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Construction of monodispersed single-crystalline hierarchical ZSM-5 nanosheets via anisotropic etching

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ABSTRACT

Hierarchical zeolites and single-crystalline zeolite nanosheets (NSs) have been recognized as two separate types of targeting porous materials to overcome the diffusion limitations of traditional bulk zeolites. The synthesis of uniform single-crystalline hierarchical zeolite NSs featured with NS morphology and interconnected mesoporosity, remains rarely reported. In this work, we prepared ZSM-5 zeolites with the above microstructural features via simple alkaline etching. Moreover, both their microstructure and acid strength could be accurately tuned with this approach, resulting in not only higher conversion rate and BTX selectivity but also superior anti-coking performance in the subsequent methanol aromatization reaction.

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Zeolites have been widely utilized in petroleum refining, gas adsorption, and ion exchange due to their robust framework, suitable pores, and high acidity [1–4]. However, sub-nanometer-sized channels in bulk zeolites may lead to undesirable diffusion limitations [5]. A variety of approaches have been developed to address the above concerns. Among them, the preparation of zeolite nanosheets (NSs) has been proven as an effective approach for shortening the diffusion path length in the framework [6]. For instance, di-functional surfactants, e.g., C₂₂H₄₅-N(CH₃)₂-C₆H₁₂-N $(CH_3)_2$ -C₆H₁₃, have been utilized to direct the synthesis of 2-4 nm-thick ZSM-5 zeolite NSs [7–9]. Recently, C₁₆H₃₃-N⁺(CH₃)₂- $C_4H_8-N^+(CH_3)_2$ -benzylamine was designed to direct the synthesis of 11 nm-thick MOR NSs [10]. The hydrophilic multi-ammonium head group was responsible for the formation of MFI-type zeolites, while the hydrophobic alkyl tail served as a barrier against excessive growth of crystals along the *b*-axis, resulting in the formation of lamellar zeolite NSs containing intercrystal mesopores [7,11]. It was further confirmed that the thickness of prepared NSs could be precisely tailored through controlling the number of multiammonium head groups [8] as well as the concentration of Na⁺ ions [12] in the reaction solution. Besides, a protocol for the design of a small organic template to synthesize self-pillared pentasil (SPP) zeolite NSs has been developed. Very recently, 10-20 nmthick SPP ZSM-5 NSs with house-of-card morphology were successfully synthesized from a novel cationic pyridine-based template [13]. Multilamellar Silicalite-1 zeolite NSs could be further exfoliated into monodispersed 3 nm-thick NSs through a polymer-melt-compounding technique integrated with density gradient centrifugation [14–16]. Recently, multilamellar Silicalite-1 was also used as the precursor to synthesize pillared Silicalite-1 NSs via a vapor-phase pillarization process [17]. Alternatively, monodispersed 2.5-unit-cell-thick Silicalite-1 zeolite NSs could be directly synthesized via seeded growth with bis-1,5 (tripropylammonium) pentamethylene diiodide (dC5) as structure-directing agents (SDAs) [18]. Bottom-up etching represented an alternative method for the preparation of robust MFI zeolite NSs. For instance, monodispersed 10 nm-thick Silicalite-1 zeolite NSs could be in-situ synthesized by simply adjusting the aging time in a fluoride media [19]; while combing preliminary aging with fluoride-assisted low-temperature crystallization resulted in the formation of Silicalite-1 zeolite NSs with tens of nanometers along the *b*-axis [20]. Very recently, we succeeded in the preparation of monodispersed 25 nm-thick Silicalite-1 zeolite NSs via anisotropic etching with TPAOH [21].

The introduction of hierarchy represents another effective approach for reducing the diffusion path length of bulk zeolites. Dealumination and desilication have been widely used in preparing hierarchical zeolites. Dealumination could be accomplished by steaming, acid treatment or selective leaching [22]. Neverthe-

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less, hierarchical mesopores thus created were commonly presented as isolated cavities without significant contribution to intracrystalline diffusion [23]. Alternatively, desilication of bulk zeolites via alkaline etching was more effective in introducing interconnected mesopores and therefore, reducing the diffusion path length [24]. Relevant studies on alkaline leaching-based desilication indicated that an optimum Si/Al ratio was required for the construction of desirable intracrystalline mesopores. Nevertheless, the addition of external pore-directing agents (e.g., $Al(OH)_4^-$ and TPAOH) or NH₄F had become necessary for the expansion of adaptable Si/Al ratios in a wide range [25–27]. In addition, hierarchical ZSM-5 zeolites with well-developed mesoporosity and preserved microporosity were prepared via successive desilication treatment and lignin-mediated reassembly [28]. In recent work, we succeeded in the creation of uniform hierarchical mesopores (4-6 nm) in Al-rich ZSM-5 zeolites (Si/Al = 14.3) through combining dealumination and desilication via consecutive steaming-alkaline treatment [29]. Very recently, a facile Al-distribution regulated desilication approach was developed to synthesize hierarchical Al-rich ZSM-5 crystals (Si/Al = 12.5) [30]. It should be addressed that, although remarkable progress has been made in the fabrication of zeolite NSs or hierarchical zeolites, facile preparation of monodispersed single-crystalline hierarchical zeolite NSs featured with both of the above microstructural advantages, remains a great challenge.

In this study, we introduced a novel progressive wall-thinning strategy for the fabrication of monodispersed single-crystalline hierarchical ZSM-5 NSs featuring high crystallinity, tunable Si/Al ratios, and excellent stability via simple anisotropic alkaline etching in aqueous TPAOH solution (Fig. 1a). It was expected that through concurrent creation of hierarchy and reduction in diffusion path length along the *b*-axis, the overall diffusion path length of guest molecules in ZSM-5 zeolites could be minimized with no comprise in other physicochemical properties (like framework robustness). The subsequent methanol aromatization reaction indicated that prepared ZSM-5 zeolites exhibited a higher conversion rate and BTX selectivity; in addition, its anti-coking performance was significantly enhanced.

Recently we prepared Silicalite-1 NSs via simple anisotropic etching of coffin-shaped bulk Silicalite-1 crystals in different types of alkaline etching agents under hydrothermal conditions [21].

Herein we took the initiative to fabricate monodispersed singlecrystalline hierarchical ZSM-5 NSs via alkaline etching of corresponding bulk ZSM-5 crystals with varying Si/Al ratios in aqueous TPAOH solution (experimental details were listed in Table S1). First, bulk ZSM-5 crystals with different Si/Al ratios (i.e., Z5-B-200, Z5-B-160, Z5-B-120, and Z5-B-80, accurate Si/Al ratios values were shown in Table S2), which were prepared by following the well-documented procedure [31], exhibited narrow crystal size distribution and uniform coffin-shaped morphology (shown in Fig. 1b-e and Table S3). Of particular note, we observed the formation of a highly roughened surface morphology along *a*/*c*-faces of Z5-B-80 (shown in Fig. 1e), which may be attributed to the overgrowth of sedimented ZSM-5 nanoparticles during the secondary nucleation process [32]. The subsequent alkaline etching with TPAOH resulted in the formation of uniform ZSM-5 NSs with varving morphology and Si/Al ratios. SEM images indicated that after hydrothermal treatment, uniform episode-shaped ZSM-5 NSs with relatively smooth surface morphology could be obtained (shown in Fig. 1f-h) in the case that the Si/Al ratio of bulk ZSM-5 crystals was above 120. In contrast, the surface roughness of prepared ZSM-5 NSs became more serious along with the formation of substantial amounts of mesoscopic defects (shown in Fig. 1i) in the case of lower Si/Al ratio of bulk ZSM-5 crystals (~80). This was in accordance with the results of previous studies implying that alkaline etching of bulk ZSM-5 crystals with higher Al content led to the enhanced concentration of removed Al atoms from the zeolite framework and therefore, ultimate formation of rough surface morphology [33]. In addition, the relationship between the Si/Al ratio of ZSM-5 NSs and corresponding bulk ZSM-5 crystals was investigated. Our results indicated that on the one hand, the Si/Al ratio of prepared ZSM-5 NSs, which fell in the range of 21.2 and 34.9, was much lower than that of corresponding bulk ZSM-5 crystals (Fig. S1), which could be attributed to preferential leaching of silicon atoms via alkaline treatment; on the other hand, the efficiency of "desilication" was found to reduce with the decrease of the Si/Al ratio of bulk ZSM-5 crystals, indicating that the leaching of Si atoms became increasingly difficult with increasing Al content of bulk ZSM-5 crystals, which was consistent with previous studies [34].

It should be noted that although the Si/Al ratio could be widely tailored via simple alkaline treatment, it remained very difficult to



Fig. 1. (a) Schematic illustration of the preparation of monodispersed single-crystalline hierarchical ZSM-5 zeolite NSs, (b-i) SEM images of bulk ZSM-5 crystals and corresponding ZSM-5 NSs. (b) Z5-B-200, (c) Z5-B-160, (d) Z5-B-120, (e) Z5-B-80, (f) Z5-HNS-200, (g) Z5-HNS-160, (h) Z5-HNS-120, and (i) Z5-HNS-80.

maintain their morphological integrity at a lower Si/Al ratio (e.g., Z5-HNS-80), which in turn may lead to lower chemical/mechanical stability and therefore, inferior catalytic performance [35]. Therefore, it became indispensable to optimize relevant etching conditions. Previous studies indicated that the addition of NaAlO₂ during alkaline etching resulted in hydrothermal alumination of zeolites and the formation of aluminum hydroxide deposits on zeolite surfaces so that excessive leaching of Si/Al atoms from the zeolite framework could be avoided [36]. By analogy, herein given amount of NaAlO₂ was added to the reaction solution during alkaline etching for concurrently maintaining the morphological integrity, lowering the Si/Al ratio, and preserving the framework robustness. Our results indicated that indeed, uniform 100 nmthick ZSM-5 NSs with smooth surface morphology could be obtained under optimized hydrothermal treatment conditions (denoted as Z5-HNS-80*, shown in Fig. 2a and b); moreover, in contrast to Z5-HNS-80 with Si/Al ratio of 21.2, not only the Si/Al ratio of Z5-HNS-80* (17.0) was considerably decreased but also its crystallinity was well maintained (shown in Fig. 2c). Relevant BET results indicated that calcined ZSM-5 NSs exhibited typical IV isotherm featuring a hierarchical pore structure (Fig. 2g), and relevant pore size distribution curves indicated a wide mesopore distribution due to excessive leaching (Fig. 2h and i) [37]. The existence of substantial amounts of mesopores in Z5-HNS-80* could be further confirmed by corresponding HR-TEM images, implying that desired hierarchical structure had been created (Fig. 2d and e). Moreover, in comparison with the corresponding Z5-B-80, both external surface areas (from 303.5 to 435.6 $m^2 \cdot g^{-1}$) and pore volumes (from 0.02 to 0.17 cm³ g⁻¹) of Z5-HNS-80^{*} were drastically increased (shown in Table S4). The corresponding SAED pattern of a randomly selected ZSM-5 NS (Fig. 2f) exhibited discrete diffraction spots, thereby providing clear evidence of the singlecrystalline nature of Z5-HNS-80*. The subsequent mechanical test confirmed that prepared Z5-HNS-80* were mechanically robust since their morphology remained intact even after being subjected to vigorous stirring (400 r/min) for 24 h. On the contrary, Z5-HNS-80 were broken into pieces under similar testing conditions (Fig. S2).

Our previous research indicated that the formation of Silicalite-1 NSs involved a dissolution-recrystallization mechanism upon hydrothermal treatment in an aqueous TPAOH solution [38]. In comparison, there existed some discrepancies during the formation of monodispersed single-crystalline hierarchical ZSM-5 NSs due to the presence of [Al(OH)]⁻ in the framework. SEM and TEM results confirmed that at the early stage, the interior of bulk ZSM-5 zeolites was preferentially dissolved because of a lower crystallinity, resulting in the formation of hierarchical hollow ZSM-5 crystals with well-preserved but roughened edges (Fig. 3a and b). Roughened edges of Z5-B-80 may largely be attributed to the Kirkendall effect, i.e., the diffusion of dissolved silicon and aluminum species to external surfaces of hollow ZSM-5 crystals, followed by the nucleation and subsequent growth of these secondary crystals on their *a*/*c*-faces [39,40]. Further prolonging the etching duration led to progressive thinning of crystal walls in all directions (Fig. 3c and d). Since crystal faces of bulk ZSM-5 crystals perpendicular to the *b*-axis direction were the largest, finally only [0 k0] faces of bulk ZSM-5 zeolites survived, resulting in the formation of hierarchical ZSM-5 NSs upon prolonging the etching duration to 18 h. Further extending the etching time to 24 h unexpectedly led to a remarkable increase of wall thickness to 150 nm along the *b*-axis (Fig. 3e and f), indicating that the recrystallization rate outbalanced the dissolution rate during this interval.

Considering the significant influence of acid site strength distribution and their respective concentrations in relevant catalytic reactions, NH₃-TPD measurement was further carried out to quan-

tify the acid site strength distribution of both Z5-HNS-80^{*} and Z5-B-80 samples. Two well-defined NH₃ desorption peaks centering at the low-temperature (LT) region (100–300 °C) and the high-temperature (HT) region (400–500 °C) could be discerned (Fig. 2j and Table S5), indicating the presence of strong-strength acid sites. Besides, Lewis acid (LA) and Brønsted acid (BA) site distributions of the above samples were studied by Py-IR. The band at 1454 cm⁻¹ could be assigned to LA, while the band at around 1546 cm⁻¹ could be assigned to BA (Fig. 2k) [41]. The B/L ratio increased dramatically from 1.2 to 359.6 via TPAOH etching probably because of the insertion of more Al species into the zeolite framework (Table S6).

To gain further insights into the above NH₃-TPD and Py-IR results, ²⁷Al and ²⁹Si MAS NMR analyses were carried out. ²⁷Al MAS NMR revealed that in comparison with Z5-B-80, Z5-HNS-80* showed a stronger peak at δ_{FAL} = 54 ppm corresponding to tetrahedrally coordinated Al atoms in the zeolite framework (Fig. 21) [42], which further demonstrated that the enhanced structural order of four-coordinated Al aluminosilicates and the increase of framework Al content with the formation of Z5-HNS-80* jointly contributed to the generation of more BA sites. While corresponding ²⁹Si NMR spectra of Z5-B-80 and Z5-HNS-80* showed four poorly separated peaks centering at -105 to -117 ppm (Fig. 2m and n), which could be assigned to Si(2Al), Si (1Al), and Si(0Al) sites, respectively [43]. Of particular note, the peak intensity of $\delta_{\text{Si(1AI)}}$ of Z5-HNS-80* was remarkably higher than that of Z5-B-80 (Fig. 2m and n), which could be ascribed to the higher content of framework Al species of the former, which was consistent with aforementioned ²⁷Al MAS NMR result. To sum up, controlled Si leaching from the zeolite framework, the incorporation of Al species derived from NaAlO₂ in the zeolite framework, and the creation of mesopores with no compromise in crystallinity during alkaline etching cooperatively contributed to a higher concentration of BA site and strengthened acidity of Z5-HNS-80^{*} [44].

Finally, methanol aromatization was chosen as the probe reaction to evaluate the catalytic performances of prepared Z5-HNS-80* (Si/Al ratio: 17.0) and commercial Z5-NK-18 (Si/Al ratio: 18.0) [45,46]. The methanol conversion (%) and BTX selectivity (%) as a function of time-on-stream (TEOS) of the zeolite catalysts were displayed in Fig. 4(a and b). It was observed that catalysts exhibited a complete methanol conversion (100%) at the initial stage (Fig. 4a), and BTX selectivity for Z5-HNS-80* (34.5%) was higher than that of the commercial sample (Z5-NK-18, 28.9%) (Fig. 4b). Furthermore, the lifetime and stability of Z5-HNS-80* were significantly higher than those of Z5-NK-18 (Fig. 4), irrespective of the fact that both samples possessed similar Si/Al ratios, i.e., similar acidity. To sum up, the above results vividly confirmed improved usage of acid sites due to established porosities within monodispersed single-crystalline hierarchical ZSM-5 NSs.

To summarize, in this study we successfully prepared monodispersed single-crystalline hierarchical ZSM-5 NSs exhibiting tailorable Si/Al ratio and excellent stability simply via anisotropic etching in an aqueous TPAOH solution. Of particular note, the addition of NaAlO₂ during alkaline etching was found to be indispensable for maintaining their morphological integrity in the range of low Si/Al ratio. Compared with bulk ZSM-5 crystals, both external surface areas and pore volumes of corresponding hierarchical ZSM-5 NSs were increased. The mechanism involving the NS formation was proven to follow a dissolution-recrystallization mechanism. Moreover, hierarchical ZSM-5 NSs exhibited higher BA site concentration and strengthened acidity, owing to the insertion of more Al species in the zeolite framework. The methanol aromatization reaction results indicated that compared with Z5-NK-18, Z5-HNS-80* exhibited enhanced methanol conversion (>99%), BTX selectivity (>31%), and catalyst stability (56.3 h), owing to better use of acid sites.



Fig. 2. Characterization of Z5-B-80 and Z5-HNS-80* prepared by alkaline etching in 0.73 M TPAOH aqueous solution containing 0.8 g of NaAlO₂ for 18 h. (a and b) SEM images, (c) XRD patterns, (d and e) TEM images, (f) SAED pattern, (g) N₂ adsorption-desorption isotherms curves at 77 K, (h) micropore distributions curves, (i) mesopore distributions curves, (j) NH₃-TPD patterns, (k) Py-IR spectra after pyridine desorption at 573 K, (l) ²⁷Al MAS NMR, and (m and n) ²⁹Si MAS NMR spectra of Z5-B-80 and Z5-HNS-80*.



Fig. 3. SEM and/or TEM images of Z5-HNS-80* obtained at etching times of (a, b) 40 min, (c) 45 min, (d) 6 h and (e, f) 24 h.



Fig. 4. Comparison of catalytic stability of Z5-NK-18 and Z5-HNS-80* in methanol aromatization reaction at WHSV = 1 h⁻¹, T_{reaction} = 400 °C, n (N₂): n (CH₃OH) = 1:1. (a) Methanol conversion, and (b) the selectivity of BTX.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jechem.2022.06.008.

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