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Research Progress on Zeolite Synthesis Using Fly Ash as Raw Material

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Abstract

Fly ash is a large-volume industrial solid waste discharged from coal-fired power plants, with an annual national emission exceeding 600 million tons. Its stockpiling not only occupies land resources but also easily causes environmental problems such as air and water pollution. Zeolites, as a class of silicoaluminate crystalline materials with regular pore structures, high specific surface areas, and excellent adsorption/catalytic properties, are widely applied in the fields of adsorption and separation, catalytic reactions, and environmental remediation. The synthesis of zeolites using fly ash as a silicon-aluminum source achieves the dual goals of "treating waste with waste" and high-value resource utilization, thus emerging as a research hotspot in the fields of materials science and environmental engineering in recent years. This paper systematically reviews the physicochemical properties of fly ash and their impacts on zeolite synthesis, elaborates in detail the principles, process parameter optimization, product performance, and research progress of mainstream preparation methods including hydrothermal synthesis, solvothermal synthesis, and microwave-assisted synthesis. Furthermore, it summarizes the current application status of fly ash-derived zeolites in wastewater treatment, gas adsorption, catalytic reactions, and other fields, and points out the existing problems in current research such as complex pretreatment, high energy consumption, and difficulties in large-scale production. This work provides a reference for the resource utilization of fly ash and the low-cost preparation of zeolites.

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1. Introduction

As one of the world's primary energy sources, coal dominates electricity production. Fly ash is fine solid particles collected by flue gas purification systems after pulverized coal is burned at high temperatures (1300-1500℃). Its main chemical components include SiO_2, Al_2O_3, along with oxides such as Fe_2O_3, CaO, MgO, Na_2O, and trace heavy metal elements (e.g., Cr, Pb, As) [1]. Statistics show that the global annual emission of fly ash exceeds 1.5 billion tons. As the world's largest coal consumer, China's fly ash emission reached 680 million tons in 2024, with the cumulative stockpiling exceeding 5 billion tons [2]. Large-scale open stockpiling or direct landfilling of fly ash not only occupies cultivated land and forestland but also may release harmful substances through wind dust, rain leaching, and other pathways, polluting the atmosphere, soil, and groundwater, and posing serious threats to the ecological environment and human health [3]. Therefore, the harmless treatment and resource utilization of fly ash have become urgent environmental and resource issues.

Zeolites are three-dimensional network crystalline materials formed by the connection of SiO_4 and AlO_4 tetrahedra through oxygen bridges, featuring regular microporous structures, high specific surface areas, excellent ion exchange performance, and catalytic activity [4]. Based on differences in framework structures, common zeolites include type A, X, Y, ZSM-5, and MCM-41, which are widely used in adsorption and separation, catalysis, ion exchange, and other fields [5].

Traditional zeolite preparation mainly uses chemical reagents as silicon-aluminum sources, which are costly and consume limited mineral resources. However, the total content of SiO_2 and Al_2O_3 in fly ash usually exceeds 70%, and its chemical composition is highly consistent with the silicon-aluminum core units of zeolite framework structures, making it an ideal low-cost raw material for zeolite preparation [6]. Replacing chemical reagents with fly ash for zeolite synthesis not only reduces the production cost of zeolites but also consumes industrial solid waste, conforming to the development concepts of "carbon neutrality" and "circular economy," and demonstrating significant environmental, economic, and social benefits [7].

Research on zeolite synthesis from fly ash began in the 1960s, when type A zeolite was first synthesized from fly ash via hydrothermal method, pioneering a new path for fly ash resource utilization. Since then, with the development of material synthesis technology, various synthesis methods such as solvothermal, microwave-assisted, and ultrasound-assisted methods have been successively applied, and the product types have expanded from traditional microporous zeolites to mesoporous and hierarchical porous zeolites [9]. In recent years, researchers have conducted extensive studies on pretreatment process optimization, green templating agents, modification technology innovation, and high-end application expansion, achieving a series of breakthrough results. This paper systematically combs the effects of the physicochemical properties of fly ash on zeolite synthesis, elaborates on the principles and progress of mainstream synthesis methods, analyzes the mechanism of modification technologies, summarizes the application status, and prospects the future development direction, providing a comprehensive reference for in-depth research in this field.

2. Main Preparation Methods and Research Progress of Zeolites from Fly Ash

2.1. Hydrothermal Synthesis Method

Hydrothermal synthesis is the most mature and widely used method for preparing zeolites from fly ash. Its principle is: in a closed high-pressure reactor, with water as the solvent, heating causes the silicon-aluminum components in fly ash to depolymerize in alkaline solutions (such as NaOH, KOH), forming homogeneous or heterogeneous solutions containing $Si(OH)_4$ and $Al(OH)_4^-$. Subsequently, under the action of templating agents or mineralizers, silicon-aluminum species polymerize and crystallize directionally according to specific structures to form zeolite crystals [10]. Typical process steps include: fly ash pretreatment, batching, aging, hydrothermal crystallization, product separation, drying, and calcination [11]. In recent years, researchers have carried out extensive optimization studies on key parameters such as pretreatment methods, alkali concentration, silicon-aluminum ratio, templating agent type, crystallization temperature, and time, aiming to improve product crystallinity, specific surface area, and adsorption/catalytic performance.

The advantages of hydrothermal synthesis are: mature technology, simple operation, scalability for large-scale production, high product crystallinity close to commercial zeolites, and the ability to synthesize various types of zeolites through parameter regulation [12]. Its main limitations include: ① long reaction cycle and high energy consumption; ② complex pretreatment steps requiring multiple acid-base leaching or calcination; ③ large amount of wastewater discharge [45]. To address these issues, researchers have achieved a reduction of more than 50% in synthesis cycle and 30%-40% in energy consumption by coupling auxiliary means such as microwave and ultrasound [13].

2.2. Solvothermal Synthesis Method

Solvothermal synthesis uses organic solvents (such as ethanol, ethylene glycol, glycerol, ionic liquids) instead of water as the reaction medium, and crystallization reactions are carried out in a closed system (120-250℃) [14]. Its core principle is: the polarity and dielectric constant of organic solvents are different from those of water, which can regulate the dissolution rate and polymerization mode of silicon-aluminum species, while reducing the surface tension of the reaction system to promote crystal nucleation and growth. This method is particularly suitable for synthesizing high-silicon or mesoporous zeolites [15].

The typical process steps are similar to hydrothermal synthesis, with the main differences being: ① replacing deionized water with organic solvents, where the mass ratio of solvent to fly ash is usually 5:1-10:1; ② higher crystallization temperature (150-250℃) and shorter reaction time (4-24 h); ③ some systems can form pores through the steric hindrance effect of solvent molecules without templating agents [16].

The key advantages of solvothermal synthesis are: organic solvents have stronger selective dissolution capacity for silicon-aluminum components in fly ash, especially suitable for high-crystalline fly ash, with silicon-aluminum dissolution rate increased by 20%-30% compared with hydrothermal method [17]; it can synthesize high-purity mesoporous or hierarchical porous zeolites. For example, Liu *et al.* [51] synthesized MCM-41 mesoporous zeolites with uniform pore size distribution and specific surface area of 1056 m^2/g using ethylene glycol as solvent, which is much higher than that of hydrothermal products; the reaction system has low corrosiveness, which can reduce the requirements for reactor material and extend equipment service life [18].

In recent years, research on ionic liquids as solvents has attracted much attention. Ionic liquids have the characteristics of low vapor pressure, good thermal stability, and strong designability. Their cations can act as templating agents to guide pore formation, and anions can regulate the polymerization rate of silicon-aluminum. The ZSM-5 zeolite synthesized using ionic liquid [BMIM]Br as solvent and templating agent has a crystallinity of 96% and a selectivity of 89% for propylene oligomerization reaction. Moreover, ionic liquids can be recycled, reducing environmental risks [19]. However, solvothermal synthesis also has limitations: organic solvents are relatively expensive, some are volatile and toxic, and supporting recovery devices are required for large-scale production, restricting its industrial application [20].

2.3. Microwave-Assisted Synthesis Method

Microwave-assisted synthesis uses the thermal and non-thermal effects of microwaves to enhance the dissolution and crystallization of silicon-aluminum components in fly ash [55]. Its principle is: the microwave frequency (usually 2.45 GHz) matches the dipole vibration frequency of water molecules and silicon-aluminum species, enabling the reaction system to heat up uniformly in a short time (heating rate up to 10-20℃/min). Meanwhile, the polarization effect can break Si-O-Al bonds, promoting the depolymerization and directional polymerization of silicon-aluminum species [21]. Typical process steps: mix pretreated fly ash with alkali solution and templating agent to form reaction slurry; place it in a microwave reactor, control power and temperature, and react for 0.5-6 h; subsequent separation, drying, and calcination steps are consistent with hydrothermal method [22].

The core advantage of microwave-assisted synthesis is high efficiency and energy saving: the synthesis cycle is shortened by 80%-90% compared with traditional hydrothermal method. For example, Wei *et al*. [23] synthesized type A zeolite via microwave-assisted method with a reaction time of only 1 h, while traditional hydrothermal method requires 24 h; energy consumption is reduced by 40%-60%, as microwave internal heating avoids heat conduction loss of traditional heating and does not require long-term heat preservation [24]; the product has high crystallinity, fine and uniform particles. Uniform microwave heating can reduce crystal agglomeration, and the synthesized ZSM-5 zeolite has a crystal particle size of 0.1-0.3 μm and a specific surface area of 426 m^2/g, with better performance than hydrothermal products. The limitations of this method are: the volume of microwave reactor is small, and the uniformity of microwave field needs to be solved for large-scale production; high-power microwaves are prone to local overheating, requiring precise control of reaction parameters [25].

2.4. Ultrasound-Assisted Synthesis Method

Ultrasound-assisted synthesis uses the cavitation effect of ultrasound to enhance mass transfer and reaction processes [26]. Its mechanism includes: cavitation effect destroys the dense structure on the surface of fly ash particles, increasing the reaction contact area; promotes alkali solution penetration, accelerates the breaking of Si-O bonds and Al-O bonds, and improves silicon-aluminum dissolution rate; uniformly disperses silicon-aluminum species to avoid impurity crystal formation caused by excessive local supersaturation [27]. The process steps are similar to microwave-assisted method, usually carried out in an ultrasonic cleaner or ultrasonic reactor with ultrasonic power of 100-500 W and reaction time of 1-12 h, which can be used alone or coupled with hydrothermal and microwave methods [28].

The main advantages of ultrasound-assisted synthesis are: enhancing pretreatment effect, achieving activation without high-temperature calcination; shortening crystallization time. When synthesizing type A zeolite via ultrasound-assisted hydrothermal method, the reaction time can be shortened from 24 h to 6 h, while maintaining crystallinity above 90% [29]; reducing reaction temperature. Traditional hydrothermal method requires above 120℃, while ultrasound-assisted method can achieve efficient crystallization at 80-100℃, further reducing energy consumption [30]. However, the limitations of ultrasound-assisted method are: the penetration depth of ultrasound field is limited, requiring the design of special reaction devices for large-scale production; long-term high-power ultrasound may damage the zeolite crystal structure, requiring matching control of ultrasound time and power [31].

2.5. Other Synthesis Methods

2.5.1. Solid-State Synthesis Method

Solid-state synthesis uses fly ash, alkali source, and templating agent as raw materials, with no solvent or a small amount of solvent. After mechanical grinding and mixing, direct calcination and crystallization are carried out at high temperature (200-400℃) [32]. Its advantages are simple process, no wastewater discharge, extremely low cost, and suitability for large-scale production; however, the product has low crystallinity and small specific surface area, requiring subsequent modification to improve performance [33].

2.5.2. Sol-Gel Method

The sol-gel method first converts fly ash into silica sol and alumina sol through acid leaching or alkali dissolution, adjusts the pH value to form a uniform sol, and then forms zeolites after gelation, aging, and calcination [34]. This method can precisely regulate the silicon-aluminum ratio and product composition, suitable for synthesizing high-purity zeolites with specific structures, but it has complex processes, long cycles, poor sol stability, and great difficulty in large-scale production [35].

5.3. Hierarchical Porous Zeolite Synthesis Method

Traditional fly ash-based zeolites mostly have a single microporous structure, limiting their adsorption and catalytic performance for macromolecular pollutants. In recent years, the synthesis of hierarchical porous zeolites has become a research hotspot, mainly realized through "hard templating method" and "soft templating method."

3. Application Fields of Fly Ash-Based Zeolites

3.1. Environmental Governance Field

Fly ash-based zeolites are widely used in environmental governance, covering three core directions: water pollution control, air pollution control, and soil remediation. In water pollution control, low silicon-aluminum ratio type X, Y, and A zeolites have excellent adsorption performance for heavy metals such as Pb^{2+} and Cu^{2+} due to their high cation exchange capacity. High silicon-aluminum ratio ZSM-5 zeolites have strong hydrophobicity, showing significant adsorption effect on organic pollutants such as phenol after modification. Those loaded with TiO_2 have a photocatalytic degradation rate of over 90% for dyes. H-type zeolites have an ammonia nitrogen adsorption capacity of 45 mg/g, and La^{3+}-loaded zeolites have a phosphate adsorption capacity of 32 mg/g, which can effectively remove nitrogen and phosphorus from water [36]. In air pollution control, zeolites loaded with metals such as Fe, Cu, and V serve as SCR catalysts, achieving NO conversion rates of 85%-95% at 200-400℃. Type X and Y zeolites have adsorption capacities for VOCs equivalent to commercial products but with lower costs. Amine-functionalized type X zeolites have a CO_2 adsorption capacity of 6.8 mmol/g and a CO_2/N_2 separation factor exceeding 50, suitable for CO_2 capture [37]. In soil remediation, adding 5% Na-P type zeolite can reduce the available Pb^{2+} content in Pb^{2+}-contaminated soil by 60% and crop absorption by more than 50%. Adding 3% zeolite can reduce the electrical conductivity of saline-alkali soil by 35% and increase crop yield by 20%-30%, realizing soil improvement by adsorbing heavy metals or Na^+ and releasing beneficial ions [38].

3.2. Catalysis Field

With adjustable acid sites and pore structures, fly ash-based zeolites have excellent catalytic performance and can replace some commercial catalytic materials. Their applications cover two major directions: petrochemical catalysis and environmental catalysis. In the petrochemical field, ZSM-5 can be used as an active component of catalytic cracking catalysts, increasing the yield of light oil from heavy oil to 78%, which is 5% higher than commercial catalysts, with coke yield reduced by 3%. Pt-loaded H-Y type zeolites have a n-butane isomerization conversion rate of 62% and isobutane selectivity of 90%. H-type zeolites can also be used as solid acid catalysts for esterification reactions, replacing traditional concentrated sulfuric acid to reduce corrosion and pollution. In environmental catalysis, fly ash-based zeolites loaded with semiconductors such as TiO_2 and ZnO have a formaldehyde degradation rate of 92% under ultraviolet light with good stability. When used as carriers for Fenton/photo-Fenton reaction catalysts, they have a phenol degradation rate of 95%, and the reaction rate constant is twice that of pure Fe_3O_4 [39].

4. Energy Storage Field

Fly ash-based zeolites have important applications in the energy storage field, mainly including adsorption energy storage and battery materials. In terms of adsorption energy storage, they can be used as adsorbents in adsorption refrigeration and heat storage systems, realizing energy conversion through adsorption-desorption cycles of adsorbates such as water and methanol. For example, fly ash-based Na-X type zeolites have a water adsorption capacity of 0.35 g/g and adsorption heat of 3000 J/g, with a coefficient of performance (COP) of 0.75 when used in solar adsorption refrigeration systems, close to the level of commercial adsorbents. In the field of battery materials, modified fly ash-based zeolites can be used as electrode materials or electrolyte additives for lithium-ion batteries and supercapacitors. For example, carbon-coated ones as anode materials for lithium-ion batteries have an initial discharge capacity of 580 mAh/g and a capacity retention rate of 85% after 50 cycles. As electrode materials for supercapacitors, they have a specific capacitance of 180 F/g with good cycle stability [40].

5. Other Applications

Fly ash-based zeolites can also be used in gas separation, food preservation, agricultural fertilizers, and other fields. For example, their shape-selective adsorption performance can be used to separate oxygen and nitrogen in air; they can be used in food packaging to adsorb moisture and ethylene, extending food shelf life; as slow-release fertilizer carriers, they can control the nutrient release rate and improve fertilizer utilization efficiency.

6. Conclusions

As a large-volume industrial solid waste, the resource utilization of fly ash is of great significance for ecological environment protection and circular economy development. Synthesizing zeolites from fly ash achieves a win-win situation between high-value utilization of solid waste and low-cost preparation of zeolites, and has become a hot academic research topic. This paper systematically reviews the research progress of fly ash-based zeolites, and the results show that:

1. The physicochemical properties of fly ash are the key factors affecting zeolite synthesis. Pretreatment technologies such as mechanical activation, alkali fusion activation, and acid leaching activation can effectively improve the reactivity of silicon-aluminum components, remove impurity interference, and the combined use of pretreatment technologies shows better effects.
2. The synthesis process has developed from traditional hydrothermal method to a pattern of multiple coexisting technologies such as microwave/ultrasound-assisted, templating, and solvent-free methods. The alkali fusion-hydrothermal method is currently the most mature process due to its high product crystallinity and stable performance. Green processes such as solvent-free method and microwave-assisted method have good development prospects, but further optimization of large-scale production technology is required.
3. Fly ash-based zeolites are widely used in environmental governance, catalysis, energy storage, and other fields, especially showing good application effects in water pollution control and air pollution control, but the high-end application fields still need further expansion.

At present, the research on fly ash-based zeolites still faces challenges such as raw material property constraints, insufficient greenness of synthesis processes, and lack of industrialization technology. In the future, it is necessary to strengthen basic theoretical research, develop efficient and green synthesis and modification technologies, expand high-end application fields, and promote their transformation from laboratory research to large-scale industrial application, providing technical support for China's solid waste resource utilization and ecological environment protection.

7. Thank-You Note

The author would like to thank previous researchers for their contributions in conducting research on the progress of zeolite synthesis using fly ash as raw material. Thanks to the research results obtained by previous researchers, I and other readers can obtain comprehensive information on the progress of zeolite synthesis using fly ash as raw material. Such information will undoubtedly serve as a solid foundation for further research aimed at developing innovative and high-value zeolite synthesis technologies, contributing to the advancement of solid waste resource utilization. I hope that the findings of this article can provide a broader perspective on the feasibility and application potential of zeolite synthesis technologies using fly ash as raw material, as well as their practical value in environmental governance and circular economy development.

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