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## SCIENTIFIC ARTICLE

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# Stepwise extraction, functionalization, and composite formation of lignin and silica from rice husk: Synthesis and characterization

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## ARTICLE INFORMATION

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## ABSTRACT

This study presents a novel approach for the stepwise extraction of lignin and silica from rice husk, followed by the synthesis of a silica-lignin hybrid composite. The rice husk was first processed to obtain rice husk ash (RHA), from which lignin and silica were sequentially extracted using alkali and acidic treatments. Lignin was isolated by acidification of the filtrate, while silica was recovered by neutralizing the remaining solution and calcining the precipitate. The silica was functionalized with amino groups via reaction with 3-aminopropyltriethoxysilane (APTES) to enhance its compatibility with lignin. The lignin-silica composite was synthesized by oxidizing the lignin with sodium periodate, followed by the addition of the amino-functionalized silica, which led to electrostatic interactions between the two components. The composite was characterized using UV-Vis, FTIR, SEM, EDX, and TGA techniques, confirming successful hybridization of lignin and silica. The obtained results indicate that the composite exhibited unique structural and thermal properties, demonstrating potential applications in areas such as adsorption, catalysis, and material science. This study provides a sustainable method for valorizing rice husk waste into high-value hybrid materials.

## Highlights

- Stepwise extraction of lignin and silica from rice husk using alkali and acid treatments.
- Amino-functionalized silica synthesized via reaction with APTES for enhanced reactivity.
- Lignin is oxidized with sodium periodate to promote interaction with modified silica.
- Composite confirmed via UV-Vis, FTIR, SEM, EDX, and TGA as structurally integrated.
- Sustainable valorization of rice husk into lignin-silica hybrid for advanced applications.

## 1. Introduction

The quest for sustainable and functional materials has become increasingly important due to the growing need for environmentally friendly alternatives to conventional synthetic materials (Moreno et al., 2016; Morimoto et al., 2023; Peralta et al., 2024). Rice husk, a byproduct of rice processing, has garnered attention as a valuable resource for the extraction of silica and lignin, two biomolecules with diverse industrial applications (Nzereogu et al., 2023; Hiloidhari and Baruah, 2011; Akhter et al., 2021; Tanveer et al., 2022; Elakkiah, 2019; Saravanan et al., 2017; Garcia et al., 2016). Silica, a highly versatile inorganic compound, has numerous applications in areas ranging from catalysis to electronics

(Elakkiah et al., 2019; Akhter et al., 2021; Gariya et al., 2023; Ong et al., 2021; Puglia et al., 2014; Wang et al., 2022), while lignin, a natural polymer, is renowned for its potential in bio-based materials, adsorption processes, and as a renewable feedstock for biofuels (Ahuja et al., 2018; Menon and Rao, 2012; Pham et al., 2023). The simultaneous extraction of both components from rice husk offers the potential to create hybrid composites that combine the advantages of both, yielding materials with enhanced properties for a range of applications.

Rice husk is primarily composed of cellulose, hemicellulose, and lignin, with silica predominantly found in the form of amorphous silica in the ash (Bhattacharyya, 2014; Quispe et al., 2017; Asadullah, 2014). Traditional methods for silica extraction from rice husk often rely on

high-temperature calcination or chemical treatments such as alkali fusion, which, while effective, can lead to a loss of structural integrity or the introduction of undesirable contaminants (Nzereogu et al., 2023; Kamari and Ghorbani, 2021; Kumari et al., 2023; Nourmoradi et al., 2019; Kamari et al., 2021). Lignin, on the other hand, can be extracted through alkaline treatments such as sodium hydroxide or ethanol-based methods, which not only remove the lignin from the plant matrix but also preserve its unique structural characteristics (Dagnino et al., 2018; da Rosa et al., 2017; Nguyen et al., 2022). By optimizing extraction methods, it is possible to obtain both silica and lignin with minimal loss of their functional properties, enabling the creation of silica-lignin hybrid composites with superior performance.

The novelty of this approach lies in the stepwise extraction and functionalization of silica and lignin directly from rice husks, eliminating the need for separate synthesis and modification steps often required by conventional methods. Traditional approaches involve preparing SiO<sub>2</sub> through processes such as sol-gel synthesis using tetraethoxysilane or precipitation using sodium silicate, followed by modification with reagents like N<sub>2</sub>-(aminoethyl)-3-aminopropyltrimethoxysilane (Arunmetha et al., 2015; Mejía et al., 2016; Supiyani et al., 2022; Shweta and Jha, 2015; Haoran et al., 2013; Jesionowski et al., 2010; Nowacka et al., 2013). These methods then combine the synthesized silica with pre-treated lignin, often oxidized with sodium periodate (Jesionowski et al., 2010; Nowacka et al., 2013; Xiong et al., 2015). However, such composites frequently result in aggregated materials, limiting their functionality. In contrast, this new method activates the functional groups of lignin and aminates the silica before combining them, leading to more efficient and effective hybrid materials with improved properties, such as enhanced adsorption, thermal stability, and mechanical strength (Pham et al., 2023; Jesionowski et al., 2010).

The use of sodium periodate as an activator for lignin is crucial in enhancing its reactivity and functionality. Sodium periodate selectively oxidizes the phenolic groups in lignin, introducing reactive aldehyde groups that can effectively interact with other materials, thus improving bonding and compatibility with silica (Sun et al., 2023; Kozhevnikov et al., 2017; Sun and Jiang, 2024). Additionally, the use of APTES (3-aminopropyltriethoxysilane) for silanization of silica particles provides a significant advantage over traditional methods. APTES forms covalent bonds between silica and the functional groups of lignin, creating a stable and homogeneous composite structure, improving dispersibility, and reducing aggregation (Budnyak et al., 2018; Klapiszewski et al., 2012; Klapiszewski et al., 2013; Klapiszewski et al., 2013; Klapiszewski et al., 2015). This approach overcomes limitations of earlier methods that relied on untreated lignin and poorly integrated silica, allowing for the creation of more uniform, stable, and functional composites.

In this study, rice husk was processed into rice husk ash (RHA) by acidic dissolution of alkaline metals and alkali oxides, followed by sequential extraction of lignin and silica using acidic and alkali treatments. Lignin was precipitated by acidification with 1.0 M HCl, and silica was recovered by treating the supernatant with NaOH to form sodium silicate, which was filtered, washed, dried, and calcined at 600 °C for 4 h to yield pure nanosilica. To enhance the compatibility of silica with lignin, the surface of the silica was modified with amino groups using APTES. The lignin-silica composite was synthesized by activating the functional groups of lignin with sodium periodate, followed by the addition of the amino-functionalized silica, resulting in electrostatic interactions between the two components. The morphological, thermal, and compositional properties of lignin, silica, and their composite were

confirmed using UV-Vis, FTIR, SEM, EDX, and TGA analyses. The current study demonstrates a sustainable method for converting rice husk waste into valuable hybrid materials.

## 2. Materials and Methods

### 2.1 Raw Materials

Sodium hydroxide pellet (NaOH), ferric chloride hexahydrate (FeCl<sub>3</sub>·6H<sub>2</sub>O, 99.5%) were purchased from Merck. Absolute ethanol, hydrochloric acid (HCl), and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) were supplied by Pascal Scientific Limited, Nigeria. All chemicals were of analytical grade and used directly without purification. The rice husk (RH) used as a starting resource for this study was harvested in 2023 and was graciously received from the Igbimo Rice Processing Facility in Ekiti State, Nigeria.

### 2.2 Rice husk ash preparation

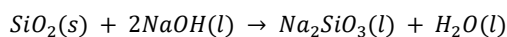
The rice husk (RH) was sieved through a 100 mesh 304 Stainless Steel sieve to remove dust, soils, stones, and other foreign materials. It was then washed multiple times with tap and distilled water, dried under sunlight for three days, and then ground into powder using a commercial grinder. Approximately 30.0 g of the dried RH was refluxed at 120°C in 750 mL of 1.0 M HCl for 24 h to remove alkaline metals and alkali oxides from the rice husk. After cooling to room temperature, the RH was washed thoroughly with distilled water until a constant pH was achieved, and dried at 60 °C for 24 h to obtain rice husk ash, and was labeled as RHA.

#### 2.2.1 Preparation of lignin

Lignin was extracted from RHA following the method reported elsewhere (Ahuja et al., 2018). Briefly, 5.0 g of RHA was weighed and added into a round-bottom flask containing an aqueous solution of NaOH (200 mL and 1.0 M), and heated for 24 h at 100 °C under. After cooling down to room temperature, the mixture was separated, and the cellulose and hemicellulose-rich residues were discarded, while the filtrate, which contains lignin and silica, was collected for isolation of lignin and silica at different pH levels. For the extraction of lignin, the filtrate was acidified with 5 M HCl until pH 1 was reached to allow lignin precipitation, which was recovered by centrifugation at 10,000 rpm for 10 min, washed with deionized water to remove metal oxides, including CaO, P<sub>2</sub>O<sub>5</sub>, MgO, and K<sub>2</sub>O. The recovered lignin was freeze-dried to obtain pure lignin.

#### 2.2.2 Preparation of silica

The supernatant obtained after lignin recovery was treated with alkali solution to adjust the pH from 1 to 4 and refluxed at 90°C under constant stirring for 4 h, facilitating the solubilization of silica and its conversion into sodium silicate, Eqn. 1. The resulting solid was purified through three centrifugation cycles at 10,000 rpm for 10 min using a deionized water-ethanol mixture to remove water-soluble salts formed during neutralization. The purified silicate was oven-dried at 100°C for 24 h and thermally calcined at 600°C for 5 h to eliminate residual organic matter from the silica nanospheres.

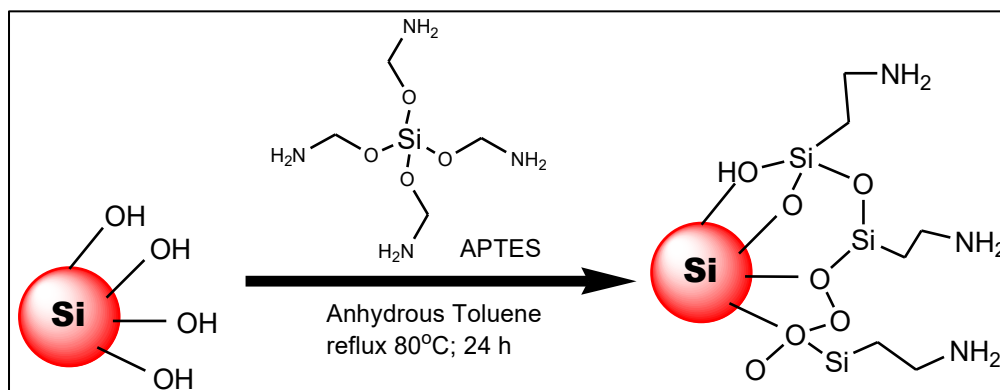


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### 2.3 Amino functionalization of silica nanospheres

To functionalize silica nanospheres with amino ( $\text{-NH}_2$ ) groups, 3 g of silica nanospheres were first dispersed in 150 mL of toluene by sonication. Subsequently, 1.5 mL of APTES was added dropwise to the

mixture, and the solution was refluxed under constant stirring at 80 °C for 24 h. After the reaction, the functionalized silica ( $\text{SiO}_2\text{-NH}_2$ ), Scheme 1, was then separated from the mixture by filtration, washed multiple times with ethanol, and dried at 100 °C for 24 h.

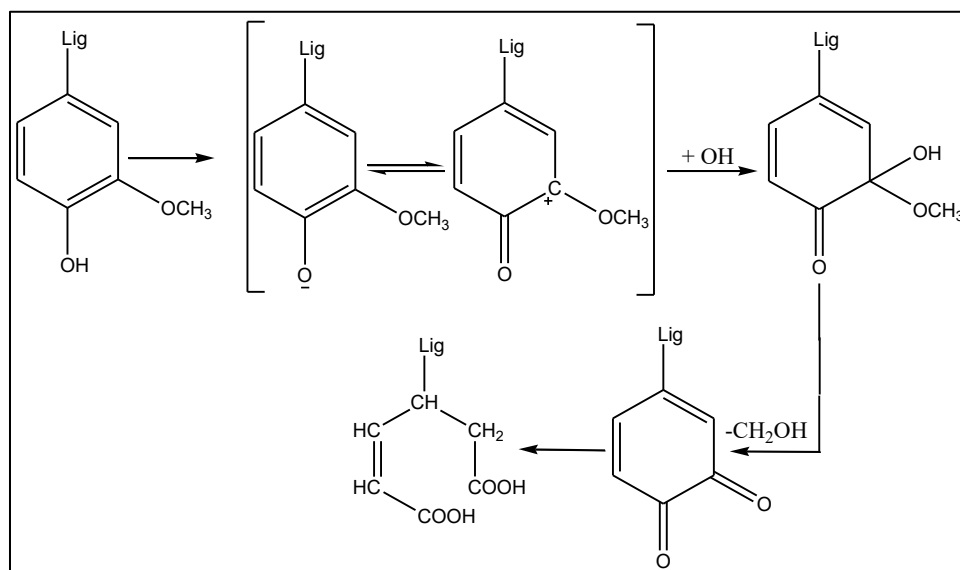


**Scheme 1:** Synthetic pathway for APTES-modified silica.

### 2.4 Synthesis of lignin/silica hybrid composite

To prepare the lignin/silica hybrid composite, 5 g of lignin was first dispersed in 100 mL of a 1:1 (w/w) dioxane/water mixture. Then, 0.5 M sodium periodate in 50 mL was added to the solution to oxidize the surface functional groups of lignin, Scheme 2. Subsequently, 5 g of  $\text{SiO}_2\text{-NH}_2$

was added, and the mixture was stirred in the dark for 2 h at room temperature to facilitate electrostatic interaction between the activated lignin and the positively charged aminated  $\text{SiO}_2$ . The resulting composite was recovered by a filtration process, washed three times with ethanol, and dried under vacuum to obtain LS.



**Scheme 2:** The mechanism of the activation/oxidation reaction of lignin with sodium periodate.

### 2.5 Material characterization

**(a) UV-Vis Spectroscopy:** The solid reflectance spectra of Lignin, silica, and L/S were recorded on a Shimadzu UV-VIS-NIR Spectrophotometer UV-3100 with an MPCF-3100 sample compartment with samples mounted between two quartz discs, which fit into a sample holder coated with barium sulfate. The spectra were recorded over the wavelength range of 800–250 nm, and the scans were conducted at a medium speed using a 20 nm slit width.

**(b) Scanning electron microscope (SEM)/energy dispersive X-ray:** The surface morphology and elemental composition of the samples were analyzed using an SEM equipped with energy dispersive analysis of X-ray equipment (EDAX) (XL 30 FEG ESEM).

**(c) Fourier Transmitted Infra-Red Spectroscopy (FTIR):** The surface functional groups on the samples were recorded on a Thermo Fisher Scientific FTIR spectrophotometer, using pressed KBr pellets.

**(d) Thermogravimetric analysis:** Thermogravimetric analysis was employed for the thermal stability of the samples using a Mettler Toledo TGA/SDTA 851 thermal analyzer apparatus at a heating rate of 10 °C/min from 40 to 800 °C under the protection of a N<sub>2</sub> atmosphere.

### 3. Results and Discussions

#### 3.1. Material characterization

##### 3.1.1 FT-IR spectroscopy studies

Figure 1(a-c) displays the FTIR spectra for extracted lignin (a), silica (b), and the lignin/silica composite (c). In the lignin spectrum, the band between 3330–3400 cm<sup>-1</sup> corresponds to the stretching vibrations of O-H of phenolic and aliphatic groups (Balasubramanian et al., 2018). Vibrational peaks at 2854.65 and 2924.09 cm<sup>-1</sup> are associated with sp<sup>3</sup> C-H stretching in methyl (-CH<sub>3</sub>), methoxy (-OCH<sub>3</sub>), and methylene (-CH<sub>2</sub>-) groups of lignin backbones. The small but visible weak band at 1720.50 cm<sup>-1</sup> indicates C=O stretching of carbonyl/carboxyl groups, while the band at around 1635.64 cm<sup>-1</sup> confirms C=C stretching in conjugated p-substituted aryl ketones (Cherif et al., 2020). An aromatic skeleton vibration appears at 1507.39 cm<sup>-1</sup>, while the band at 1396.48 cm<sup>-1</sup> is due to the combination of aromatic C-H in-plane deformation and aliphatic C-H stretching in CH<sub>3</sub>. The FTIR result also reveals spectral features consistent with GSH-type lignin, including bands at 1134 cm<sup>-1</sup> and 864.11 cm<sup>-1</sup> (Cherif et al., 2020). In the silica spectrum from RHA, peaks at 794.12 cm<sup>-1</sup> and 1085.70 cm<sup>-1</sup> correspond to asymmetric Si-O stretching vibrations, while the 502.54 cm<sup>-1</sup> band represents Si-O-Si bending vibration (Herth et al., 2016). Notable absorption peaks at 3245.69–3548.94 cm<sup>-1</sup>, 1623.54 cm<sup>-1</sup>, and 1358.50 cm<sup>-1</sup> in the lignin/silica composite are attributed to OH (from lignin), NH (from aminated silica), C=O, and C-C (from lignin), respectively. The comparison of spectra 1a and 1b shows that the characteristic peaks observed in the composite (c) are present, confirming successful composite formation.

##### 3.1.2 Optical absorption analysis

UV-Vis spectroscopy was used to analyze the absorption characteristics of extracted silica, lignin, and their composite, as shown in Figure 2. The extracted silica from rice husk displayed two distinct absorption bands at 232 and 304 nm, confirming the formation of silicon dioxide particles (Biradar et al., 2020). Extracted lignin exhibited a strong absorption band at 237 nm and a smaller shoulder at 339 nm, attributed to extended conjugation and the presence of various  $\alpha$ -carbonyl substituents on the benzene ring, respectively (Ruwoldt et al., 2022). The silica/lignin composite showed two absorption bands at 239 and 345 nm, corresponding to the peaks observed in the individual spectra of silica and lignin, thus confirming the successful integration of both components (Joshi et al., 2019; Ruwoldt et al., 2022). The absorption profile of the composite suggests an interaction between silica and lignin, resulting in a unique combined absorption pattern. Notably, the absorption profile of lignin is more redshifted compared to both silica and the composite, covering the entire UV region, which indicates its broad absorption

capacity due to ionization of the phenolic moieties in the lignin structure (Balasubramanian et al., 2018).

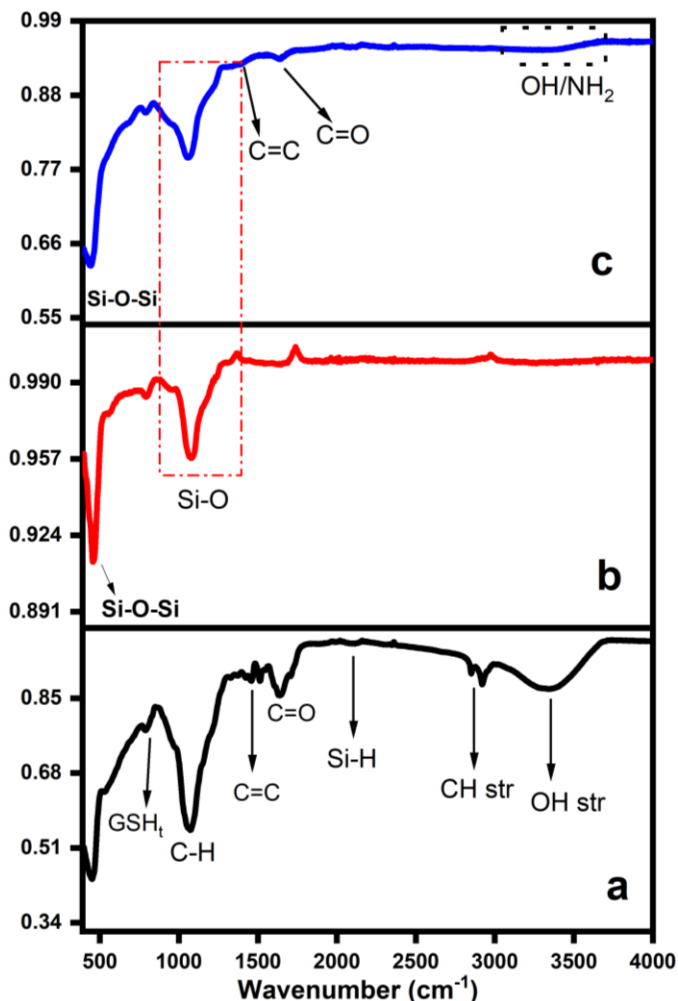


Figure 1: FT-IR spectroscopy of (a) lignin, (b) silica, and (c) lignin-silica.

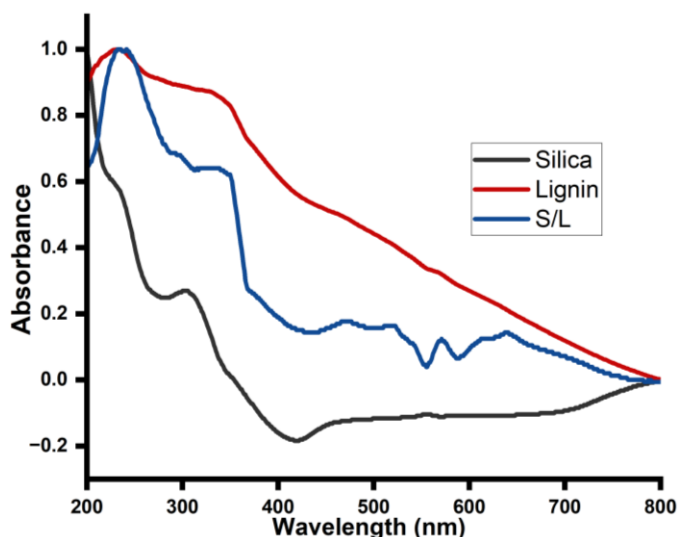


Figure 2: UV-Vis diffuse reflectance spectra of lignin, aminated silica, and L/S.

### 3.1.3 Elemental Composition

The EDX analysis of aminated silica, lignin from rice husk, and their composite reveals the elemental composition of each material (Figure 3). Pure silica consists of Si, O, C, and N, with the at. wt% of Si being 22.77%, indicating the presence of silicon dioxide with some nitrogen from the aminated groups, Figure 3a. The lignin contains C, Si, O, Cl, Na, S, and N, highlighting the complex structure of lignin, which includes various substituents such as Cl, Na, S, and N (Figure 3b). The at. wt% of Si in lignin is 4.97%, reflecting its lower Si content. The composite shows the presence of Si, O, C, N, and I, with iodine (I) being a distinct element not found in either pure silica or lignin (Figure 3c). The at. wt% of Si in the composite is 18.47%, indicating successful incorporation of silica into the lignin matrix. The presence of I in the composite is attributed to the use of sodium periodate ( $\text{NaIO}_4$ ) as the activating agent during lignin modification. This suggests that the modification process facilitated the integration of silica and lignin, resulting in a composite with a distinct elemental profile.

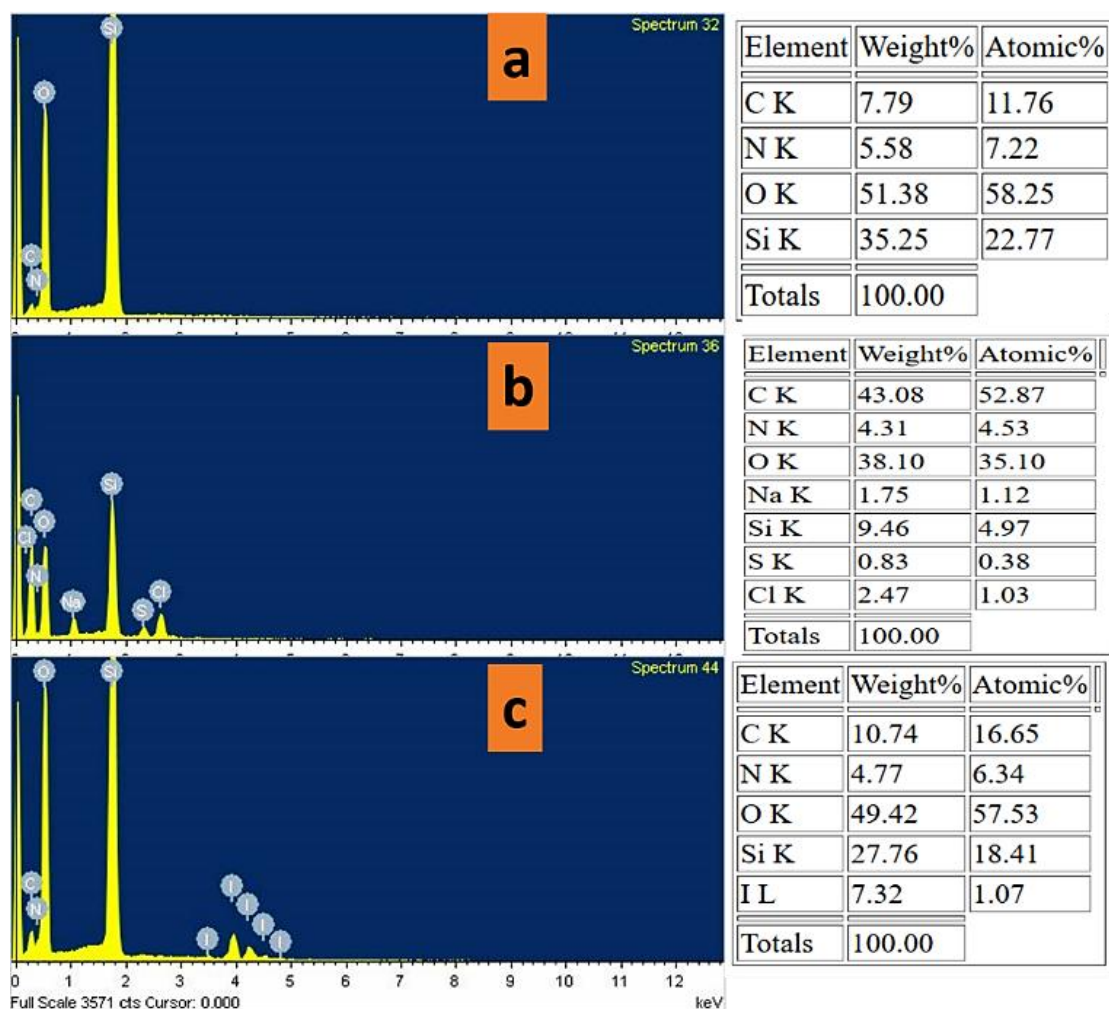
### 3.1.4 Morphology

The SEM micrograph of extracted lignin in Figure 4 shows that the

morphology has a rough, granular structure with irregular, porous aggregates, suggesting that the lignin retains a complex, heterogeneous texture after extraction. The image at the left (magnification 2.94k) shows slightly larger particles with visible surface texture, while the right side (magnification 8.26k) highlights finer features with more pronounced granular or fibrous characteristics, indicating a high level of surface irregularity.

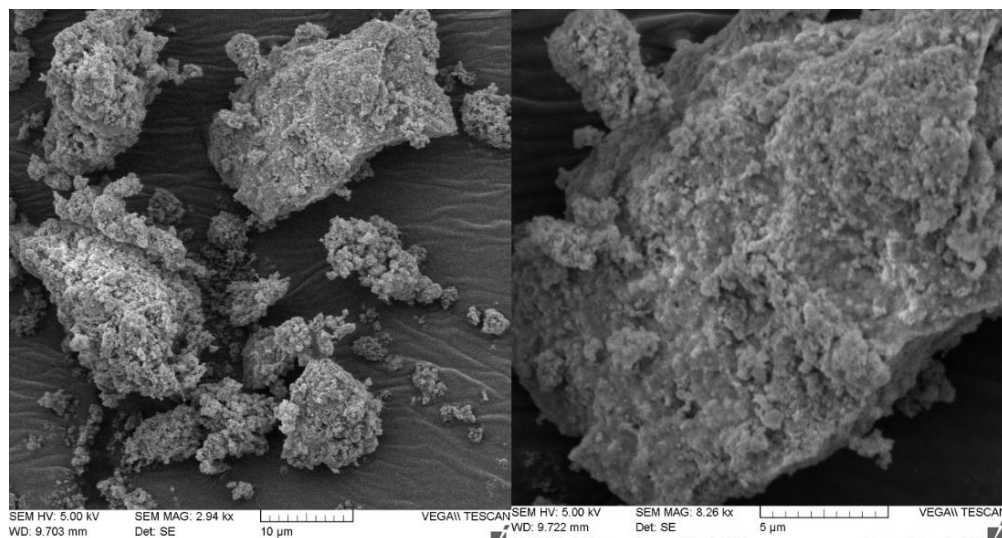
The SEM micrograph of isolated silica (Figure 5) extracted from rice husk shows a porous, agglomerated structure with spherical particles, typical of silica obtained through processes such as alkaline extraction or sol-gel methods. The particles are fine and exhibit a granular texture, with some degree of clustering, suggesting a high surface area, which is characteristic of amorphous silica.

Figure 6 shows that the morphology of a silica/lignin composite has a heterogeneous structure, where the lignin matrix appears as larger, irregular, layered features interspersed with smaller, spherical silica particles. The aminated silica contributes to a rougher and more interconnected surface morphology, which likely enhances interfacial interaction between the components. This structural integration suggests the potential for improved adsorption, catalytic, or mechanical properties in applications requiring composite materials with high surface area and active functional groups.

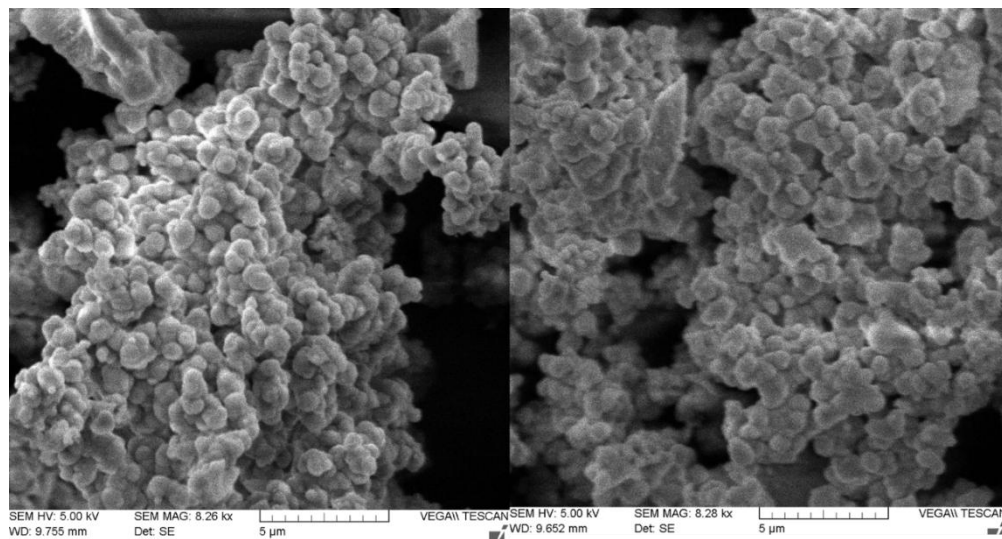


**Figure 3:** EDX analysis of (a) aminated silica, (b) lignin, and (c) lignin/silica, and the corresponding atomic and weight percentages of elemental composition.

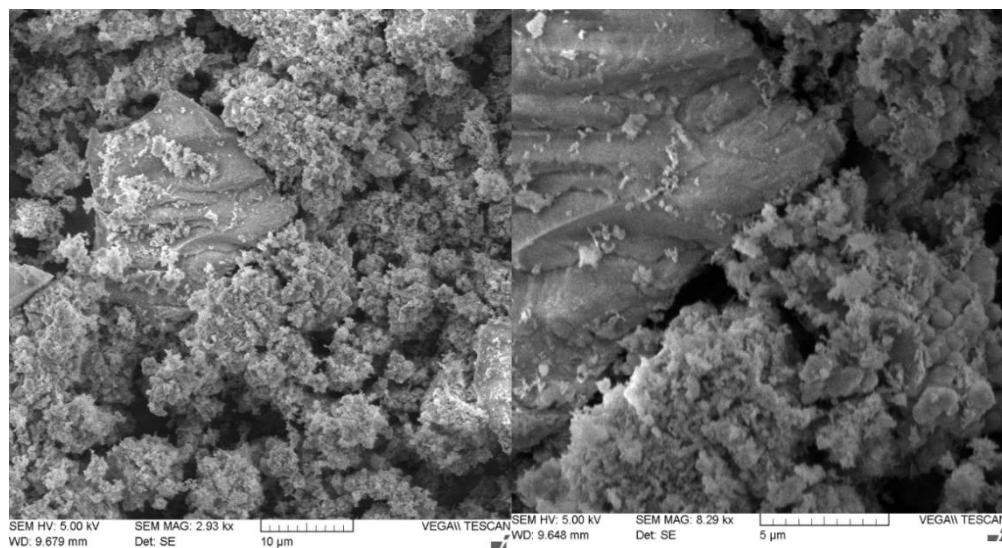




**Figure 4:** SEM image of extracted lignin.



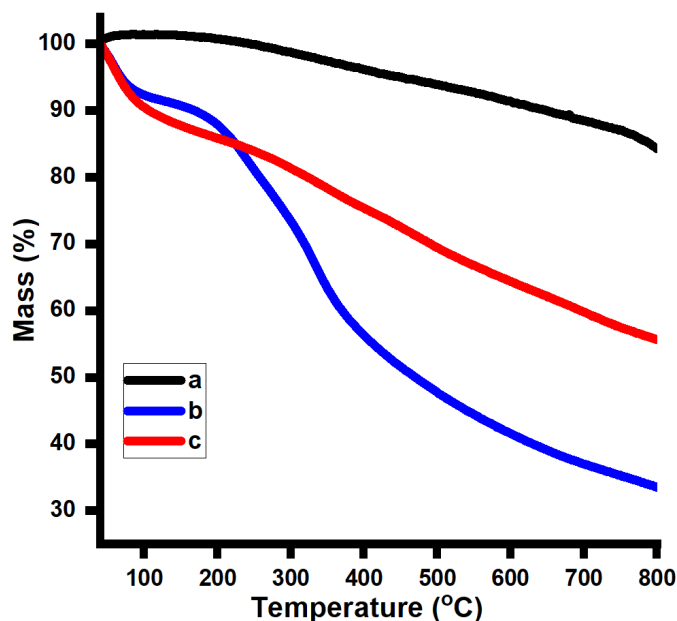
**Figure 5:** SEM image of extracted silica.



**Figure 6:** SEM image of silica-lignin composite.

### 3.1.5 Thermal analysis

Figure 6 depicts the TGA thermograph of pristine silica, lignin, and their composite (Figure 7). The thermograph of aminated silica shows minimal mass loss across the temperature range 40–800 °C, with decomposition starting at an onset temperature of 220 °C and gradually decreasing to a minimum at 800 °C, resulting in a total weight loss of 15.4%. This loss is attributed to the removal of amino groups and other organic components from the coupling agents used in modifying the silica, confirming successful salinization of silica particles. The lignin thermograph reveals three stages of thermal oxidative degradation. The first stage, occurring at a decomposition temperature of 173 °C, exhibits a weight loss of approximately 7.3%, due to the evaporation of moisture and volatile impurities. A sharp decline follows between 187 °C and 380 °C, with a total weight loss of 32.16%, attributed to cleavage of aliphatic side chains, dehydration, and depolymerization of lignin's aromatic structure (Silva et al., 2011). The final stage continues up to 800 °C, with a total weight loss of 66.5%, primarily due to the oxidation of carbonaceous residues. The TGA profile of the silica/lignin composite shows two significant degradation steps, similar to lignin but at a slower rate. The first mass loss occurs between 100–150°C, attributed to the evaporation of moisture or residual solvents, followed by a second mass loss above 200°C, reaching a minimum of 65% at 800°C due to the thermal degradation of lignin and oxidation of carbon residues (Balasubramanian et al., 2018). Beyond 200°C, the presence of silica stabilizes the composite, reflecting the thermal stability of silica particles and its contribution to preventing complete degradation of the composite. This profile suggests a synergistic effect, where silica enhances the thermal stability of the lignin component, contributing to the slower loss rate observed for the composite.



**Figure 7:** TGA curves for (a) silica, (b) lignin, and (c) silica/lignin composite.

## 4. Conclusion

This study presents an eco-friendly approach for the stepwise extraction of lignin and silica from rice husk, followed by the synthesis

of a lignin-silica hybrid composite. Characterization using imaging, thermal, and spectroscopy techniques confirmed the structure and functionality of the composite. FTIR spectroscopy showed the integration of lignin and silica, with characteristic peaks indicating functional groups from both components. UV-Vis spectra revealed distinct absorption bands for silica and lignin, with the composite exhibiting a combined absorption profile, confirming successful hybridization. The findings highlight the unique structural, thermal, and optical properties of the composite, suggesting potential applications in adsorption, catalysis, and bio-based functional materials. This method offers a sustainable route for valorizing rice husk waste into high-value materials, advancing green chemistry and waste management strategies.

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## Competing Interest

The author affirms that there are no identifiable conflicts of interest, either financial or personal, that could be perceived as influencing the work presented in this paper.

## Data Availability Statement

All data generated or analyzed during this study are included in this published article.

## Ethics Approval

This study did not involve human participants or animals and therefore did not require ethics approval.

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