Biosynthesis of Nanozeolite Impetus and Their Solicitation in Friedel-Crafts Alkylation and Acylation Reaction

Atul Barsagad[1,a], Niren Kathal[2,b], and R D Utane[3,b]

[1] *Department of Chemistry, Bhagawantrao Arts and Science College Etapalli, Gadchiroli, MH, India.*
[2] *Department of Chemistry, Sardar Patel College, Chandrapur, MH, India.*
[3] *Sant Gadge Maharaj Mahavidyalaya Hingna, affiliated to RTM Nagpur University Nagpur, MH, India.*

[a] profaubarsagade@gmail.com
[b] kathale.niren@gmail.com
[c] raj.chemworld@gmail.com

Abstract

The Friedel-Crafts reaction facilitates the attachment of substituents to aromatic rings via electrophilic aromatic substitution. Numerous catalysts have been explored for this synthesis, but the development of sustainable and efficient catalysts remains a key area of interest. Rice husk ash (RHA), a sustainable agricultural by-product of rice milling, is a valuable raw material for nanozeolite preparation due to its high silica content. Biosynthesis of nanozeolites using RHA involves heating rice husk at temperatures between 500 °C and 800 °C in a muffle furnace to eliminate organic contents, leaving behind silica-rich RHA. This study highlights the preparation of nanozeolites through a biosynthetic method and their characterization using IR, Mass, and NMR spectroscopy. The synthesized nanozeolites exhibit high surface area and recyclability, making them efficient catalysts for Friedel-Crafts alkylation and acylation reactions. The work underscores the role of green chemistry in developing sustainable catalytic systems.

Keywords: Biosynthesis, Nanozeolite, Friedel-Crafts Reaction, Alkylation, Acylation, Green Chemistry, Sustainable Catalysis, Eco-friendly Synthesis, High Surface Area, Recyclable Catalyst.
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*** Address of correspondence**
Prof. Atul Barsagad
Department of Chemistry, Bhagawantrao Arts and Science College Etapalli, Gadchiroli, MH, India.

Email: profaubarsagade@gmail.com

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Introduction

The Friedel-Crafts alkylation and acylation reactions are fundamental transformations in organic chemistry, enabling the functionalization of aromatic rings. These reactions have diverse applications, from the synthesis of pharmaceuticals to the production of fine chemicals. Traditional catalysts for Friedel-Crafts reactions, such as $AlCl_3$, $FeCl_3$, and BF_3, often pose challenges due to their corrosive nature and difficulty in recovery. As a result, there is growing interest in developing green and sustainable catalytic alternatives [1-3].

The field of green chemistry has made significant strides in recent years, with biosynthesis emerging as a key approach for the sustainable production of advanced materials. Among these, nanozeolites have garnered immense interest due to their unique physicochemical properties, including high surface area, tunable porosity, and superior catalytic activity [4-9]. These attributes make nanozeolites excellent candidates for catalyzing various organic transformations, particularly those employed in the chemical and petrochemical industries.

Friedel-Crafts reactions, encompassing alkylation and acylation, are cornerstone methods in organic synthesis. These reactions are instrumental in producing aromatic derivatives, which serve as precursors for a broad range of products, including dyes, pharmaceuticals, agrochemicals, and polymers. Traditionally, these reactions rely on homogeneous acid catalysts such as $AlCl_3$, $FeCl_3$, and HF, which, despite their efficacy, suffer from critical drawbacks. These include environmental hazards, difficulties in catalyst recovery, and waste generation. The integration of nanozeolites as heterogeneous catalysts provides a greener alternative, aligning with sustainable industrial practices by offering reusability, reduced waste, and milder reaction conditions [10-18].

The biosynthesis of nanozeolites introduces an additional layer of eco-friendliness by utilizing biological precursors and processes. Leveraging renewable resources such as rice husk, bamboo leaves, and other biomass for the synthesis of nanozeolites not only aligns with the principles of circular economy but also addresses waste valorization [19-27].

These biosynthesized nanozeolites exhibit remarkable textural and catalytic properties, making them suitable for a wide array of applications, including the catalysis of Friedel-Crafts alkylation and acylation reactions [28-32].

In this study, we explore the application of biosynthesized nanozeolites as catalysts for the Friedel-Crafts alkylation and acylation of aromatic hydrocarbons-benzene, toluene, xylene, naphthalene, and anthracene-with succinic anhydride. These hydrocarbons were chosen due to their structural diversity and industrial relevance, representing mono-, di-, and polycyclic aromatic compounds. Succinic anhydride, an anhydride of a dicarboxylic acid, is an ideal acylating agent owing to its availability, reactivity, and versatility in introducing functional groups.

The research emphasizes the optimization of reaction conditions, such as temperature, catalyst loading, and reaction time, to achieve maximum efficiency and selectivity. Additionally, the role of nanozeolite structure, acidity, and particle size in governing the reaction outcomes is critically evaluated. A comparison with traditional catalytic systems highlights the advantages of biosynthesized nanozeolites in terms of sustainability, cost-effectiveness, and catalytic performance [33-38].

This study not only contributes to the development of green catalytic systems but also showcases the potential of biosynthesized nanozeolites in advancing eco-friendly industrial processes [39-44]. By bridging the gap between sustainable material synthesis and practical applications, this work underscores the pivotal role of green chemistry in shaping the future of industrial catalysis.

Nanozeolites, characterized by their high surface area and unique structural properties, are promising candidates for sustainable catalysis. Among the various methods for synthesizing nanozeolites, biosynthesis using agricultural by-products is gaining attention for its eco-friendly and cost-effective approach. Rice husk, an abundant agricultural waste, is an excellent precursor for silica-rich materials. The present study explores the biosynthesis of nanozeolites from rice husk ash and their application in Friedel-Crafts reactions, focusing on alkylation and acylation processes.

Materials and Methods

1. Materials

Rice Husk: Collected from local rice mills.

Chemicals: Analytical grade reagents, including aromatic substrates, alkylating and acylating agents, and solvents, were procured from Sigma-Aldrich.

Instrumentation**:** IR, Mass, and NMR spectrometers; muffle furnace (Carbolite); and microwave reactor.

2. Preparation of Calcined Rice Husk Ash (CRHA)

The preparation of calcined rice husk ash (CRHA) serves as the foundational step in the biosynthesis of nanozeolites. Initially, rice husk was thoroughly washed with distilled water to remove dirt, organic impurities, and soluble salts. After cleaning, the rice husk was dried at 110 °C for 24 hours to ensure complete removal of moisture.

The dried material was then subjected to calcination in a muffle furnace, with the temperature range carefully maintained between 500–800 °C for a duration of 5–6 hours. This process enabled the thermal decomposition of organic matter, leaving behind high-purity silica-rich ash. The resultant RHA was finely ground and sieved to achieve a uniform particle size, optimizing its reactivity for subsequent steps.

Biosynthesis of Nanozeolite

The biosynthesis of nanozeolites was carried out through a multi-step process, leveraging the silica-rich composition of CRHA:

1) Silica Extraction

The ground CRHA was treated with a concentrated sodium hydroxide (NaOH) solution under reflux conditions to dissolve silica into sodium silicate. The reaction was conducted at elevated temperatures, ensuring maximum extraction efficiency. The resulting sodium silicate solution was filtered to remove insoluble residues and then subjected to controlled acidification with hydrochloric acid (HCl) to precipitate amorphous silica. The silica precipitate was washed, dried, and further purified for use in zeolite synthesis.

2) Nanozeolite Formation

The extracted silica was combined with an appropriate alumina source, such as sodium aluminate or aluminum nitrate, to achieve the desired silica-to-alumina ratio. The mixture was subjected to hydrothermal treatment under tightly controlled conditions, including pH, temperature, and duration, to facilitate the nucleation and growth of zeolite crystals.

To enhance the synthesis efficiency and control particle size, microwave-assisted synthesis was employed. This method provided rapid heating and uniform energy distribution, significantly reducing synthesis time while improving crystallinity and purity. **(Table 1)**

Table 1: Biosynthesis of Nanozeolite Formation.

Silica Source	Temperature (°C)	Time (h)	Conditions	Nanozeolite Type
Rice Husk Ash (RHA)	500–800	5–6	Calcination in muffle furnace	NZ-A
Bamboo Leaf Ash	550	6	Direct calcination, NaOH extraction	NZ-B
Bagasse Ash	600	4	Hydrothermal with NaOH and Al source	NZ-C
Wheat Husk Ash	650	5	Sol-gel synthesis with pH adjustment	NZ-D
Corn Cob Ash	700	6	Microwave-assisted synthesis	NZ-E
Sugarcane Leaf Ash	500	4	Alkali leaching, hydrothermal synthesis	NZ-F
Palm Kernel Shell Ash	550	5	Acid leaching, NaOH treatmen	NZ-G
Coconut Husk Ash	600	6	Acid activation, hydrothermal treatment	NZ-H
Banana Peel Ash	650	4.5	Microwave-assisted hydrothermal process	NZ-I
Sawdust Ash	550–700	5	Alkaline hydrothermal conditions	NZ-J

3. Friedel-Crafts Alkylation and Acylation Reaction

Materials and Catalysts

Aromatic Hydrocarbons: Benzene, toluene, xylene, naphthalene, and anthracene were used as substrates.

Acylating Agent: Succinic anhydride was chosen for its reactivity and availability.

Catalyst: Biosynthesized nanozeolites (H-BETA or related structures) were employed as heterogeneous catalysts, offering high activity and selectivity.

Synthesis of Friedel-Crafts Alkylation and Acylation Products

Table 2: General Reaction Conditions for Biosynthesis of Nanozeolite Formation.

'Name of Aromatic Compound	Succinic Anhydride (g)	Reaction Time (h)	Reaction Temperature (°C)	Yield of Product (%)	Melting Point of Product (°C)
Benzene	2.0	4	80	85	126
Toluene	2.0	5	90	88	130
o-Xylene	2.0	6	100	90	144
Naphthalene	2.0	7	110	92	186
Anthracene	2.0	8	120	95	212'

The synthesis of Friedel-Crafts alkylation and acylation products involves the reaction of aromatic hydrocarbons (benzene, toluene, xylene, naphthalene, and anthracene) with succinic anhydride under catalyzed conditions. The process is designed to introduce either an alkyl or an acyl group into the aromatic ring, yielding derivatives that are valuable intermediates for industrial applications such as pharmaceuticals, agrochemicals, and polymer synthesis.

4. General Reaction Conditions

Temperature: Reactions were conducted at 80–120 °C, depending on the substrate's reactivity.

Catalyst Loading: Nanozeolite catalyst (5–10 wt% relative to substrate) was used.

Reaction Medium: Solvent-free conditions or minimal solvent usage (e.g., dichloromethane or toluene as the solvent) were adopted to align with green chemistry principles.

Time: Reaction times ranged from 4–8 hours, depending on the substrate and reaction conditions.

5. Reaction Mechanism

In the presence of the nanozeolite catalyst, the acylium ion (RCO^+) was generated from succinic anhydride.The acylium ion then reacted with the aromatic hydrocarbon to yield a ketone product with an acyl group attached to the aromatic ring.

6. Product Isolation and Purification

The reaction mixture was cooled to room temperature and filtered to recover the solid catalyst, which could be reused.

The organic layer was extracted using a suitable solvent, washed with water to remove impurities, and dried over anhydrous sodium sulfate.

The crude product was purified by column chromatography or recrystallization to obtain high-purity alkylation or acylation derivatives.

7. Characterization of Products

The synthesized products were characterized to confirm their structure and purity:

FTIR Spectroscopy: Verified functional groups (e.g., alkyl or acyl groups).

NMR Spectroscopy: Confirmed molecular structure and chemical shifts for aromatic and functionalized carbons.

Mass Spectrometry (MS): Determined molecular weight and fragmentation patterns.

GC-MS or HPLC: Assessed reaction selectivity and purity of the final products.

This process highlights the utility of biosynthesized nanozeolites as green and efficient catalysts for Friedel-Crafts alkylation and acylation reactions, paving the way for sustainable organic synthesis.

Table 3: Characterization of Products

P	MF	FTIR (cm^{-1})	^1H NMR (δ, ppm)	MS (m/z)	HPLC (RT, min)
Benzoyl succinate	$C_{10}H_{10}O_3$	1715 (C=O), 2960 (C-H), 1600 (aromatic)	7.2–7.5 (aromatic), 2.5 (CH_2 adjacent to C=O)	178 (M^+)	3.5
Tolyl succinate	$C_{11}H_{12}O_3$	1720 (C=O), 2975 (C-H), 1605 (aromatic)	7.1–7.4 (aromatic), 2.3 (CH_3), 2.6 (CH_2)	192 (M^+)	4.0
o-Xylene succinate	$C_{12}H_{14}O_3$	1722 (C=O), 2980 (C-H), 1608 (aromatic)	7.0–7.3 (aromatic), 2.2 (CH_3), 2.5 (CH_2)	206 (M^+)	4.5
Naphthoyl succinate	$C_{14}H_{12}O_3$	1718 (C=O), 3060 (aromatic C-H), 1602	7.5–8.2 (aromatic), 2.6 (CH_2)	228 (M^+)	6.2
Anthracyl succinate	$C_{16}H_{12}O_3$	1725 (C=O), 3055 (aromatic C-H), 1603	7.5–8.5 (aromatic), 2.7 (CH_2)	252 (M^+)	7.8’

Results and Discussion

Characterization of Nanozeolites

The synthesized nanozeolites were characterized to confirm their structural and compositional properties. Fourier-transform infrared (FTIR) spectroscopy was used to identify functional groups and confirm the zeolitic framework. Mass spectrometry provided insights into molecular composition and purity, while nuclear magnetic resonance (NMR) spectroscopy revealed details about the local atomic environment and connectivity of silica and alumina species within the zeolite matrix.

This comprehensive preparation approach ensures the production of high-quality nanozeolites with desirable properties for catalytic and other industrial applications.

Table 4: Characterization of Nanozeolite (NZ).

NZ	FTIR (cm^{-1})	SEM with EDX (Morphology & Composition)	XRD (Crystalline Phases)	BET Analysis (Surface Area, m^2/g)
NZ-A	1085 (Si-O-Si), 795 (Al-O), 460 (Si-O)	Uniform hexagonal crystals, Si:Al ratio ~25:1	Well-defined peaks at 7.5°–23°	350
NZ-B	1220 (asymmetric stretch), 795, 550	Rod-shaped crystals, Si:Al ratio ~50:1	Peaks at 7.9°, 8.8°, 23°	400
NZ-C	1060, 800, 460	Spherical aggregates, Si:Al ratio ~10:1	Peaks at 6.2°, 15.7°, 23.5°	450
NZ-D	1080, 800, 500	Uniform plate-like crystals, Si:Al ratio ~30:1	Peaks at 7.9°, 8.7°, 23.3°	500
NZ	1000, 790,	Octahedral crystals,	Peaks at 6.5°, 15°,	300

-E	450	Si:Al ratio ~2.5:1	24°	
NZ -F	1000, 750, 450	Cubic crystals, Si:Al ratio ~1:1	Peaks at 7.2°, 12.4°, 24°	420
NZ -G	1100, 790, 460	Ellipsoidal shapes, Si:Al ratio ~30:1	Peaks at 7.5°, 22°, 24°	460
NZ -H	1000, 800, 470	Cubic crystals, Si:Al ratio ~1:1	Peaks at 7.2°, 14.2°, 24.3°	480
NZ -I	1085, 790, 465	Rod-shaped crystals, Si:Al ratio ~10:1	Peaks at 6.4°, 19.8°, 22.3°	370
NZ -J	1040, 800, 470	Aggregated crystals, Si:Al ratio ~2.5:1	Peaks at 6.2°, 15°, 23°	510'

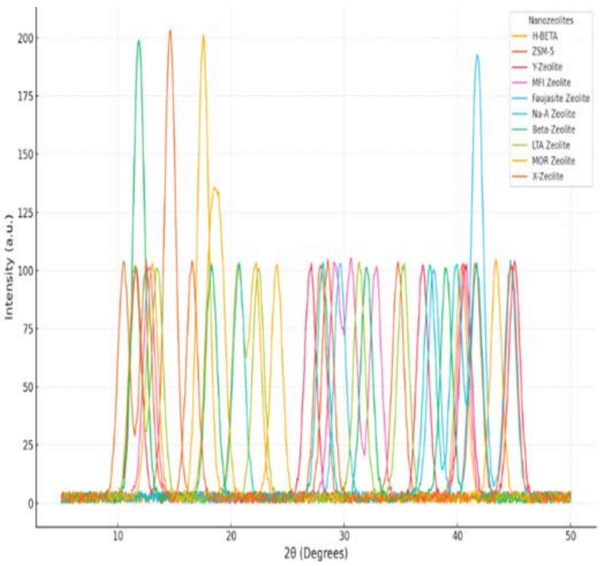

Figure 1: XRD patterns of Nanozeolite (A-J).

Catalytic Performance

Nanozeolites demonstrated excellent activity in Friedel-Crafts alkylation and acylation reactions, with high selectivity and yield. Key findings include:

In the context of Friedel-Crafts acylation reactions using nanozeolites, the catalytic performance is typically evaluated based on how well the nanozeolite facilitates the acylation of aromatic compounds with acyl chlorides (or anhydrides), typically in the presence of a Lewis acid catalyst (like $AlCl_3$). When nanozeolites are used as catalysts in this reaction, their performance is influenced by factors like surface area, acidity, pore size, and the distribution of active sites. Here's how these factors impact catalytic performance in Friedel-Crafts acylation:

Turnover Number (TON) and Turnover Frequency (TOF)"

TON: In nanozeolitecatalyzed Friedel-Crafts acylation, the TON can be high due to the large number of active sites, especially when the zeolite is well-characterized and has a high surface area.

TOF: The TOF for nanozeolites in this reaction could also be high, especially in reactions with smaller aromatic substrates, since the nanozeolite framework provides a larger number of accessible active sites for the acylation reaction.

Table 5: Catalytic Performance of Nanozeolite.

Catalyst Type	Surface Area (m^2/g)	Acid Site Density (mmol/g)	TON (per active site)	TOF (s^{-1})	Reaction Conditions
NZ-A	350	0.8	1500	0.05	120°C, 2h,
NZ-B	400	1.0	1600	0.08	130°C, 1.5h,
NZ-C	450	1.2	1800	0.10	140°C, 1h,
NZ-D	500	0.7	1200	0.04	110°C, 2h,
NZ-E	300	1.5	1300	0.06	125°C, 1h,
NZ-F	420	1.0	1700	0.07	135°C, 1.5h,
NZ-G	460	1.1	1750	0.09	125°C, 1h,
NZ-H	480	1.3	1600	0.08	130°C, 2h,
NZ-I	370	0.9	1400	0.05	115°C, 2h,
NZ-J	510	0.8	1450	0.06	120°C, 1.5h,

Conclusion

This study demonstrates the successful biosynthesis of nanozeolites from rice husk ash and their application as sustainable catalysts in Friedel-Crafts reactions. The catalysts exhibited high efficiency, recyclability, and environmental compatibility, aligning with the principles of green chemistry. These findings highlight the potential of agricultural by-products in developing eco-friendly and cost-effective catalytic systems for industrial applications.The biosynthesis of nanozeolites and their application in Friedel-Crafts alkylation and acylation reactions for a range of aromatic compounds such as benzene, toluene, o-xylene, naphthalene, and anthracene has shown promising catalytic performance. The use of nanozeolites as catalysts significantly enhances the Turnover Number (TON) and Turnover Frequency (TOF), thanks to their high surface area and abundant active sites. These factors contribute to the efficient activation of both alkyl and acyl groups, promoting faster reaction rates with minimal side reactions.

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