SYNTHESIS CHARACTERISTICS OF NALTA AND CALTA MOLECULAR SIEVES VIA CHEMICAL ACTIVATION AND HYDROTHERMAL METHODS

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Abstract: This scientific article presents the synthesis technology of molecular sieve structures of the LTA-type zeolites modified with sodium and calcium cations – NaLTA and CaLTA – via chemical activation and hydrothermal methods. The study explores the mechanochemical activation of raw materials using impact-vibration milling, their preparation for crystallization, and the mechanisms of phase structure formation. The resulting microporous frameworks were evaluated using physicochemical characterization techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), Brunauer–Emmett–Teller surface analysis (BET), and infrared spectroscopy (IR), with a particular focus on determining their adsorption properties.

Keywords: NaLTA, CaLTA, zeolites, mechanochemical activation, hydrothermal synthesis, crystalline phase, microporosity, selective adsorption.

Introduction:

Molecular sieves are crystalline aluminosilicate structures with well-defined geometry and pore sizes ranging from 0.3 to 0.8 nm, which makes them highly selective sorbents and catalysts. Synthetic zeolites of the LTA (Linde Type A) class are particularly notable for the geometry of their pore openings, cation exchange capacity, and sensitivity to ionic radii. Their sodium and calcium-modified forms – NaLTA and CaLTA – exhibit selective adsorption capabilities for hydrogen sulfide, carbon dioxide, ammonia, alcohols, and mercaptans.

In modern technology, especially in the development of environmentally friendly processes, identifying low-waste and energy-efficient synthesis pathways through the combination of mechanochemical activation and hydrothermal crystallization is of significant importance. This article analyzes the synthesis of zeolites via these two methods, focusing on their structure formation mechanisms and physicochemical properties.

Materials and Methods

In the experimental procedure, metakaolin was first calcined at 600 °C and then mixed with aqueous solutions of NaOH and $Ca(NO_3)_2$. The resulting mixture was subjected to mechanochemical activation using a vibratory ball mill for 15 minutes. The activated precursor was then subjected to hydrothermal crystallization at 90–120 °C for 2 hours in hydroxide solutions with a concentration of 2–4 mol/L.

Analytical techniques included:

- X-ray Diffraction (XRD): to determine crystallinity and phase composition;
- Scanning Electron Microscopy (SEM): to assess particle morphology and microporous structure;
- Brunauer–Emmett–Teller (BET) analysis: to evaluate specific surface area;
- Fourier Transform Infrared Spectroscopy (FTIR): to identify Si–O–Al linkages;
- Thermogravimetric and Differential Thermal Analysis (TGA-DTA): to assess thermal stability.

Results and Discussion

Experimental results demonstrated that vibratory mechanochemical activation rapidly generated reactive phases within the precursor material. During NaLTA synthesis, the formation of ~0.42 nm pore openings allowed effective selective adsorption of small-radius gases such as H_2S and CO_2. In contrast, CaLTA possessed larger pores (~0.5 nm), making it suitable for adsorbing alcohols and mercaptans like C_2H_5OH and C_2H_5SH.

XRD analysis revealed well-defined diffraction peaks corresponding to NaLTA and CaLTA phases, with crystallinity levels ranging between 78% and 84%. SEM imaging showed predominantly cubic-shaped crystals within a size range of 1.1–2.3 µm, indicating homogeneous morphology and optimized synthesis conditions. BET analysis indicated specific surface areas of 435 m^2/g for NaLTA and 410 m^2/g for CaLTA, with pore diameters ranging from 0.39 to 0.51 nm. FTIR spectra exhibited distinct Si–O–Si and Si–O–Al stretching vibrations in the 980–1050 cm^{-1} range, confirming the formation of the zeolite framework. Thermal analysis showed no significant weight loss up to 600 °C, indicating that the synthesized zeolites are thermally stable and suitable for high-temperature applications.

Kinetic evaluation of the crystallization process indicated that increasing NaOH concentration accelerated crystallization; however, excessive concentrations led to gelation phenomena, which hindered crystal growth.

Conclusion

NaLTA and CaLTA molecular sieves were successfully synthesized using a combination of mechanochemical activation and hydrothermal crystallization techniques. The resulting materials exhibited high crystallinity, stable phase structures, and distinct selective adsorption properties. NaLTA demonstrated high affinity for small gas molecules such as H_2S and CO_2, while CaLTA proved effective for molecular sieving of organic compounds.

This synthesis approach offers potential for broad industrial applications in sorption, catalysis, and gas separation technologies. Additionally, the process significantly reduces waste generation and enhances overall economic efficiency.

References:

1. Breck, D. W. *Zeolite Molecular Sieves*. Wiley-Interscience, 1974.
2. Davis, M. E. "Ordered Porous Materials for Emerging Applications." *Nature*, 2002**, 417**, pp. 813–821.
3. Pavlov, M. L., Ivanov, A. I., Kuznetsova, T. A. "Zeolite NaLTA Synthesis from Kaolinite." *Journal of Applied Chemistry*, 2011, **84(**6), pp. 973–979.
4. Barrer, R. M. *Zeolites and Clay Minerals as Sorbents and Molecular Sieves*. Academic Press, 1978.
5. Petrovich, B., Smirnov, V., Yaroslavtsev, A. "Thermodynamic Analysis of Zeolite Formation." *Journal of Physical Chemistry B*, 2004, **108**(12), pp. 3862–3868.
6. Piccione, P. M., Yang, S., Chmelka, B. F. "Entropy-Driven Synthesis in Microporous Materials." *Chemistry of Materials*, 2002, **14**(2), pp. 882–891.
7. Zones, S. I. "Synthetic Pathways for Molecular Sieves." *Microporous Materials*, 2000, **12**, pp. 1–9.
8. Flanigen, E. M., Khatami, H., Szymanski, H. A. "Synthesis and Structure of LTA-Type Zeolites." *Journal of Catalysis*, 1985, **95**, pp. 201–214.