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SPECIAL ISSUE ARTICLE

Direct conversion of rice husk into hydrogen and sodium silicate via alkaline thermal treatment with low-CO_2 emission

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Abstract

This study presents a novel single-step method for producing sodium silicate and high-purity hydrogen from rice husk biomass with reduced CO_2 emission using alkaline thermal treatment (ATT). Conventional methods extract amorphous silica from rice husk through an energy-intensive, CO_2-emitting process, followed by a separate reaction with sodium carbonate to synthesize sodium silicate. In contrast, the proposed method eliminates this additional step by directly generating sodium silicate while simultaneously producing hydrogen, ensuring a reduced carbon-emission process. ATT enables interconnected reactions, including in-situ CO_2 capture, enabling decarbonized hydrogen production as an energy carrier and sodium silicate for high-value applications. The use of NaOH promotes hydrogen generation at lower temperatures while capturing CO_2 as solid carbonates, distinguishing this process from conventional gasification. Additionally, sodium carbonates formed in situ react with SiO_2 in rice husk ash, enabling direct sodium silicate synthesis. Experimental results demonstrate that ATT at 950°C, with a rice husk-to-NaOH ratio of 1:3 and 1.9 g of SiO_2, yields up to 5.1 g of sodium silicate and 51 mmol of hydrogen per gram of rice husk, with reduced CO_2 emission. Optimizing reaction conditions through elemental analysis enhances hydrogen yield and improves sodium silicate synthesis, highlighting the sustainability of this approach.

KEYWORDS

alkaline thermal treatment, biomass conversion, carbon-neutral process, hydrogen, rice husk, sodium silicate

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1 | INTRODUCTION

Amorphous silica with controlled size and high purity plays a crucial role in various industries, including eco-friendly tire manufacturing, coatings, paints, cosmetics, catalysts, and pharmaceuticals.[1,2] However, naturally occurring amorphous silica is limited, necessitating synthetic production methods.[3] The conventional acid precipitation process synthesizes silica by first producing sodium silicate from quartz or rice husk ash, followed by acid precipitation.[4] Using rice husk biomass waste as a silica source offers multiple benefits, such as utilizing agricultural waste and generating high-purity amorphous silica. However, traditional extraction methods pose significant environmental concerns, including high carbon emissions and challenges in managing unburned carbon residues.

Beyond material synthesis, the need for low-carbon and carbon-neutral energy solutions has become increasingly urgent in mitigating climate change. Hydrogen is widely regarded as a clean and efficient energy carrier that can replace fossil fuels while reducing CO_2 emissions.[5] Unlike conventional fossil-based energy sources, hydrogen enables efficient energy storage and can be produced sustainably, making it a promising solution to the intermittent issues associated with renewable energy sources like wind and solar power.

Current hydrogen production technologies, such as water electrolysis and methane reforming, have distinct advantages and limitations.[6] Water electrolysis generates hydrogen without emitting greenhouse gases, but remains economically constrained due to high energy consumption.[7] Conversely, methane reforming is more cost-effective but depends on fossil fuels, making carbon neutrality difficult to achieve.[8] Biomass-based hydrogen production has emerged as an attractive alternative, as biomass absorbs CO_2 during its growth, contributing to a more sustainable energy cycle.[9] However, conventional biomass gasification still generates greenhouse gases, limiting its overall environmental benefits.[8] Therefore, developing an approach that enables hydrogen production with minimal CO_2 emissions while simultaneously generating valuable byproducts is of significant interest.

This study explores alkaline thermal treatment (ATT) as a novel method for hydrogen production, offering a highly efficient process that significantly suppresses CO_2 emissions while generating high-purity hydrogen. Beyond achieving carbon neutrality, ATT has the potential for carbon negativity by integrating bioenergy with carbon capture and storage. A key advantage of this approach is its ability to convert rice husk waste directly into hydrogen and sodium silicate in a single-step reaction, eliminating the need for a separate processing stage.

Rice husk, an abundant agricultural byproduct in rice-producing regions, consists primarily of organic carbon and silica, making it a promising feedstock for this conversion process.[4] While rice husk has traditionally been considered a biomass energy source, its high ash content (comprising approximately 50% cellulose, 25%–30% lignin, 15%–20% silica, and 10%–15% moisture) presents challenges for direct combustion or co-firing applications. With a calorific value nearly half that of coal, rice husks are a relatively inefficient fuel source.[10] However, well-combusted rice husk ash has found applications in construction materials, such as concrete reinforcement, and in advanced technologies, such as lithium-ion battery anodes,[11] where its silica content is transformed into silicon. Despite these applications, broader utilization strategies remain limited.

This study proposes an innovative ATT process that simultaneously converts rice husk waste into high-purity bio-hydrogen and sodium silicate in a single reaction step, without CO_2 emissions. In this process, Na_2CO_3 formed in situ directly reacts with rice husk ash (SiO_2) to produce sodium silicate, eliminating the need for an additional reaction. This breakthrough not only enhances the sustainability of hydrogen production but also expands the potential applications of rice husk biomass waste, contributing to both clean energy generation and advanced material synthesis. To validate the feasibility of ATT, this study systematically examines its efficiency in hydrogen and sodium silicate production, explores the underlying reaction mechanisms, and optimizes process conditions. The findings offer a sustainable alternative for biomass utilization, paving the way for carbon-neutral energy and material synthesis.

2 | EXPERIMENTAL

2.1 | Materials

Rice husk, the outer protective layer of rice grains, is a byproduct of rice milling and consists primarily of cellulose, hemicellulose, lignin, and silica, which contribute to its structural integrity. Cellulose provides mechanical strength as a key component of plant cell walls, while hemicellulose reinforces the fibrous network. Lignin, a complex aromatic polymer, enhances rigidity and improves biodegradation resistance. The rice husk was obtained from a local farm in Boeun County, Chungcheongbuk-do, South Korea. Sodium hydroxide (NaOH) was procured from Daejung Chemicals & Metals with ≥98% purity and used without further purification. Silicon dioxide (SiO_2) powder, with a purity of ≥99% and particle sizes ranging from 50 to 70 mesh, was procured

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from Sigma-Aldrich and utilized without any further purification.

2.2 | Reaction procedure

Experiments were conducted using a horizontal reactor consisting of a quartz tube (2.2 cm internal diameter, 2.5 cm outer diameter, and 100 cm length) placed inside a furnace (Figure S1). A thermocouple was inserted into the quartz tube to monitor the reaction temperature. Rice husk and alkali materials were placed in an alumina boat (3 mL capacity) positioned at the center of the furnace. NaOH served as the alkali catalyst, while nitrogen (N_2) was introduced as the carrier gas at a flow rate of 50 mL min^{-1}, controlled by a mass flow controller. Gas products were collected in a Tedlar bag for analysis.

Real-time gas analysis was performed using an Agilent 490 Micro gas chromatograph (GC) equipped with Molsieve 5A and PoraPLOT Q columns. A control experiment was conducted for steam gasification, using only the reactants without NaOH. The furnace temperature was gradually increased from ambient temperature to the target temperature at a rate of 4°C/min. Preheated water vapor was introduced at a rate of 0.023 mL/min to sustain the ATT reaction, and gas composition was continuously monitored using GC.

2.3 | Analysis

Gas composition was determined in real-time using an Agilent 490 Micro GC. The RT-Molsieve 5A column was used to separate and quantify H_2, N_2, CH_4, and CO, while the PoraPLOT Q (RT-Qbond) column analyzed CO_2, C_2H_4, C_2H_6, C_2H_2, and C_3H_8. Gas samples were directly injected into the micro-GC, with N_2 used as an internal standard. The measured gas compositions were normalized to molar units to ensure accuracy and reproducibility. The carbon content of the biomass was distributed among gaseous carbonaceous compounds (CH_4, CO, and CO_2), liquid-phase tar components (C^{2+}), and solid residues.

Elemental composition analysis of the dried samples (C, H, O, and N) was conducted using a Thermo Flash 2000 Elemental Analyzer. Samples were oxidized at approximately 1800°C, converting the elements into their respective gaseous oxides. These gases were processed through a reduction stage to remove excess oxygen, then separated by GC and detected using a thermal conductivity detector. The resulting elemental analysis (EA) data provided precise quantification of the elemental composition, facilitating stoichiometric calculations for the ATT reaction.

X-ray diffraction (XRD) analysis was performed using a Rigaku Miniflex 600 X-ray generator to investigate the crystalline structure and phase composition of the reaction products. The prepared sample was placed in the sample holder for optimal measurement conditions. X-rays were directed onto the sample, and the resulting diffraction patterns were recorded for phase identification. Data were collected over a specified 2θ range, with an optimized step size and scan rate to enhance resolution. Diffraction patterns were compared with reference standards, and quantitative phase analysis was performed using the reference intensity ratio method to determine the relative proportions of crystalline phases. XRD analysis was essential for characterizing reaction products formed during the ATT process, particularly sodium silicate and other crystalline phases.

Inductively coupled plasma optical emission spectroscopy (ICP-OES) was conducted using a ThermoFisher iCAP Pro ICP-OES to determine elemental composition with high precision. Solid samples were digested in hydrochloric acid, nitric acid, or a combination thereof to ensure complete dissolution. The resulting solution was diluted to maintain analyte concentrations within the instrument's calibration range. Calibration standards were prepared in a matrix comparable to the sample to ensure analytical accuracy. The digested solution was introduced into plasma via a nebulizer and spray chamber system, where it was atomized and ionized under high-temperature plasma conditions. Emission spectra were recorded, and elemental concentrations were quantified by comparing measured intensities with calibration standards. ICP-OES provided precise quantification of Si and Na concentrations, offering key insights into the compositional characteristics of the reaction products.

3 | RESULTS AND DISCUSSION

3.1 | H_2 production from rice husk via ATT

Hydrogen production from rice husks was investigated using ATT to achieve CO_2-free hydrogen generation, in contrast to conventional gasification methods. Previous studies on biomass-ATT reactions suggest the following mechanism (Equation (1)),[12,13] where hydrogen from biomass is extracted as pure H_2, while carbon and oxygen are captured by sodium to form Na_2CO_3, preventing CO_2 emissions:

$$C_xH_yO_z + (2x)NaOH + (x-z)H_2O \rightarrow (2x + y/2 - z)H_2 + xNa_2CO_3 \quad (1)$$

Based on the elemental composition of rice husk, a specific reaction mechanism is proposed:

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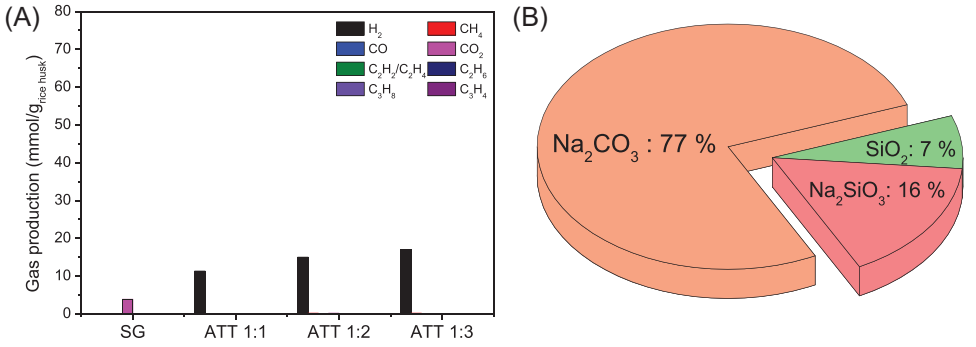


FIGURE 1 (A) Hydrogen production via alkaline thermal treatment (ATT) at different rice husk-to-NaOH mass ratios (SG: no NaOH, 1:1, 1:2, and 1:3). (B) Inorganic product composition from ATT at a 1:3 rice husk-to-NaOH ratio. Reactions were conducted at 230°C, where NaOH and SiO_2 interaction typically occurs (85–200°C).

$$C_{3.18}H_{5.67}O_{2.44} + 6.36NaOH + 0.74H_2O \rightarrow 6.76H_2 + 3.18Na_2CO_3\ (200–700^{\circ}C) \quad (2)$$

The theoretical optimum molar ratio of rice husk to NaOH is 1:6.36, corresponding to a weight ratio of approximately 1:3.07. To optimize hydrogen production, ATT reactions were conducted using various rice husk-to-NaOH weight ratios, including the optimal 1:3 ratio.

Rice husk also contains amorphous SiO_2, which comprises 10.3 wt% of its total composition, as determined by EA (Table S1). SiO_2 can react with both NaOH (introduced as a reactant) and Na_2CO_3 (a byproduct of ATT), forming sodium silicate through the following reactions:

$$2xNaOH + SiO_2 \rightarrow (Na_2O)_xSiO_2\ (\text{mostly } Na_2SiO_3) + xH_2O\ (200^{\circ}C) \quad (3)$$

$$xNa_2CO_3 + SiO_2 \rightarrow (Na_2O)_xSiO_2 + xCO_2\ (850^{\circ}C) \quad (4)$$

According to Equations (2)–(4), NaOH can either react with rice husk to generate H_2 and Na_2CO_3 (Equation (2)) or interact with SiO_2 to form sodium silicate (Equation (3)) at temperatures below 700°C.[14,15] If NaOH preferentially reacts with SiO_2, hydrogen generation from rice husk will be limited, as NaOH—an essential reactant in ATT—will be consumed in the SiO_2 reaction. This would also suppress Na_2CO_3 formation, thereby hindering the primary sodium silicate production pathway (Equation (4)).[16] Additionally, in the absence of NaOH, rice husk undergoes conventional gasification, resulting in CO_2 emissions, which contradicts the study's objective of achieving carbon-free hydrogen production. To determine whether Equations (2) or (3) dominate at temperatures around 200°C, ATT reactions were performed at 230°C, and the results are presented in Figure 1.

Figure 1A confirms that the ATT reaction generates highly pure H_2 (> 97%), while Figure 1B verifies the formation of sodium silicate (Na_2SiO_3), indicating that both Equations (2) and (3) proceed at low temperatures. To further support these findings, FT-IR analysis was conducted (Figure S2). As shown in Figure S2A, the untreated rice husk exhibits a characteristic SiO_2 peak at 1100 cm^{-1}. After reaction under ATT conditions with a 1:3 rice husk-to-NaOH ratio, the spectrum in Figure S2B shows a distinct peak at 1450 cm^{-1} corresponding to Na_2CO_3, along with additional peaks at 880 and 590 cm^{-1}, attributed to sodium silicate (Na_2SiO_3) formed through the reaction of NaOH with SiO_2in the rice husk. These results collectively confirm that Equations (2) and (3) proceed at a low-temperature ATT process. To determine which reaction is preferred, the proportion of NaOH reacting with SiO_2 versus rice husk biomass was calculated. Based on XRD analysis, the NaOH consumption for Na_2SiO_3 formation via SiO_2 reaction is estimated at 0.076 g. Given that 1.5 g of NaOH was initially added in the 1:3 ATT reaction, the majority of NaOH (1.424 g) reacted with rice husk biomass, confirming that Equation (2) dominates over Equation (3) at low temperatures. Furthermore, Na_2CO_3 formation via ATT is unlikely to react with SiO_2 to form sodium silicates (Equation (4)) under these conditions, as this process typically requires high temperatures (~850°C).

Next, ATT reactions were performed at an elevated temperature (950°C) to evaluate whether H_2 is produced exclusively over CO_2 and to determine whether sodium silicates form via the reaction between Na_2CO_3 (an ATT byproduct) and SiO_2 (from rice husk). Figure 2 presents the gaseous product distribution for various rice husk-to-NaOH ratios, using steam gasification (SG) as a reference. Unlike conventional ATT, which typically occurs at ~500°C, this reaction was conducted at an exceptionally high temperature (950°C). This temperature was selected because the reaction between Na_2CO_3 and SiO_2, which yields sodium silicates, occurs above 850°C,[17] as shown in Equation (4).

In SG, both H_2 and CO_2 are produced in substantial amounts, along with small quantities of CH_4 and CO.

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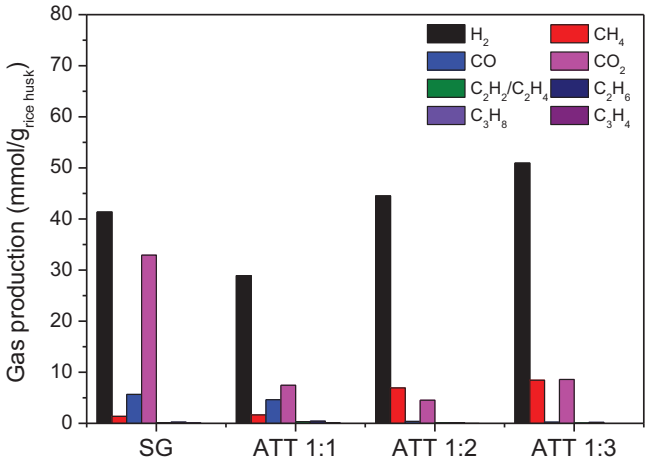


FIGURE 2 Rice husk-alkaline thermal treatment (ATT) reaction results at different rice husk-to-NaOH weight ratios (steam gasification [SG]: no NaOH, 1:1, 1:2, and 1:3). The reaction temperature was increased from room temperature to 950°C to facilitate the reaction between Na_2CO_3 (ATT product) and SiO_2, which occurs above 850°C (Equation (4)).

However, when NaOH is introduced (ATT reaction), H_2 is generated almost exclusively, while CH_4, CO_2, and CO are detected only in minor amounts. At 950°C, a maximum H_2 yield of 50.96 mmol/g-rice husk was achieved using a 1:3 rice husk-to-NaOH ratio. Previous studies on ATT reactions with other biomass sources (e.g., wheat straw, grass, and seaweed) have shown that alkali materials enhance H_2 selectivity compared to SG.[18,19] However, in contrast to these findings, SG of rice husk also produced a significant amount of H_2. To explore this deviation, gas production trends were analysed as a function of temperature, as shown in Figure 3.

Figure 3 presents the gas formation rates for different rice husk-to-NaOH ratios as a function of temperature. In the reference SG experiment, no H_2 was produced at low temperatures. However, at temperatures exceeding 500°C, H_2, CO_2, and a small amount of carbon monoxide (CO) were generated, consistent with conventional high-temperature SG reactions. In contrast, when the ATT reaction was conducted, increasing the NaOH content significantly altered the gas production profile. At high temperatures (above 500°C), H_2 formation from SG decreased substantially, while H_2 generation at lower temperatures (~300°C) increased significantly. Additionally, CO_2 emissions were markedly reduced. In the 1:1 ATT reaction, hydrogen production occurred in two distinct temperature ranges—one at lower temperatures (~300°C) and another at higher temperatures (~700°C)—accompanied by small amounts of methane (CH_4), CO, and CO_2. As the rice husk-to-NaOH ratio increased from 1:1 to 1:3, two key trends emerged:

1. H_2 production at lower temperatures was further enhanced.
2. CO_2 formation at very high temperatures (above 850°C) also increased.

The observed enhancement in low-temperature H_2 production aligns with findings from previous ATT studies,[20] which demonstrated that NaOH promotes H_2 release from biomass at lower temperatures while simultaneously capturing CO_2 as carbonates. The increasing H_2 yield at lower temperatures with higher NaOH concentrations (1:2 and 1:3) can thus be attributed to the ATT effect, where the ATT reaction (Equation (1)) becomes dominant over SG, mirroring behavior seen in other biomass systems.

While the ATT reaction exhibits high selectivity for H_2 production, small amounts of CH_4 indicate that a fraction of the rice husk undergoes thermal decomposition at elevated temperatures. Additionally, CO_2 emissions observed at very high temperatures in rice husk-ATT originate from Na_2CO_3 rather than from the rice husk itself.[21] In the ATT process, CO_2 released from biomass is captured by Na to form Na_2CO_3,[18] preventing direct atmospheric emissions. This is evident in Figures 3C and 3D, which show no CO_2 formation below 700°C. However, at higher temperatures, Na_2CO_3 can react with SiO_2 to form sodium silicates, releasing CO_2 in the process (Equation (4)). This explains the increased CO_2 emissions at high NaOH concentrations, where the ATT reaction dominates over SG and leads to Na_2CO_3 accumulation.[12] These results suggest that the key reactions governing rice husk-ATT are represented by Equations (2) and (4). Compared to SG, ATT offers clear advantages, including higher H_2 yields at lower temperatures and significantly lower CO_2 emissions, reinforcing its potential as a cleaner hydrogen production method.

The theoretical maximum hydrogen yield from 1 g of rice husk is 82.1 mmol based on the proposed ATT reaction stoichiometry. Experimentally, ~50 mmol of H_2 was produced (~60.9% yield), along with ~9 mmol of CH_4, indicating carbon diversion from the ATT pathway. If this carbon had undergone the ATT reaction, an additional 19.2 mmol of H_2 could have been generated, raising the potential yield to 69.2 mmol (84.3%). These results highlight that methane formation suppresses hydrogen production, and improving ATT selectivity is crucial to maximizing H_2 yield. To further investigate carbon distribution, carbon analysis of the rice husk after reaction under ATT at 950°C with a 1:3 rice husk-to-NaOH ratio was conducted. As shown in Figure S3, 72.14% of the initial carbon was converted to inorganic carbon (mainly Na_2CO_3), 22.2% to gaseous carbon (e.g., CH_4), and 5.66% remained as residual organic carbon. This result is in good agreement with

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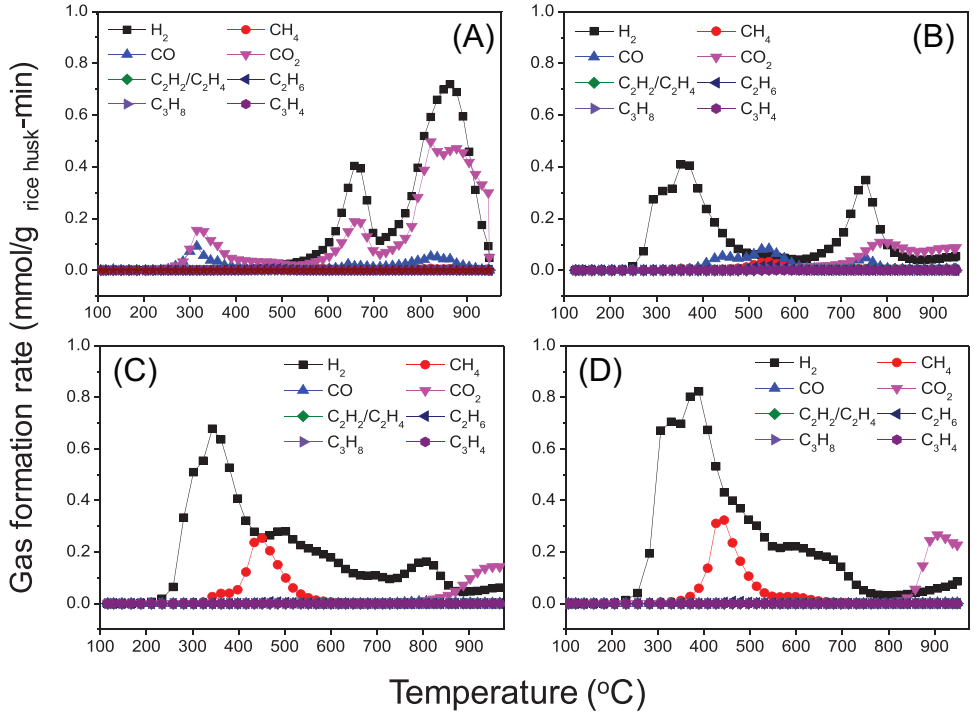


FIGURE 3 Gas formation from rice husk at different alkaline thermal treatment (ATT) ratios as a function of temperature: (A) steam gasification (SG), (B) ATT (1:1), (C) ATT (1:2), and (D) ATT (1:3).

the trends observed in Figure 2 and further emphasizes that suppressing CH_4 formation and enhancing solid–solid contact between NaOH and the silica-rich biomass are key strategies for improving the efficiency of the ATT reaction.

3.2 | Sodium silicate formation in rice husk-ATT

This study demonstrates the simultaneous production of sodium silicate and hydrogen during the ATT process. In this reaction, silica derived from rice husk reacts with sodium carbonate, forming sodium silicates with varying Na_2O-to-SiO_2 ratios,[14] which have high-value applications. Rice husk contains approximately 10.3 wt% amorphous silica, which interacts with Na_2CO_3—a byproduct of ATT—to produce sodium silicate.[16] During ATT, NaOH first reacts with CO_2 released from rice husks to generate Na_2CO_3. The subsequent reaction between Na_2CO_3 and SiO_2 leads to the formation of sodium silicates, as represented by Equation (4).

The specific type of sodium silicate formed depends on the Na_2O-to-SiO_2 ratio, resulting in compounds such as Na_2SiO_3 (Na_2O:SiO_2 = 1:1), $Na_2Si_2O_5$ (1:2), and $Na_2Si_3O_7$ (1:3). Figure 4 presents the composition of inorganic products obtained from rice husk-ATT at 950°C under two conditions: (a) when only rice husk and NaOH are used as reactants and (b) when an additional 1.9 g of SiO_2 is introduced at a rice husk-to-NaOH ratio of 1:3. In Figure 4A, the primary products are ~85.2% Na_2CO_3 and ~10% sodium silicates, with a minor amount of unreacted SiO_2. This confirms that sodium silicates are generated alongside hydrogen (as discussed earlier). Additionally, the substantial presence of Na_2CO_3 indicates that excess SiO_2 is required to drive its complete conversion into sodium silicates. To verify this, an additional 1.9 g of SiO_2 was introduced, and the ATT reaction was repeated at 950°C.

As shown in Figure 4B, when extra SiO_2 was added, the final product contained almost no Na_2CO_3, with ~81.3% sodium silicates and ~18.7% unreacted SiO_2. This confirms that most of the Na_2CO_3 reacted with SiO_2 to form sodium silicates. The average Na_2O-to-SiO_2 ratio in the produced sodium silicates was approximately 1:2, as further validated by ICP-OES analysis (Na:Si molar ratio ~1) in Table S2. These findings demonstrate the successful synthesis of sodium silicate mixtures through the reaction of Na_2CO_3 with SiO_2. However, about one-third of the additional SiO_2 remained unreacted, likely due to mixing limitations between solid reactants or insufficient reaction temperature.

To further investigate the role of excess SiO_2 in rice husk-ATT, we examined its influence on the reaction mechanism. As previously confirmed (Figure 2), SiO_2 can

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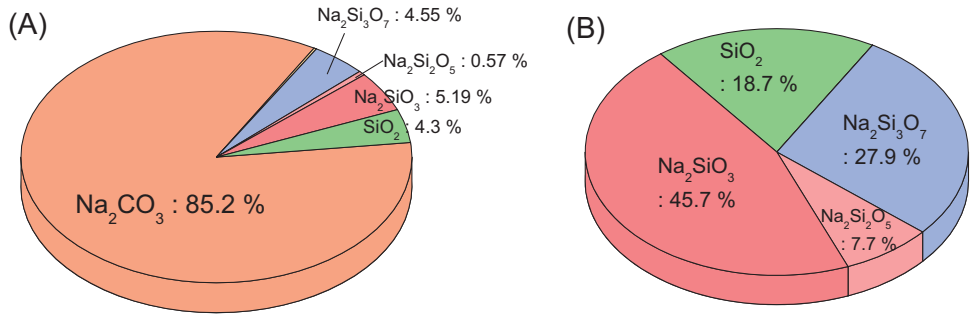


FIGURE 4 Composition of inorganic products from rice husk-alkaline thermal treatment (ATT) at 950°C with a rice husk-to-NaOH ratio of 1:3. (A) Without additional SiO_2. (B) With 1.9 g of additional SiO_2.

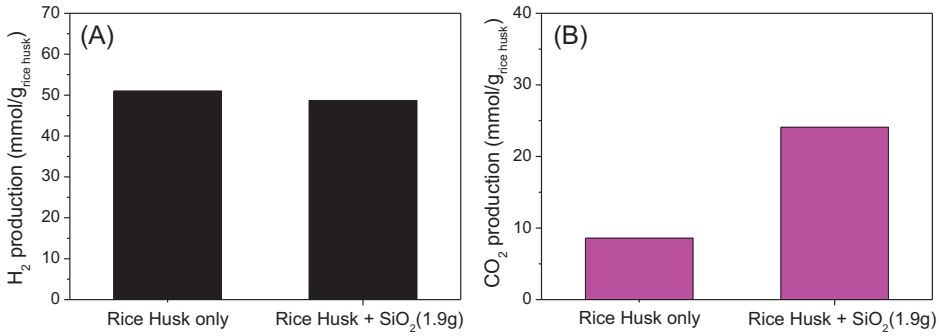


FIGURE 5 Effect of additional SiO_2 on hydrogen and CO_2 formation in rice husk-alkaline thermal treatment (ATT).

react with NaOH at lower temperatures (~200°C) to form Na_2CO_3, though this occurs to a limited extent. However, when a large amount of SiO_2 is introduced—such as the 1.9 g addition—it raises the possibility that this reaction (Equation (3)) could become more dominant than the primary ATT reaction (Equation (2)). To assess the preferred reaction pathway under these conditions, we conducted ATT experiments with excess SiO_2 and analyzed H_2 and CO_2 production (Figure 5).

The results in Figure 5 indicate that hydrogen production remained unchanged despite the addition of SiO_2, while CO_2 emissions increased. In the rice husk only sample, heating to 900°C led to the evolution of a small amount of CO_2, which can be attributed to the partial thermal decomposition of Na_2CO_3. Nevertheless, the majority of Na_2CO_3 remained thermally stable at this temperature and reacted with added SiO_2 to form sodium silicates. This suggests that the additional SiO_2 promotes sodium silicate formation while concurrently releasing CO_2, consistent with Equation (4). Based on this observation, we conclude that Equations (2) and (4) are the dominant reaction pathways governing rice husk-ATT, as initially proposed in the introduction.

4 | CONCLUSION

This study introduces a novel hydrogen–sodium silicate co-production process that simultaneously addresses two critical challenges: sustainable hydrogen generation and high-value material synthesis. By integrating these reactions into a single-step, reduced carbon emission process, this approach eliminates the need for conventional multi-step methods, enhancing both environmental sustainability and economic viability. Utilizing agricultural waste as a feedstock, this method offers a practical and scalable pathway for producing clean hydrogen while repurposing rice husk-derived silica into valuable sodium silicates. Given the increasing demand for renewable hydrogen energy and eco-friendly silica-based materials, the proposed ATT process presents a promising, industrially adaptable solution for sustainable energy and material production. Although nitrogen was employed as a carrier gas for gas analysis in this study, industrial implementation can benefit from using only steam—the actual reactant—as the transport medium. This not only simplifies the system by eliminating the need for external carrier gases but also enhances overall process efficiency and integration.

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