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Rapid synthesis of fault-free GME zeolite†

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The optimization of the synthesis of known zeolite structures and the discovery of new applications for them are continuously hot topics in the field of zeolite chemistry. One of the typical examples is the GME zeolite structure. Herein, we for the first time report a rapid synthesis of fault-free GME zeolite by the combined strategy of seeding and aging. The fault-free GME zeolite can be obtained at 160 °C in only 2.5 h. Various characterization techniques, such as X-ray diffraction (XRD), scanning electron microscopy (SEM), high-resolution transmission electron microscopy (HRTEM) and nuclear magnetic resonance (NMR) have been used, which show that the obtained product has good crystallinity, perfect hexagonal morphology, fully 4-coordinated Al species and fault-free features. In addition, after the post-treatment of the as-made GME zeolite, Na–GME zeolite with a large BET surface area and high porosity could be successfully obtained. More importantly, the obtained Na–GME zeolite is used for the first time for CO₂ capture, giving a high CO₂ adsorption of 5.37 mmol g^{−1} and excellently selective CO₂ adsorption from CO₂/N₂ (15/85, v/v) with a separation coefficient of 58.8. The fault-free GME zeolite used for CO₂ capture might be of potential significance for industrial applications.

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1. Introduction

Zeolites, particularly aluminosilicate zeolites, have been widely employed in the fields of catalysis, ion-exchange, separation and adsorption because of their high surface area, large pore volume, uniform micropore distribution, and excellent stability.^{1–10} Currently, over 260 types of zeolite have been given three-letter codes by the International Zeolite Association-Structural Commission (IZA-SC).¹¹ However, only about 20 types of zeolite have been used for industrial applications.^{12–18} Thus, optimizing the synthesis of known zeolite structures and discovering new applications for them is of great significance, and would be very helpful to promote their practical application in industry. Among them, one of the typical examples is GME zeolite.

GME zeolite is composed of double six-membered rings (6-MRs) and GME cages, which form a three-dimensional micropore channel with an aperture size of 12 × 8 × 8-MRs.^{19,20} Initially, GME zeolite was discovered in nature, where it always exhibits stacking faults and CHA intergrowth,

leading to significantly reduced porosity.^{20–22} Thus, great efforts have been made to reduce the faults and improve the purity.^{19,22–31} For example, Daniels *et al.* showed that synthetic GME zeolite could be obtained after crystallization for at least 3 days in the presence of dabco-polymer, giving a greater adsorption capacity of cyclohexane than that of natural GME zeolite.²² Chiyoda *et al.* showed that GME zeolite could be synthesized from the transformation of Sr–Y zeolite.¹⁹ Notably, the crystallization of GME zeolite was successful at 240 °C for 14 d. Mielby *et al.* displayed that a steam-assisted interzeolite transformation from Y zeolite could be successfully achieved to synthesize GME zeolite in 20 h.²³ However, these synthetic GME zeolites also have stacking faults, which could still block the micropores and thus reduce the porosity. Recently, Dusselier *et al.* for the first time showed a fault-free synthesis of GME zeolite in the presence of 3,5-dimethylpiperidinium, which gave a micropore volume of 0.17 cm³ g^{−1} after ozone-treatment at 150 °C.²⁴ Notably, the crystallization would be completed after at least 110 h. Mitani *et al.* also reported that GME zeolite with high porosity could be successfully synthesized from the transformation of FAU zeolite using a dual template route, in which it was fully crystallized after 2 d.²⁵ However, despite the great progress that has been made in the successful synthesis of fault-free GME zeolite, it is still not very efficient due to the long crystallization time. Currently, it is still highly desirable to synthesize fault-free GME zeolite in a rapid manner.

More recently, we showed for the first time a rapid synthesis of high silica SSZ-13 zeolite by the combined strategy of short aging and zeolite seeding, which gives crystallization in only

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4 h.³² The addition of zeolite seeds would produce more basic building units during the short aging, which would lead to the successful synthesis of high silica SSZ-13 zeolite rapidly. However, the rapid synthesis of fault-free GME zeolite at the hour level has still not been reported using this method.

Herein, we demonstrate a rapid synthesis of fault-free GME zeolite for the first time. Notably, GME zeolite with high crystallinity could be fully crystallized at 160 °C in only 2.5 h after the addition of zeolite seeds and then aging at room temperature for 3 h. After the post-treatment of the as-made GME zeolite, a Na-GME zeolite with high porosity could be obtained successfully. More importantly, the obtained Na-GME zeolite gives a high CO₂ adsorption of 5.37 mmol g⁻¹ and excellently selective CO₂ adsorption from CO₂/N₂ (15/85, v/v) with a separation coefficient of 58.8.

2. Experimental

Rapid synthesis of GME zeolite at 160 °C for 2.5 h

As a typical example, 1.14 g of deionized water and 0.1156 g of sodium aluminate were mixed in a beaker. After the dissolution of sodium aluminate, 1.7 g of *N,N*-dimethyl-3,5-dimethylpiperidinium hydroxide solution, 0.48 g of sodium hydroxide, and 3.93 g of silica sol were added into the above solution one by one. After stirring for 1 h at room temperature, 0.024 g of GME seeds was added. After aging for 3 h at room temperature under stirring conditions, the mixture was transferred to an autoclave and sealed. After crystallization at 160 °C for 2.5 h under rotation conditions (50 rpm), a fully crystalline GME zeolite product was obtained with a yield of 22%. Notably, the aging time is very critical for the successful synthesis of the target product with good crystallinity. If the aging time is too short, such as 10 min, the obtained product would always have GIS zeolite as an impurity phase.

Synthesis of the conventional GME zeolite

According to the literature report by Mielby *et al.*,²³ the conventional GME zeolite with some faults from interzeolite transformation was successfully synthesized at 120 °C for 3 d, and was named as T-GME.

Preparation of K-GME and Na-GME

The as-made GME zeolite was mixed with 2 mol kg⁻¹ KCl solution (solid : liquid = 1 mg : 2 mg) and aged for 12 h at room temperature. The resulting thick paste was calcined at 500 °C for 5 h. The obtained sample was fully washed with warm distilled water to remove excess salt. Then, a second calcination was performed at 580 °C for 6 h, which gave the partially K⁺-exchanged GME, named as KS-GME. To obtain Na-GME or K-GME, KS-GME was exchanged three times with 1 mol L⁻¹ NaCl or KCl at 80 °C for 2 h.

Performance tests

The CO₂ adsorption experiments were carried out using high-purity CO₂ (99.999%) at 298 K over a pressure range of

0–101.325 kPa by a JW-ZQ100. The samples were placed inside the balance and outgassed at 473 K under vacuum conditions for 4 h, and then cooled down to the experimental temperature. The experiments started with the gas pressure increasing stepwise until 101.325 kPa. Before achieving the equilibrium condition, the mass variation was recorded constantly.

CO₂/N₂ breakthrough separations were performed in a multi-component adsorption breakthrough curve analyzer (Beishide Instrument Technology (Beijing) Co., Ltd, China). Before the test, each sample (0.4–1.0 g) was pre-processed in a He atmosphere (5 mL min⁻¹) for 2 h at 573 K in quartz tubing (6 mm diameter). The test started when the gas mixture (CO₂/N₂: 15/85, v/v) was introduced (3 mL min⁻¹) at 298 K after cooling to ambient temperature.

3. Results and discussion

The as-made GME zeolite was firstly characterized in detail by the X-ray diffraction (XRD), scanning electron microscopy (SEM), nuclear magnetic resonance (NMR), and thermogravimetric-differential thermal analysis (TG-DTA) techniques, as displayed in Fig. 1. Fig. 1A shows the XRD pattern of the as-made GME zeolite, giving a series of typical peaks associated with the GME structure, which were in good agreement with the simulated XRD pattern from IZA-SC (Fig. S1†). Fig. 1B shows the SEM image of the as-made GME zeolite with hexagonal features, giving the crystal size at about 1 μm. Fig. 1C displays the ²⁹Si MAS NMR spectrum of the as-made GME zeolite,

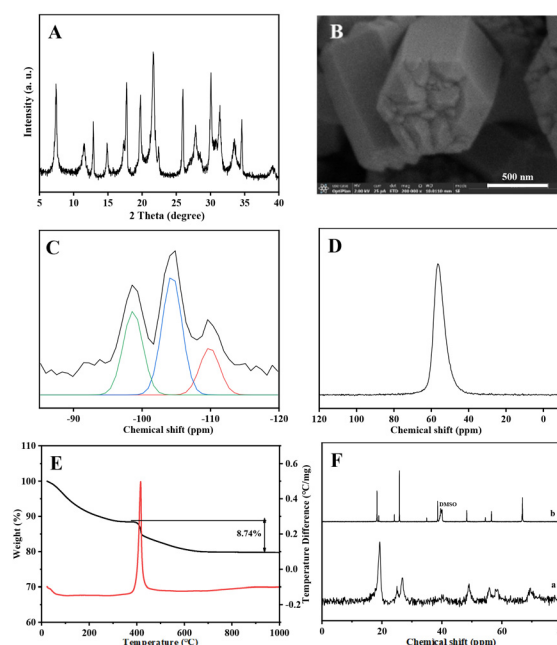


Fig. 1 (A) XRD pattern, (B) SEM image, (C) ²⁹Si MAS NMR spectrum, (D) ²⁷Al MAS NMR spectrum, and (E) TG-DTA curves of the as-made GME zeolite. (F) ¹³C MAS NMR spectrum of the as-made GME zeolite (a) and ¹³C NMR spectrum of the organic template in a dimethyl sulfoxide solution (b).

giving three peaks with chemical shifts at about -109.8 , -104.4 and -98.5 ppm, which were reasonably attributed to Si (4Si), Si(3Si) and Si(2Si) species, respectively. The calculated Si/Al ratio of the as-made GME zeolite product was about 3.5 based on the ^{29}Si MAS NMR spectrum, which was very similar to that (Si/Al ratios at 3.6 and 3.1) of the XRF and ICP results, respectively. Fig. 1D displays the ^{27}Al MAS NMR spectrum of the as-made GME zeolite, showing only one peak with the chemical shift at about 55.6 ppm, which means that there are no extra-framework aluminum species. Fig. 1E shows the TG-DTA curve of the as-made GME zeolite, which displays an obvious exothermic peak between 380 and 450 $^{\circ}\text{C}$, resulting in weight loss associated with the decomposition of the organic template at about 8.74%. The above results also show that the organic template was indeed in the GME zeolite micropores. Fig. 1F displays the ^{13}C MAS NMR spectrum of the as-made GME zeolite and ^{13}C NMR spectrum of the organic template, which showed that the organic template in the GME zeolite was intact, in good agreement with the liquid organic template. The FTIR spectrum of the as-made GME zeolite is shown in Fig. S2,[†] giving C–H stretching peaks at 2800–3000 cm^{-1} . Thus, according to the results of TG-DTA, NMR, and FTIR, it can be reasonably concluded that the organic template exists in the as-made GME zeolite micropores and directs the formation of the GME zeolite structure.

As we known, in the GME zeolite structure (Fig. S3[†]), faults always appear in the 12-MR channel along the c -axis and thus partially block the micropores.^{20,23,24,33} Dusselier *et al.* used rotation electron diffraction to determine the extent of disorder of the GME zeolite (named as CIT-9).²⁴ It could be observed that CIT-9 did not have streaking along the c -axis, indicating fully open 12-MR micropores. Thus, the TEM technique has been used for assessing the extent of the faults in the GME zeolite, as shown in Fig. 2. Fig. 2A and B show the TEM image of the GME zeolite and its corresponding SAED

image, where it can be clearly seen that no streaking appeared along the c -axis, indicating that the 12-MR channels were fully open without any faults. In addition, the orderly 12-MR channels could be clearly observed from the HRTEM images and fitted well with the GME structure, which further proves that no faults formed in the GME zeolite product, as shown in Fig. 2C and D. Based on the results of XRD and TEM, it can be concluded that the GME zeolite product synthesized in this work is fault-free.

However, the GME zeolite is not resistant to calcination at high temperature, and direct calcination would cause transformation from GME to AFI, as shown in Fig. S4.[†] After the treatment of the as-made GME zeolite with KCl under suitable conditions, a stable GME zeolite could be successfully obtained, as shown in Fig. S4 and S5.[†] Moreover, a Na-GME zeolite could also be successfully obtained by ion-exchange of NaCl, as shown in Fig. S6.[†] Fig. 3 shows the N_2 sorption isotherms of the Na-GME zeolite, which displayed the typical Langmuir curves. Simultaneously, the BET surface area and

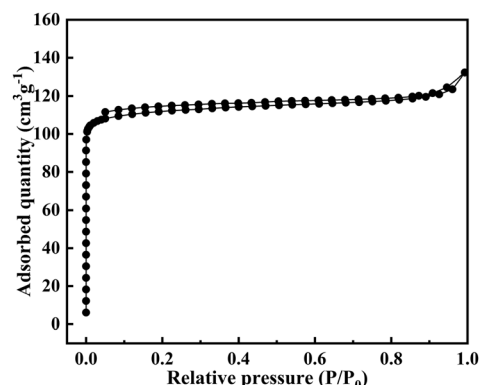


Fig. 3 N_2 sorption isotherms of the Na-GME sample.

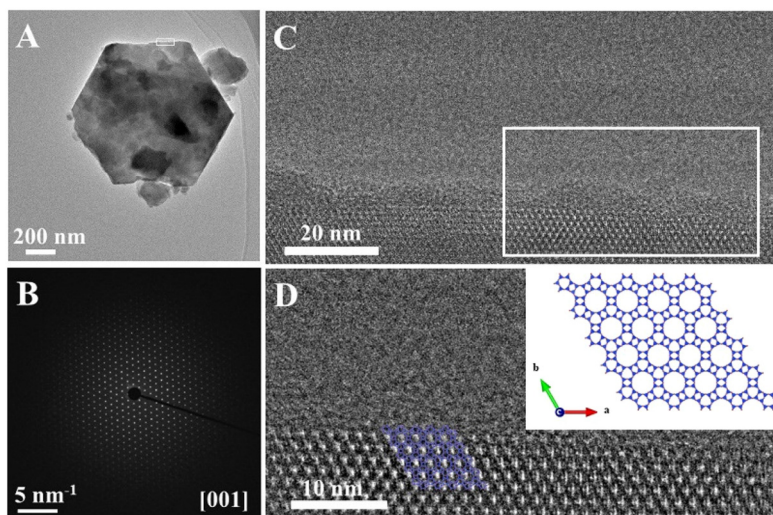


Fig. 2 (A) TEM image of the GME zeolite, (B) SAED image of the [001] crystal plane viewed along the c -axis, (C) HRTEM image of the selected area in (A), and (D) enlarged HRTEM image of the selected area in (C); the inset is the GME framework viewed along c -axis.

micropore volume were determined to be $341 \text{ m}^2 \text{ g}^{-1}$ and $0.16 \text{ cm}^3 \text{ g}^{-1}$, respectively, which also showed that the obtained GME zeolite was fault-free.

Notably, to obtain the optimized conditions for synthesizing fault-free GME zeolite, various synthetic factors were investigated, including $\text{Na}_2\text{O}/\text{SiO}_2$ ratio, OSDA/SiO_2 ratio, $\text{H}_2\text{O}/\text{SiO}_2$ ratio, Si/Al ratio, and the number of seeds in the initial gel. It was found that the $\text{Na}_2\text{O}/\text{SiO}_2$ ratio was very important to synthesize pure GME zeolite with good crystallinity. When the $\text{Na}_2\text{O}/\text{SiO}_2$ ratio was 0.34, a GME zeolite with good crystallinity could be obtained successfully (run 4 in Table 1 and Fig. 1A); when the $\text{Na}_2\text{O}/\text{SiO}_2$ ratio was lower than 0.31, the products were GME zeolite together with an amorphous phase (run 1–3 in Table 1 and Fig. S7a–c†); when the $\text{Na}_2\text{O}/\text{SiO}_2$ ratio was higher than 0.37, the products were GME zeolite together with GIS or ANA zeolites as impurities (run 6–7 in Table 1 and Fig. S7e and f†). Thus, the suitable $\text{Na}_2\text{O}/\text{SiO}_2$ ratio for the successful synthesis of the GME zeolite was 0.34. In addition, the most suitable OSDA/SiO_2 ratios were 0.096–0.136, which could obtain GME zeolite with high crystallinity (run 4, 9, and 10 in Table 1 and Fig. S8b–d†). If the OSDA/SiO_2 ratio was lower than 0.076, the obtained products were GME zeolite with GIS zeolite as an impurity (run 8 in Table 1 and Fig. S8a†); if the OSDA/SiO_2 ratio was higher than 0.156, ANA zeolite as an impurity could be observed in the final product (run 11 in Table 1 and Fig. S8e†). Therefore, the OSDA/SiO_2 ratio should be carefully adjusted during the whole synthesis. Moreover, when the $\text{H}_2\text{O}/\text{SiO}_2$ ratio in the starting gel was 5, the final product was GME zeolite together with an amorphous phase (run 12 in Table 1 and Fig. S9a†); when the $\text{H}_2\text{O}/\text{SiO}_2$ ratio was increased to 15, GME zeolite with good quality could be obtained successfully (run 4 in Table 1 and Fig. 1A); further increasing the $\text{H}_2\text{O}/\text{SiO}_2$ ratio to 30, the obtained product was

GME zeolite together with an amorphous phase (run 13 in Table 1 and Fig. S9c†). Additionally, when the Si/Al ratios were increased from 20 to 40, GME zeolite with high crystallinity was obtained successfully (run 4 and 15 in Table 1 and Fig. S10b and c†); when the Si/Al ratio was lower than 10, the obtained product was GME zeolite together with ANA zeolite as an impurity (run 14 in Table 1 and Fig. S10a†); when the Si/Al ratio increased from 60 to 80, AEI zeolite started to appear in the final product (run 16 and 17 in Table 1 and Fig. S10d and e†). Furthermore, when the zeolite seeds were absent in the starting gel, the product would always be GME zeolite together with GIS zeolite (run 18 in Table 1 and Fig. S11a†). Addition of 2% zeolite seeds was enough to get a GME product with good crystallinity (run 4 in Table 1 and Fig. 1A). Further addition of 4% zeolite seeds would not shorten the complete crystallization time, indicating that the addition of 2% zeolite seeds was enough for complete crystallization of the GME zeolite at 160°C for 2.5 h (Fig. 4 and 5).

Additionally, the crystallization temperature was also very important for rapid crystallization. When the crystallization temperature was at 140°C , the complete crystallization of the GME zeolite with good crystallinity would take about 6 h, as shown in Fig. S12.† The corresponding dependence of the zeolite crystallinity on the crystallization time is shown in Fig. S13.† When the crystallization temperature was at 160°C , the crystallization of the GME zeolite could be completed in 2.5 h. The details are provided in the following text. More importantly, the aging of initial gel was very crucial for the successful synthesis of GME zeolite with good quality. When the aging time after the addition of zeolite seeds was 10 min, the

Table 1 GME zeolite synthesized under various conditions

Run ^a	$\text{Na}_2\text{O}/\text{SiO}_2$	OSDA/SiO_2	$\text{H}_2\text{O}/\text{SiO}_2$	Si/Al	Seed ^c (%)	Product ^d
1	0.25	0.136	15	20	2	Amor + GME
2	0.28	0.136	15	20	2	GME + Amor
3	0.31	0.136	15	20	2	GME + Amor
4	0.34	0.136	15	20	2	GME
5 ^b	0.34	0.136	15	20	2	GME
6	0.37	0.136	15	20	2	GME + GIS
7	0.40	0.136	15	20	2	GME + ANA
8	0.34	0.076	15	20	2	GME + GIS
9	0.34	0.096	15	20	2	GME
10	0.34	0.116	15	20	2	GME
11	0.34	0.156	15	20	2	GME + ANA
12	0.34	0.136	5	20	2	Amor + GME
13	0.34	0.136	30	20	2	GME + Amor
14	0.34	0.136	15	10	2	GME + ANA
15	0.34	0.136	15	40	2	GME
16	0.34	0.136	15	60	2	GME + AEI
17	0.34	0.136	15	80	2	AEI + GME
18	0.34	0.136	15	20	0	GIS + GME

^aThe products were synthesized at 160°C for 4 h. ^bThe products were synthesized at 160°C for 2.5 h. ^cMass ratio of zeolite seeds to the silica source. ^dThe phase that appears first is dominant.

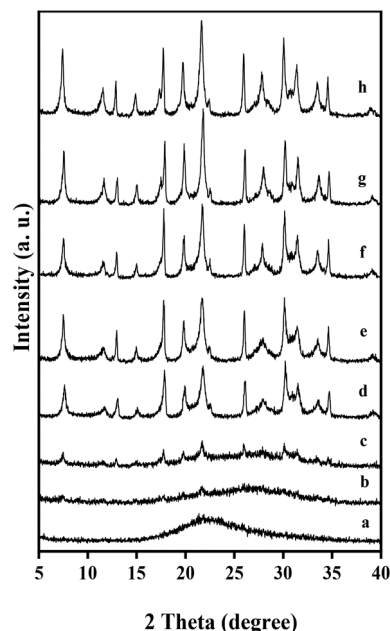


Fig. 4 XRD patterns of the GME zeolite crystallized for (a) 0, (b) 0.5, (c) 1, (d) 1.25, (e) 1.5, (f) 2, (g) 2.5 and (h) 4 h when aging of the initial gel was performed for 3 h at room temperature after the addition of zeolite seeds.

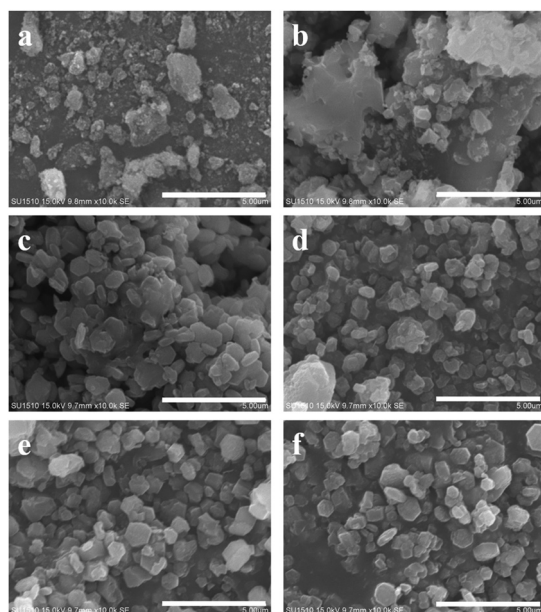


Fig. 5 SEM images of the GME zeolite crystallized for (a) 0, (b) 1, (c) 1.25, (d) 2, (e) 2.5, and (f) 4 h when aging of the initial gel was performed for 3 h at room temperature after the addition of zeolite seeds. The scale bar is 5 μm .

obtained product always contained GIS phase (Fig. S14a†); when the aging time after addition of zeolite seeds was 3 h, pure GME zeolite could be successfully obtained (Fig. 1A). When the aging time was 12 h, the crystallization of the GME zeolite still required 2.5 h (Fig. S14c†). In summary, GME zeolite with good crystallinity could be rapidly synthesized only under appropriate initial ratios, aging and crystallization conditions.

The crystallization process of the GME zeolite was monitored in detail by the XRD and SEM techniques, as shown in Fig. 4 and 5. Before crystallization, almost no peaks associated with the GME structure could be observed from the XRD pattern, even if zeolite seeds were added into the starting gel (Fig. 4a), indicating the dissolution of the zeolite seeds after aging of the starting gel for 3 h. After crystallization for 0.5 h, very weak characteristic peaks associated with the GME structure started to appear (Fig. 4b). Increasing the crystallization time to 1 h, the intensity of the peaks became stronger (Fig. 4c). Correspondingly, small amounts of GME zeolite crystals could be seen *via* the SEM image, as displayed in Fig. 5b. When the crystallization time was 1.25 h, the characteristic peaks and the hexagonal crystals associated with the GME zeolite structure could be clearly observed, as shown in Fig. 4d and 5c. When the crystallization time increased from 1.5 to 2.5 h, the intensity of the XRD patterns constantly increased (Fig. 4e–g). At the same time, more zeolite crystals could be formed, as shown in Fig. 5d and e. Further increasing the crystallization time to 4 h, the intensity of the XRD pattern and the crystal morphology remained similar (Fig. 4h and 5f), meaning that a GME zeolite with high crystallinity could be

already successfully obtained at 2.5 h. The corresponding dependence of the zeolite crystallinity on the crystallization time at 160 $^{\circ}\text{C}$ is displayed in Fig. S13.†

It is well-known that CO_2 is a main greenhouse gas, leading to environmental issues such as global warming and ocean acidification.^{34–38} Thus, great efforts have been devoted to capturing CO_2 using efficient technologies. The key to efficiently capturing CO_2 is the employment of adsorbents. Current adsorbents, such as ethanol amine, CaO , zeolites and MOFs, have been widely researched.^{39–47} Among them, zeolites are potentially very important to capture CO_2 due to their excellent features, such as large-scale commercialization and large adsorption capacity. Recently, zeolites such as KFI, CHA, FAU, and MOR have been reported for capturing CO_2 .^{48–52} For example, Zhou *et al.* for the first time showed that an Fe-containing MOR zeolite could be efficiently used for capturing CO_2 from flue gas.⁵² However, GME zeolite has never been reported for capturing CO_2 efficiently.

Fig. 6 shows the CO_2 adsorption isotherms of Na–GME, K–GME, and T–GME. It can be clearly observed that the above three zeolite samples have the ability to capture CO_2 . Notably, the Na–GME zeolite had a high CO_2 adsorption capacity (5.37 mmol g^{-1}), while the K–GME zeolite displayed a low CO_2 adsorption capacity (3.73 mmol g^{-1}). The above phenomenon could be reasonably attributed to the fact Na–GME has a larger micropore volume and BET surface area than the K–GME zeolite, as shown in Table S1 and Fig. S15.† However, the T–GME displayed the lowest CO_2 adsorption capacity (2.45 mmol g^{-1}), which might be caused by the stacking-faults of the micropore channels (Fig. S16†).

To further explore the potential of the GME zeolite for the separation of a binary gas, a CO_2/N_2 (15/85, v/v) mixed gas breakthrough experiment was conducted on the above GME zeolite samples. As shown in Fig. 7, the retention time of CO_2 for the Na–GME zeolite was 160 min, while N_2 would penetrate rapidly. In comparison, the K–GME and T–GME zeolites displayed retention times of only 120 and 80 min, respectively (Fig. S17†), meaning that Na–GME had the best separation per-

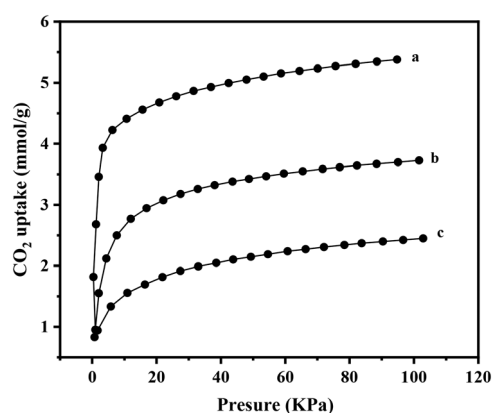


Fig. 6 CO_2 adsorption isotherms of Na–GME (a), K–GME (b), and T–GME (c) samples.

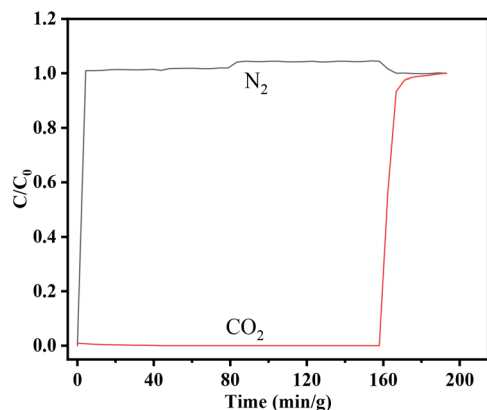


Fig. 7 CO₂/N₂ (15/85, v/v) mixture breakthrough curves of the Na-GME sample.

formance. In addition, based on the breakthrough curve in Fig. 7, the separation coefficient of Na-GME zeolite for CO₂/N₂ (15/85, v/v) could be calculated to be about 58.8, also showing the excellent ability of CO₂/N₂ separation compared with other zeolites reported in the literature (Table S2†).

4. Conclusions

In summary, the rapid synthesis of fault-free GME zeolite has been successfully shown for the first time. By combining both seeding and aging strategies, a GME zeolite with good crystallinity could be synthesized at 160 °C in 2.5 h. The obtained GME zeolite was characterized in detail by HRTEM and N₂ sorption techniques, confirming the fault-free feature in the micropore channels. As a result, after the post-treatment of the as-made GME zeolite, a Na-GME zeolite with high porosity could be obtained, giving the abilities of high CO₂ adsorption and excellently selective CO₂ adsorption for CO₂/N₂ (15/85, v/v). The rapid synthesis of fault-free GME zeolite, as well as its good CO₂ capture performance, might offer a novel door for its practical application in the foreseeable future.

Author contributions

This manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

Data availability

The data supporting this article have been included as part of the ESI.†

Conflicts of interest

There are no conflicts to declare.

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