSB's high BET surface area may be located in finer or less-connected porosity that is accessible to N₂ during physisorption but less accessible to the larger, hydrated Fe and Zn ions under aqueous adsorption conditions, consistent with the progressive pore densification and reduced connectivity observed by TEM/SEM at higher CTAB loading (see Morphology of Mesoporous Silica). The narrower, more uniform mesopores in MSC instead offer a higher density of accessible silanol binding sites together with nanoconfinement effects that can enhance ion–surface interactions in narrower channels, favoring more effective heavy-metal uptake despite its lower bulk surface area. In these experiments, maximum removal efficiencies of approximately 79% for Fe and 87.5% for Zn were achieved, consistent with reported adsorption capacities of mesoporous silica-based materials for heavy metal removal.^{28,29,30}

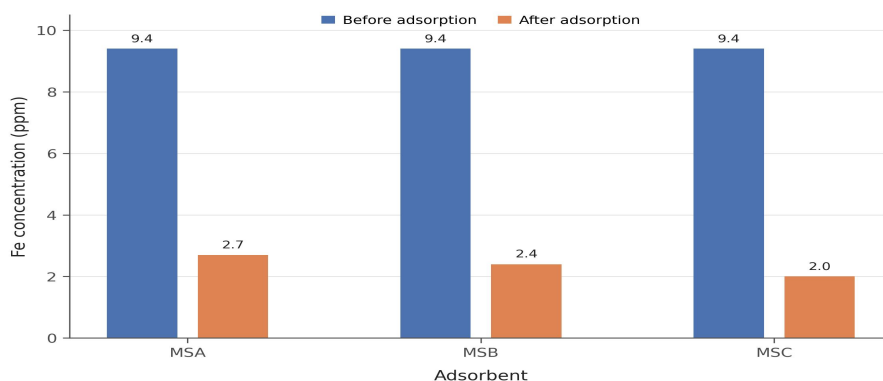


Fig.-6: Fe Concentration in Landfill Leachate Before and After Adsorption Treatment by MS (MSA, MSB, MSC)

The values are in the same range as recent reports for other biomass- and biosorbent-derived adsorbents applied to heavy-metal-laden wastewater and seawater, including crab-shell-based adsorbents for

Cu(II)/Cd(II) and Ni(II)/Zn(II) and chitosan-coated carbon biosorbents for Cd(II).^{11,12,13,14} The leachate results also support the SDG-6 link, since the adsorbent lowers Fe and Zn levels in a real waste-derived water stream before discharge.

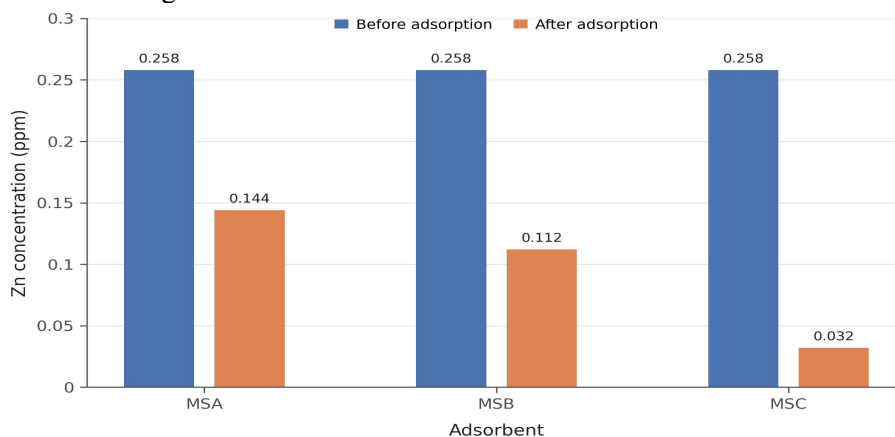


Fig.-7: Zn Concentration in Landfill Leachate Before and After Adsorption Treatment by MS (MSA, MSB, MSC)

CONCLUSION

The present work converts snake fruit peel into high-purity mesoporous silica through acid-alkali extraction and CTAB-templated sol-gel synthesis. CTAB templating increased surface area and pore volume while producing smaller, more uniform mesopores than the non-templated silica. When applied to real landfill leachate, the material removed about 79% of Fe and 87.5% of Zn, and SEM-EDX mapping supported metal retention on the silica surface. The results indicate that the mesostructure, especially pore accessibility, is important for adsorption in this leachate matrix. Further work should examine CTAB concentration more narrowly, regeneration and reuse of the adsorbent, and performance toward other metals in different wastewater matrices. Because the adsorbent is prepared from agricultural waste and applied to contaminated water, the work is directly connected to SDG 6 (Clean Water and Sanitation).

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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