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Green Synthesis of ZSM-5-Like Material from Rice Husk: Structural and Morphological Characterization

Aasthiya bharathinathan[*1], **Karthikeyan Duraisamy** [2] **Sethuraman Narayanan** [3]
[1] *Research Scholar, Department of Mechanical Engineering, Annamalai University, Chidambaram.*
[2]*Associate Professor, Department of Mechanical Engineering, Annamalai University, Chidambaram,*
[3]*Associate Professor, Department of Mechanical Engineering, IFET college of Engineering, Villupuram*
Corresponding Author, Email: aaasthiyabharathi96@gmail.com

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Abstract
This study proposes the derivation of SiO_2 from rice husk ash and reports the properties of in-house made zeolite ZSM-5, correlated with the commercial one. The rice husk ash is treated with an alkaline solution followed by hydrothermal treatment (850°C). The synthesized samples were characterized by XRF, XRD, FESEM and FTIR. The XRF analysis on the synthesized rice husk ash confirms the presence of 78.36% silica, while the XRD analysis shows the formation of amorphous structure and crystalline form it was observed the presence of crystalline silica in the form of cristobalite. in addition, the field-emission scanning electron microscope (FE-SEM), shows the presence of micro scale shapeless morphology and varying SiO_2/Al_2O_3 molar ratios from 20 to 40, the cubic crystals appears zeolite-like material. Fourier transform infrared (FT-IR) appears to be a technique of the outcome of double-ring tetrahedral vibration and in-house zeolite prepared from the rice husk ash possesses the asymmetric stretching of the Si tetrahedral within the zeolite framework. It is inferred that the extraction of silica from raw rice husk is an economical and environmentally beneficial substitute. In addition, this novel approach proposes the development of green chemical techniques for ZSM-5 production from agricultural byproducts.

1. Introduction

Utilizing renewable resources is becoming more popular as a result of the search for ecologically acceptable and sustainable procedures in material synthesis [1]. A renewable resource, encompasses organic materials derived from plants and animals, including wood, crop residues, and organic waste. Through processes like combustion, gasification, and fermentation, biomass can be converted into heat, electricity, biofuels, and other valuable products, offering a sustainable alternative to fossil fuels while reducing greenhouse gas emissions. Organic materials, such as agricultural residues, food waste, and forestry byproducts, represent valuable resources for sustainable development. With proper management and utilization, these materials can be converted into biofuels, fertilizers, and biodegradable plastics, contributing to resource efficiency and environmental stewardship [2-3]. Agricultural residues serve as valuable resources for sustainable development by offering renewable sources of energy, such as biofuels, and serving as organic soil amendments to enhance fertility and carbon sequestration. Through efficient utilization and management, these residues contribute to reducing greenhouse gas emissions, promoting soil health, and fostering agricultural resilience in the face of climate change [4]. Rice husk waste, a prominent agricultural residue, holds significant potential as a renewable energy source through biomass conversion processes like gasification or combustion. Additionally, its silica content renders it valuable for diverse applications, including as a precursor for synthesizing zeolites, contributing to waste valorization and sustainable resource management[5]. Using this silica to create zeolites is a promising direction for green chemical applications. Zeolites find wide-ranging applications, including catalysis in petrochemical processes, adsorption for water purification, and as molecular sieves in gas separation, owing to their unique porous structure and ion-exchange properties. Their advantages lie in their high selectivity, stability, and environmentally friendly nature, offering efficient solutions for various industrial and environmental challenges while minimizing energy consumption and waste generation. ZSM-5 is distinct from other zeolites since it can be utilized for catalysis, adsorption, and molecular sieving. [6-7]. The unique characteristics of ZSM-5 viz., high surface area, shape-selective catalysis, and Microporous structure, have led to its extensive use in a variety of industrial processes. In petrochemical processes, it is widely used as a catalyst to convert methanol to hydrocarbons, such as olefins and gasoline [8]. Furthermore, its capacity to adsorb molecules selectively according to their size and shape makes it indispensable for separation procedures like the purification of liquid and gas streams [9]. Owing to its ability to adsorb organic contaminants and heavy metals from wastewater, ZSM-5 is employed for environmental recovery. Additionally, ZSM-5 finds application in environmental remediation, as it can effectively adsorb heavy metals and organic pollutants from wastewater.

Its versatility extends to the pharmaceutical industry, where it serves as a catalyst in organic synthesis reactions [10]. Current research focusses towards the creation of environmentally safe processes for ZSM-5 zeolite synthesis, with a particular focus on the use of silica obtained from RHA [11-14]. To reduce their negative effects on the environment and improve sustainability, researchers across the world attempted green synthesis at temperatures ranges between 100 up to 1000°C.To customize the characteristics of ZSM-5 for particular applications, research has looked into a number of factors affecting the synthesis process, including the make-up of the reaction mixture, crystallization settings, and post-synthesis treatments. To clarify the structural and morphological characteristics of the synthesized zeolites, crystalline nature of the synthesized sample (JCPDS no. 42-0024). It can be noted that the intensity of crystalline peak is slightly reduced in modified ZSM-5.[15-16]. Despite significant progress in the green synthesis of ZSM-5 zeolite from RHA-derived silica, several research gaps remain to be addressed. Firstly, there is a need for a deeper understanding of the mechanisms involved in the nucleation and growth of ZSM-5 crystals, particularly under green synthesis conditions. This knowledge is crucial for optimizing synthesis parameters and controlling the properties of the resulting zeolite. Moreover, the scalability of green synthesis routes and their economic viability warrant further investigation to facilitate industrial adoption. Additionally, exploring the potential synergies between ZSM-5 zeolite and other green materials or processes could lead to innovative applications and enhanced environmental benefits.

The increase in use of automobiles has been recognized to have a range of adverse effects on the atmosphere including congestion, noise, accident, increased energy consumption and also air pollution. Various researchers identified reducing air pollution to be a key priority of the future. Prior to now, the pollution brought on by CO, unburned HC, and CO2 received the majority of the attention[17-20]. Recently, it was found that automobile pollution amplified the effect of NOx on health issues. Lean burn petrol and diesel engines are popular mostly because of their fuel efficiency and durability[21-22].

Flyash consisting of SiO_2 and Al_2O_3 with high thermal stability is obtained and it has good catalyst support. Other minor components of metal oxides such as Fe_2O_3, CaO, MgO, K_2O etc., can also be used as effective catalyst components[23-25]. The fly ash is considered as a good catalyst support due to their similarity in composition in volcanic material and precursor of natural zeolite.[26] Ashes produced on burning anthracite or bituminous coal has more silica and alumina and less amount of lime[27]. Ash from lignite or sub-bituminous coal contains more lime than other types of ashes[28]. Due to its high lime concentration, the latter not only exhibits pozzalanic qualities but also possesses cementitious capabilities. Ash with a SiO_2 + Al_2O_3 + Fe_2O_3 content of more than 70 wt% and a high lime concentration has been defined as Class F by the American Society for Testing Materials (ASTM C618), whereas Ash with a SiO2 + Al_2O_3 + Fe_2O_3 content of between 50 and 70 wt% and a high lime content is categorized as Class C. Precisely, burning of low-rank coals produce high calcium class C fly-ashes with cementitious properties[29-31].

2. Materials and Methodology

Raw Rice husk was obtained from a rice mill near Annamalai University, Chidambaram, İndia. Sodium hydroxide (NaOH pellets) and Aluminum oxide (Al_2O_3) were purchased from Spectrum reagents and chemıcals Pvt Ltd Cochin. Tetrapropyl ammonıum bromıde (TPABr: $C_{12}H_{28}BrN$)was purchased from (Hi Media Laboratories Pvt Ltd,Mumbai,India) was used as a structure- directing substance and Hydrochloric acid (HCL) (35 %, Merck, India) was utilized to eject the minerals impurities from the rice husk. and distilled water were used as a starting materials in the initial mixture for the synthesis of ZSM-5 zeolite.

Raw rice husks (RRH) were sieved to eliminate clay and dust particles. The sieved RRH was then soaked in 10M of concentrated HCl Solution. Subsequently, the mixture underwent washing, filtering, and air-drying, followed by calcination at 850°C for 5 hours. 50g of acid treated rice husk and 30 g of NaOH were dissolved in 500ml of distilled water to obtain solution A. Solution A was stırred contınuously for 4hours at room temperature. In addition, 10g of TPABr and 30g of sodium aluminate ($NaAlO_2$) was suspended in 500ml of distilled water to obtain solution B. The Solution B was stırred contınuously for 4hours at room temperature. The solutıon A and B was mixed and stirred contınuously for 4hours at room temperature. An aluminosilicate gel was formed .the stirring was continued for 24 hours at room temperature for the dispersion of the solution. The above mentioned dispersed solution was transferred into Teflon line stainless steel autoclave. The autoclave was placed in muffle furnace and heated at 180° C for 24 hours crystallization after this autoclave was cooled and the reaction mixture was filtered, washed several time with copious amount of distilled water until pH had reached 7, dried overnight at 110° C for 12 hours and heated at 550° C for 6 hours in muffle furnace. The TPABr is used for structural directing agent and NaOH was used to get Na form of ZSM-5 zeolite.

2.1 Synthesis Process:

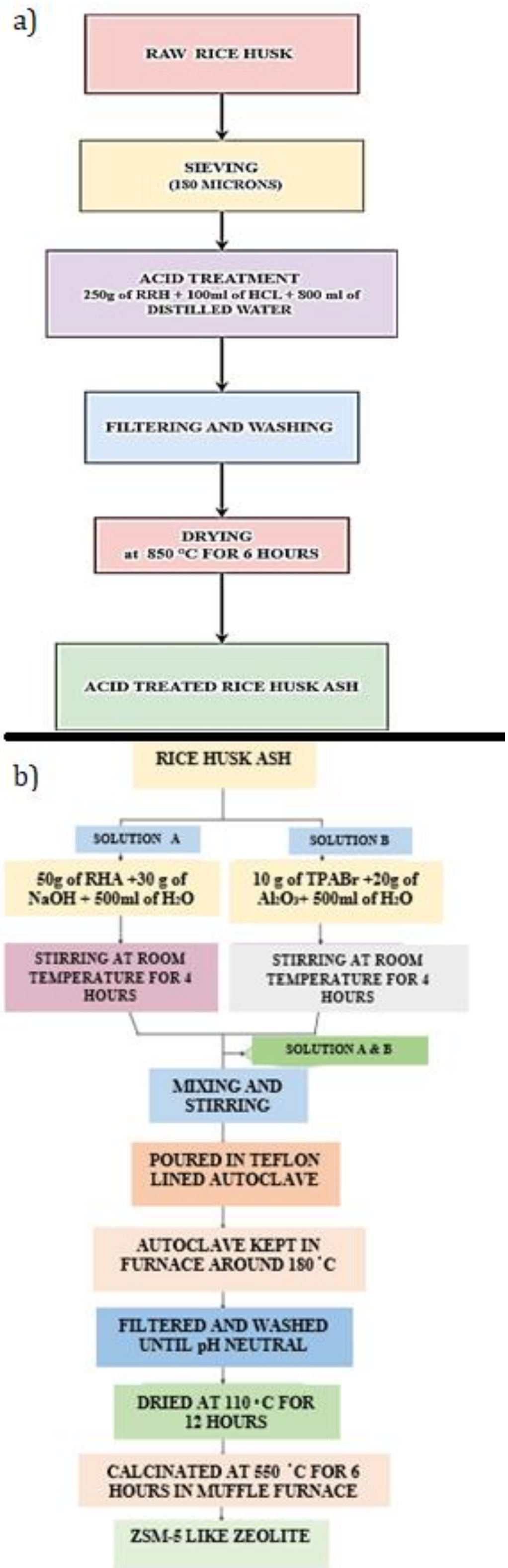


Figure 1 a) and b) synthesis of Rice husk ash and synthesis of ZSM-5 like zeolite from Rice husk ash

From the figure1 reveals that the synthesis of raw rice husk and ZSM-5 rice husk. The following are the details

a) Acid Treated Rice Husk Ash Processing

Raw Rice Husk: The entire process starts with taking the raw rice husk itself as the source material.

Sieve (180 microns): Rice husk is sieved by taking 180 microns as the size of the mesh for maintaining evenness in the size of the particles.

Acid Treatment :

250g of sieved rice husk is subjected to a mixture of 100 mL HCl and 500 mL distilled water.

Filtration and Washing: The mixture, after the treatment of acid, is filtered and washed.

Drying: The material, after being treated, is dried at 850°C for 6 hours.

Acid Treated Rice HusK Ash: The product obtained after this process is acid-treated rice husk ash.

b) Process for Synthesizing ZSM-5 Like Zeolite from Rice Husk Ash

Rice Husk Ash: Acid-treated rice husk ash obtained from process (a) is used in this process.

Solution A:

In this, 50 g of RHA was added to 30 g of NaOH in 500 mL of water. The solution was stirred at room temperature for 4 hrs.

Solution B: 10 g of TPABr [Tetra-propyl ammonium bromide] and 20 g of Al_2O_3 were added in 500 mL of water. This was also stirred at room temperature for 4 hrs. Mixing Solutions A & B: These two solutions were mixed together by stirring.

Poured into Teflon-lined autoclave: The mixture is poured into a Teflon-lined autoclave.

Autoclave kept in a furnace at 180°C: The autoclave is placed inside a furnace, heated to 180°C.

Filtered and washed until pH neutral: The heated mixture is filtered and washed up to pH neutral.

Dried at 110°C for 12 h: The filtered material is dried at 110°C for 12 hours.

Calcination at 550°C for 6 Hours: Finally, calcination is carried out at 550°C in a muffle furnace for 6 hours. The final product obtained is a ZSM-5 like zeolite.

2.2 CHARATERIZATION

The synthesized in-house made ZSM-5 like zeolite obtained from the rice husk ash, was characterized by the following techniques:

XRF (X-Ray Fluorescence): The chemical composition was determined by X-ray fluorescence spectroscopy (XRF (Bruker S8 Tiger).

XRD (X-Ray Diffraction): X-ray Diffraction (XRD) was performed using an ARL EQUINOX 3000 X with a Cu-Ka radiation source operating at 40 kV and 30 mA in a scanning range of 5–115 (2θ).

FESEM (Field -Emission Scanning Electron Microscopy):

The microstructure of extracted ZSM-5 like zeolite (in house made) was investigated with Carl ZEISS -SIGMA

300 Field Emission Scanning Electron Microscopy equipped with EDAX.

FTIR (Fourier Transform Infrared Spectroscopy): Functional groups within the ZSM-5 zeolite structure were identified by FT-IR (Bruker Alpha II, wavelength 350 to 8000) using KBr as a medium. IR spectra were scanned in the range of 400 cm^{-1}– 4,000 cm^{-1} with a resolution of 4 cm^{-1}.

3 RESULT AND DISCUSSIONS:

3.1 X- Ray Fluorescence (XRF)

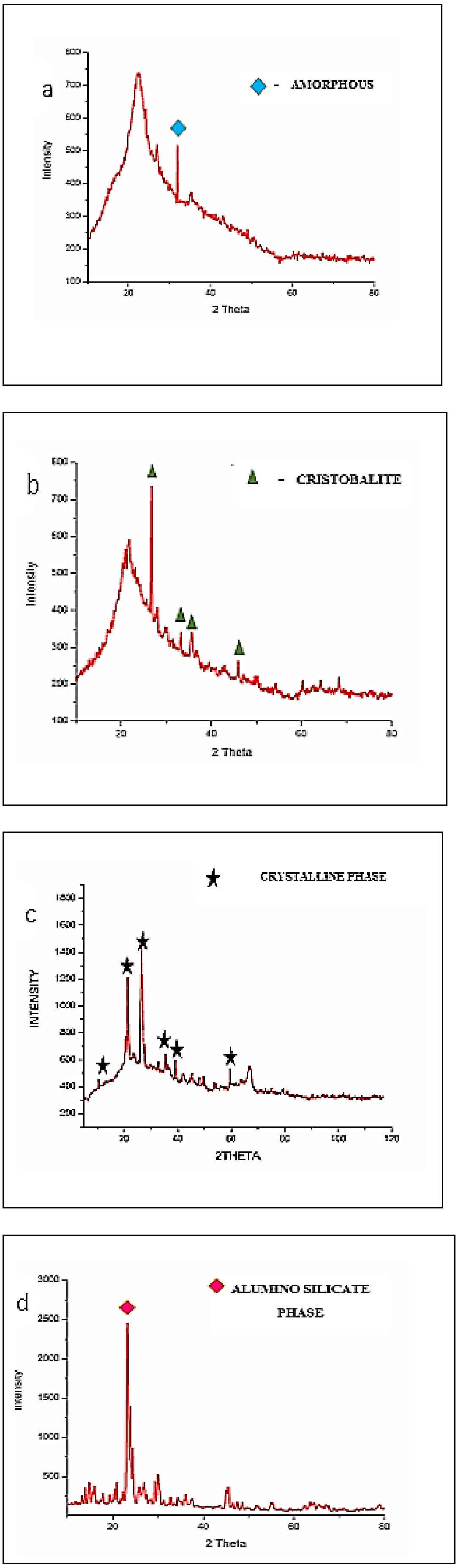
The below provides the elemental composition of a sample. The table 1 lists various oxides (compounds of oxygen with other elements) and their respective percentages in different categories: Raw rice husk (RRH), Rice husk ash (RHA), in-house made, and Commercial ZSM-5. The acıd treated rıce husk ash represents a hıgher weıght of SiO_2 (78.36%) compared to RRH (59.57%). The weight of Al_2O_3 is found to be higher (3.58% to 6.41%) due to the application of a structural directing agent.

Table:1 Elemental composition of Raw rice husk (RRH), Rice husk ash (RHA), in house made and Commercial ZSM-5 (weight %)

METAL OXIDES	RRH %	RHA %	IN HOUSE MADE ZSM-5%	COMMERCIAL ZSM-5 %
SiO_2	59.57	78.36	71.91	76.10
Al_2O_3	3.58	4.47	6.41	7.20
Na_2O	0.58	10.37	14.2	16.20
P_2O_5	18.85	3.45	0.90	0.00
MgO	4.59	2.47	1.31	0.00
K_2O	6.86	0.45	0.95	0.00
CaO	1.95	0.24	0.21	0.00
TiO_2	0.24	0.10	1.11	0.00
Fe_2O_3	0.14	0.07	0.00	0.00
Cl	0.53	0.03	0.00	0.00
SO_3	3.08	0.00	0.00	0.00
MoO_3	0.02	0.00	0.00	0.00

3.2 X-ray diffraction (XRD)

The figures illustrate the evolution of XRD diffractogram for RHA, RRH, and in-house made and commercial ZSM-5. While the XRD diffractogram of RRH shows fig4a) a broad peak between 2θ values of 27° and 57°, indicative of an amorphous phase, RRH exhibits distinct peaks such as a broad hump and predominant peaks. This suggests a more ordered atomic arrangement in RHA. A prominent peak at 2θ = 22° in the RRH diffractogram corresponds to the cristobalite phase, indicating poor long-range order.

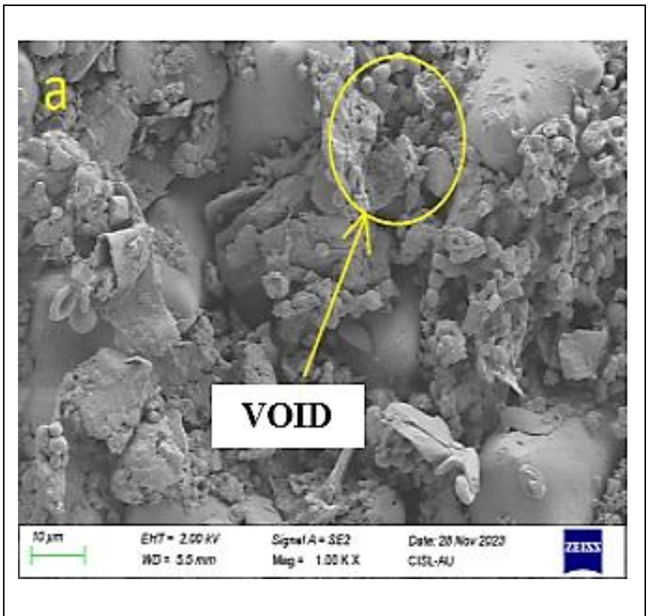


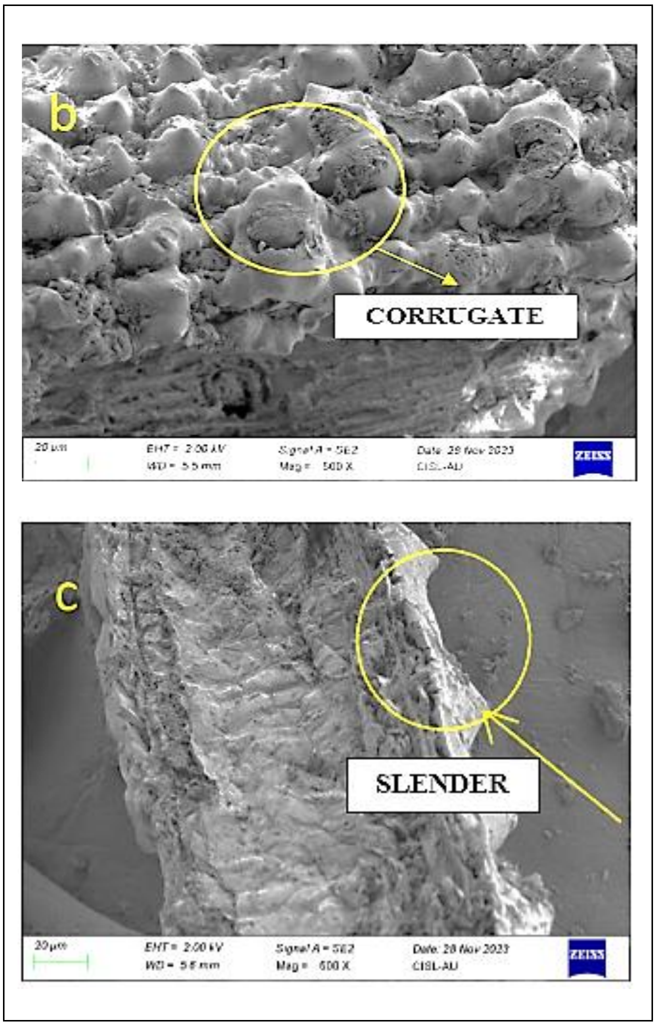
The XRD analysis in Figure 4 reveals the structural characteristics of (a) raw rice husk, (b) rice husk ash, (c) in-house synthesized ZSM-5, and (d) commercial ZSM-5.

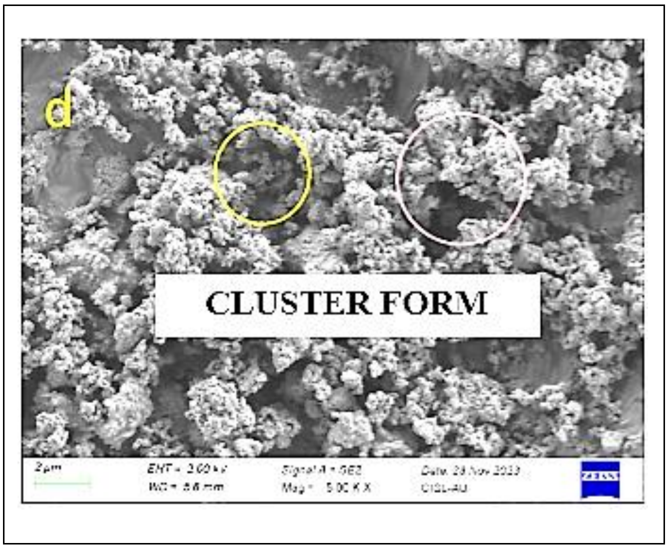
Rice husk ash (RHA) fig4b) presents additional XRD data at specific diffraction angles. Sharp peaks appear at 2θ values of 22°, confirming the presence of crystalline silica in the form of cristobalite. This phase is known to form when rice husk is calcined above 800°C. Small, sharp peaks at 2θ values of 35° and 37° suggest the early formation of the ZSM-5 crystal structure. The presence of these peaks depends on the early formation of the ZSM-5 phase provided XRD diffractogram is indicative of fig: 4c) a highly crystalline in-house made-ZSM-5 material. The presence of sharp peaks at specific 2θ angles denotes the initiating crystalline material, despite of increasing the SiO2/Al2O3 ratio from 80 to 400, promoting the crystalline phase, a crystalline material developed, while the relative peak intensities suggest a well-crystallized and oriented sample. The low background intensity further indicates a high-quality material with minimal impurities. This XRD diffractogram is consistent with the expected diffraction pattern for in-house made ZSM-5, suggesting successful synthesis of the in-house material. The XRD pattern of fig: 4d) provided for the commercial ZSM-5 sample exhibits a series of sharp, well-defined peaks, indicative of a highly crystalline material. These peaks are characteristic of the ZSM-5 structure, confirming its presence in the sample. The relative intensities of the peaks suggest a well-oriented crystal lattice, and the absence of significant background noise indicates a high purity of the material. This XRD pattern is consistent with the expected diffraction pattern for commercial ZSM-5, confirming the successful identification of the material.

3.3 Field emission scanning electron microscopy (FE-SEM)

FESEM analysis was conducted to examine the surface morphology, particle shape, and structure of raw rice husk, rice husk ash, and both in-house and commercially produced ZSM-5. As depicted in Figure 5a, raw rice husk exhibits an efficient structure with a compacted and clustered shape on its surface. This arrangement leads to the presence of numerous voids.





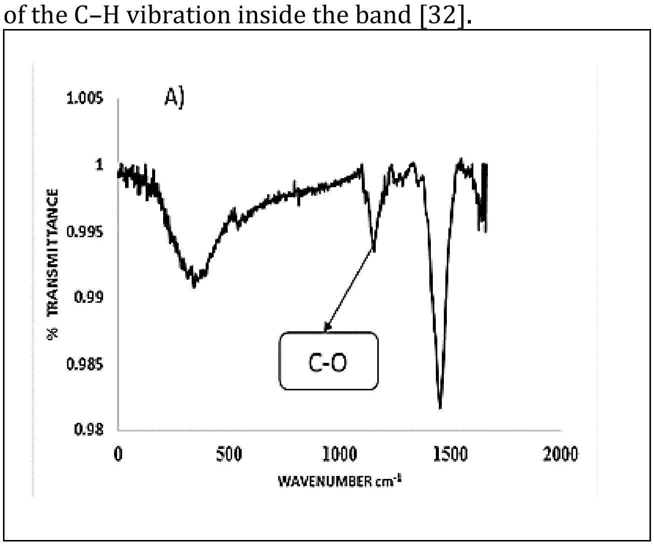


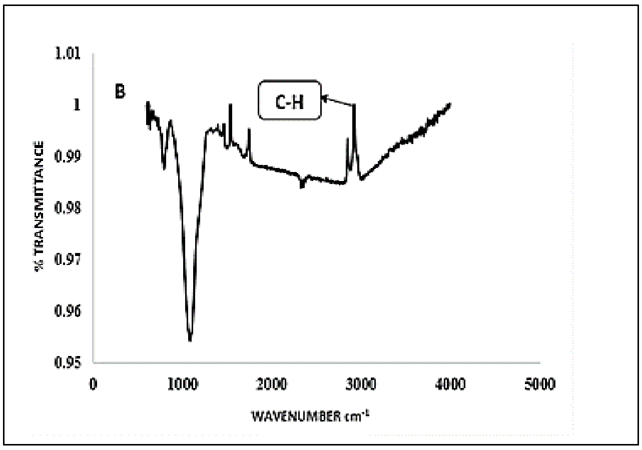
The FESEM analysis in Figure 5 reveals the surface morphology and microstructure of (a) raw rice husk, (b) rice husk ash, (c) in-house synthesized ZSM-5, and (d) commercial ZSM-5.

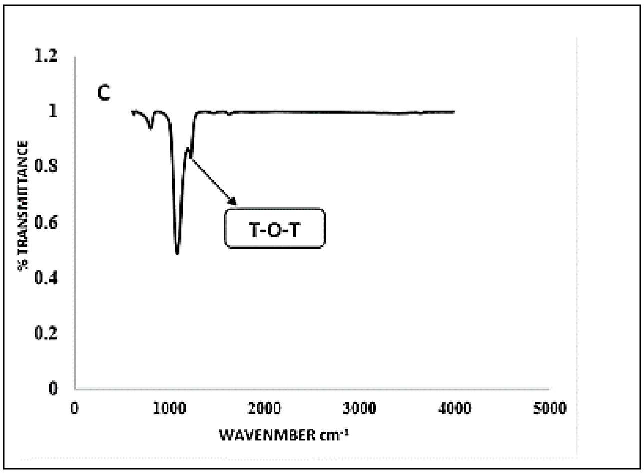
FESEM analysis was conducted to examine the surface morphology, particle shape, and structure of raw rice husk, rice husk ash, and both in-house and commercially produced ZSM-5. As depicted in Figure 5a, raw rice husk exhibits an efficient structure with a compacted and clustered shape on its surface. This arrangement leads to the presence of numerous voids. The silica extracted from rice husk, referred to as RHA, underwent carbonization at approximately 850°C for six hours. Following carbonization, the transformed rice husk displayed a cross-sectional. When analyzed using XRD, rice husk ash exhibits a structural resemblance to plant stem anatomy while maintaining its original amorphous structure. The structural composition of rice husk ash figure: 5b reveals discernible patterns within the stem, characterized by elongated, linear configurations. Figure 5c presents FESEM micrographs of an in-house-made ZSM-5 zeolite-like material with varying SiO_2/Al_2O_3 molar ratios. As the SiO_2/Al_2O_3 molar ratio increases from 20 to 40, the cubic crystals of the zeolite-like material also grow larger. Synthesis parameters, such as temperature, time, and alkalinity, significantly influence the formation and crystallinity of the zeolite product. Additional characterization techniques, like XRD and FTIR, can provide further insights into the structural properties of the synthesized material. Commercial ZSM-5, as shown in Figure 5d, exhibits multiple crystalline particles clumping together to form larger aggregates. These aggregates are characterized by well-defined hexagonal or cubic shapes with distinct facets. Commercial ZSM-5, a Microporous aluminosilicate, often exhibits a similar morphology with crystalline particles forming aggregates. Its unique structure, characterized by a 10-membered ring pore system, makes it a valuable catalyst in various industrial processes such as hydrocarbon isomerization, alkylation, and methanol-to-hydrocarbons conversion.

3.4 Fourier Transform Infrared Spectroscopy (FTIR)

The FT-IR spectrum of the raw rice husks is presented in Fig.6a. The spectra show multiple prominent bands at 455, 1000, and 1500 cm^{-1} are attributed to in-plane stretching, or symmetric stretching, of the Si-O and C-O vibrations. Molecular water or hydroxyl-related bonds can be linked to the distinct vibration modes seen at 1200, 1500, and 4500 cm^{-1} in the rice husk ash spectrum Fig. 6b, which are attributed to both symmetric and asymmetric stretching of the C–H vibration inside the band [32].







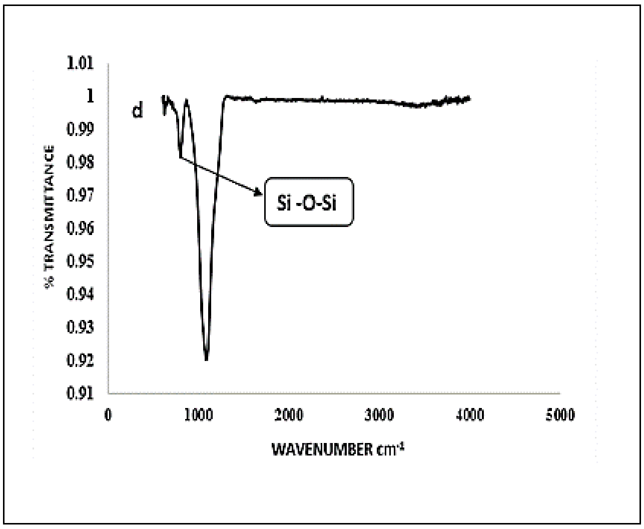


Figure 6 presents the FTIR spectra of (a) raw rice husk, (b) rice husk ash, (c) in-house synthesized ZSM-5, and (d) commercial ZSM-5.

In hydrothermal treated of in house made ZSM-5 fig3c, the peak around 980 and 1000cm^{-1} appeared Si-O-Si functional group. A symmetric stretch is seen at 980 and 1000 cm^{-1} in the bending vibration of adsorbed T-O-T (T=Si, Al) in the House made. The MFI-structured zeolite seen in Fig. 6c is the outcome of double-ring tetrahedral vibration and asymmetric stretching of the Si tetrahedral within the zeolite framework [32]. Furthermore, (Fig.6d) the bands at 1102 and 848 cm^{-1} highlight the crystalline ZSM-5 phase's unique properties. These bands correlate to the symmetric

Si−O−Si stretching vibration linked to the five-membered ring. Hence, in the pentasil unit of the MFI framework, asymmetric Si−O−Si stretching vibration of the two five-membered rings .

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Conflıcts Of Interest

The authors declare no conflicts of interest.

Authors Contrıbutıon

Aasthiya B: conceptualization, methodology, investigation, resources, writing- original draft. **Dr.D.Karthikeyan:** review & editing. N Sethuraman: review & editing

CONCLUSION

The green synthesis of silica from rice husk ash leads to the following salient conclusions.

1. The in-house made ZSM-5 extracted from rice husk, exhibit properties similar to the commercial zeolite. By proper treatment, rice husk can be utilized instead of commercial silica.
2. XRD analysis revealed both amorphous and crystalline structures. Crystalline silica was observed in the form of cristobalite.
3. FE-SEM analysis revealed micro-scale shapeless morphology and varying SiO_2/Al_2O_3 ratios.
4. Fourier Transform Infrared (FT-IR) spectroscopy analysis indicated double-ring tetrahedral vibration and in-house zeolite exhibited asymmetric stretching of Si tetrahedral.

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