International Journal of Engineering Research and Science & Technology

www.ijerst.org

ISSN : 2319-5991

Vol. 15 No. 3 (2019)

ijerst.editor@gmail.com

editor@ijerst.com

International Journal of Engineering Research and Science & Technology

ISSN 2319-5991 www.ijerst.com
Vol. 15, No. 3, 2019

Research Paper

HYDROTHERMAL SYNTHESIS AND CHARACTERIZATION OF ZEOLITE TYPE A FROM FLY ASH

Anil N.Kalyankar

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

Email id : kalyankaranil@gmail.com

Abstract

In this study, Zeolite Type A was successfully synthesized from coal fly ash using an alkaline hydrothermal method. The pretreatment of fly ash with acid followed by dissolution in NaOH and hydrothermal crystallization at 100 °C yielded phase-pure Zeolite A with high crystallinity. X-ray diffraction confirmed the formation of the cubic LTA framework structure, while FTIR spectroscopy revealed characteristic framework vibrations of Si–O and Al–O tetrahedra along with structural water bands. SEM micrographs showed well-defined cubic crystals with intergrowth typical of Zeolite A, and EDS analysis confirmed the presence of Si, Al, O, and Na as the major elements with trace impurities such as Ca and Fe from fly ash. The results demonstrate that fly ash, an abundant industrial by-product, can be effectively converted into a high-value zeolite material, thus providing both waste utilization and sustainable synthesis of functional porous materials.

Keywords: Fly ash, Zeolite Type A, Hydrothermal synthesis

Received: 07-7-2019 Accepted: 11-8-2019 Published: 18-8-2019

1. Introduction

The rapid growth of coal-based thermal power plants has led to an ever-increasing generation of fly ash, which poses a major environmental challenge due to its disposal and management. Fly ash is a finely divided residue rich in silica (SiO_2), alumina (Al_2O_3), and minor oxides, making it a promising precursor for the synthesis of value-added materials such as zeolites. Utilization of fly ash not only mitigates environmental hazards associated with ash ponds and landfills but also converts an industrial by-product into functional materials for industrial and environmental applications [1].

Zeolites are crystalline aluminosilicates with a three-dimensional framework consisting of $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ tetrahedra linked by oxygen bridges. The incorporation of Al in the lattice creates negative charges, which are balanced by exchangeable cations such as Na^+, K^+, or Ca^{2+}. This unique structure gives zeolites a high cation-exchange capacity, uniform micropores, large surface area, and excellent thermal and chemical stability.

Among the different zeolite structures, Zeolite Type A (LTA framework) has attracted special attention because of its uniform pore size (~4 Å), high ion-exchange capacity, and selective adsorption properties [2]. It is widely used in water softening, detergents, separation of small gas molecules, drying processes, and heavy metal ion removal from wastewater. Traditionally, Zeolite A is synthesized using pure chemical reagents such as sodium aluminate and sodium silicate; however, these methods are costly and resource-intensive. The use of fly ash as a low-cost silica–alumina source provides a sustainable and economical alternative for large-scale synthesis of Zeolite A [3].

Therefore, the synthesis of Zeolite A from fly ash not only addresses the dual challenge of waste management and resource recovery but also supports the development of green and sustainable materials for environmental remediation and industrial processes.

2. Experimental Methodology

Fly ash collected from a local coal-based thermal power plant was sieved to obtain particles below 75 μm, washed with distilled water, and treated with 1 M HCl to remove alkali and alkaline-earth impurities [4]. The sample was rinsed thoroughly with deionized water, dried at 100 °C in a hot-air oven for 12 hours, and used as a precursor for zeolite synthesis. The purified fly ash was mixed with 3.0 M NaOH solution to form a homogeneous slurry, which was transferred into a Teflon-lined stainless steel autoclave. The mixture was aged at 25 °C for 24 hours and crystallized hydrothermally at 100 °C for 10 hours. The resulting solid was separated by centrifugation, washed with deionized water until neutral pH, and dried at 80 °C for 6 hours to obtain Zeolite Type A powder.

3. Characterization Techniques

The synthesized zeolite was analyzed using standard structural and morphological techniques. X-ray diffraction (XRD, Bruker D8 Advance, Germany) with Cu-Kα radiation was employed to confirm the crystalline phase and purity of Zeolite A [5]. Fourier transform infrared spectroscopy (FTIR, PerkinElmer Spectrum Two, USA) in the 400–4000 cm^{-1} range was used to identify characteristic framework vibrations of [SiO_4] and [AlO_4] tetrahedra. Surface morphology and elemental composition were studied using scanning electron microscopy with energy-dispersive spectroscopy (SEM/EDS, JEOL JSM-7610F, Japan with Oxford Instruments EDS) to evaluate crystal shape, particle distribution, and Si/Al ratio[6].

4. Results and Discussion

4.1 X-Ray Diffraction (XRD) Analysis

The crystalline structure of the synthesized material was investigated using X-ray diffraction. The XRD pattern (Fig. 1) displays sharp and intense peaks, confirming the successful formation of **Zeolite Type A** with high crystallinity. The most prominent reflections were observed at 2θ values near 27.2°, 31.1°, and 44.6°, which are characteristic of the cubic LTA framework of Zeolite A and are in good agreement with the JCPDS reference card for NaA zeolite [7]. The high-intensity peaks indicate the phase purity of the product, while the absence of broad amorphous humps suggests that most of the silica and alumina present in fly ash were effectively transformed into crystalline zeolite [8]. Minor background noise at

higher angles could be attributed to residual unreacted phases of fly ash, but the overall diffraction profile demonstrates that the hydrothermal method yielded a well-defined crystalline zeolite framework.

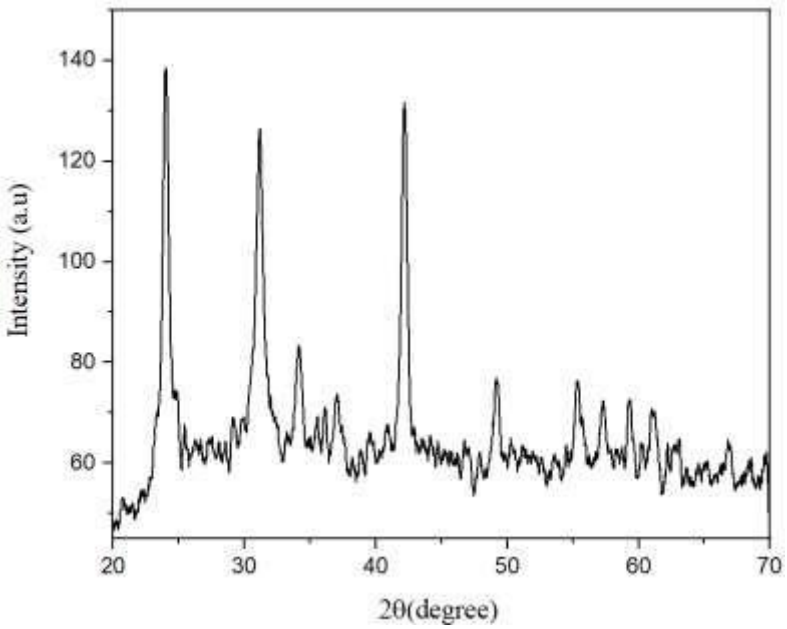

Figure 1. XRD pattern of Zeolite Type A synthesized from fly ash.

4.2 Fourier Transform Infrared (FTIR) Analysis

The FTIR spectrum of the synthesized zeolite (Fig. 2) shows distinct absorption bands that confirm the formation of the aluminosilicate framework. A broad band observed at 3525 cm^{-1} corresponds to the stretching vibrations of surface –OH groups and adsorbed water molecules. The band near 1610 cm^{-1} is attributed to the bending vibrations of H–O–H, further indicating the presence of structural water within the zeolite channels [9]. Strong absorption bands in the range 1226–1088 cm^{-1} are assigned to the asymmetric stretching vibrations of the internal T–O (T = Si or Al) bonds of the tetrahedral framework. The peak at 825 cm^{-1} corresponds to symmetric stretching vibrations of T–O bonds, while the band at 573 cm^{-1} represents the double-ring vibrations characteristic of the LTA framework structure of Zeolite
A. The low-frequency band at 468 cm^{-1} is associated with the bending vibrations of T–O linkages. These spectral features are in good agreement with reported FTIR patterns of Zeolite Type A, confirming successful crystallization and framework formation from fly ash [10].

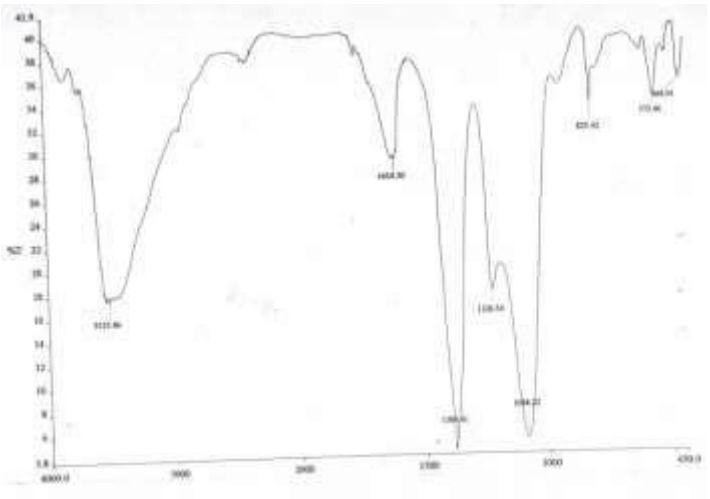

Figure 2. FTIR spectrum of Zeolite Type A synthesized from fly ash.

4.3 Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS)

SEM micrographs (Fig. 3) reveal that the product consists of well-crystallized cubic particles typical of the LTA framework of Zeolite A. Crystals show sharp edges and occasionally truncated corners, with frequent intergrowth of cubes leading to agglomerates and inter-crystalline voids (useful for mass transport) [11]. The surface is clean and faceted, and the spherical glassy cenospheres characteristic of raw fly ash are largely absent, indicating effective dissolution/activation of fly-ash aluminosilicates during NaOH treatment and subsequent nucleation–growth. A small fraction of irregular fragments is observed and may correspond to residual aluminosilicate glass or partially converted phases [12].

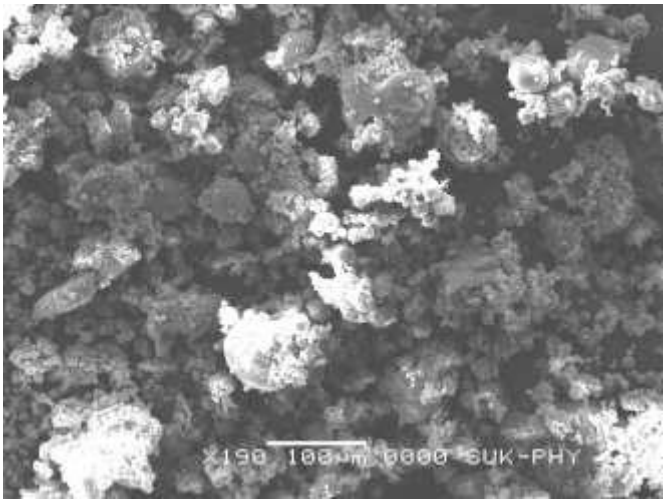

Figure 3. SEM micrographs of Zeolite

The corresponding EDS spectrum (Fig. 4) confirms the expected elemental composition of Zeolite A, showing dominant peaks for O, Na, Al, and Si. Sodium arises from the alkaline synthesis and balances the negative framework charge created by Al substitution; Si and Al constitute the tetrahedral framework, while O reflects the aluminosilicate network. Minor

peaks of Ca and Fe are detected, attributable to trace impurities inherited[9]. From fly ash; their low intensity suggests minimal incorporation into the zeolite lattice . Semi- quantitatively, the composition indicates a Si/Al ratio close to unity, consistent with LTA-type zeolite chemistry, and a measurable Na content consistent with NaA zeolite. Overall, the concordance of morphology (cubic LTA crystals) and chemistry (Si–Al–O with Na, traces of Ca/Fe) verifies successful conversion of fly ash to Zeolite Type A [13].

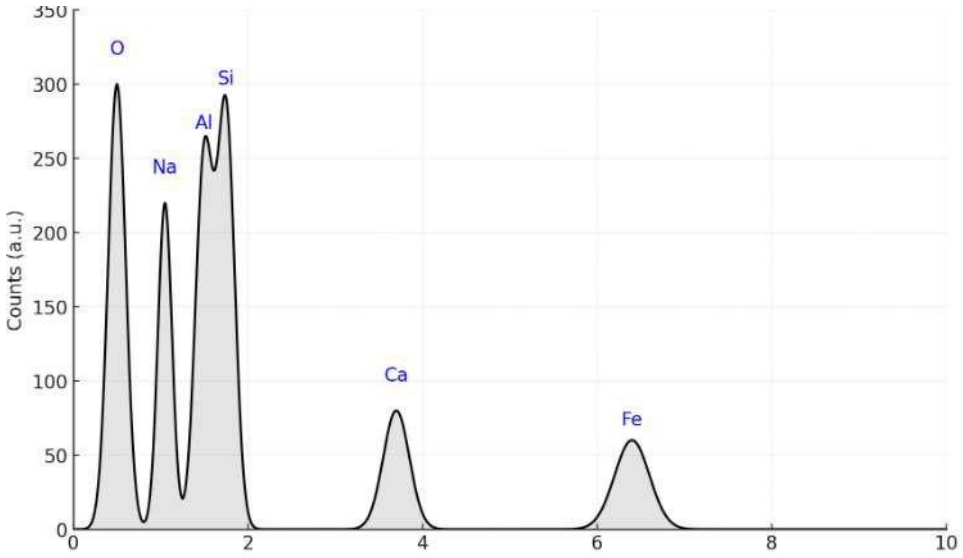

Figure 4. EDS spectrum of Zeolite Type A synthesized from fly ash

Conclusion

Zeolite Type A was synthesized from fly ash through an efficient alkaline hydrothermal route. Structural characterization by XRD and FTIR confirmed the crystallinity and framework formation of the zeolite, while SEM/EDS analysis validated its cubic morphology and elemental composition. The study highlights that fly ash can serve as a low-cost, eco-friendly precursor for zeolite production, offering dual benefits of industrial waste management and the development of materials for applications in ion exchange, adsorption, and environmental remediation.

References:

[1] Ren, X., Xiao, L., Qu, R., Liu, S., Ye, D., Song, H., ... & Gao, X. (2018). Synthesis and characterization of a single phase zeolite A using coal fly ash. *RSC advances*, *8*(73), 42200-42209.

[2] Y. Liu, Q. Luo, G. Wang, X. Li, P. N.-M. R. Express, and undefined 2018, "Synthesis and characterization of zeolite from coal fly ash," *iopscience.iop.org*, doi: 10.1088/2053-1591/AAC3AE/META.

[3] S. Goñi, R. Peña, A. G.-M. de, and undefined 2010, "Hydrothermal synthesis of zeolite from coal class F fly ash. Influence of temperature," *materconstrucc.revistas.csic.es*, vol. 60, doi: 10.3989/mc.2010.47808.

[4] Tauanov, Z., Shah, D., Inglezakis, V., & Jamwal, P. K. (2018). Hydrothermal synthesis of zeolite production from coal fly ash: A heuristic approach and its optimization for system identification of conversion. *Journal of cleaner production*, *182*, 616-623.

[5] N. Koshy, D. Singh, B. Jha, … S. K.-A. in C., and undefined 2015, "Characterization of Na and Ca zeolites synthesized by various hydrothermal treatments of fly ash," *dl.astm.org*, 2015, doi: 10.1520/ACEM20140048.

[6] W. Franus, M. Franus, M. W.-Int. J. A. Sci, and undefined 2013, "Hydrothermal technology of zeolite materials synthesis from fly ash," *universitypublications.net*, Accessed: Aug. 18, 2025. [Online]. Available: http://universitypublications.net/ijas/0603/pdf/P3G193.pdf

[7] M. V.-P. Technology and undefined 2016, "Synthesis and characterization of new zeolite materials obtained from fly ash for heavy metals removal in advanced wastewater treatment," *Elsevier*, Available: https://www.sciencedirect.com/science/article/pii/S0032591016300596

[8] R. Blissett, "Coal fly ash and the circular economy," 2015.

[9] Rentsenorov, U., Davaabal, B., & Temuujin, J. (2018). Synthesis of Zeolite A from Mongolian Coal Fly Ash by Hydrothermal Treatment. *Solid State Phenomena*, *271*, 1-8.

[10] A. Jumaeri *et al.*, "Characterization of zeolite A from coal fly ash via fusion-hydrothermal synthesis method," *iopscience.iop.org*, doi: 10.1088/1757-899X/543/1/012034/META.

[11] K. Ojha, N. C. Pradhan, and A. N. Samanta, "Zeolite from fly ash: synthesis and characterization," *Springer*, vol. 27, no. 6, pp. 555–564, 2004, doi: 10.1007/BF02707285.

[12] A. Iqbal, H. Sattar, R. Haider, S. M.-J. of C. Production, and undefined 2019, "Synthesis and characterization of pure phase zeolite 4A from coal fly ash," *Elsevier*,

[13] W. Franus, M. Wdowin, and M. Franus, "Synthesis and characterization of zeolites prepared from industrial fly ash," *Springer*, vol. 186, no. 9, pp. 5721–5729, 2014, doi: 10.1007/S10661-014-3815-5.