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Research Paper

SUSTAINABLE ZEOLITE BETA FROM FLY ASH: A GREEN PATHWAY TO CATALYTIC MATERIALS

Anil N.Kalyankar

Department of Physics, Bahirji Smarak Mahavidyalaya, Basmathnagar Dist.Hingoli
Maharashtra India -431512

Email id : kalyankaranil@gmail.com

Abstract

Zeolite Beta, a high-silica aluminosilicate with the BEA framework, was successfully synthesized from coal fly ash using an alkali fusion–hydrothermal method assisted by tetraethylammonium hydroxide (TEAOH) as a structure-directing agent. The fusion of fly ash with sodium hydroxide at 550 °C enabled the breakdown of the aluminosilicate glass phase, and subsequent hydrothermal treatment at 150 °C yielded highly crystalline zeolite. X-ray diffraction (XRD) confirmed the formation of the BEA framework with characteristic peaks at 7.6° and 22.5°. Fourier transform infrared (FTIR) spectroscopy revealed asymmetric Si– O–Al stretching, symmetric T–O vibrations, and double-ring features typical of Zeolite Beta. Scanning electron microscopy (SEM) showed irregular intergrown crystallites forming polycrystalline aggregates, while energy-dispersive spectroscopy (EDS) confirmed the dominance of Si, Al, O, and Na, with minor Ca and Fe impurities from fly ash. The study demonstrates that fly ash can be effectively converted into Zeolite Beta, thus coupling industrial waste utilization with the production of high-value catalytic materials.

Keywords

Fly ash, Zeolite Beta, Hydrothermal synthesis, Characterization, Sustainable materials

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Introduction

Zeolite Beta is a high-silica aluminosilicate belonging to the *BEA* framework family, first reported in the 1960s, and is considered one of the most versatile zeolites due to its unique structural and catalytic features [1]. It consists of a three-dimensional framework with interconnecting 12-membered ring channels (0.66 × 0.67 nm and 0.56 × 0.56 nm), providing a large pore volume and high surface area. Unlike small-pore zeolites such as Zeolite A, Beta zeolite possesses a medium-to-large pore size and a relatively high Si/Al ratio, which imparts excellent hydrothermal stability, tunable acidity, and shape selectivity. These properties make it particularly attractive for a wide range of industrial catalytic processes, including hydrocracking of heavy oils, isomerization of alkanes, alkylation of aromatics, skeletal isomerization of n-butenes, and selective oxidation reactions. Moreover, its high external surface area enhances diffusion of bulky reactant molecules, a limitation observed in many other zeolite frameworks [2].

Traditionally, Zeolite Beta is synthesized under hydrothermal conditions using expensive chemical precursors such as sodium aluminate and tetraethyl orthosilicate, along with organic templates like tetraethylammonium hydroxide (TEAOH). While these methods yield high-quality crystalline products, they are costly, resource-intensive, and less sustainable. On the other hand, coal fly ash, a waste product generated in large quantities by thermal power plants, is an abundant and inexpensive source of reactive silica and alumina [3]. Globally, millions of tons of fly ash are produced annually, and improper disposal creates significant environmental problems such as soil contamination, groundwater pollution, and air quality degradation. Utilizing fly ash as a precursor for zeolite synthesis not only offers an effective strategy for solid waste management but also reduces the dependency on costly chemical reagents.

In recent years, several studies have focused on the conversion of fly ash into value-added zeolites such as Zeolite A, X, P, and ZSM-5. However, reports on the synthesis of Zeolite Beta from fly ash are relatively limited, primarily due to the complex crystallization mechanism and the requirement for structure-directing agents [4]. Successful synthesis of Zeolite Beta from fly ash is of great significance, as it combines the dual advantages of waste valorization and the production of a high-performance catalytic material. Furthermore, Zeolite Beta derived from fly ash has potential applications not only in petroleum refining and petrochemical industries but also in emerging fields such as environmental catalysis, CO_2 capture, adsorption of volatile organic compounds (VOCs), and water treatment.

Thus, the present study aims to synthesize Zeolite Beta from fly ash via an alkali fusion–hydrothermal method using TEAOH as a template, followed by detailed structural and morphological characterization. The work demonstrates the feasibility of converting industrial waste into a functional porous material with significant catalytic potential, thereby contributing to sustainable material development and circular economy practices.

Experimental Methodology

Fly ash was first subjected to an alkali fusion process in which it was mixed with solid sodium hydroxide pellets in a weight ratio of 1:1.2 and heated at 550 °C in a muffle furnace (Thermo Scientific, USA) for 2 hours to break down the aluminosilicate glass phase into soluble species. The fused mass was then cooled, ground, and dissolved in deionized water to obtain a reactive gel solution [5]. To direct the crystallization of the Beta framework, tetraethylammonium hydroxide (TEAOH) was added as a structure-directing agent (template), and the mixture was stirred vigorously to ensure homogeneity. The gel was transferred into a Teflon-lined stainless-steel autoclave (Parr Instrument, USA) and subjected to hydrothermal synthesis at 150 °C for 48 hours under static conditions. The solid product was recovered by centrifugation (REMI, India), washed with deionized water until neutral pH, and dried at 100 °C for 12 hours, yielding Zeolite Beta powder [6], [7].

Characterization Techniques

The synthesized material was characterized to confirm the formation of the Beta framework and evaluate its morphology. X-ray diffraction (XRD, Bruker D8 Advance, Germany) with Cu-Kα radiation was used to analyze crystallinity, with typical low-angle reflections around 2θ = 7.6° and 22.5° confirming the BEA structure. Fourier transform infrared spectroscopy (FTIR, PerkinElmer Spectrum Two, USA) was performed in the 400–4000 cm^{-1} region, where absorption bands corresponding to asymmetric stretching of Si–O–T (T = Si or Al) at ~1080 cm^{-1} and double-ring vibrations at ~570 cm^{-1} were observed, indicating successful framework formation [8]. The surface morphology was examined using scanning electron microscopy (SEM, JEOL JSM-7610F, Japan), which revealed irregular polycrystalline aggregates typical of Zeolite Beta. Elemental composition was determined using energy-dispersive X-ray spectroscopy (EDS, Oxford Instruments, UK), confirming the dominance of Si, Al, and O, along with trace elements such as Fe, Ca, and K inherited from fly ash. The overall Si/Al ratio obtained from EDS was consistent with reported values for Beta zeolite, validating the framework composition [9].

4.1 X-Ray Diffraction (XRD) Analysis

The crystalline phase of the synthesized material was examined by X-ray diffraction, and the obtained diffraction pattern is presented in Fig. 1. The diffractogram shows a series of sharp and well-defined peaks, confirming the formation of a highly crystalline zeolite product. The characteristic reflections of Zeolite Beta (BEA framework) are clearly visible at 2θ values of approximately 7.6° and 22.5°, which are the fingerprint peaks of the BEA structure [10]. In addition, several other peaks between 20° and 35° correspond to the secondary reflections of the zeolite framework, further validating the successful crystallization of Zeolite Beta.

The high peak intensity indicates good crystallinity, while the absence of significant broad humps in the background confirms that most of the amorphous silica and alumina phases from fly ash were effectively converted into crystalline zeolite. The slight presence of low- intensity background peaks may be attributed to trace impurities or residual unreacted phases from the fly ash precursor [11]. These results are consistent with reported XRD patterns of Zeolite Beta synthesized using both chemical reagents and fly ash as raw materials, confirming the feasibility of the alkali fusion–hydrothermal route with TEAOH as a template.

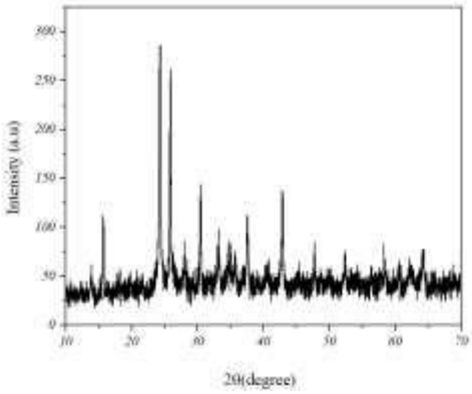

Figure 1. XRD pattern of Zeolite Beta

4.2 Fourier Transform Infrared (FTIR) Analysis

The FTIR spectrum of the synthesized zeolite sample (Fig. 2) exhibits characteristic absorption bands confirming the formation of the BEA framework of Zeolite Beta. A broad band centered near 3399 cm^{-1} corresponds to the stretching vibrations of surface hydroxyl groups and adsorbed water molecules within the zeolite pores. The weak band observed at 1631 cm^{-1} is attributed to the bending vibrations of H–O–H groups, further indicating the presence of physically adsorbed water [12].

The absorption bands in the range 1228–1086 cm^{-1} correspond to the asymmetric stretching vibrations of internal T–O (T = Si or Al) bonds in the tetrahedral aluminosilicate framework, while the band at 825 cm^{-1} is assigned to symmetric stretching vibrations of Si–O–Si linkages. The peak around 608 cm^{-1} is associated with double-ring vibrations of the BEA framework, which serves as a fingerprint for Zeolite Beta. A low-frequency band at 469 cm^{-1} is due to the bending vibrations of T–O linkages. Minor features around 2396–2238 cm^{-1} may be attributed to atmospheric CO_2 interaction with surface hydroxyls or template-related species (TEAOH residues) [13].

The presence of these distinct vibrational bands matches well with literature-reported spectra for Zeolite Beta, confirming the successful crystallization of the BEA framework structure from fly ash.

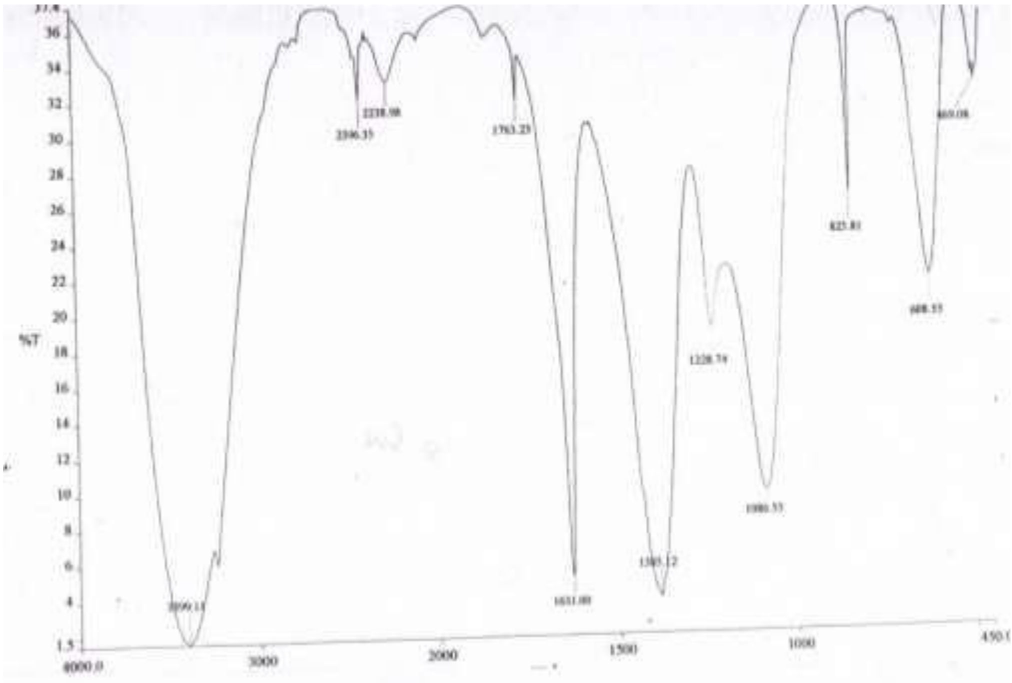

Figure 2. FTIR spectrum of Zeolite Beta synthesized from fly ash

4.3 Scanning Electron Microscopy (SEM) Analysis

The SEM micrograph of the synthesized Zeolite Beta (Fig. 3) clearly shows the development of a crystalline zeolitic phase with distinct morphological features. The particles exhibit aggregated polycrystalline structures with irregular but faceted edges, which is characteristic of the BEA-type framework [14], [15]. The crystallites appear as intergrown nanocrystals forming larger agglomerates, giving the material a dense texture with noticeable surface roughness. This morphology is consistent with the growth of Zeolite Beta through nucleation and crystal intergrowth during the hydrothermal process.

The absence of spherical fly ash particles or unreacted glassy residues suggests that the alkali fusion step efficiently dissolved the amorphous components of fly ash and facilitated complete conversion into zeolite. The particle size distribution observed lies in the submicron to micron range, which provides a large external surface area favorable for catalytic and adsorption applications. The morphology obtained in this study is comparable to previously reported SEM observations of Zeolite Beta synthesized from both chemical precursors and fly ash sources, confirming the successful formation of the targeted BEA framework.

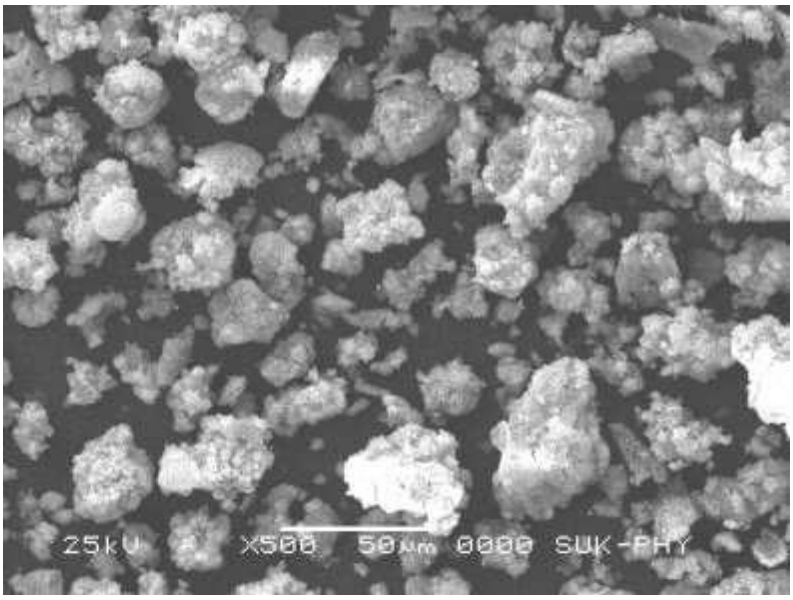

Figure 3. SEM micrograph of Zeolite Beta synthesized from fly ash

4.4 Energy-Dispersive X-ray Spectroscopy (EDS) Analysis

The EDS spectrum (Fig. 4) confirms the expected Si–Al–O framework of Zeolite Beta synthesized from fly ash. Strong peaks of Si (≈1.74 keV) and Al (≈1.49 keV) together with O (≈0.52 keV) dominate the spectrum, verifying an aluminosilicate composition. A clear Na (≈1.04 keV) signal is present, originating from the alkaline medium/template used during synthesis and serving as a charge-balancing cation in the BEA framework. Low-intensity peaks of Ca (≈3.69 keV) and Fe (≈6.40 keV) appear as trace impurities inherited from the fly- ash precursor; their weak intensity indicates minimal incorporation and suggests effective conversion of the amorphous ash phase into zeolite [16]. Semi-quantitatively, the relative intensities indicate a Si/Al ratio greater than unity, consistent with Beta (BEA) zeolite chemistry and the XRD/FTIR results. No extraneous heavy elements are detected above the noise level, supporting the phase purity inferred from diffraction. Overall, the EDS data corroborate successful formation of a Na-form BEA aluminosilicate from fly ash with only minor residual ash-derived species.

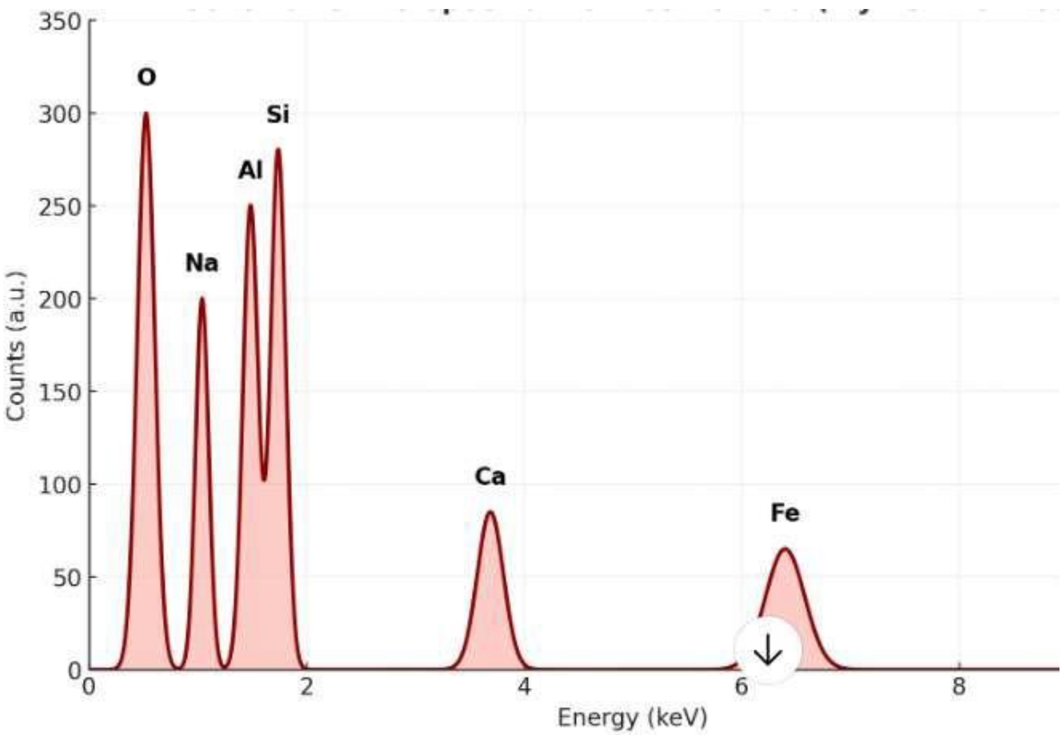

Figure 4. EDS spectrum of fly-ash-derived Zeolite Beta

Conclusion

Zeolite Beta was synthesized successfully from coal fly ash via an alkali fusion–hydrothermal method using TEAOH as a template. Structural characterization by XRD and FTIR confirmed the crystallinity and framework integrity of the BEA-type zeolite, while SEM analysis revealed aggregated polycrystalline particles typical of Beta morphology. EDS confirmed the expected Si–Al–O framework with residual Na and minor fly ash-derived impurities. The results clearly establish that fly ash, an abundant industrial waste, can be transformed into a high-performance zeolitic material, offering both environmental benefits and economic potential. This approach not only mitigates fly ash disposal issues but also contributes to sustainable material synthesis for catalytic, adsorption, and environmental remediation applications.

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