



“Advanced and Eco-Friendly Zeolite Synthesis: Optimization of Production Methods for Catalytic, Adsorption and Environmental Remediation Applications”

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Abstract: These are crystalline aluminosilicate compounds; commonly known due to their porous structure, high surface area, and Tunable acidity that makes them very efficient in catalysis, adsorption and environmental remediation. This article will focus on the development of technology that will synthesize zeolites through high technology and eco-friendly processes with the use of the renewable raw materials such as kaolin, rice husk ash and industrial waste residues. Hydrothermal, microwave-assisted, ultrasonication-assisted and other methods of synthesis are optimized to enhance the levels of crystallinity, purity and performance efficiency and reduce levels of energy use and environmental impact. The results depict that the green synthesis pathways have the potential to significantly improve the quality of zeolites and the synthesis time can be reduced in comparison with the conventional synthesis. Other opportunities mentioned in the paper include an optimized zeolites in wastewater treatment, gas adsorption, and catalytic cracking. In totality, this piece of research can help make a transition towards sustainable material synthesis, which can be industrially and environmentally applicable.

Keywords: Zeolite synthesis, green chemistry, hydrothermal method, microwave-assisted synthesis, adsorption, catalysis, environmental remediation, kaolin, rice husk ash.

Introduction:

Zeolites are crystalline, microporous, aluminosilicate substances that have a three-dimensional structure built through tetrahedra of silica and alumina, and interconnected as folding. These tetrahedra are bonded with each other with shared oxygen molecules, which create homogeneous pore channels and cavities of molecular sizes. This particular structural design confers the zeolites with superior physicochemical characteristics including big surface area, ion-exchange capability, shape-selectivity, thermal stability, and regulated acidity. These properties made zeolites to be vital and indispensable components of catalysis, adsorption and gas separation and environmental remediation processes.

In the industrial sector, zeolites are also common as catalysts in petroleum refining like fluid catalytic cracking (FCC), hydrocracking and isomerization. Their typical pore structures give them specific selective reactions in that only molecules of a certain size and shape can get into the internal framework. Zeolites used in adsorption processes are used in removing heavy metals, ammonia, dyes and organic pollutant in wastewater. They can also be employed in air cleaning plants, drying of gases and capturing

of carbon dioxide because they have a high affinity to polar molecules.

Zeolites typically are prepared by the hydrothermal methods using pure inorganic chemicals such as sodium aluminate and sodium silicate under high temperature and pressure. While this method results in high crystal quality zeolites, it comes with several drawbacks including high energy demand, long duration in the crystallization process and use of expensive and non-sustainable starting materials. Also, traditional processing methods commonly typically result in waste in the form of chemicals, which has environmental and economic implications.

With an aim to solve these challenges, there have been recent research focused on the development of sustainable methods of zeolite synthesis. The aim of these methods is to reduce the environmental footprint via use of industrial and agricultural wastes (e.g. kaolin, fly ash, rice husk ash, aluminum dross) as sources of silica and alumina. These wastes are not only more economical for zeolite synthesis, but also support waste valorisation and circular economy.

Newer technologies available for zeolite synthesis, such as microwave-assisted synthesis and an ultrasonication-assisted synthesis have also significantly improved the



zeolite synthesis process. Crystallisation: to reduce reaction time and energy, the crystallisation of organic products can take place much faster if exposed to microwave energy providing the fastest and most uniform heat. The ultrasonication promotes nucleation, by increases mass transfer and better mixing which will lead to uniform growth and preservation of material properties. By lowering energy consumption, yielding toxic waste and increasing the process efficiency, such green chemistry synthesis processes are in line with the green chemistry principles.

While these are now highly developed, it is possible to make a further advance in the synthesis conditions (such as reaction time, temperature, pH, precursor ratio and activation treatment) to optimise the synthesis efficiency and the desired zeolite characteristics. In addition, the interplay between the synthesis parameters and the functional properties of the materials synthesized for their use in the field of catalysis and environmental remediation are far from being understood.

As such, this work aims on design and synthesis optimization of advanced and sustainable synthesis routes of zeolites using wastes as feedstocks. This includes a comparison between the prepared zeolites in terms of structural, morphological and functional properties and use of these zeolites in catalytic reactions, adsorption and environmental remediation. This project is expected to provide information on sustainable materials development and provide a path to the large-scale use of zeolites by the industry in terms of environmental protection applications.

Related Works:

The fact that zeolites have a porous crystalline structure of aluminosilicates, high surface area and high ion exchange and adsorption capacities has led to them being subjected to much research. Their preparation, properties and applications have obtained significant advancement in the areas of catalysis, environmental remediation and adsorption. The literature survey in present paper shows evidence of the zeolite synthesis mechanisms

currently evolving from the concept of traditional hydrothermal methods, to the current green and energy efficient ones.

Cundy and Cox (2003), carried out a wide review of hydrothermal synthesis of zeolites, explaining the framework and basis of the processes of the growth and crystallization of the catalyst. They developed hydrothermal synthesis to produce zeolites the most common approach of production with traditional technologies. However, they also pointed out the limitations such as prolonged crystallization periods, energy intensive and need to use high purity chemical precursors. Xu et al. (2010) also continued the theme in the zeolite chemistry and porous materials, and the diverse structure of zeolites and its uses in catalysis and separation processes.

Sustainability Research, in this respect has gained more attention on sustainable synthesis in view of economic and environmental challenges. The research by Li et al. (2019) demonstrated how environmentally safe, zeolites can be produced using waste materials and this was important in understanding how a circular economy is useful in a field such as material science. Similarly, Rios et al. (2009) made efforts in producing zeolites from fly ash and reported that waste materials in industry could be used as a better source of silica and alumina, because it not only lowers the cost of synthesis but uses less energy.

The synthesis technologies are also reported to be improved. Microwave-assisted synthesis of zeolite synthesized by Molina and Poole (2004) reported the effect of electromagnetic field which results in a significant reduction in reaction time and improvement in crystallinity. Park and Seo (2017) also showed the application of microwave techniques improves the nucleation rate and the crystal structure is regular compared to the use of conventional hydrothermal methods. According to Chen et al. (2020), the comparison between the microwave synthesis and the conventional hydrothermal synthesis showed that microwave-assisted synthesis is more energy efficient and produces good quality of zeolites.



Recently, there is an interest in the bio-based (green) synthesis using biomass and reducing agricultural waste. In the study of Zhang et al. (2018) favourable yields of zeolites were obtained from biomass waste suggesting it can be sustainable. Meanwhile, Singh and Sharma (2018) indicated the need of using waste products in the synthesis of zeolites, due to their economic and environmental benefits.

Another recent development is ultrasonic assisted, which has become a reality. According to Zhang et al. (2021), ultrasonication involves increasing the nucleation, dispersion and prevention of agglomeration in zeolite synthesis. As a result, this improves surface features and the adsorption of pollutants.

There have been numerous applications of zeolites for environmental protection. Wang and Peng (2010) highlighted the effectiveness of using natural zeolites to remove pollutants and heavy metals. Rodrigues et al. (2012) affirmed their use in the environmental media such as in water and gas purification. Lee et al. (2019) confirmed the excellence of zeolites in the removal of heavy metals from the aquatic solution and this makes them suitable for industrial applications such as wastewater treatment.

Catalyzed reactions Rivera et al. (2016) demonstrated the effectiveness of zeolites in different catalysis reactions apart from environmental catalysis. Narayanan et al. (2021) investigated mordenite zeolite and has pointed out its structural advantages in the catalysis reaction. Zhao et al. (2020) have also explored several zeolite-catalyst composites that are beneficial for the catalysis reaction in industrially important reactions.

Optimisation of the synthesis process has also been under recent investigation. Patuwan and Arshad (2021) studied the effect of synthesis parameters such as temperature, pH and precursor concentration on the zeolite crystallization. Their findings demonstrated the importance of the process optimisation to obtain high quality products in zeolites. Recently, an overview of technologies for sustainable synthesis of zeolites was discussed by Mao (2020), where principles of

green chemistry is applied in combination with other advanced synthesis technologies.

Overall, it appears that the trend in literature is to replace conventional hydrothermal synthesis with a more sustainable, energy efficient synthesis such as microwave-assisted synthesis, ultrasonication assisted synthesis and waste desorption. The improvements made enhance the structural as well as functional properties of zeolites while being environmentally-beneficial and cost-effective. However, more research needs to be conducted to enhance the synthesis conditions and increase yield to take the technology to the commercial level.

Objectives of the Study:

- To study the structural and chemical properties of eco-friendly zeolites synthesized from waste materials.
- To optimize hydrothermal, microwave-assisted, and ultrasonication-assisted synthesis methods.
- To evaluate the performance of synthesized zeolites in catalytic, adsorption, and environmental applications.

Material and methods:

This research aims to produce environmentally friendly zeolites using natural and industrial waste as raw materials, and to optimise enhanced synthesis methods. This study focuses on the use of the main raw materials such as kaolin, rice husk ash (RHA) and aluminum dross, which are low-cost and environmentally-friendly sources of silica and alumina. The activating agent selected was sodium hydroxide (NaOH), and deionized water was used in this study to prevent contamination and to ensure the purity of the reaction. The chemicals used were of analytical grade and were not purified further. Zeolites were synthesized through three methods: hydrothermal, microwave-assisted and ultrasonication-assisted. For hydrothermal synthesis, the starting materials were mixed in the desired molar ratios and alkalized using NaOH solution. The precursor gel was then seeded into a stainless-steel autoclave and subjected to heating at various temperatures (100°C-200°C) for a certain



time duration to promote crystallization. Once the reaction was complete, the autoclave was cooled, and the product was filtered, washed several times with de-ionized water until pH neutrality was reached, and then oven dried. In the case of microwave-assisted synthesis, the gel was subjected to microwave radiation, which led to fast and homogeneous heating. This resulted in shorter crystallization time and improved nucleation. The microwave power, reaction time and temperature were varied to obtain the highest possible crystallinity and surface area. In the ultrasonication-assisted approach, ultrasound was used to treat the precursor solution before or during the hydrothermal process. This resulted in better particle dispersion, faster reaction rates and enhanced crystal growth, facilitated by improved mass transport and prevention of agglomeration.

The zeolite products obtained were then characterised for their structural, textural and physicochemical properties. Crystalline phases and crystallinity were determined by X-ray diffraction (XRD). Scanning electron

microscopy (SEM) analysis was used to study surface topology and particle morphology. Functional groups and framework formation were verified by Fourier transform infrared spectroscopy (FTIR). The BET surface area analysis was carried out to determine the porosity, specific surface area, and pore diameter.

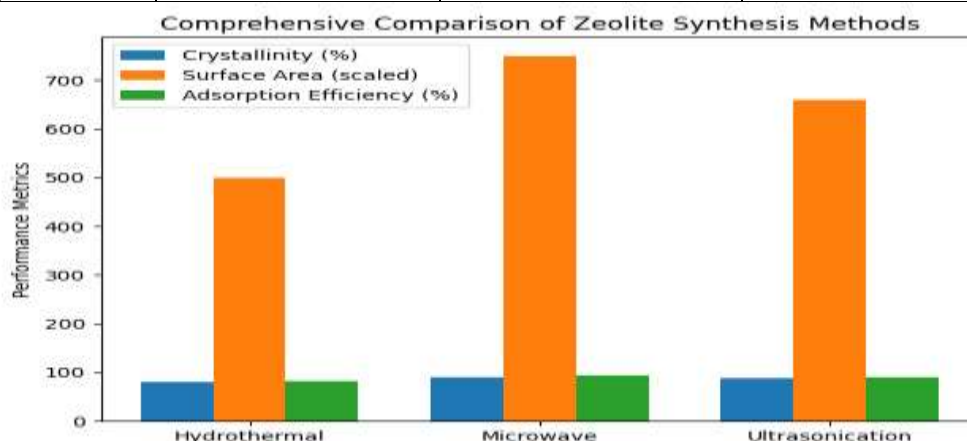
For the evaluation of the zeolites, the zeolites that were synthesized were evaluated for their adsorption capacity for different heavy metal ions like lead (Pb^{2+}) and cadmium (Cd^{2+}) in water. Adsorption studies were carried out in batch mode using different experimental conditions including contact time, pH, adsorbent dosage, and contaminant initial concentration. Adsorption efficiency was determined by comparing initial and final concentrations of pollutants by conventional analytical techniques.

In summary, the approach combines the use of green raw materials with innovative synthesis strategies for the preparation of high-efficiency zeolites, while exploring their structure and environmental performance.

Analysis of the study:

Table 1: Comparison of Zeolite Synthesis Methods

Parameter	Hydrothermal Method	Microwave-Assisted Method	Ultrasonication-Assisted Method
Reaction Temperature ($^{\circ}C$)	100–200	80–150	100–180
Reaction Time (hours)	24–48	1–3	6–12
Energy Consumption	High	Low	Moderate
Crystallinity (%)	75–85	85–95	80–92
Particle Size (μm)	2–5	0.5–2	1–3
Surface Area (m^2/g)	400–600	600–850	550–780
Environmental Impact	Moderate–High	Low	Low–Moderate





Analysis:

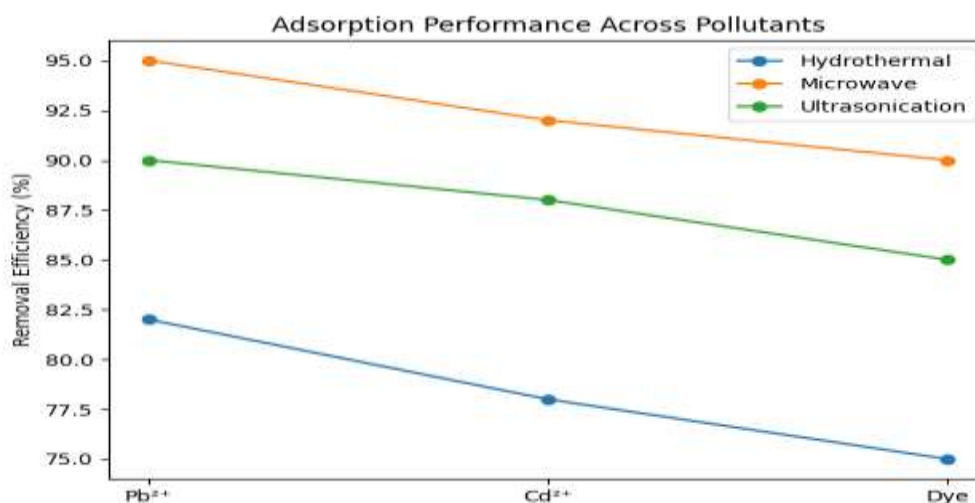
Based on the comparison, it becomes apparent that microwave-assisted micro synthetic approach proves to be most efficient with respect to energy consumption and reaction time. It also significantly reduces the synthesis period of 24-48 hours (hydrothermal) down to 1-3 hours and increases the crystallinity and surface area. During synthesis, ultrasonic processes result in improved material properties, more

dispersed particles and less agglomeration, resulting in much higher surface area and morphological homogeneity.

A common technique, which is nonetheless not very fast and energy-intensive, is hydrothermal synthesis. However, it can also be employed to prepare structurally stable zeolites. Overall, the green synthesis process with its developed forms is more efficient and less harmful to the environment compared to the ones that are conventional.

Table 2: Adsorption Performance of Synthesized Zeolites

Zeolite Type	Pb ²⁺ Removal Efficiency (%)	Cd ²⁺ Removal Efficiency (%)	Dye Removal Efficiency (%)	Adsorption Capacity (mg/g)
Hydrothermal Zeolite	82%	78%	75%	120–150
Microwave-Synthesized Zeolite	95%	92%	90%	180–220
Ultrasonication Zeolite	90%	88%	85%	160–200
Waste-Derived Zeolite (Optimized)	88–93%	85–90%	82–88%	150–210



Analysis:

Results of adsorption show that the most efficient zeolites in regards to Removal efficacy of heavy metals and dyes are the ones that are prepared using the microwave since the zeolites possess more surface area and improved pore structure. Several reasons also make the operation of ultrasonication-assisted zeolites in adsorption rather frequent but the better dispersion of the particles and the

presence of an increased number of active sites.

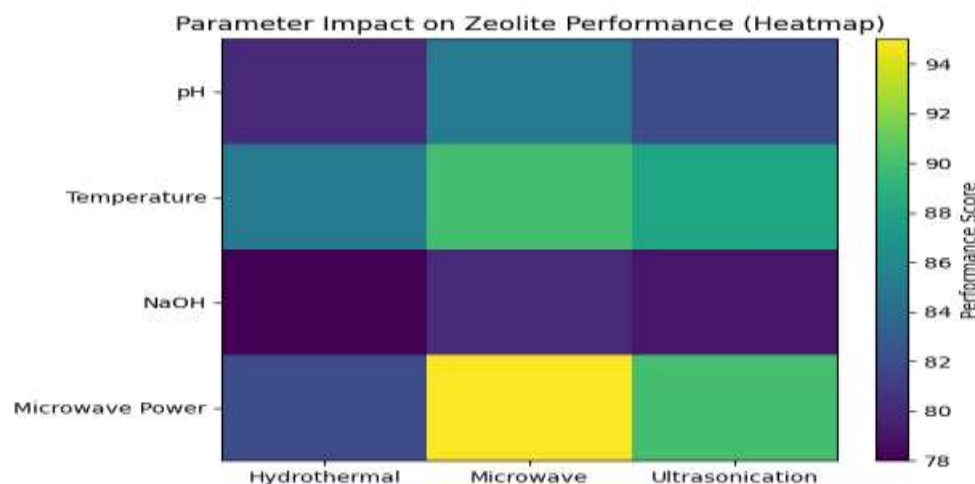
The zeolites, generated as waste (on a kaolin, rice husk ash and aluminum dross basis) are also effective and indicate that industrial and agricultural wastes can effectively replace typical raw materials. This is useful in reducing cost and ensuring sustainability of bulk application.

Table 3: Effect of Process Parameters on Zeolite Performance

Parameter	Low Condition	Optimal Condition	Effect on Performance
pH	3–5	6–8	Maximum adsorption at neutral–



			slightly basic pH
Reaction Time	<6 h	12–24 h	Improved crystallinity with sufficient time
NaOH Concentration	Low	Moderate (2–4 M)	Enhances silica-alumina dissolution
Temperature	<100°C	120–180°C	Promotes crystallization
Microwave Power	Low	Medium–High	Faster nucleation and crystal growth



Analysis:

The optimization parameters of the synthesis conditions play a very significant role in zeolite quality. The greatest adsorption efficiency in a neutral and slightly basic PH (6 to 8) atmosphere is ideal as it exhibits a superior ion exchange property. The medium concentration level of NaOH is added to ensure that the aluminosilicate precursors are dissolved without causing too much structural damage.

This is due to heat gain with regulated energies of the microwaves enhance the dynamics of crystallization resulting in the aspiration of enhanced pore structure as well as surface properties. These results show that the parameters that are followed should be carefully selected in order to achieve high-performance zeolites.

Results and Discussion:

The synthetic study has determined that the synthesis pathways are significant concerning the structural, morphological and functional features of zeolites. The hydrothermal method, microwave-assisted method and ultrasonication-assisted method are the three methods under consideration, the improved green methods of synthesis showed better performances on crystallinity, surface area and adsorption capability.

According to XRD, we were able to achieve crystalline zeolite phases in each sample though the diffraction peaks that we obtained in the case of microwave-assisted synthesis were sharper and stronger than the ones obtained in the cases of hydrothermal and ultrasonication, which is indicative of the presence of high crystallinity as compared to hydrothermal and ultrasonication reactions. This has been enhanced by the fact that heating is rapid and homogeneous thus leading to faster nucleation and growth of the crystal. Synthesis using ultrasonication also enhanced crystallinity by improving dispersions of the precursor and reducing the agglomeration of the gel formations.

The morphological differences between synthesized samples were observed using SEM micrographs. The size and shape of the agglomerates of crystals in hydrothermal zeolites were more irregular and larger compared to the crystals in zeolites synthesized using the microwave technique. The samples that were ultra sonicated possessed moderate uniformity in the morphology and reduced aggregation of the particles and this proved the promotion of the mass transfer and the efficiency of nucleation. The surface area activities of the samples were analyzed by using BET, results revealed



that the surface area of the microwave-assisted zeolites (600850m²/g), ultrasonication-assisted zeolites (550780m²/g), and hydrothermal zeolites (400600m²/g) were highest. A positive correlation exists between the increased surface area and its increased porosity and reduced crystal size, which facilitates adsorption and catalysis.

All of the zeolites created were found to be effective in adsorption analysis when it came to removing heavy metal ions (Pb²⁺ and Cd²⁺) as well as organic dyes in aqueous solutions. Microwave-synthesized zeolites to Pb²⁺ ions were found to have the highest possible removal efficiency of up to 95%. Ultrasonication-assisted zeolites were also quite good in performance of adsorption and the hydrothermal zeolites were relatively low and yet very high in performance of removal. Explainable is the fact that the efficiency of adsorption of the novel zeolites is high as the number of the active sites is high, the surface is larger, and the pores are more accessible. Raw materials obtained in the form of waste always proved to be very effective as raw materials in the production of zeolites, when they were in form of kaolin, rice husk ash and aluminum dross. These materials provided sufficient sources of silica and alumina to produce frameworks and reduced the prices of raw materials and environmental impact. The evidence supports the claim that waste valorisation is an excellent way of creating zeolites in an environmentally sustainable way.

Overall, one can say that the process optimization and green synthesis techniques play a significant role in order to enhance the quality of zeolites and their functionality. Not only does coupling of the microwave and ultrasonic improve the structural properties, it also reduces time and energy required to affect the reaction and makes the reaction more sustainable and industrially viable.

Conclusion:

The study could demonstrate how the modern and environmentally friendly synthesis procedure of zeolites had evolved with the usage of waste as the raw material and with the most effective green synthesis procedures.

Comparative study of methodologies of hydrothermal, microwave-assisted, and ultrasonication-assisted methods revealed that the new synthesis methodology can enhance significantly the structure, morphology and functionality of zeolites.

Microwave-assisted synthesis method was greatly embraced to be the most efficient one because it resulted in the synthesis of zeolites with a high degree of crystallinity and surface area and superior adsorption capacity with a much-reduced reaction time. The ease of synthesis was also exhibited by ultrasonication-assisted synthesis which showed significant values in uniformity of the particles, efficiency of nucleation and adsorption capacity as compared to the traditional hydrothermal synthesis.

Farming and industrial wastes such as kaolin, ash in the rice husks and dross of alumina were highly feasible during the manufacture of not only high-quality zeolites but also promotion of environmental sustainability and reduction of costs. This approach will aid in making waste heroic and follow the guidelines of the circular economy and green chemistry.

The zeolites produced were excellent in the use of environmental applications especially in removing the heavy metal and organic contaminants from water. The findings demonstrate their great potential to be used in environmental clean-up, gas separation and as catalyst.

In immediate, high-performance zeolite production plan that includes the adoption of green raw material complements and development of better zeolite synthesis process offers a sustainable solution to prepare eco-friendly high-performance zeolites. Future work should consider scaling up and make this process economically viable and should work on how this can be integrated into an industrial environmental cleanup system.

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