

# Research progress on synthesis of zeolites from coal fly ash and environmental applications

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## HIGHLIGHTS

- Up-to-date information on the preparation of zeolite from CFA were summarized.
- The applications of CFA zeolites in environmental protection field were reviewed.
- The feasibility analysis of industrial production of CFA zeolites were discussed.

## ARTICLE INFO

### Article history:

Received 17 February 2023  
Revised 9 May 2023  
Accepted 6 June 2023  
Available online 20 July 2023

### Keywords:

Coal fly ash  
Zeolite  
Synthetic method  
Environmental application  
Industrialization

## GRAPHIC ABSTRACT



## ABSTRACT

The by-product of coal combustion, coal fly ash (CFA), has become one of the world's most emitted solid wastes, and bulk utilization while achieving high value-added products is the focus of current research. Using CFA to prepare zeolite cannot only reduce environmental pressure, but also obtain high value-added products, which has a good market prospect. In this paper, the research progress of hydrothermal synthesis method of CFA zeolites is reviewed in detail and summarized several other synthetic methods of CFA zeolites. This review also presents an overview of CFA zeolites application in environmental applications like water treatment, gas adsorption and soil remediation. However, a considerable number of literature data have documented using CFA zeolites for water treatment, whereas research on CFA zeolites application to gas adsorption and soil remediation is still limited. In addition, the current status of basic research on the industrial production of CFA zeolites is briefly summarized, and the development trend of the synthetic zeolite of CFA is prospected. After the feasibility analysis of the industrial production of CFA zeolite, it is concluded that the only two methods with high feasibility for industrial application are two-step hydrothermal and alkali melting methods, and the industrial production technology still needs to be studied in depth.

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## 1 Introduction

In the 21st century, the world has witnessed an alarming rise in energy, resource, and environmental crises, which has prompted a renewed focus on energy conservation, environmental protection, and waste recycling. Coal fly

ash (CFA) is a major industrial solid waste that has become a pressing environmental concern globally. In 2020, the China production of CFA was estimated to be approximately 650 million tons. The large-scale accumulation of CFA has led to significant environmental impacts, such as soil land occupation (Zhang et al., 2021a), air pollution from CFA blowing into the atmosphere (Ahmaruzzaman and Gupta, 2012), and potential health and ecological risks to local communities

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(Makgabutlane et al., 2020; Ju et al., 2021). Therefore, there is a critical need to develop effective strategies for the management and utilization of CFA to mitigate its adverse effects on the environment. Unutilized CFA has become a severe obstacle to sustainable development, making its disposal and utilization crucial. Despite CFA extensive use in cement production, wall materials, refractories, road construction, backfilling, and other low-end fields (Qin et al., 2021; Stoy et al., 2021), there is an urgent need to recycle CFA efficiently and with added value. Indeed, the synthesis of zeolites from CFA can broaden the ways of resource utilization of CFA with economic and environmental benefits effectively (Amrhein et al., 1996).

The treatment of water, gas pollution and soil has emerged as an urgent concern due to the rapid development of industry and social economy (Zhao et al., 2021). In this context, the development of low-cost and efficient adsorption materials constitutes a critical aspect of pollution control. Zeolites, a kind of inorganic aluminosilicate mineral crystal that first discovered and named by Swedish mineralogist Cronstedt in 1756 (Cronstedt, 1756), have gained widespread application as industrially relevant adsorbents owing to their regular pore structure (Shi et al., 2020; Kwon et al., 2021), high cation exchange capacity (Abdelrahman et al., 2021; Kumar et al., 2021) and good thermal stability (Fan et al., 2008; Ma et al., 2014). Commercial zeolites are typically synthesized using pure chemical raw materials (Zhang et al., 2011b; Ren et al., 2012; Lou et al., 2016), which can be costly and limit their application in the field of environmental protection. For instance, zeolite A, commonly used for adsorption materials, can cost between 9000 to 15000 yuan/t. The high cost of these materials can be a significant barrier to their widespread use in environmental protection applications. Therefore, there is a need for alternative, cost-effective methods for synthesizing zeolites that can be used in environmental protection. Finding suitable, widely sourced, and cheap raw materials is urgently desired. The F-class CFA are usually rich in  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ , and include a small amount of  $\text{Fe}_2\text{O}_3$ ,  $\text{CaO}$  and other oxides (Chen et al., 2018a), rendering it a suitable raw material for synthesizing zeolites due to its analogous chemical composition. Achieving the strategic objective of high-value utilization of CFA by combining it with the pertinent issue of environmental pollution control to realize “treating waste with waste and turning waste into treasure” is a very worthwhile research topic (Yu et al., 2014; Ju et al., 2023). This endeavor has remarkable significance for promoting energy conservation and emission reduction, and fostering harmonious economic and social development.

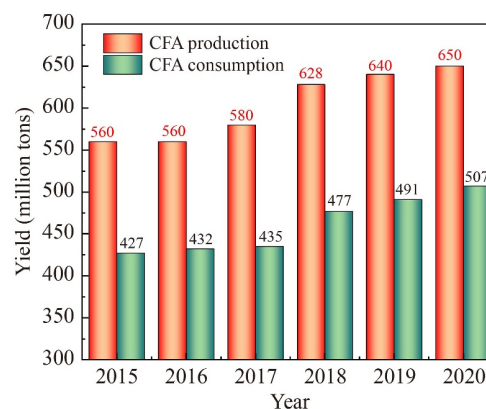
The synthesis of CFA zeolite has a history of more than 30 years (Chen et al., 2018b). Starting from the most traditional hydrothermal method, researchers have improved the method of synthesizing zeolite from CFA

by using a variety of new approaches, and various types of zeolite products have been developed (Belviso, 2020). To fully exploit the potential of CFA as a precursor for zeolite synthesis and expand its application in the environmental field, this study provides a comprehensive review of the synthesis technology and status of CFA-based zeolites. The associated challenges in the synthesis of synthetic zeolite are also discussed in detail, along with future recommendations. The review aims to provide a comprehensive understanding of the advantages and limitations of CFA-based zeolites, and to identify areas where further research is needed.

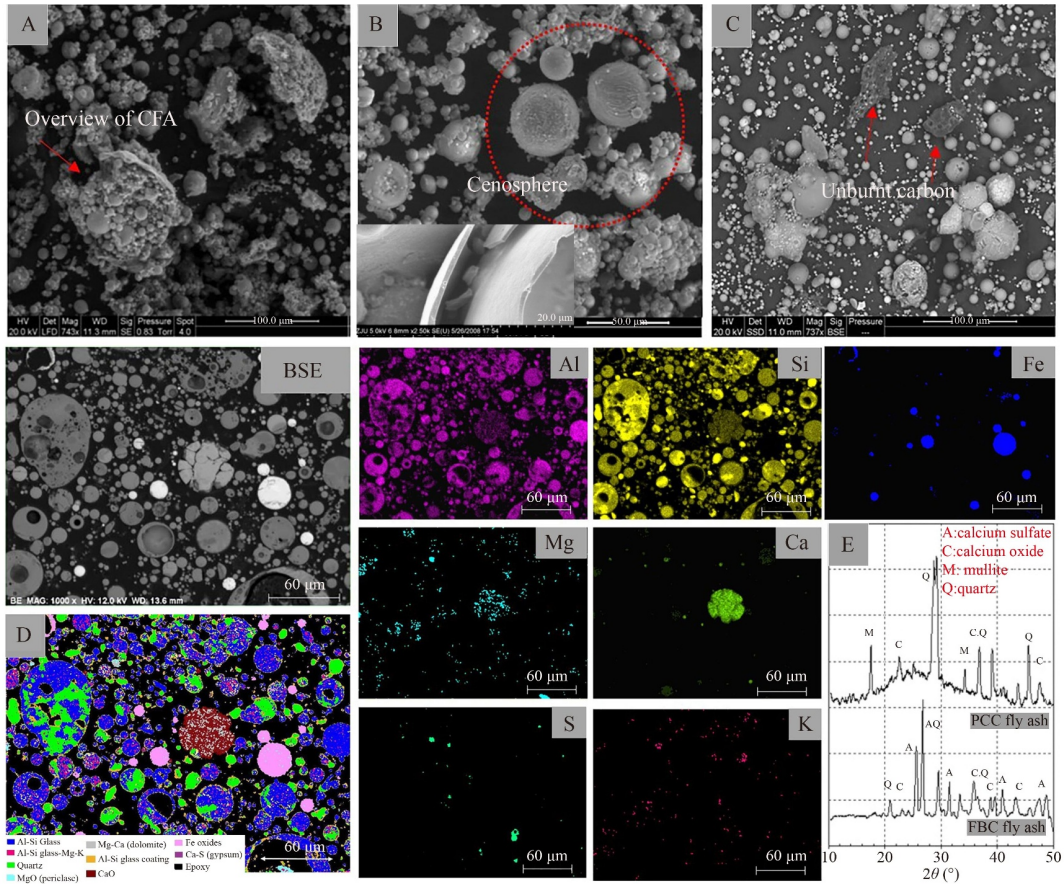
## 2 Properties of CFA

CFA, also known as fly ash, is produced by the combustion of pulverized coal in the furnace at 1200–1300 °C (pulverized coal furnace) or 800–950 °C (circulating fluidized bed boiler), which is the dust collected from coal-fired flue gas in thermal power plants (Xu et al., 2022). Fig. 1 provides CFA production and utilization in China, which has been consistently increasing with an annual output of 650 million tons in 2020. CFA utilization is about 3/4th of the total production. CFA displays a range of colors, from light gray to dark brown, which are determined by combustion conditions, composition, particle size, and water content. Generally, anthracite and bituminous CFA possess higher carbon residues and appear darker, while sub-bituminous or lignite CFA contains less carbon remains and appear light gray (Gollakota et al., 2019). In terms of physico-chemical properties, CFA has a bulk density of 0.5–2.8 g/cm<sup>3</sup>, particle size ranging from 2.5–300 μm, and a specific surface area of 300–500 m<sup>2</sup>/kg.

The pH of CFA is influenced by the Ca/S molar ratio in the ash, with most ashes tend to be alkaline (Yao et al., 2015). The SEM images (Figs. 2A–2C) reveals that the CFA particles are primarily spherical and composed of



**Fig. 1** Production and comprehensive utilization of CFA in China from 2015 to 2020.



**Fig. 2** The characterization results of CFA, (A–C) the SEM images, (D) The cluster map showing distinction of phases considering major and minor elements, (E) XRD patterns of FBC and PCC-fly ashes, and EDS elemental maps of CFA (Yao et al., 2015; Mohebbi et al., 2022; Chindaprasirt et al., 2011).

cenospheres, irregular-shaped debris and porous particles (unburnt carbon), and the cenospheres are hollow spheres formed in the process of combustion, with a wall thickness to diameter ratio limited to 2.5%–10.5% (Ju et al., 2021). The EDS elemental maps and cluster map (Fig. 2) provide a comprehensive view of the major, minor, and trace elements distribution, as well as the main phases present in the sample, including Al-Si glass, periclase, quartz, gypsum, calcium and iron oxides.

The chemical composition of CFA is mainly affected by the properties of coal combustion. Table 1 presents the chemical compositions of CFA obtained from different coal, including  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{Fe}_2\text{O}_3$  and a small amount of unburnt carbon. Compared with bituminous coal and anthracite, the content of  $\text{CaO}$  and  $\text{SO}_3$  in CFA after combustion of sub-bituminous coal and lignite is higher, and the content of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  is lower. Furthermore, CFA also contains trace elements, such as Zn, Pb, Cr, Ni, Ba, V and Sr, which are known to have negative impacts on the environment. The American Society for Testing Materials standard (ASTM) divides CFA into two classes according to the  $\text{CaO}$  content: F ( $\text{CaO} < 10\%$ ,  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 > 70\%$ ) and C ( $\text{CaO}$

**Table 1** Chemical compositions of CFA obtained from different coals (Ahmaruzzaman, 2010; Sahoo et al., 2016)

Composition (wt.%)	Bituminous	Subbituminous	Lignite	Anthracite
$\text{SiO}_2$	20–60	40–60	15–45	28–57
$\text{Al}_2\text{O}_3$	5–35	20–30	10–25	18–36
$\text{Fe}_2\text{O}_3$	10–40	4–10	4–15	3–16
$\text{CaO}$	1–12	5–30	15–40	1–27
$\text{MgO}$	0–5	1–6	3–10	1–4
$\text{Na}_2\text{O}$	0–4	0–2	0–6	0–1
$\text{K}_2\text{O}$	0–3	0–4	0–4	0–4
$\text{P}_2\text{O}_5$	0–4	0–2	0–10	0–5
LOI	0–15	0–3	0–5	1–8

$> 20\%$ ,  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 > 50\%$ ) (American Society for Testing and Materials, 2005). The mineral composition of CFA is related to coal formation, deposition, geological factors, and combustion conditions. Approximately 188 mineral groups and 316 individual minerals have been identified in various CFA samples (Vassilev et al., 2003; Vassilev and Vassileva, 2005). CFA is mainly divided into two types: pulverized coal combus-

tion (PCC) fly ash and fluidized bed combustion (FBC) fly ash, as the difference in combustion methods. Fig. 2E shows the XRD diffraction patterns of fly ash obtained by two combustion routes, PCC fly ash consists mainly of mullite, quartz and some glass phases (amorphous aluminosilicate and silica), and FBC fly ash is mainly composed of calcium sulfate, quartz, calcium oxide and a small amount of dicalcium silicate (Chindaprasirt et al., 2011). CFA usually have higher contents of silicon and aluminum components (50%–70%) (Zaheer, 2022), which exhibit effective chemical activity under alkaline conditions. In the context of converting CFA into zeolites, a high degree of amorphous aluminosilicate glass is beneficial, as this form can dissolve in the most efficient way in a basic synthesis environment. Kunecki et al. (2021) reported that applying a grinding process before synthesizing zeolites significantly improves their potential applications at relatively low costs.

In the realm of dual carbon, reduction of CO<sub>2</sub> emissions and development of capture technology have emerged as a significant research focus. Verrecchia et al. (2020) have conducted a study on the adsorption capacity of CFA zeolite for CO<sub>2</sub>. The CO<sub>2</sub> adsorption experiment performed in this study have revealed that CFA exhibits null CO<sub>2</sub> adsorption capacity, thereby indicating it's not a proper material for CO<sub>2</sub> capture. In contrast, CFA zeolite has demonstrated a CO<sub>2</sub> adsorption capacity of 3.84 molCO<sub>2</sub>/kgzeo, thereby establishing its potential as a low-cost material for CO<sub>2</sub> capture. Furthermore, CFA zeolite has demonstrated promising adsorption capacity in heavy metal wastewater treatment (Joseph et al., 2020) and soil remediation (Zheng et al., 2020) compared to the original CFA. These findings hold significant relevance in the

ongoing efforts toward realizing sustainable and eco-friendly industrial processes.

### 3 Method for synthesizing zeolite from CFA

Zeolites produced from CFA is gaining notice as one of high value-added application, possibly due to the similar chemical compositions of CFA and contains some volcanic substances (Boycheva et al., 2021). The geothermal action of volcanic craters converts the glass phase solution in volcanic material into zeolite, providing a potential route for zeolite synthesis from CFA (Tanaka et al., 2008). The first attempt to synthesize zeolite from CFA via a hydrothermal method was reported by Höller and Wirsching (1985), and subsequent articles have proposed various methods and conditions for zeolite development from CFA (Franus et al., 2014; Panek et al., 2016).

#### 3.1 Conventional hydrothermal method

The hydrothermal method is an effective method for producing artificial crystal materials by introducing suitable precursors (typically oxides or gels) into a closed, high-temperature and high-pressure reaction system (Tauanov et al., 2018). This process dissolves and recrystallizes solid substances that are typically difficult to dissolve under normal conditions. In the initial stage of exploring methods for synthesizing zeolite from CFA, researchers primarily investigated relatively simple processes such as the one-step method, two-step method, and alkali melting method. Fig. 3 portrays a schematic

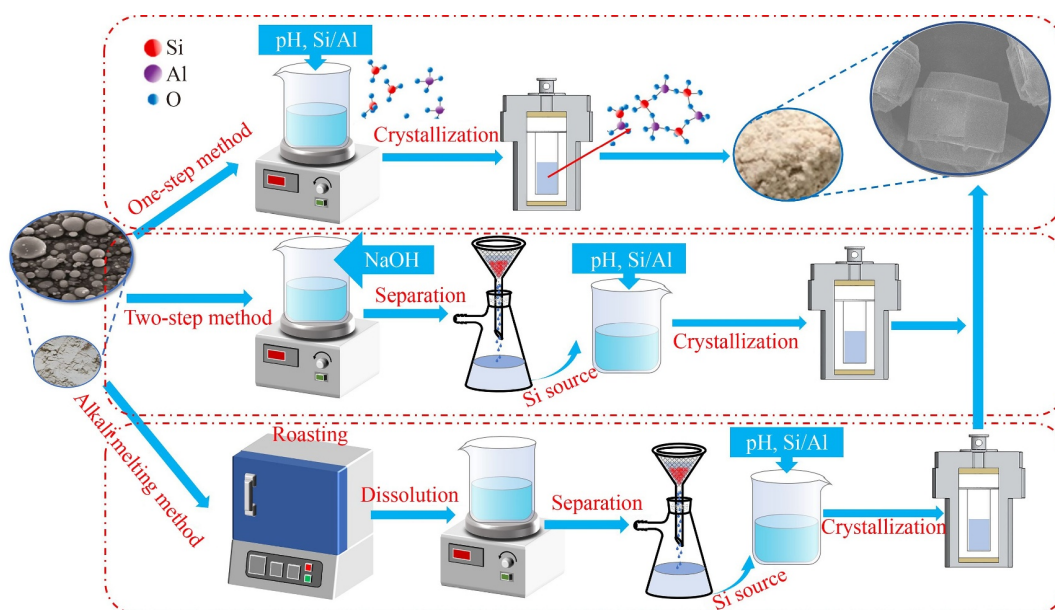
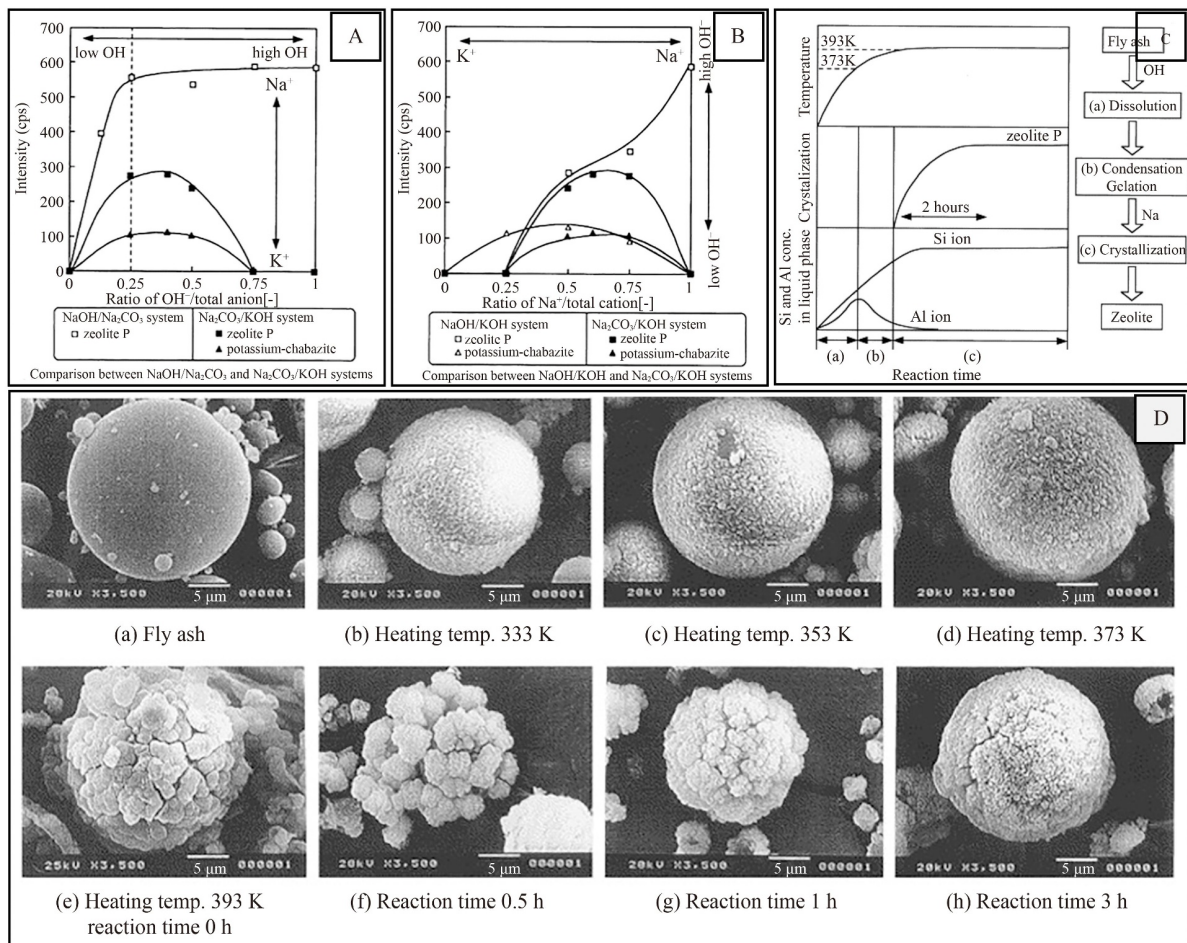


Fig. 3 Schematic illustration of one-step method, two-step method and alkali melting method synthesis of zeolites from CFA.

illustration of these techniques used for synthesizing zeolites from CFA.

**One-step method:** the one-step method is a method in which CFA and lye are directly added into the reactor according to a suitable ratio for hydrothermal synthesis. [Murayama et al. \(2002\)](#) investigated the reaction mechanism and role of alkali in this process. As shown in [Figs. 4A–4C](#), there exist three steps in the zeolite synthesis process: 1) the dissolution reaction of CFA occurs in the temperature range of 293–393 K as the first step; 2) condensation of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  in alkali solution leads to the formation of Si-Al gel that deposits as large flakes on the particle surface ([Fig. 4D\(e\)](#)), and 3) polycondensation of the Si-Al gel results in the crystallization of zeolite crystals. The  $\text{OH}^-$  in alkali solution plays a crucial role in the crystallization of zeolites ([Nada and Larsen, 2017](#)), significantly improving the growth rate of zeolite crystals and influencing the crystal phase composition of the product. [Figs. 4A](#) and [4B](#) illustrates the X-ray diffraction intensities of zeolite P prepared in various alkali, elucidating the three roles of anions and cations in zeolite synthesis: 1) the main factor

determining the total reaction rate of zeolite synthesis is the concentration of  $\text{Na}^+$  in the reaction solution; 2)  $\text{OH}^-$  in alkali solution contributes to the dissolution of CFA and accelerates the depolymerization reaction, while  $\text{Na}^+$  is helpful for the crystallization of CFA zeolite; 3) the crystallization rate decreases with the increase of  $\text{K}^+$  concentration under the condition of  $\text{Na}^+$  and  $\text{K}^+$  coexist in the alkaline solution. [Musyoka et al. \(2012\)](#) further monitored the formation of A-type zeolite from CFA precursor using an *in situ* ultrasonic system and showed that the formation of zeolite A from CFA follows a solution- and solid-phase transition mechanism. Whereas the amorphous gel structure is continuously destroyed during the crystallization process, suggesting the amorphous gel acts as both nutrient reservoirs and hosts for nucleation sites. Researchers generally believe that in hydrothermal synthesis systems, the formation of zeolites is mainly determined by the formation of aluminosilicate gels in solution. Although one-step hydrothermal method is easy to operate, it needs long hydrothermal reaction time, leading to high energy consumption ([Musyoka et al., 2013](#)), meanwhile, the growth mechanism of



**Fig. 4** (A, B) X-ray diffraction intensities of the zeolite P synthesized in various alkali; (C) Proposed reaction mechanism for zeolite synthesis; (D) SEM images of CFA and the products obtained in various synthesis parameters ([Murayama et al., 2002](#)).

zeolites synthesized with CFA needs further study due to the complexity at present, and zeolites synthesized with CFA still display some technical shortcomings such as low purity and crystallinity, poor controllability of crystal form, and so on.

**Two-step method:** the two-step method was developed based on one-step method, and involves two main steps: 1) mixing CFA with alkali liquor according to the appropriate solid-liquid ratio, the shell on the surface of CFA is broken to promote the dissolution of active substances, a suspension of CFA dispersed in this alkali liquor is employed as the starting suspension; 2) the Si/Al ratio and OH<sup>-</sup>/CFA ratio in the filtrate are adjusted before heating the gel in an autoclave for a period of time to promote crystallization. This results in the extraction of various types of zeolites such as P- (Xie et al., 2013; Kumar and Jena, 2022; Kunecki et al., 2023), A- (Tanaka et al., 2006; Hui and Chao, 2008; Hong et al., 2017; Iqbal et al., 2019; Amoni et al., 2022), LTA- (Tanaka et al., 2004), MOR- (Zhou et al., 2021), and MFI-type zeolites (Missengue et al., 2017). For instance, Zhu et al. (2018) used CFA as the main precursor material to prepare a uniform defect-free NaA zeolite membrane layer with a thickness of about 6.0 mm on the inner surface of Al<sub>2</sub>O<sub>3</sub> hollow fiber carrier via two-step hydrothermal method. This method proved effective in treating low concentration Pb<sup>2+</sup> wastewater, achieving a Pb<sup>2+</sup> removal efficiency of up to 99.9% at 0.1 MPa after 12 h of filtration. The two-step hydrothermal method has emerged a promising approach for fully utilized fully utilizing waste liquid from the one-step method, resulting in high conversion rates and zeolite purity. However, this method has been limited by its technological complexity, high cost, and difficulty in controlling reaction conditions (Tanaka and Fujii, 2009). To overcome these challenges, further optimization and development of this method are necessary to achieve high-quality zeolite production.

**Alkali melting method:** the alkali melting activation method is a technique used to enhance the activity of CFA through the destruction of inert materials at high

temperature under solid strong alkali roasting conditions (Park et al., 2019; Ma et al., 2010). The schematic illustration of alkali melting method synthesis of zeolites is shown in Fig. 3. This method involves calcining CFA with solid strong alkali, followed by grinding and adjusting the Si/Al ratio of the mixed solution with distilled water, and extracting zeolites from the resulting solution. There are three different types of zeolites (zeolite X, zeolite Y, and zeolite SSZ-31) are synthesized by alkali melting method at different NaOH/CFA weight ratio and crystallization condition (Sivalingam and Sen, 2018). The crystallinity of zeolite X can reach 76%, and the removal rate of Crystal Violet (CV) can be as high as 99.62%. Research suggests that in the process of alkali calcination, alkali can dissolve the glass body in CFA at high temperature and destroy the inert crystal phase structure, thereby stimulating the activity of CFA, which is conducive to the conversion of CFA to zeolite (He et al., 2016). The alkali melting method can effectively stimulate the activity of CFA, improve the conversion rate of zeolite, and obtain zeolite with high purity and good performance in a short time. As a result, it has been widely used.

In summary, numerous studies have been conducted on the synthesis of zeolites using the conventional hydrothermal method from CFA. It has been found that the type of zeolite is mainly determined by the synthesis conditions, including the physical and chemical properties of the reactant (Si/Al ratio), the base solution (solid/liquid and NaOH/CFA), and the synthesis conditions (temperature, time, pH, etc.), as summarized in Table 2. These findings highlight the importance of controlling the synthesis parameters to achieve desired zeolite properties.

### 3.2 New composite hydrothermal method

The conventional hydrothermal method has been the mainstream approach for synthesizing CFA zeolites. However, this strategy suffers from several drawbacks, including low product purity, poor repeatability, long

**Table 2** Zeolite synthesized by conventional hydrothermal method from CFA

Method	Zeolite	Hydrothermal conditions				Ref.	Remarks
		NaOH or NaOH/CFA	Solid/liquid (g/cm <sup>3</sup> ) or Si/Al	T (°C)	t (h)		
One-step method	Na-P1	2 mol/L	1/4	120	3–24	Murayama et al. (2002)	NaOH, Na <sub>2</sub> CO <sub>3</sub> and KOH
	Na-X	2–13	–	90–100	8–48	Mondragon et al. (1990)	Na-P1
	Blend	2.5–3.5	0.88–1.10	159	3.8	Moriyama et al. (2005)	Na-A, Na-G
	Na-P1	2.8–5.0	0.28–0.5	25	48	Ma et al. (1998)	–
	Na-P1	2 mol/L	–	90–150	12	Steenbruggen & Hollman (1998)	–
	K-CHA	3 mol/L	–	160	3	Murayama et al. (2008)	KOH
	Na-P1	2 mol/L	–	150	24	Querol et al. (2001)	SOD, TOB
	Na-P1	2/3.5 mol/L	1/4–1/8	100	24	Inada et al. (2005)	–

(Continued)

Method	Zeolite	Hydrothermal conditions				Ref.	Remarks	
		NaOH or NaOH/CFA	Solid/liquid (g/cm <sup>3</sup> ) or Si/Al	T (°C)	t (h)			
Two-step method	Na-P1	2 mol/L	2.0	90	48	Hollman et al. (1999)	–	
	Na-X	2 mol/L	1.8	90	48		–	
	Na-A	2 mol/L	1.2	90	67		SOD	
	Na-P1	3 mol/L	2/9	80	36	Wdowin et al. (2014)	–	
	Na-P1	3 mol/L	2/9	80	26	Kunecki et al. (2018)	–	
	Na-P1	2 mol/L	1.5/9	95	24	Xie et al. (2013)	Na-A	
	Na-P1	1 mol/L	1/10	150	60	Kumar et al. (2022)	–	
	SOD	5 mol/L	1/10	150	20		–	
	Analcime	3 mol/L	1/10	150	20		–	
	Na-A	2 mol/L	Si/Al=1	90–95	1.5–2.5	Iqbal et al. (2019)	Aging time: 12 h	
	Na-A	4 mol/L	Si/Al=1.93	100	4	Amoni et al. (2022)	–	
	Na-A	2 mol/L	–	90	1.5	Hui et al. (2008)	Second reaction T: 95 °C 2.5 h	
	Na-A	–	Si/Al=0.9	85	24	Tanaka et al. (2004)	SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> ≥ 1.7, Na-X	
	MOR	–	Si/Al=3.92	150	24	Zhou et al. (2021)	–	
	MFI	–	Si/Al=5.8	160	72	Missengue et al. (2017)	Structure-directing agent: TPABr	
	Na-A	2.2 mol/L	Si/Al=0.5	85	24	Tanaka et al. (2009)	Trace amount of sod	
	Na-A	2.2 mol/L	Si/Al=1.0	85	24		Single phase	
	Na-X	2.2 mol/L	Si/Al=4.5	85	24		2 ≤ SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> < 4.5, Blend	
	Na-P1	3 mol/L	Si/Al=1.43	105	24	Derkowski et al. (2006)	–	
	SOD	5 mol/L	Si/Al=1.21	105	24		Add: 3 mol/L NaCl	
	Na-X	3 mol/L	Si/Al=1.1	75	24		–	
	NaA-X	Na <sub>2</sub> O/SiO <sub>2</sub> =1.4	Si/Al=2.1	90	8	El-Naggar et al. (2008)	Ageing T: 24 h	
	Alkali melting method (Fusion: 550–600 °C for 1–2 h)	Na-X	1.2	1/10	90	7	Verrecchia et al. (2020)	NaOH/CFA > 1.2, SOD
		Na-Y	0.9	Si/Al=4, 5	90	12	Ren et al. (2020)	Ageing for 24 h at 55 °C
		Na-X	1.0–1.2	Si/Al < 5	90	6	Molina & Poole (2004)	–
Na-X		–	1.6–2.4	90	4–8	Boycheva et al. (2021)	–	
Na-A		1.5	Si/Al=1	100	24	Hong et al. (2017)	–	
Na-X		1.2	Si/Al=1.78	90	15	Zhang et al. (2017)	–	
SOD		1.0	Si/Al=1.25	105	24	He et al. (2016)	–	
Na-X		0.5	Si/Al=2.25	90	2	Kalvachev et al. (2016)	60% conversion	
Na-A		0.33	Si/Al=1.22	100	24	He et al. (2020)	With addition 3% HCl	
Na-P1		2 mol/L	Si/l=2.01	100	13	Ye et al. (2008)	Fusion: 830 °C, 1 h	
Na-A		2 mol/L	Si/l=0.91	100	5		10% Na-A seed	
Na-X		2 mol/L	Si/l=0.91	100	5		2 vol.% directing agents	
Na-A		2–1.2	Si/Al=1.6	100	12	Fotovat et al. (2009)	Si/Al=3, FAU	
Na-X		N.A.	–	35–60	48	Belviso et al. (2010)	ZK-5, using sea water	
Na-P1		0.2 mol/L	Si/Al=2.0	120	4	Kazemian et al. (2010)	–	
Na-X		1.2	Si/Al=1.0–1.2	100	24	Izidoro et al. (2013)	–	
Na-A		1.2	–	100	7		–	
Na-X	1.25	–2	80–100	5–10	Sivalingam et al. (2018)	NaOH/CFA=1.0, Na-Y		
CZC	2	–	750	1 (N <sub>2</sub> )	Zhao et al. (2022)	Carbon-zeolite composite (CZC)		

reaction cycle, and insufficient utilization of reactants (Chang and Shih, 2000; Moreno et al., 2001; Belviso et al., 2019). To overcome these limitations, researchers have proposed new explorations, as summarized in Table 3, which combine traditional hydrothermal methods with other technologies. Novel composite methods, such as microwave-assisted (Chen et al., 2021b), seed-assisted (Wang et al., 2015), Molten-salt, ultrasound-assisted and supercritical hydrothermal method have been developed (He et al., 2021). These approaches offer promising alternatives to traditional hydrothermal methods and have the potential to improve product quality, reduce reaction time, and enhance reactant utilization.

Microwave-assisted hydrothermal conversion: microwave-assisted synthesis offers unique advantages over conventional heating due to the energy transfer

mechanism of microwaves, which involves the interaction of dielectric molecules with molecular weights (Behin et al., 2014). Querol et al. (1997) first proposed the preparation of zeolites from CFA by microwave-assisted methods in 1997, which significantly reduced crystallization period compared to the conventional hydrothermal method. However, Inada et al. (2005b) found that delayed nucleation hindered the formation of zeolites in the intermediate stage of microwave heating, suggesting the need for a combination of early-stage microwave heating and later-stage conventional heating to promote the zeolitization of CFA. This conclusion is well aligned with the reports of Fukui et al. (2007) and Chen et al. (2021a). Fukasawa et al. (2018) synthesized K-zeolite by microwave hydrothermal treatment of CFA using biomass incineration ash extract and investigated

**Table 3** Zeolite synthesized by new composite hydrothermal method from CFA

Method	Zeolite	Hydrothermal conditions				Ref.	Remarks
		NaOH or NaOH /CFA	Solid/liquid (g/cm <sup>3</sup> ) or Si/Al	Heating	Time		
Microwave-assisted hydrothermal conversion	Na-A	1	Si/Al=1	100–300 W	10–30 min	Behin et al. (2014)	Crystallinity [%]: 67.24
	Na-P1	2 mol/L	–	500 W	15 min	Inada et al. (2005)	–
	Phillipsite	2 mol/L	–	2.45 GHz	Less 2 h	Fukui et al. (2007)	Rice husk ash as a silica source
	K-MER	4 mol/L	Si/Al=7	1000 W	5 min	Chen et al. (2021)	Ultrasound frequency was 25 kHz
	Phillipsite	KOH 1.5 mol/L	–	2.45 GHz	2–32 h	Fukasawa et al. (2018)	KCl and K <sub>2</sub> CO <sub>3</sub>
Seed-assisted method	ZSM-5	–	30	90	72 h	Zhang et al. (2020)	Seeds was 3 wt% to Si
	FAZ-A	2 mol/L	1–1.1	90–105 °C	3 h	Rayalu et al. (2001)	Zeolite-A was added as seed
	MRE	0.125	–	150	7 d	Ma et al. (2022)	Yield (%): 73
	SFE	0.125	–	150	7 d		Yield (%): 71
	MWW	0.125	–	150	4 d		Yield (%): 90
	MFI	0.125	–	150	5 d		Yield (%): 88
	IWV	0.125	–	150	7 d		Yield (%): 89
	TON	0.3	–	150	4 d		Yield (%): 45
	ERI	0.3	–	150	6 d		Yield (%): 44
	EUO	0.3	–	150	7 d		Yield (%): 53
Molten-salt method	Cancrinite Sodalite	–	–	350	12 h	Choi et al. (2001)	NaOH-NaNO <sub>3</sub>
	Cancrinite Sodalite Na-P	3 mol/L	–	350±5	24 h	Park et al. (2000)	NaOH-NaNO <sub>3</sub> (KNO <sub>3</sub> )
	Cancrinite Sodalite	Na <sub>2</sub> CO <sub>3</sub> /CFA=0.5:1	NaCl/CFA=0.3:1	790–950	5–10 h	Yao et al. (2011)	Na <sub>2</sub> CO <sub>3</sub> , NaCl
Ultrasound-assisted hydrothermal conversion	Na-A Sodalite	1–2 h = 60 °C; 3 h = 65 °C and 4 h = 75 °C	–	(240 W; 35 kHz)	4 h	Belviso (2018)	–
	Na-A	–	4	80–94	6 h	Hums et al. (2016)	(Fusion: 550–600 °C for 1–2 h)
	Na-A	2.4 mol/L	–	100	2 h	Ojumu et al. (2016)	
	ZSM-5	–	–	24 kHz	95–145 h	Chen et al. (2020)	Irradiation powers (97, 194, 323 W)
Supercritical hydrothermal method	Sodalite	8 mol/L	15:1	400	5 min	Wang et al. (2015)	–
	Cancrinite	2–4 mol/L	–	–	–	–	–
	Cancrinite	5:1	2.5:1	400	5 min	Zhao et al. (2016)	Na <sub>2</sub> CO <sub>3</sub> as activator

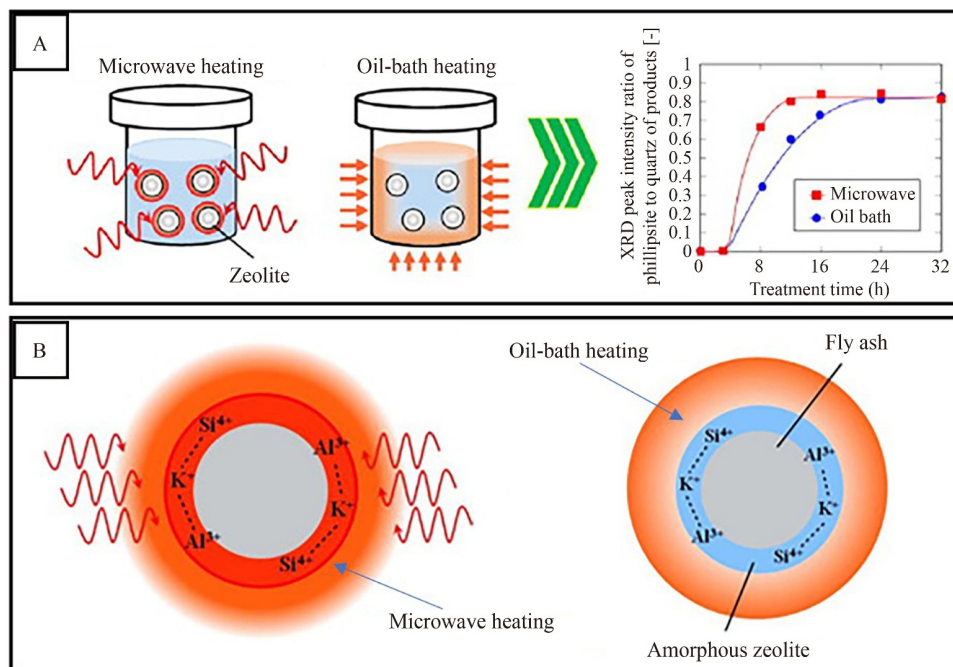


the effect of microwave heating on the rate of crystalline zeolite synthesis by comparison with oil bath heating. Fig. 5A clearly shows that the microwave heating significantly reduced the synthesis time of K-zeolite compared to using oil-bath heating. This is due to the higher rate constant ( $k$ ) of zeolite crystal formation with microwave heating, which was more than five times that of external heating. While microwave-assisted hydrothermal conversion is simple, convenient, and greatly reduces the crystallization time, it still has some issues, such as low conversion rate of high-quality zeolites and low crystallinity, which require further research.

Seed-assisted method: the addition of a small number of crystals, known as “seeds,” to the synthesis material can induce nucleation growth and increase the crystallization rate (Zhang et al., 2020a). Zeolites are stable crystals, making it difficult to synthesize a single crystal form of zeolite in a single crystallization reaction without a structure-directing agent. In fact, multiple forms of zeolite may be formed in a single reaction (Bukhari et al., 2015). Optimizing the reaction conditions can create favorable conditions for zeolite production, and seed-assisted methods are a strategy for pushing the reaction to specific zeolites (Rayalu et al., 2001). Larosa et al. (1992) used natural zeolite as seed crystal to synthesize zeolite P and Zeolite Y from Class F CFA, which not only shortened the crystallization time, but also improved the purity of zeolite. Moreover, the study showed that in the presence of Phillipsite seeds, zeolite P can be formed at a lower crystallization temperature of 60 °C compared to samples

without seed added. Crystal kinetics studies have shown that adding seed crystals can significantly reduce the induction period of CFA zeolites (Kacirek and Lechert, 1976). Slangen et al. (1997) synthesized A-type zeolite using a microwave-assisted method, where the mixture was aged for different times (50, 60, 120, 180, 240, 1200 min), resulting in varying degrees of zeolite crystallinity (0%, 0%, 0%, 80%, 90%, 100%). Microwave heating for only 1 min was sufficient to produce zeolite A, compared to conventional heating (2 h). Interestingly, when aged samples were mixed with unaged samples, the unaged samples produced zeolite A with better crystallinity and shorter crystallization times, indicating that aging the sample prior to heat treatment is critical for generating zeolite crystals, possibly because seeds formed in the aged solution (Bukhari et al., 2015). Adding seed crystals can shorten the nucleation induction period, reduce the crystallization reaction time, simultaneously, suppress the generation of miscellaneous crystals, which is helpful for selecting and synthesizing specific types of zeolites (Rayalu et al., 2001). Seed-assisted method has certain development prospects in large-scale applications, but the induction mechanism requires further theoretical support for its promotion.

Molten-salt method: the molten-salt method offers an alternative approach to convert coal fly ash (CFA) into zeolites by employing (e.g., NaOH-NaNO<sub>3</sub> or NaOH-KNO<sub>3</sub>) as the reaction medium without the addition of water (Choi et al., 2001). This method has been proposed as a means to reduce waste liquid and improve product purity by forming cancrinite and sodalite using alkali and



**Fig. 5** (A) Relationships between the ratio of XRD peak intensities and treatment time for microwave and oil-bath heating; (B) Schematic representations of the temperature distributions around CFA and zeolite (Fukasawa et al., 2018).

salt instead of a solution (Park et al., 2000). Compared to the hydrothermal method, the zeolite material produced via the molten-salt method has higher alkali metal content, lower pH values, and a higher Si/Al ratio. NaCl has been found to function as a melting aid, reducing the combustion temperature of carbon and improving the conversion rate of sodalite (Yao et al., 2011). Although adding a certain proportion of NaNO<sub>3</sub> to the mixture of CFA and alkali can facilitate the formation of zeolite grains at temperatures below the melting point of the salt and base in the NaOH-NaNO<sub>3</sub> system, the conversion and purity of zeolite are still suboptimal (Choi et al., 2001). Despite the lack of water usage during the preparation process, a significant amount of water is still required for post-treatment and washing. Otherwise, the molten-salt method requires a higher temperature, larger quantities of salt, and may result in more impurities in the final product, thereby limiting its widespread application.

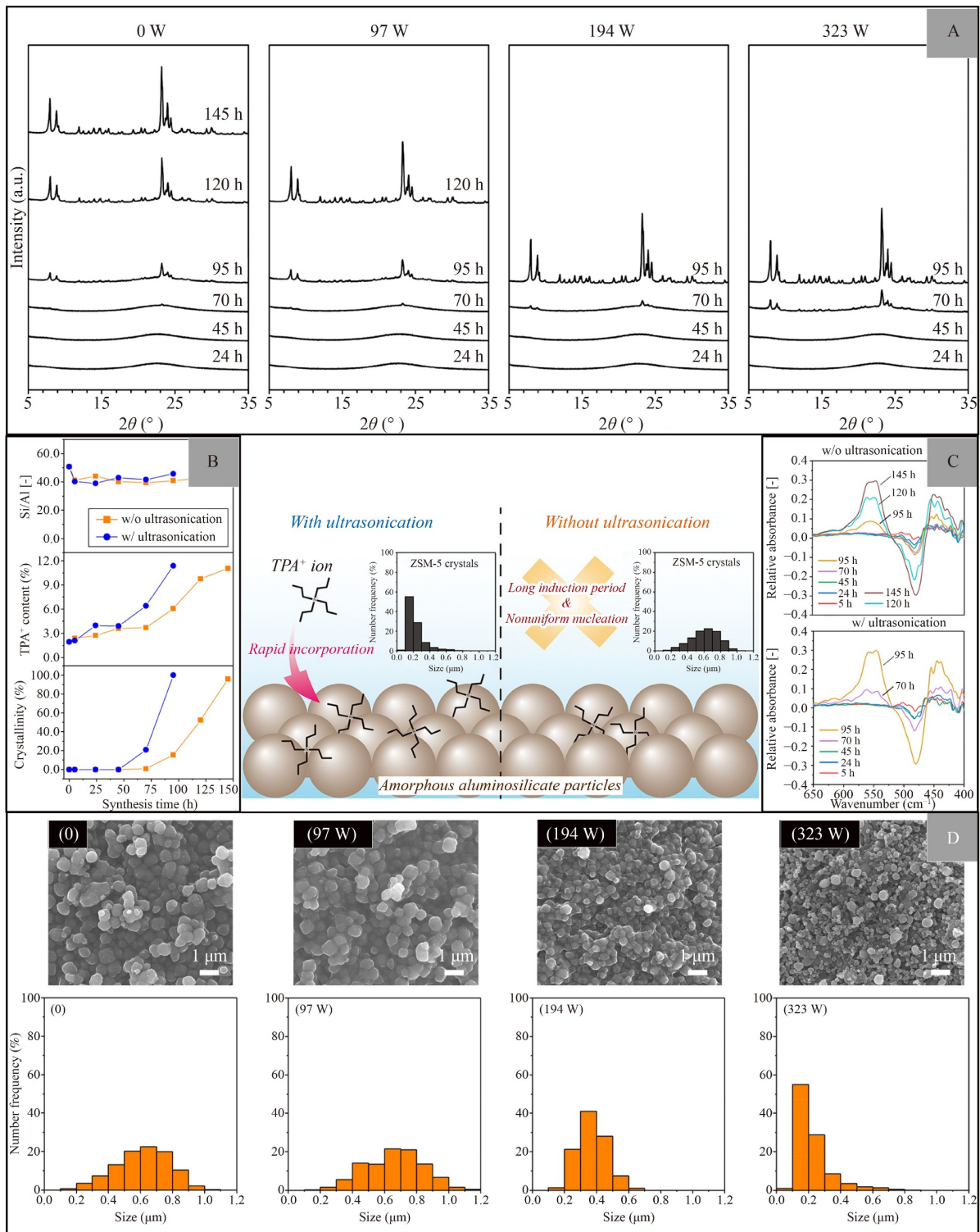
**Ultrasound-assisted hydrothermal conversion:** CFA is inherently stable and requires substantial energy input to activate and undergo structural changes for zeolite synthesis. Ultrasound is an ideal choice due to its high energy density and extra features, such as mechanical stirring, reducing reaction temperature and shortening reaction time (Hums et al., 2016; Belviso, 2018). There are three main applications of ultrasound for the synthesis of zeolites (Ju et al., 2021). 1) Firstly, it is used for raw material pretreatment to accelerate the dissolution of raw materials. Ojumu et al. (2016) reported that ultrasound is a viable alternative to the pre-fusion step, reducing the pretreatment time from 1.5 h to 10 min. 2) Secondly, ultrasound is an effective measure for enhancing the crystallization process. Aldahri et al. (2016) successfully converted CFA to zeolite Na-P through a 4 h thermo-sonochemical treatment (1 h conventional heating followed by 3 h of sonochemical treatment). Comparison between pre-ultrasound and post-ultrasound treatments showed that ultrasound promotes dissolution and crystallization, with pre-ultrasound accelerating dissolution but the crystallization rate not matching, while post-ultrasound promotes crystallization and accelerates zeolite formation but partially inhibits nucleation. 3) Finally, ultrasound is used throughout the hydrothermal process to inject energy for dissolution, condensation, nucleation, and crystallization. Ultrasound's high energy density and additional features, such as mechanical stirring, reduce reaction temperature, and shorten reaction time, make it an ideal choice for zeolite synthesis. Chen et al. (2020) investigated the impact of ultrasound on the crystallization behavior of ZSM-5. The study revealed that increasing the irradiation power decreased the crystallization time (Fig. 6A: it took about 145 h to obtain fully crystallized ZSM-5 without ultrasonication, while 95 h under the irradiation power of 323 W), which also modulates the nucleation and crystal growth of ZSM-5 to

synthesize crystals of different sizes (Fig. 6D). Fig. 6B demonstrated that ultrasound facilitated the formation of inorganic-organic complexes (IOCs) by enhancing the binding of organic cations to inorganic substrates. The appearance of a five-membered ring was confirmed by the splitting of the 550 cm<sup>-1</sup> band to a doublet after 70 h of reaction (Fig. 6C). In the ultrasonic system, the crystal sizes were small (Fig. 6D, particle size < 200 nm), leading to an increase in silanol groups on the external surface. The higher concentration of silanol groups facilitated the condensation reaction, creating a suitable environment for nucleation and crystal growth of zeolites. Overall, this study highlights the potential of ultrasound as an effective method for controlling the crystallization behavior and size of zeolites.

**Supercritical hydrothermal method:** supercritical hydrothermal synthesis is a promising approach to produce zeolite from CFA by utilizing the gas-liquid transport properties and ultra-low surface tension of supercritical water (Jin et al., 2010). This method has been shown to yield various types of zeolites, such as cancrinite and sodalite (Wang et al., 2015), in a significantly shorter time than conventional hydrothermal synthesis. For instance, ultrasound-assisted hydrothermal conversion can achieve complete crystallization within 1–10 min at 400 °C, compared to typical crystallization times of 12 to 48 h. Moreover, the synthesized zeolite exhibits excellent performance for Hg removal from simulated coal gas. Alkali fusion pretreatment of CFA prior to supercritical hydrothermal synthesis further enhances the efficiency of zeolite production (Zhao et al., 2016). The optimal crystallization conditions for cancrinite synthesis include a liquid to solid ratio of 5:1, a silicon aluminum ratio of 2.5:1, and a short crystallization time of 5 min at 400 °C. The fast reaction rates and low metal oxide solubility of supercritical hydrothermal synthesis enable high nucleation rates, leading to rapid zeolite formation.

### 3.3 Other methods for zeolites synthesizing

As the in-depth study of zeolite synthesis from CFA recent years, the synthesis methods have shown diversity: solvent-free, dialysis hydrothermal and gradual heating method have come out in succession. Xiao and coworkers (Ren et al., 2012b; Meng and Xiao, 2013; Wu et al., 2014) are the first to report a solvent-free method for zeolite synthesis, which can completely avoid the use of solvents by directly mixing, grinding, and heating the initial solid material. Recently, our research group have successfully prepared ZSM-5 zeolite with iron tailings and rice husk ash as raw materials by solvent-free method (Zhang et al., 2020b). The resulting product meets commercial-grade requirements, affirming that solid wastes like CFA can be utilized effectively in zeolite preparation. In summary, our findings demonstrate that



**Fig. 6** (A, C, D) XRD patterns, FT-IR spectra, SEM images and particle size distributions of the products obtained different ultrasonic irradiation powers; (B) Variation of Si/Al, amount of TPA<sup>+</sup>, and relative crystallinity with synthesis time under w/o ultrasonication ( $\blacksquare$ ) and 323 W ultrasonic ( $\bullet$ ) systems (Chen et al., 2020).

the solvent-free method is a promising approach to achieve efficient and eco-friendly zeolite synthesis using waste materials. Tanaka et al. (2006) employed the hydrothermal dialysis method, which utilizes a semi-

permeable membrane to infiltrate the silicon-alumina ions dissolved in CFA into NaOH, and adjusts the appropriate silicon-alumina ratio to prepare Na-A zeolite. The product synthesized by dialysis method has high relative

crystallinity and purity, but it suffers from two defects: high recycling costs of approximate 0.02\$ per gram of single-phase Na-A zeolite, and a long recycling period of 48 h to synthesize Na-A zeolite. The gradual heating method involves obtaining zeolite products after crystallization of materials at low and high temperatures for a period of time. Hui et al. (2006) synthesize zeolite 4A samples by step-change of synthesis temperature during hydrothermal treatment of CFA prepared solution. After heating at 90 °C for 1.5 h and then at 95 °C for 0.5–8 h, a single crystalline phase zeolite 4A with relative crystallinity of 80% was obtained. This approach can yield zeolite with high relative crystallinity and purity in a relatively short time, but the conversion and utilization rate of CFA is relatively low.

In summary, the process of synthesizing zeolite from CFA primarily considers factors such as CFA utilization rate, product purity, and process route. This work provides a comprehensive overview of the methods for preparing zeolite from CFA and their respective advantages, disadvantages and provide some instructive suggestion, as summarized in Table 4.

## 4 Environmental applications of CFA zeolites

The zeolite exhibits a well-defined pore structure, excellent thermal and hydrothermal stability, making it a promising catalysts and adsorbents for various chemical

industry applications (Asl Hosseini et al., 2018). CFA zeolite, due to its low purity and impurities (Iqbal et al., 2019), is limited applications in fine chemicals. However, it has been proven to be a highly effective adsorbent in environmental protection. Accordingly, this research exclusively concentrates on the environmental application of CFA zeolite, particularly in water treatment, gas adsorption, and soil remediation. Although CFA zeolite displays several potential uses, our study aims to examine its efficiency in addressing environmental challenges.

### 4.1 Application for water treatment

CFA zeolites possess regular pore channels and high ion exchange capacity, rendering them a promising option for water and wastewater pollutant treatment (Koshy and Singh, 2016). Synthesizing zeolites from CFA presents an economically viable and efficient adsorbent solution. Recent studies have demonstrated the remarkable effectiveness of CFA zeolites in removing heavy metals (Cardoso et al., 2015), ammoniacal nitrogen (Ding and Sartaj, 2015) and organic matter (Wang et al., 2021) from wastewater. As a result, CFA zeolites have emerged as a promising solution for wastewater treatment, with the potential to significantly enhance water resources quality and promote sustainable development.

**Application for heavy metal removal:** CFA zeolites have demonstrated effective removal of individual heavy metal ions such as Ni<sup>2+</sup> (He et al., 2020), Cu<sup>2+</sup> (Yang et al., 2019a), and As<sup>5+</sup> (Yang et al., 2019b), as well as

**Table 4** Characteristics of zeolites synthesized by different methods from CFA

Method	Advantage	Disadvantage	Advise
One-step method	Simple synthetic procedure	The more side reactions, long hydrothermal time, and lower purity	Selecting high activity CFA as raw materials and produce products with low purity requirements
Two-step method	High conversion and crystallinity	Technological complexity, high cost and The reaction conditions are difficult to control	Develop a more cost-effective and simplified protocol for the second step, while maintaining high conversion and crystallinity
Alkali melting method	High yield and purity	Procedures trival, and high energy consumption	It is currently the most promising solution to achieve industrial production, further studying the synthesis mechanism of zeolite, and improving conversion rate and crystallinity
Microwave-assisted method	Fast reaction rate and low energy consumption	More by product is produced and lower purity	Optimize reaction parameters to minimize byproduct formation and improve purification methods to increase purity
Seed-assisted method	Simple synthetic procedure and fast reaction rate	Technological complexity and lower purity	Simplify the procedure and improve purification methods to increase yield and purity
Molten-salt method	Avoids the use of water solvents	High temperature, high salt dosages and impurity substance	Explore ways to reduce salt dosages and improve purification methods to increase product purity
Ultrasound-assisted method	Shortening reaction time, and reducing reaction temperature	Complicated infrastructure, high costs	Develop more cost-effective infrastructure and adjust parameters to maximize efficiency and reduce costs
Supercritical hydrothermal method	Very fast reaction rate and high crystallinity	The reaction mechanism is complex and high cost	Further investigate the mechanism of the reaction to optimize conditions and develop more cost-effective ways to run the process
Solvent-free method	High yield, better utilization of autoclaves, and reduction of pollutants	The raw material pretreatment process is complex, and lower purity	Streamline raw material pretreatment and develop more efficient purification methods to increase product purity
Dialysis hydrothermal	The relative crystallinity and purity of the product are both high	Dialysis requires a long reaction period, and the solution cannot be recovered	Investigate new materials for dialysis membranes to reduce reaction time and improve solution recovery
Gradual heating method	Relatively high crystallinity and high purity	Conversion rate and utilization rate of fly ash are both low	Develop new catalysts or additives to increase fly ash utilization rate and improve conversion efficiency

simultaneous removal of multiple heavy metal ions simultaneously (Joseph et al., 2020). Recent research efforts have focused on producing zeolites from CFA and their applications in water adsorption, as summarized in Table 4. The absorption rate of heavy metal ions is dependent on various parameters, including zeolite type, initial concentration and type of heavy metal ions, dosage (liquid/solid ratio), and operating conditions (temperature, interaction time and pH, etc.) (Remenárová et al., 2014). Wu et al. (2008) reported that the smaller pore size hydroxysodalite exhibits higher adsorption capacity for  $\text{Cr}^{3+}$  than NaP1 zeolite due to the prevention of competition from larger-sized innocuous cations (mainly  $\text{Na}^+$  and  $\text{Ca}^{2+}$ ), highlighting the selective adsorption of heavy metals by different types of zeolites based on their pore structures. Table 5 shows that increasing contact time enhances the removal efficiency of heavy metal ions

due to greater interaction between pollutants and adsorbents. The adsorption time varies for different heavy metal ions, with  $\text{Pb}^{2+}$  reaching equilibrium within 25 min, and  $\text{Cr}^{3+}$  requiring 4 h when CFA zeolite is used to treat heavy metal-containing waters (Visa et al., 2012). Moreover, the pH of the system affects the performance of pollutant-zeolite interactions, which can be adjusted by adding an appropriate concentration and volume of acid or alkali during adsorption experiments (Apiratikul and Pavasant, 2008). Generally, CFA zeolites exhibit better removal efficiency of heavy metal ions at high pH ( $> 10$ ) due to the precipitation of hydroxides out of solution and cation exchange in the zeolite matrix (Sui et al., 2008; Deng and Ge, 2015).

Application for ammoniacal nitrogen removal: Ammonia nitrogen (free  $\text{NH}_3$  and  $\text{NH}_4^+$  in water in equilibrium), a common wastewater pollutants, is

**Table 5** Heavy metal ions uptake characteristics of CFA zeolites

Ionic species	Zeolite	CEC (meq/100 g)	$\eta$ (%)	$q_e$ (mg/g)	Operating conditions					Ref.
					pH	$T$ ( $^{\circ}\text{C}$ )	$t$ (min)	$C_i$ (mg/L)	Dosage (g/mL)	
$\text{Cr}^{3+}$	SOD, Na-P1	–	75.5	–	3.4	20	240	–	400	Wu et al. (2008)
	Na-A	105	100	–	–	–	24 h	–	0.2	Koukouzas et al. (2010)
	Phillipsite	–	100	–	1.55	20	60	1000	0.02	Itskos et al. (2015)
	Thomsonite	–	98	–	1.55	20	60	1000	0.02	Itskos et al. (2015)
	Na-A	–	87.3	35.6	3	25	240	200	0.005	Hui et al. (2005)
	ZSM-18	120	100	–	–	–	24 h	–	0.02	Koukouzas et al. (2010)
$\text{Cu}^{2+}$	Na-A	500	99.7	–	3	25	240	200	0.5	Wang et al. (2009)
	FAU	–	100	39.7	–	25	90	100	5	Joseph et al. (2020)
	FAA-CS	–	71	119.1	10.2	–	120	350	0.002	Visa & Chelaru (2014)
$\text{Zn}^{2+}$	Na-A	560	93.2	29.7	3	25	240	200	0.5	Wang et al. (2009)
	FAU	–	100	34.7	–	25	90	100	5	Joseph et al. (2020)
	Phillipsite	–	95	–	1.55	20	60	1000	0.02	Itskos et al. (2015)
	Thomsonite	–	60	–	1.55	20	60	1000	0.02	Itskos et al. (2015)
	Na-A	500	–	22.4	3	25	240	200	–	Wang et al. (2009)
	Na-P1	290	80.8	–	303	–	30	0.13	0.01	Cardoso et al. (2015)
$\text{Ni}^{2+}$	Na-A	105	99.9	–	–	–	24 h	–	0.2	Koukouzas et al. (2010)
	Na-A	–	94	47	–	25	180	100	2	He et al. (2020)
	Na-X	270.4	91	15.9	5	30	30	100	1	Zhang et al. (2018)
	Na-P1	228.7	48.3	–	4.6	–	–	0.52	0.08	Prasad et al. (2011)
$\text{Pb}^{2+}$	FAU	–	100	40	–	25	90	100	5	Joseph et al. (2020)
	Na-A	–	99.9	–	5.8	25	12 h	50	–	Zhu et al. (2018)
	CZC	–	–	185.68	7	–	40	–	0.1	Zhao et al. (2022)
	Phillipsite	–	–	48	1.55	20	60	1000	0.02	Itskos et al. (2015)
$\text{Cd}^{2+}$	FAU	–	100	40.3	–	25	90	100	5	Joseph et al. (2020)
	FAA-CS	–	60	156.2	10.2	–	120	515	0.002	Visa et al. (2014)
	Na-P1	290	94.1	–	3.3	–	30	0.22	0.01	Cardoso et al. (2015)
$\text{As}^{5+}$	Na-X	–	83	27.8	2.14	25	240	–	0.1	Yang et al. (2019)
	Na-P1	290	100	–	3.3	–	30	0.04	0.01	Cardoso et al. (2015)

typically removed through conventional methods such as activated sludge process. However, residual concentrations of  $\text{NH}_4^+$  remaining after treatment (30–50 ppm) can pose environmental concern if discharged into lower flow rate watercourse, contributing to eutrophication (Nguyen and Tanner, 1998). To address this issue, CFA zeolites with high ion exchange capacity provide an ideal material for  $\text{NH}_4^+$  incorporate additional treatment. From the perspective of reducing the cost of adsorbents for ammonia nitrogen wastewater treatment, protecting the environment, and achieving waste reuse, the use of CFA synthetic zeolite for treating ammonia nitrogen wastewater has tremendous advantages and has become a research hotspot for researchers. Ji et al. (2015) compared the efficiency of zeolites synthesized from low-calcium ( $\text{CaO} < 5\%$ ) and high-calcium ( $\text{CaO} > 15\%$ ) CFA and found that the former exhibited a higher ammonium removal rate due to the competition between  $\text{Ca}^{2+}$  and  $\text{NH}_4^+$ . The maximum removal efficiency of ammonium is 76.78%, achieved at an initial ammonium concentration of 5 mg/L, dosage 12 g/L, and pH of 8.0. Similarly, Cardoso et al. (2015) evaluated the efficacy of CFA produced zeolites in swine wastewater treatment and found Na-P1 have higher total ammonia nitrogen adsorption capacity (31 mg/g), and the removal rate is up to 50%. Furthermore, Juan et al. (2009) synthesized three types zeolites (NaP1, K-F and K-Chabazite/K-Phillipsite) from CFA for ammonium uptake and concluded that K-F zeolite has less effective in ammonia nitrogen treatment. It is worth noting that powdered zeolites exhibited a higher removal rate of  $\text{NH}_4^+$  from wastewater (80%) than granular zeolite (70%). These findings suggest that using low-calcium CFA zeolites holds promise for ammonium removal from wastewater and merits further exploration.

CFA zeolite also prove a very perfect effect in the treatment of organic contaminant (Atun et al., 2011), fluoride (Chen et al., 2022), and radioactive substances (Khaleque et al., 2020) etc. in wastewater. However, it's the limited yield and adsorption capacity have hindered its industrial application in water treatment. Current studies on CFA zeolite materials mainly revolve around their modification with acids, alkalis, rare earths, iron ions etc., and research on developing new, efficient, and robust wastewater adsorbents in combined with other advantageous water treatment agents is inadequate. Therefore, achieving efficient adsorption, broadening the application scope of CFA zeolites in wastewater treatment, and integrating them with the existing wastewater treatment technologies will be crucial research areas in the future.

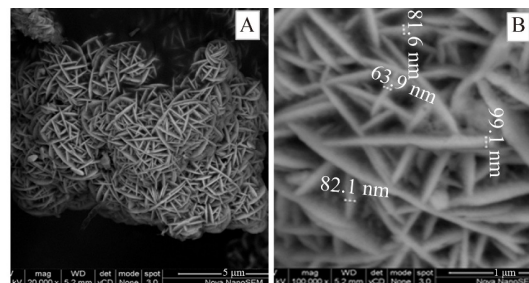
#### 4.2 Application for gas adsorption

Carbon dioxide ( $\text{CO}_2$ ), hydrogen sulfide ( $\text{H}_2\text{S}$ ), sulfur dioxide ( $\text{SO}_2$ ), nitrogen oxides ( $\text{NO}_x$ ), and volatile organic compounds (VOCs) are hazardous gases

commonly emitted from coal and fossil fuel combustion, volcanic activity, and transportation, contributing to air pollution. CFA zeolites have demonstrated exhibit excellent adsorption and purification capabilities for these pollutants in various studies (Srinivasan and Grutzeck, 1999; Muriithia et al., 2020; Ren et al., 2020). Notably, CFA zeolites exhibit unique adsorption capacity at relatively lower temperatures, as shown in Table 5. Furthermore, CFA, which contains silicon, aluminum, and unburned carbon, is a promising material for synthesizing hydrogen sorbents. Under cryogenic conditions and high pressure, CFA zeolites can adsorb up to 8.5 wt.% of hydrogen, making them a promising option for hydrogen storage applications.

Up till now, a variety types of zeolites (Zeolite A, X, P1, Y, NaX, Na-P1, Sodalite) have been synthesized from CFA (Ren et al., 2020). These zeolites have been widely used as adsorbents for air pollution, and their adsorption/desorption parameters are summarized in Table 5. Muriithia et al. (2020) developed a novel hierarchical morphology NaX-type zeolite ( $\text{NaX}_{\text{FA}}$ ) from CFA, which consists of plate-like structures arranged in spherical clusters (Fig. 7A) with a crystal width of between 63.9 and 99.1 nm (Fig. 7B).  $\text{NaX}_{\text{FA}}$  exhibited excellent physical adsorption performance for  $\text{CO}_2$ . The role of alkali metal ions in the carbonization and zeolization efficiency of CFA has also been investigated. Monasterio-Guillot et al. (2020) reported that  $\text{Na}^+$  played a key role as a “structure builder” in the converting 60% CFA to zeolite. resulting in the highest calculated value for carbonation efficiency (79.26%). In contrast, using  $\text{K}^+$  as the “structure builder” resulted in a zeolite yield and carbonation efficiency of 30% and 50%, respectively. These results suggests that the use of a sodium-alkaline solution for the preparation of CFA zeolite, followed by its use in  $\text{CO}_2$  capture, is the most effective option.

VOCs are a type of greenhouse gas that poses a threat human health. Adsorption has been identified as an effective method for capturing VOCs. Ren et al. (2020) used CFA-derived zeolite (FAZ-Y3) as an adsorbent for acetone adsorption and found that the adsorption capacity of FAZ-Y3 reached 134 mg/g with good cyclic stability. Popova et al. (2020) synthesized Co-modified NaX



**Fig. 7** Microstructure of hierarchical morphology NaX-type zeolite made from CFA (Muriithia et al., 2020).

zeolite (FAZ) from CFA and investigated its catalytic degradation activity for VOCs with different functionalities, including acetone, toluene, 1,2-dichlorobenzene, and n-hexane. As shown in Table 6, the Co modification of FAZ significantly improved its catalytic activity for the degradation of VOCs. The use of CFA-derived zeolites as effective adsorbents for VOCs has promising potential for air pollution control. The development of Co-modified FAZ zeolites demonstrates the potential for enhancing the catalytic activity of CFA-based zeolites for VOCs degradation. Further research is needed to optimize the synthesis and modification methods of CFA-based zeolites for VOCs control applications.

There are also many reports on the adsorption of other harmful gases. Wdowin et al. (2015) converted CFA into Na-X zeolite and modified it with Ag (Ag-X) for Hg adsorption. Although Ag-X exhibited high adsorption efficiency for Hg (up to 87%). Its adsorption capacity was lower in the presence of other gases, such as SO<sub>x</sub>, HCl, NO<sub>x</sub>, and CO. Maiti et al. (2018) synthesized Na-P1 zeolite from CFA for the adsorption of NO<sub>2</sub>, NO, N<sub>2</sub>O

and CO pollutants. Quantitative research revealed that the maximum adsorption capacity of each harmful gas on Na-P1 zeolite was N<sub>2</sub>O (64.77 at%), followed by NO (24.60 at%), CO (12.94 at%) and NO<sub>2</sub> (10.63 at%). These studies demonstrate the potential of CFA-based zeolites as effective adsorbents for a variety of harmful gases. However, it is important to consider the presence of other gases that may interfere with the adsorption capacity of the zeolites. Further research is needed to optimize the synthesis and modification methods of CFA-based zeolites for the efficient removal of harmful gases from the environment.

Currently, there is a wealth of literature on the use of CFA zeolite for water treatment, but relatively few studies have explored its potential for exhaust gas adsorption. Furthermore, while CFA zeolite has demonstrated the ability to adsorb various ions simultaneously during wastewater treatment, it is limited to adsorbing only a single gas during exhaust gas treatment. This is due to the potential impact of multi-gas adsorption on the overall adsorption efficiency.

**Table 6** CFA zeolites adsorbents for exhaust gas removal

Exhaust gas	Zeolite	Sorption capacity	BET (m <sup>2</sup> /g)	Conditions	Ref.	Remarks
CO <sub>2</sub>	Na-A	About 100%	257	Flow rate of 20 mL/min; Degassed 200 °C for 1 h (He); Adsorption <i>t</i> : 4 h	Muriithi et al. (2020)	–
	Zeolite A	–	145	Pretreatment: 1 h at 350 °C; Adsorption: 25 °C; Desorption: 130 °C	Soe et al. (2016)	Comparable to commercial zeolite A and 13X
	MOR zeolite	1.92 mmol/g	266.49	Flow rate of 50 mL/min; Degassed 200 °C for 1 h (N <sub>2</sub> ); Sample: 12 mg	Zhou et al. (2021)	–
	Na-Ca-X	3.16 mmol/g	486	0.40 g sample and 3 vol% CO <sub>2</sub> from the CO <sub>2</sub> /N <sub>2</sub> mixture at a flow rate of 30 mL/min	Boycheva et al. (2021)	CO <sub>2</sub> adsorption in dynamic conditions
	Zeolite X	60 mg/g	–	Pretreatment: 320 °C in Ar flow at rate of 5 °C/min; Sample: 12 mg; Adsorption: 22 °C	Kalvachev et al. (2016)	CO <sub>2</sub> sorption capacity about 3 times lower than commercial zeolite
	Zeolite A	2.8 mmol/g	486	0.40 g sample of 20–80 mesh and 10% CO <sub>2</sub> /N <sub>2</sub> at a flow rate of 30 mL/min	Popova et al. (2019)	CO <sub>2</sub> adsorption in dynamic conditions
SO <sub>2</sub>	Zeolite 4A-X	74 mg/g	–	Activation <i>T</i> : 200 °C; Adsorption <i>T</i> : –78 °C	Querol et al. (2002)	–
	Zeolite Y	46 mg/g	102.4	Pretreatment conducted at 200 °C under N <sub>2</sub> flow of 100 mL/min for 2 h	Pedrolo et al. (2017)	K <sup>+</sup> as the “structure builder”
NH <sub>3</sub>	Zeolite 4A-X	297 mg/g	–	Activation <i>T</i> : 400 °C; Adsorption <i>T</i> : 25 °C	Querol et al. (2002)	Activation and adsorption <i>T</i> : 200/–10 °C; 22 mg/g
	Zeolite 4A-X	111 mg/g	–	Activation <i>T</i> : 200 °C; Adsorption <i>T</i> : –23 °C	Querol et al. (2002)	Activation and adsorption <i>T</i> : 400/25 °C; 72 mg/g
NO	SSZ-13 zeolites	Nearly 100%	630	The feed gas containing 500 ppm NO, 500 ppm NH <sub>3</sub> , 5% O <sub>2</sub> , <i>T</i> : 100–700 °C	Wang et al. (2021)	Ion exchange with Cu
	MOR zeolite	93.58 %	122.89	The feed gas contained 400 ppm NO, 440 ppm NH <sub>3</sub> , 5% O <sub>2</sub> , <i>T</i> : 200 °C	Zhou et al. (2021)	Modified with Mn
Acetone adsorption	Zeolite Y	134.0 mg/g	386.9	A sample (0.18 g, 40–60 meshes) was used and the total flow rate was 500 mL/min	Ren et al. (2020)	Exhibits cyclic adsorption stability
Toluene	Zeolite X	525.03 µg/g	990.3	Pretreatment: 2 h 250 °C Adsorption temperature: 50–170 °C	Zhu et al. (2019)	Zeolite X had better adsorption performance than commercial, activated carbon
o-Xylene		545.26 µg/g				
m-Xylene		563.82 µg/g				
p-Xylene		582.91 µg/g				
Cyclohexane		117 mg/g				

Therefore, there is a need for further research and development of zeolites capable of effectively adsorbing multiple gases simultaneously to improve their potential for exhaust gas treatment applications.

### 4.3 Application for soil remediation

Soil pollution caused by toxic metals and organic pollutants is a significant global environmental problem that poses a threat human health, food safety, and soil eco-services (Cheng et al., 2020). Adsorption technology is a commonly used methods for remediating soils contaminated by both toxic metals and organic pollutants (Rajendran et al., 2022). Studies have shown that zeolites synthesized from silica-alumina source such as CFA (or any other source of Si, Al) can be added to contaminated soils in admixture with other components to promote the stabilization of toxic metals or catalytic degradation of organic exogenous compounds. There are three primary processes through which zeolites immobilize soil toxic metals: 1) CFA zeolites provide alkalinity to acid-contaminated soils, leading to the precipitation of the insoluble phase; 2) the increase in alkalinity promotes the adsorption of metals by surface complexation processes related to surface charge; and 3) cation exchange in zeolites may also lead to metal retention. While zeolites have been extensively studied as adsorbents in water treatment applications, research on their use for stabilizing and removing contaminants from soil is limited (Belviso, 2020). However, there are many challenges hindering the use of zeolite materials for soil remediation applications. This study lists the advantages and limitations of zeolite applications in soil remediation, as shown in Fig. 8.

In recent years, there has been increasing interest in using solid waste for the preparation of zeolites for soil remediation, in line with the concept of resource recycling. Terzano et al. (2007) synthesized zeolites X, P and A directly in artificially Cu- and Cd-polluted soil by melting CFA. Studies have shown that direct synthesis of zeolites in soil can capture heavy metals (Cu, Cd) in the form of hydroxide/oxide precipitation by the newly formed zeolite structure, thus reducing the mobility and effectiveness of heavy metals. Zhao et al. (2021) synthesized zeolite using a simplified hydrothermal method with CFA as raw material for Cd reduction in the paddy soil. The study demonstrated that application of CFA zeolite promoted rice growth (rice production increased by 29.8% and 31.7% in 2017 and 2018, respectively), while bio-available Cd decreased by 26% and 28% in 2017 and 2018, respectively. In addition to immobilization, zeolites, due to their high porosity, can reduce toxic metal forms to fewer toxic forms, such as  $Cr^{4+}$  to  $Cr^{3+}$  (Antoniadis et al., 2017). Reduced ions can be trapped in zeolites and slowly released, preventing them from being taken up by plants. However, the pH of CFA used to prepare zeolites is high, and the use of large amounts of CFA zeolites can lead to a significant increase in soil alkalinity, with potential negative effects on both soil and plant ecosystems. More details about the effect of zeolite on soil remediation can be found in Table 7.

## 5 Feasibility analysis of industrial production of CFA zeolites

At present, there are numerous methods to synthesize zeolites, but most of them are only applicable at

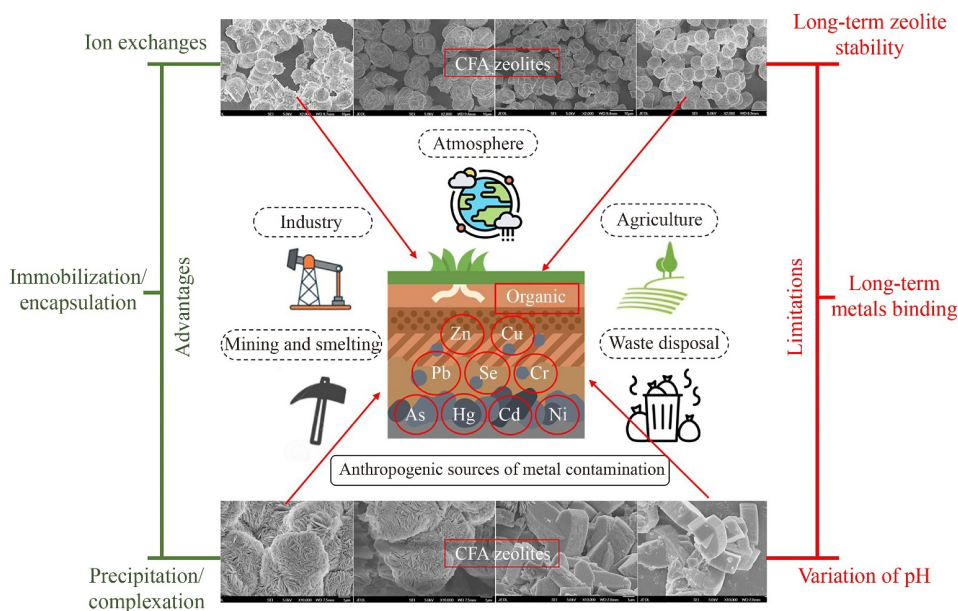


Fig. 8 The advantages and limitations of CFA zeolite in soil remediation.



**Table 7** Effect of zeolite amendments on soil remediation

Zeolite	Pollution	Type of soil	Type of experiment	Effect	Ref.
Natural zeolite	Cd, Pb, As, W	Vegetable field	Laboratory experiment	The combination agent was a mixture of zeolite and biochar with a weight ratio of 1:1	Zheng et al. (2020)
Clinoptilolite	Ni	Wheat field	Plot experiment	Biochar and zeolite	Shahbaz et al. (2021)
Faujasite	Cu, Pb, Zn, Cd	Including wastewater	Plot experiment	The zeolites were regenerated by NaCl solution (1 mol/L)	Ji et al. (2017)
Zeolite from fly ash	Cd	Paddy soil	Plot experiment	Addition of natural zeolite strongly increased the soil pH, reduced the adsorption and transport of Cd in rice organs, as well as enhanced the rice growth and yield	Zhao et al. (2020)
Zeolite from fly ash	Cu	Loamy sand	Plot experiment	Application of zeolite had a significant effect on soil properties; zeolite reduced the content of total nitrogen and widened the C:N ratio	Wyszkowski (2019)
Natural zeolite added in amounted to 3.0% of the soil mass	Cr, Zn, Pb, Cu, Ni	Light loamy sand, Ni-contaminated soil	Plot experiment	Natural zeolite significantly reduced the average content of chromium	Radziemska & Mazur (2016)
Zeolite X, P, and A	Cu, Cd	Soil samples were artificially	Laboratory experiment	Zeolite X and zeolite P for the Cu-contaminated soils, and zeolite X and zeolite A for the Cd-contaminated samples	Terzano et al. (2007)
Zeolite from fly ash	Cd, Pb, Zn	Loamy sand	Plot experiment	Zeolite used individually as well as in a mixture with biochar effectively immobilized pollutants	Mierzwa-Hersztek et al. (2019)

laboratory scale, and few methods can be used for scaling up production. Furthermore, the presence of many elements in CFA, especially highly toxic chemical components such as As, Pb, makes it difficult to scale up the study of CFA synthetic zeolite. To date, the only two methods, two-step hydrothermal and alkali melting methods, have demonstrated high feasibility for industrial application. Hong et al. (2017) prepared Na-A zeolite phase via the alkali melting method from CFA and further designed and simulated an up-scale production plant with 20 years of operation and 5000 kg/h production capacity using Aspen Plus program. The results showed that the payback period is 7.1 years with both high environmental and economic benefits. Du Plessis et al. (2014) proposed breaking the scale between the microscopic and pilot scale of hydrothermal and alkali melting methods for the synthesis of CFA zeolite by establishing a main reactor. The ultrasonic method was also tried to reduce the crystallization time during zeolite synthesis, and the supernatant produced in the synthesis process was efficiently recovered and recycled, achieving a more ideal situation for industrial production. Despite the progress made in these methods, further research is still needed to address the challenges associated with scaling up zeolite production from CFA.

Given the diverse types of CFA zeolites, different technological processes should be designed for each type. Several scholars have proposed optimization solutions for the process flow, as presented below: Yan and Chang (2017) optimized and developed a co-production process for extracting alumina and 4A zeolite from high-alumina CFA. This process provides a more suitable alkali supply

scheme to ensure stable production of 4A zeolite while reducing alumina energy consumption. This technology is of great significance for the up-scale production of 4A zeolite. Belviso (2018) introduced ultrasonic energy in the synthesis of CFA zeolite and adopted a two-stage optimization measure to determine the process conditions for rapidly producing a stable zeolite. Tauanov et al. (2017) systematically optimized the process conditions, such as reaction time, reaction temperature, and adsorbent loading, to reduce the effect of magnetite in CFA on the synthesized zeolite. This led to the production of high-purity zeolite products. These optimization solutions demonstrate the potential for developing efficient and sustainable processes for synthesizing CFA zeolites. However, further research is necessary to explore other optimization measures and to determine the optimal processes for different types of CFA zeolites.

One of the main challenges associated with the industrial production of zeolites from CFA is the variability of the raw material. CFA is a complex mixture of different compounds, and its properties can vary significantly depending on the source and composition of the coal. Therefore, it is important to develop robust and reliable synthesis methods that can be adapted to different types of CFA. Another challenge is the energy requirements of the synthesis process. The production of zeolites from CFA typically involves high-temperature treatments and long reaction times, which can be energy-intensive. Therefore, it is important to develop energy-efficient synthesis methods that can minimize the environmental impact of the production process. Despite these challenges, there are several opportunities

associated with the large-scale production and commercialization of zeolites from CFA. For example, these materials have a wide range of potential applications in different industries, such as water treatment, catalysis, and gas adsorption. Moreover, the production of zeolites from CFA could provide a sustainable and cost-effective alternative to traditional sources of zeolites, which are often obtained from non-renewable resources.

In conclusion, the industrial production of zeolites from CFA presents both challenges and opportunities. However, with the development of robust and energy-efficient synthesis methods, and the identification of new and innovative applications for these materials, the large-scale production and commercialization of zeolites from CFA could become a viable and sustainable option for the future.

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## 6 Conclusions

The enormous emissions of CFA pose a significant threat to the global environment and human health. As a result, disposing and utilizing CFA has become an essential point of focus. Preparing zeolites from CFA can effectively broaden resource utilization method for CFA, providing economic and environmental benefits. Furthermore, water, gas and soil pollution treatment have become pressing issues due to the rapid development of industry and social economy. Developing low-cost and efficient adsorption materials is therefore crucial for pollution control. This study provides a brief overview of the method for synthesizing zeolite from CFA and their application in the environmental field.

1) After an extended period of exploration, the hydrothermal method for synthesizing zeolites from CFA has rapidly developed, and other synthesis methods such as solvent-free, microwave-assisted and supercritical technique nowadays has also been increasingly mature. The preparation process is selected based on the origin of CFA, considering different physicochemical properties, to obtain zeolites with higher added-value. In our opinion, more attention should be paid to the preparation of zeolite A, P1, X and Y. Optimization of existing process conditions and improvement of synthesis methods have been crucial research directions for zeolite synthesis from CFA. These efforts aim to enhance the efficiency and reduce the cost of the synthesis process, as well as to improve the quality of the synthesized zeolites.

2) The synthesis of zeolites from CFA is an effective way to broaden the resource utilization of CFA, providing both economic and environmental benefits. However, due to the complexity of the growth mechanism of zeolites synthesized with CFA, further study are required. Despite the progress made in synthesizing zeolites from CFA, the synthesized zeolites still display some technical

shortcomings. For instance, low purity and crystallinity, poor controllability of crystal form, and other issues hinder their practical applications. Therefore, it is essential to explore the synthesis mechanism to improve the quality of the synthesized zeolites.

3) Synthetic CFA zeolites have demonstrated a wide range of promising applications and possess great potential for environmental applications. While there is a substantial body of literature documenting the use of CFA zeolites for water treatment, research on their application in gas adsorption and soil remediation is still limited. Therefore, future research should prioritize exploring the potential of CFA zeolites in these fields.

4) While some studies have been conducted on the commercial-scale production of CFA zeolite, industrial production technology requires in-depth investigation. (i) Currently, two methods that exhibit high feasibility for industrial application are currently available, namely the two-step hydrothermal method and the alkali fusion method. By leveraging new composite technology such as microwave-assisted, seed-assisted and supercritical techniques, the limitations of traditional methods can be effectively overcome, expediting industrialization. (ii) Developing industrial technology for CFA synthetic zeolite with high product purity, low process cost and high practicality is the focus for future research. (iii) Additionally, it is necessary to explore subsequent treatment methods of saturated zeolites (after adsorption) in environmental remediation applications. (iv) It is important to manage the residues left after the synthesis of zeolites from high pH aqueous solutions. Proper management of these residues will not only minimize environmental impact but also ensure sustainable production of zeolites. Therefore, it is necessary to develop effective strategies for the treatment and disposal of these residues to mitigate potential negative effects on the environment and human health.

**Acknowledgements** This work was supported by the National Natural Science Foundation of China (No. 51904278), the Fundamental Research Program of Shanxi Province (No. 202103021223277), the Taiyuan University of Science and Technology Doctoral Research Fund (No. 20212025), and the Natural Science Foundation of Shanxi Province (No. 20210302123218).

**Conflict of Interest** The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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