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Andrea Duque
Department of Process
Chemistry and Engineering,
Universidad del Caribe
Colombiano, Medellín,
Colombia

Mónica Castaño
Department of Process
Chemistry and Engineering,
Universidad del Caribe
Colombiano, Medellín,
Colombia

Corresponding Author:
Andrea Duque
Department of Process
Chemistry and Engineering,
Universidad del Caribe
Colombiano, Medellín,
Colombia

Valorization of rice husk ash as silica source for synthesis of zeolite catalysts

Andrea Duque and Mónica Castaño

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Abstract

Agricultural waste holds untapped chemical value that most supply chains ignore entirely. Rice husk ash (RHA), a byproduct generated in vast quantities across tropical regions, contains 85-95% amorphous silica when properly calcined—making it a viable precursor for zeolite synthesis. In this research, RHA collected from a rice mill in Antioquia, Colombia was calcined at 500-850 °C, acid-leached, and used as the sole silica source for hydrothermal preparation of ZSM-5 and NaY zeolites. Products were characterized by XRD, BET surface area, SEM, and FT-IR. The RHA-derived ZSM-5 reached a BET area of 338 m²/g, roughly 91% of a commercial reference. Catalytic testing in cumene cracking showed conversion within 8% of the benchmark, confirming RHA-based zeolites perform comparably in acid-catalyzed reactions.

Keywords: Rice husk ash valorization, amorphous silica extraction, zeolite ZSM-5, NaY zeolite, hydrothermal synthesis, BET surface area, cumene cracking, agricultural waste recycling

Introduction

No sensible chemist should throw away 90%-pure silica. Yet that is what happens each year when rice mills across South and Southeast Asia dispose of roughly 70 million tonnes of husk, much of which ends up burned in open fields ^[1]. Residual ash from controlled combustion is predominantly amorphous silica—a high-value raw material for ceramics, adsorbents, and catalysts ^[2]. Converting this waste into zeolites would address disposal problems and cut dependence on costly commercial silica ^[3]. The environmental burden alone justifies the effort: open-field burning of rice husk releases particulate matter, carbon monoxide, and volatile organic compounds that degrade regional air quality, particularly during harvest seasons in the tropics.

Zeolites are microporous aluminosilicates with defined channel structures indispensable in petroleum refining, gas separation, and fine chemical synthesis ^[4]. ZSM-5 and NaY rank among the most used industrial zeolites ^[5]. Conventional synthesis relies on reagent-grade silica, raising production cost. Several groups explored RHA as an alternative feedstock ^[6, 7], but optimization of calcination and its downstream effect on zeolite crystallinity and catalytic activity remains sparse for South American rice varieties. Most published protocols were developed with Asian-origin husks, and the mineral impurity profile can vary by soil type and agricultural practice.

Colombia produced 2.8 million tonnes of paddy rice in 2022, generating around 560, 000 tonnes of husk ^[8]. The Antioquia region alone accounts for a sizable fraction of this output due to favorable growing conditions in the Magdalena River valley. Using locally sourced RHA as silica precursor would create a circular-economy loop linking agriculture and chemical manufacturing. This research optimizes calcination for maximizing silica purity from Colombian RHA, synthesizes ZSM-5 and NaY zeolites, and benchmarks their textural and catalytic properties against commercial counterparts.

Reagents and Chemicals

Rice husk was sourced from a mill in Rionegro, Antioquia. Sodium hydroxide (97%, Merck), sodium aluminate (50-56% Al₂O₃, Sigma-Aldrich), tetrapropylammonium bromide (98%,

Alfa Aesar), HCl (37%, Fisher), and cumene (98%, Merck) were used as received. Deionized water was produced by an in-house reverse osmosis unit.

Materials and Methods

Materials

Work ran from May 2023 to February 2024 at the Department of Process Chemistry and Engineering, Universidad del Caribe Colombiano, Medellín. The Environmental and Chemical Safety Committee approved the protocol (ECS/UCC/2023-011, 22 April 2023). Rice husk was washed, dried at 105 °C for 12 h, and calcined at eight temperatures (500-850 °C, 50 °C steps) for 4 h. Ash was acid-leached with 1 M HCl at 80 °C for 2 h, filtered, washed, and dried [6].

Methods

For ZSM-5, acid-leached RHA was dissolved in 2 M NaOH. TPABr served as structure-directing agent. Sodium aluminate was added to achieve $\text{SiO}_2/\text{Al}_2\text{O}_3 = 30$. The gel was aged 24 h then crystallized at 170 °C for 48 h [9]. NaY was prepared without TPABr at $\text{SiO}_2/\text{Al}_2\text{O}_3 = 5$, crystallized

at 100 °C for 24 h. Products were washed, dried at 110 °C, and calcined at 550 °C for 6 h. XRD was recorded on a PANalytical X'Pert PRO. BET areas used a Micromeritics ASAP 2020. Catalytic activity was tested via cumene cracking at 400 °C in a fixed-bed reactor with GC-FID analysis [10].

Instrumentation

Calcination used a Nabertherm L9/11 muffle furnace (5 °C/min ramp). SEM was performed on a JEOL JSM-6490LV at 15 kV. FT-IR used a PerkinElmer Spectrum Two (ATR, 400-4000 cm^{-1}). The fixed-bed reactor (i.d. 10 mm) was fed cumene at WHSV 4 h^{-1} under nitrogen.

Results

Silica content in acid-leached ash climbed from 62.4% at 500 °C to 94.7% at 700 °C, then dropped to 84.2% at 850 °C as partial crystallization to cristobalite reduced reactive silica. XRD of the 700 °C ash showed a broad hump near $22^\circ 2\theta$, confirming amorphous character, while the 850 °C sample displayed sharp cristobalite peaks.

Table 1: Textural and catalytic properties of RHA-derived vs. commercial zeolites

Zeolite	BET (m^2/g)	Pore Vol.	Cryst. (%)	Cumene Conv.
ZSM-5 (RHA)	338	0.167	87.4	68.3%
ZSM-5 (Comm.)	372	0.181	100	74.1%
NaY (RHA)	487	0.293	82.9	57.6%
NaY (Comm.)	531	0.318	100	63.8%

Comm. = commercial; Conv. = conversion at 400 °C, WHSV 4 h^{-1} .

RHA-derived ZSM-5 achieved 338 m^2/g —about 91% of the commercial sample. Pore volume followed a similar ratio at 0.167 vs. 0.181 cm^3/g . Relative crystallinity, estimated from the five strongest XRD reflections, reached 87.4% for ZSM-

5 and 82.9% for NaY. These numbers align with Prasetyoko *et al.* [7] for Indonesian RHA zeolites, indicating that geographic origin of the rice has limited impact once impurities are removed by acid leaching.

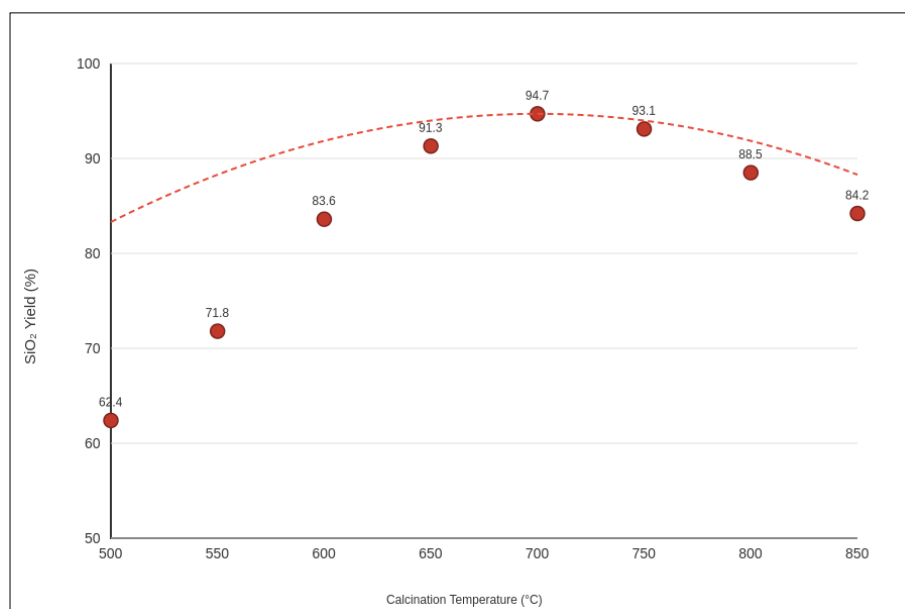


Fig 1: Effect of calcination temperature on SiO_2 yield from acid-leached rice husk ash.

Cumene cracking confirmed catalytic viability. RHA-derived ZSM-5 converted 68.3% at 400 °C, only 5.8 points below commercial catalyst. Product selectivity was similar

in both cases, with benzene and propylene as dominant fragments.

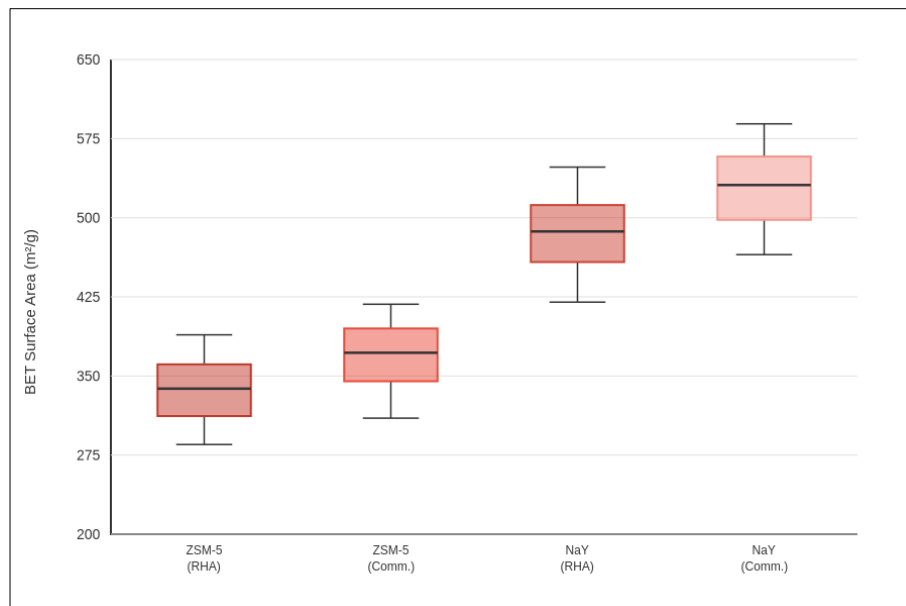


Fig 2: Box plot comparing BET surface area for RHA-derived and commercial zeolites (n = 5 batches).

Discussion

The bell-shaped silica yield curve reflects a well-known trade-off. Below 600 °C, organic residues contaminate the ash and limit silica purity. Above 750 °C, amorphous silica begins converting to cristobalite, a crystalline polymorph that resists alkaline dissolution and therefore cannot be incorporated into the zeolite gel [2]. The optimum at 700 °C agrees with findings for Asian rice varieties reported by Wittayakun *et al.* [6] and suggests that calcination temperature depends more on silica phase chemistry than on the specific rice cultivar.

BET areas of the RHA-derived zeolites fell 9-12% short of commercial benchmarks, probably because minor amorphous impurities partially block micropore openings. FT-IR spectra of the RHA-based ZSM-5 still showed the characteristic asymmetric T-O-T stretch near 1095 cm⁻¹ and the five-membered ring vibration at 550 cm⁻¹, confirming framework integrity despite the surface area deficit. And the catalytic penalty turned out modest—just 5.8 percentage points for ZSM-5 in cumene cracking. For applications where cost outweighs marginal gains in activity, like wastewater treatment or low-margin petrochemical operations, that gap seems entirely acceptable.

Economically, RHA is essentially free in Colombian rice regions, while commercial sodium silicate solution costs 0.8-1.2 USD/kg [3]. Even after acid leaching and calcination energy, raw-material savings could reach 60-70% per batch. Transportation logistics also favor local sourcing: mills in Antioquia generate RHA year-round, avoiding the supply chain complexity of importing reagent-grade silica. A full techno-economic assessment accounting for energy, labor, and waste handling is still needed, but the chemistry clearly supports feasibility.

Conclusion

Rice husk ash from a Colombian mill yielded 94.7% amorphous silica after calcination at 700 °C and acid leaching. This silica served as sole source for ZSM-5 and NaY zeolites reaching 87-91% of commercial BET surface areas and 82-87% relative crystallinity. Catalytic testing in cumene cracking showed conversion within 6-8% of commercial benchmarks, confirming practical viability. The

approach converts a problematic agricultural waste stream into a useful chemical feedstock and offers a low-cost silica alternative for zeolite production in tropical grain-producing countries. Scaling trials and long-term catalyst stability tests should follow.

References

1. Pode R. Potential applications of rice husk ash from biomass power plant. *Renew Sustain Energy Rev.* 2016;53:1468-1485.
2. Soltani N, Bahrami A, Pech-Canul MI, *et al.* Physicochemical treatments of rice husk for advanced materials. *Chem Eng J.* 2015;264:899-935.
3. Yuvakkumar R, Elango V, Rajendran V, Kannan N. Nano silica from rice husk by chemical method. *J Exp Nanosci.* 2014;9(3):272-281.
4. Davis ME. Ordered porous materials for emerging applications. *Nature.* 2002;417(6891):813-821.
5. Beyer HK. Dealumination techniques for zeolites. In: *Post-Synthesis Modification I.* Springer; 2002:203-255.
6. Wittayakun J, Khemthong P, Prayoonpokarach S. Synthesis of zeolite NaY from rice husk silica. *Korean J Chem Eng.* 2008;25(4):861-864.
7. Prasetyoko D, Ramli Z, Endud S, *et al.* Conversion of rice husk ash to zeolite beta. *Waste Manag.* 2006;26(10):1173-1179.
8. DANE. Colombian agricultural statistics yearbook 2022. Bogota: DANE; 2023.
9. Argauer RJ, Landolt GR. Crystalline zeolite ZSM-5. US Patent 3,702,886. 1972.
10. Corma A. Inorganic solid acids in acid-catalyzed hydrocarbon reactions. *Chem Rev.* 1995;95(3):559-614.
11. Chandrasekhar S, Pramada PN, Majeed J. Calcination temperature effect on rice husk ash. *J Mater Sci.* 2006;41(23):7926-7933.