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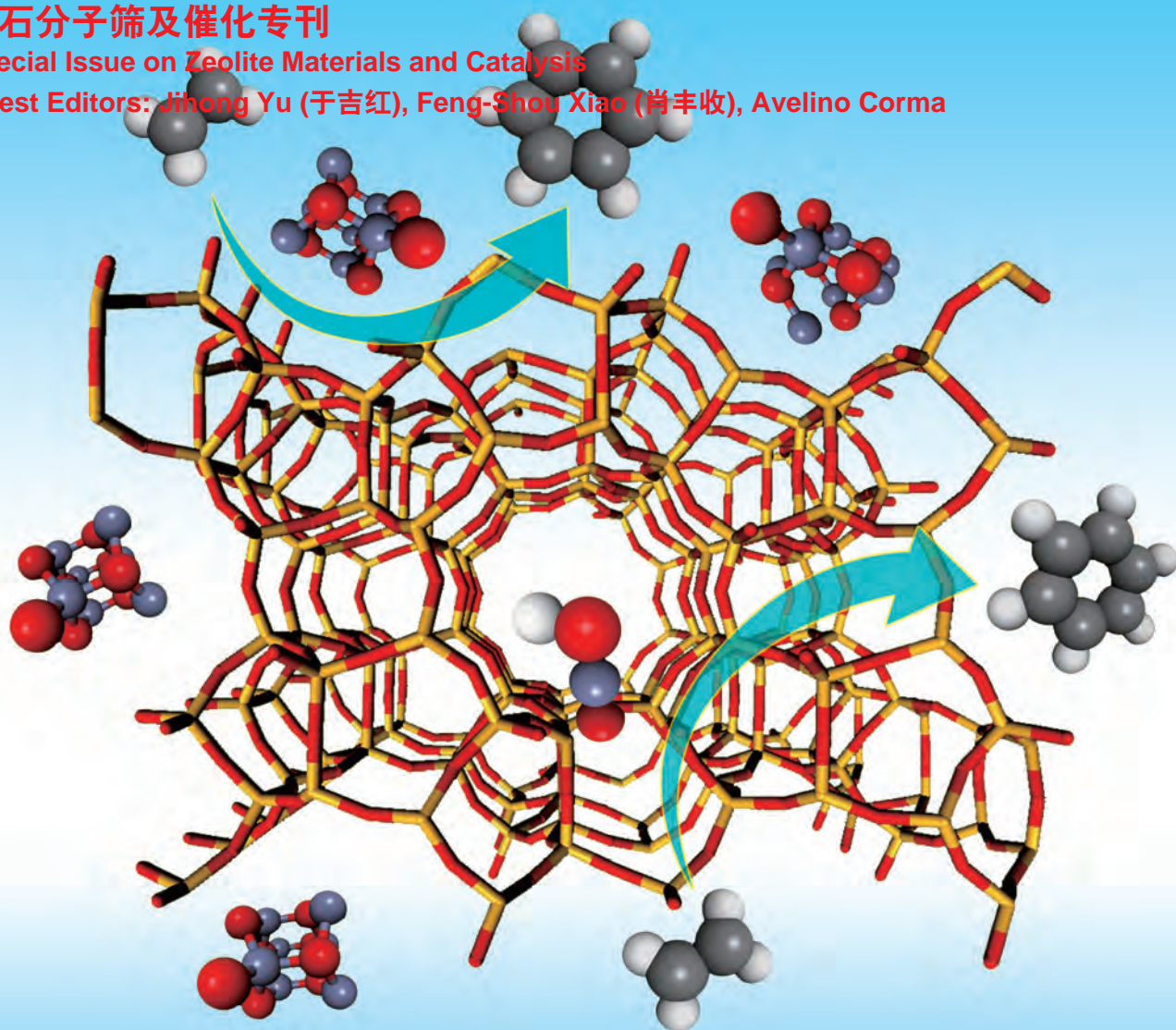
2015

Vol. 36 No. 6

沸石分子筛及催化专刊

Special Issue on Zeolite Materials and Catalysis

Guest Editors: Jihong Yu (于吉红), Feng-Shou Xiao (肖丰收), Avelino Corma



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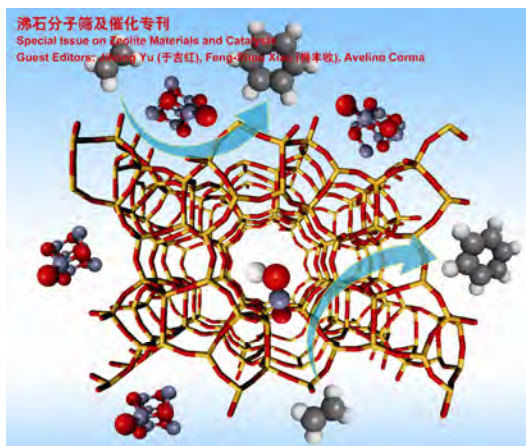
CHINESE JOURNAL OF CATALYSIS

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In This Issue



封面: 樊卫斌等采用离子交换法和物理混合法制备了 Zn/HZSM-5 催化剂, 将该催化剂用于乙烯芳构化反应. 发现催化剂中 Zn 物种以 ZnO 晶粒、ZnO 团簇和 Zn(OH)⁺ 三种状态存在, ZnO 物种促进低碳烷烃脱氢, Zn(OH)⁺ 促进烯烃芳构化. 见本期第 880–888 页.

Cover: Fan et al. studied the catalytic properties of Zn/HZSM-5 prepared by ion-exchange and physically mixing methods for aromatization of ethylene in their article on pages 880–888. It was found that Zn species existed in the states of ZnO crystals, ZnO clusters and Zn(OH)⁺ cations. ZnO facilitated the dehydrogenation of light alkanes, and Zn(OH)⁺ species promoted the aromatization of alkenes.

About the Journal

Chinese Journal of Catalysis is an international journal published monthly by Chinese Chemical Society, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, and Elsevier. The journal publishes original, rigorous, and scholarly contributions in the fields of heterogeneous and homogeneous catalysis in English or in both English and Chinese. The scope of the journal includes:

- ◆ New trends in catalysis for applications in energy production, environmental protection, and production of new materials, petroleum chemicals, and fine chemicals;
 - ◆ Scientific foundation for the preparation and activation of catalysts of commercial interest or their representative models;
 - ◆ Spectroscopic methods for structural characterization, especially methods for in situ characterization;
 - ◆ New theoretical methods of potential practical interest and impact in the science and applications of catalysis and catalytic reaction;
 - ◆ Relationship between homogeneous and heterogeneous catalysis;
 - ◆ Theoretical studies on the structure and reactivity of catalysts.
- ◆ The journal also accepts contributions dealing with photo-catalysis, bio-catalysis, and surface science and chemical kinetics issues related to catalysis.

Types of Contributions

- **Reviews** deal with topics of current interest in the areas covered by this journal. Reviews are surveys, with entire, systematic, and important information, of recent progress in important topics of catalysis. Rather than an assemblage of detailed information or a complete literature survey, a critically selected treatment of the material is desired. Unsolved problems and possible developments should also be discussed. Authors should have published articles in the field. Reviews should have more than 80 references.
- **Communications** rapidly report studies with significant innovation and major academic value. They are limited to four Journal pages. After publication, their full-text papers can also be submitted to this or other journals.
- **Articles** are original full-text reports on innovative, systematic and completed research on catalysis.
- **Highlights** describe and comment on very important new results in the original research of a third person with a view to highlight their significance. The results should be presented clearly and concisely without the comprehensive details required for an original article.
- **Perspectives** are short reviews of recent developments in an established or developing topical field. The authors should offer a critical assessment of the trend of the field, rather than a summary of literatures.
- **Viewpoints** describe the results of original research in general in some area, with a view to highlighting the progress, analyzing the major problems, and commenting the possible research target and direction in the future.

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催化学报

(CUIHUA XUEBAO)

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沸石分子筛及催化专刊

客座主编: 于吉红, 肖丰收, Avelino Corma

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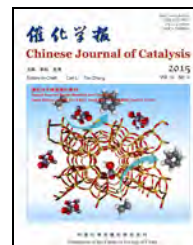
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Editorial

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Preface to Special Issue on Zeolite Materials and Catalysis

Jihong Yu, Feng-Shou Xiao, Avelino Corma
Jilin University, China; Zhejiang University, China; Universidad Politécnica de Valencia, Spain



Review

Chin. J. Catal., 2015, 36: 789–796 doi: 10.1016/S1872-2067(14)60290-4

Recent advances in solid state NMR characterization of zeolites

Shenhui Li, Lei Zhou, Anmin Zheng, Feng Deng*
Wuhan Institute of Physics and Mathematics, Chinese Academy of Sciences

Solid-state NMR

54.7

Zeolite

- Determination of topology structure*
- Characterization of acid sites*
- Investigation of host-guest interaction*

The recent research progress on advanced solid state NMR characterization of the structure, active sites (acid sites) and host-guest interaction in zeolites was reviewed.

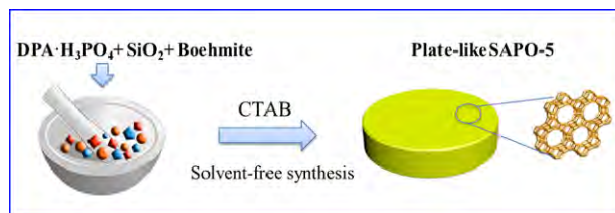
Communications

Chin. J. Catal., 2015, 36: 797–800 doi: 10.1016/S1872-2067(14)60285-0

Solvent-free synthesis of SAPO-5 zeolite with plate-like morphology in the presence of surfactants

Xian Chen, Xiangju Meng*, Feng-shou Xiao
Zhejiang University

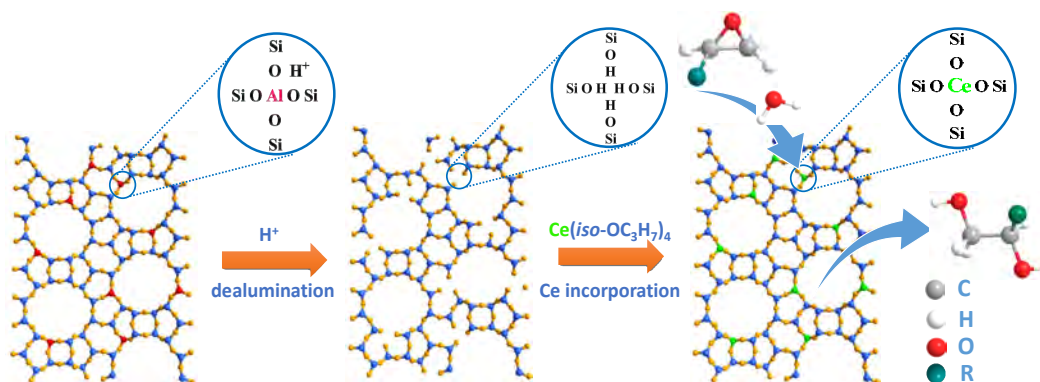
SAPO-5 crystals with plate-like morphology were synthesized in the presence of surfactants such as CTAB under solvent-free conditions. Such zeolite crystals with unique plate-like morphology are expected to be of great importance in catalytic applications.



Chin. J. Catal., 2015, 36: 801–805 doi: 10.1016/S1872-2067(14)60277-1

Incorporation of cerium atoms into Al-free Beta zeolite framework for catalytic application

Bo Tang, Weili Dai*, Xiaoming Sun, Guangjun Wu, Landong Li, Naijia Guan, Michael Hunger
Nankai University, China; University of Stuttgart, Germany



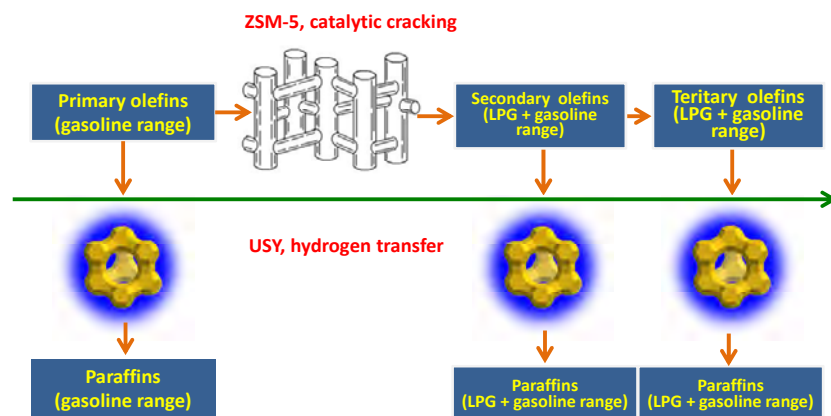
Preparation of Al-free Ce-Beta zeolite was realized for the first time by a facile and scalable two-step post-synthesis procedure and the material was used as a catalyst in a ring-opening hydration reaction.

Articles

Chin. J. Catal., 2015, 36: 806–812 doi: 10.1016/S1872-2067(14)60311-9

ZSM-5 zeolites with different SiO₂/Al₂O₃ ratios as fluid catalytic cracking catalyst additives for residue cracking

Pusheng Liu, Zhongdong Zhang, Mingjun Jia, Xionghou Gao*, Jihong Yu*
Jilin University; Lanzhou Petrochemical Research Center



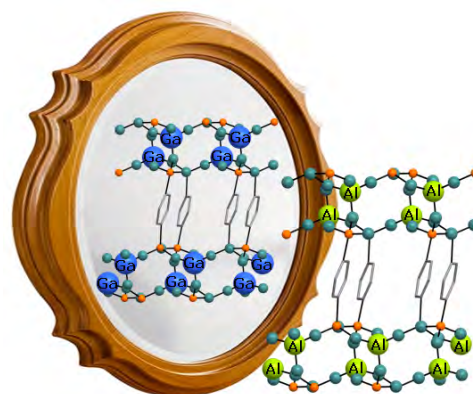
Higher SARs led to reduced cracking of primary olefins over ZSM-5 additives. Using ZSM-5 with SARs of 266 and 487 efficiently enhanced the octane number of gasoline with minimal loss of gasoline.

Chin. J. Catal., 2015, 36: 813–819 doi: 10.1016/S1872-2067(14)60296-5

Synthesis and characterization of Si/Ga Eni Carbon Silicates

Giuseppe Bellussi, Angela Carati, Stefania Guidetti, Caterina Rizzo, Roberto Millini, Stefano Zanardi, Erica Montanari, Wallace O'Neil Parker Jr., Michela Bellettato*
Eni s.p.a., Development, Operations & Technology, Downstream R&D, Italy

Hybrid organic-inorganic phenylene-gallosilicates were prepared with the same crystalline structure as their aluminum analogues, which demonstrated the possibility to tailor the crystal chemistry of the zeolite-like moiety in the framework of Eni Carbon Silicate materials.

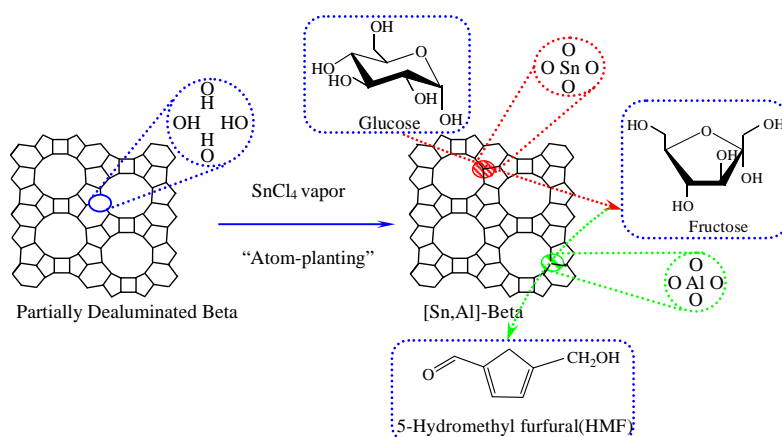


Chin. J. Catal., 2015, 36: 820–828 doi: 10.1016/S1872-2067(14)60287-4

One-pot synthesis of 5-hydroxymethylfurfural from glucose using bifunctional [Sn,Al]-Beta catalysts

Liang Li, Jianghong Ding, Jin-Gang Jiang, Zhiguo Zhu, Peng Wu*
East China Normal University

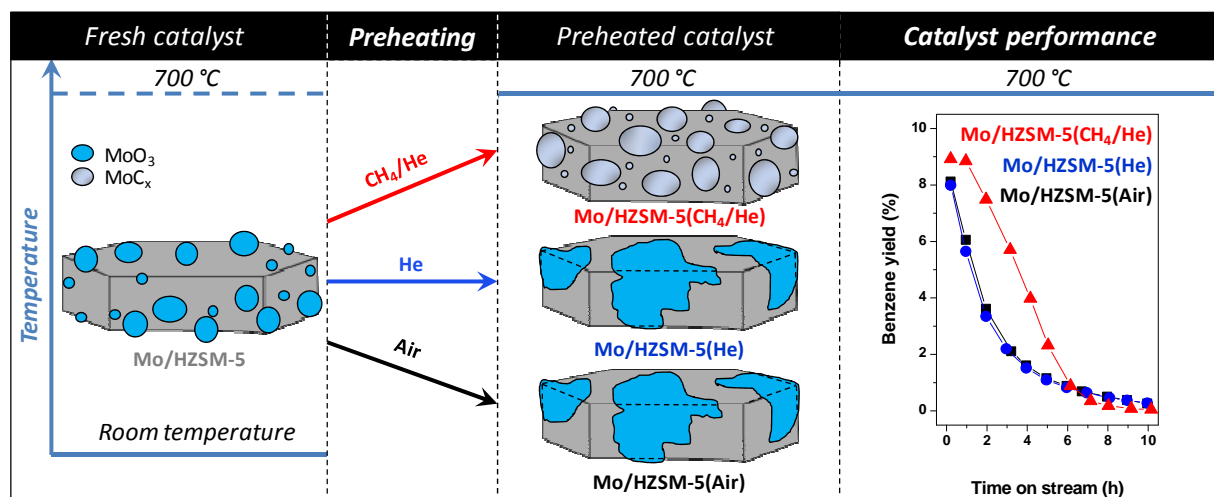
[Sn,Al]-Beta served as an integrated, bifunctional, and stable solid acid catalyst for the conversion of glucose to 5-hydroxymethylfurfural (5-HMF) in a continuous slurry reactor, giving 60% glucose conversion and 62% 5-HMF selectivity.



Chin. J. Catal., 2015, 36: 829–837 doi: 10.1016/S1872-2067(14)60301-6

Activation of Mo/HZSM-5 for methane aromatization

Christiaan H. L. Tempelman, Emiel J. M. Hensen*
Eindhoven University of Technology, the Netherlands



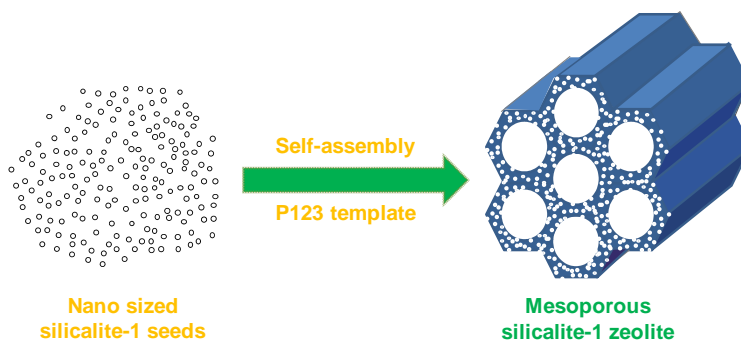
Carburization of Mo/HZSM-5 prevents dispersion of some of the molybdenum oxide particles into micropores, compared with activation in He or air. The molybdenum carbide particles formed in the micropores cause rapid deactivation of the micropore space, presumably by the formation of soft coke on the molybdenum carbides.

Chin. J. Catal., 2015, 36: 838–844 doi: 10.1016/S1872-2067(14)60255-2

Ordered mesoporous silicalite-1 zeolite assembled from colloidal nanocrystalline precursors

Fangfang Wei, Weiguo Song*, Fang Wei, Changyan Cao
Institute of Chemistry, Chinese Academy of Sciences

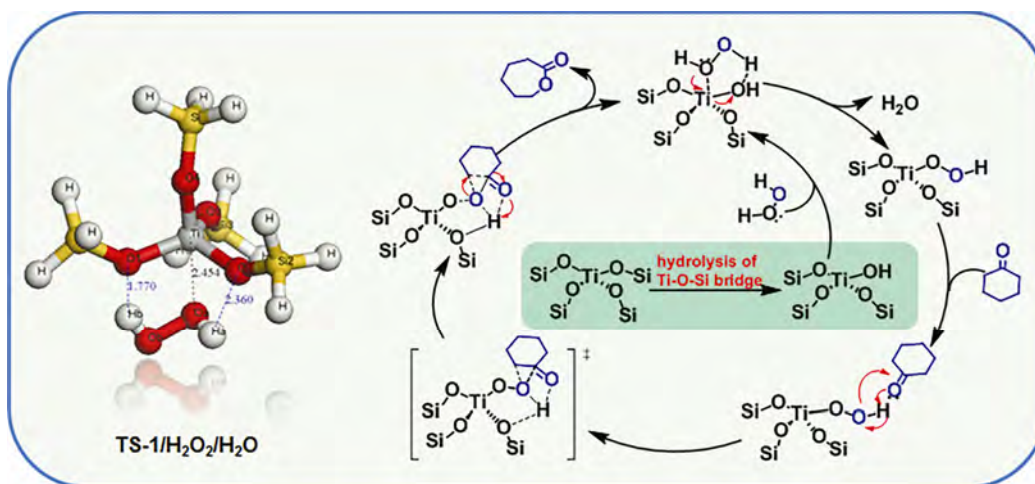
Ordered mesoporous zeolite with large surface area and pore volume is synthesized by nanosized silicalite-1 seed self-assembly and using commercial P123 triblock copolymer in acidic media.



Chin. J. Catal., 2015, 36: 845–854 doi: 10.1016/S1872-2067(15)60859-2

Heterogeneous oxidation of cyclohexanone catalyzed by TS-1: Combined experimental and DFT studies

Changjiu Xia, Long Ju, Yi Zhao, Hongyi Xu, Bin Zhu, Feifei Gao, Min Lin*, Zhenyu Dai, Xiaodong Zou*, Xingtian Shu
Stockholm University, Sweden; Research Institute of Petroleum Processing, China



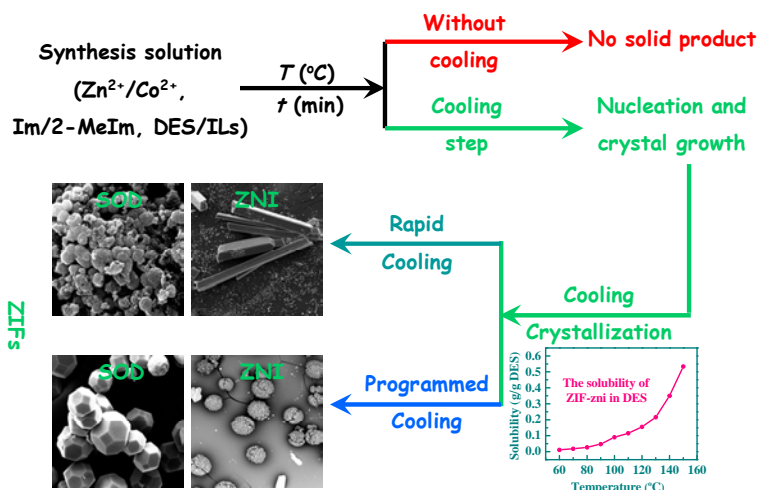
The mechanism of the oxidation of cyclohexanone catalyzed by titanium silicate zeolite using aqueous hydrogen peroxide as the oxidant was investigated by combining DFT calculations with catalytic experiments.

Chin. J. Catal., 2015, 36: 855–865 doi: 10.1016/S1872-2067(14)60278-3

Ionothermal synthesis of zeolitic imidazolate frameworks and the synthesis dissolution-crystallization mechanism

Yasong Wang, Yunpeng Xu, Dawei Li, Hao Liu, Xiaolei Li, Shuo Tao, Zhijian Tian*
Dalian Institute of Chemical Physics,
Chinese Academy of Sciences;
University of Chinese Academy of Sciences

Zeolitic imidazolate frameworks are ionothermally synthesized in an ionic liquid and a deep eutectic solvent. They precipitate from the synthesis solution by a cooling-induced crystallization process following the dissolution-crystallization mechanism.

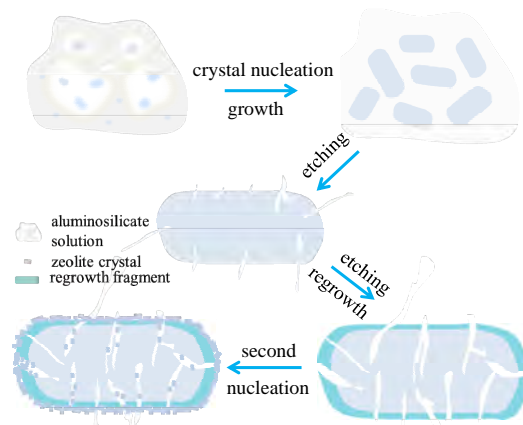


Chin. J. Catal., 2015, 36: 866–873 doi: 10.1016/S1872-2067(14)60263-1

One-pot synthesis of hierarchically structured ZSM-5 zeolites using single micropore-temple

Tongguang Ge, Zile Hua*, Xiaoyun He, Yan Zhu, Wenchao Ren, Lisong Chen, Lingxia Zhang, Hangrong Chen, Chucheng Lin, Heliang Yao, Jianlin Shi*
Shanghai Institute of Ceramics, Chinese Academy of Sciences

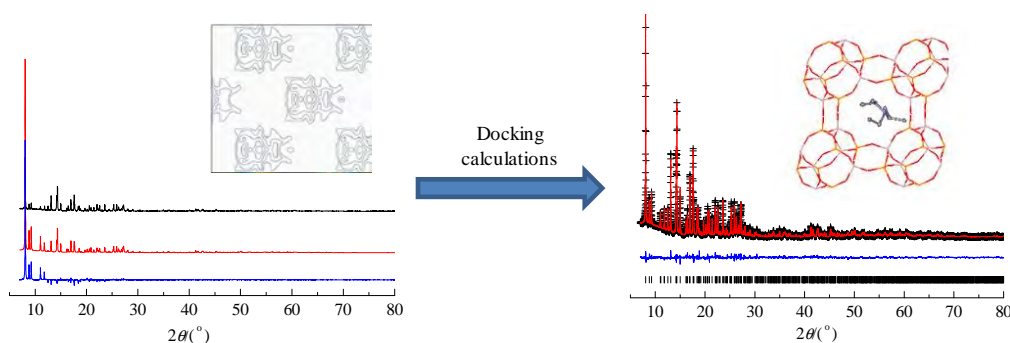
An easy one-step low temperature (100 °C) hydrothermal process has been developed for the synthesis of hierarchically structured ZSM-5 zeolites with a single micropore template, and a "nucleation/growth-demetalation/ recrystallization" mechanism is discussed.



Chin. J. Catal., 2015, 36: 874–879 doi: 10.1016/S1872-2067(14)60306-5

Location and orientation of the *N,N*-diisopropylethylamine template molecule in the $\text{AlPO}_4\text{-18}$ framework by X-ray synchrotron diffraction and molecular modelling

Christiana Zenonos, Dewi W. Lewis, Gopinathan Sankar*
University College London, United Kingdom

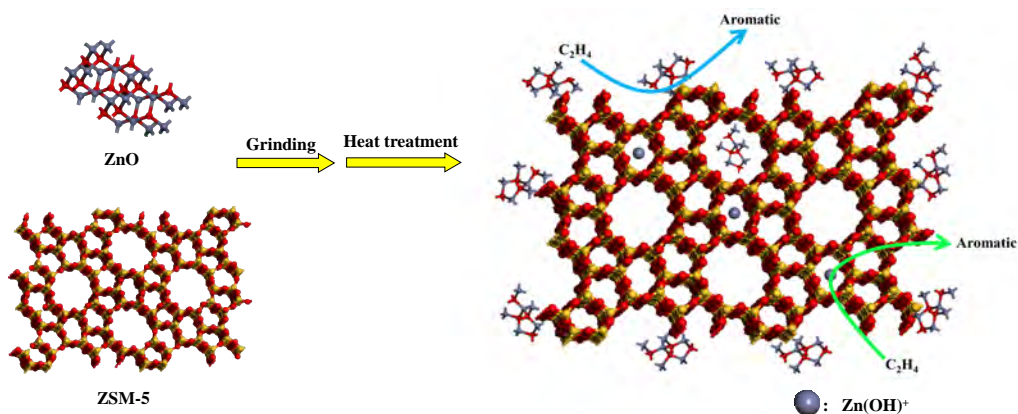


A combination of high-resolution powder diffraction and molecular modelling allowed determination of the as-synthesised structure of $\text{AlPO}_4\text{-18}$ templated with *N,N*-diisopropylethylamine.

Chin. J. Catal., 2015, 36: 880–888 doi: 10.1016/S1872-2067(14)60289-8

Influence of Zn species in HZSM-5 on ethylene aromatization

Xuchuan Chen, Mei Dong*, Xianjun Niu, Kai Wang, Gang Chen, Weibin Fan*, Jianguo Wang, Zhangfeng Qin
Institute of Coal Chemistry, Chinese Academy of Sciences; University of Chinese Academy of Sciences

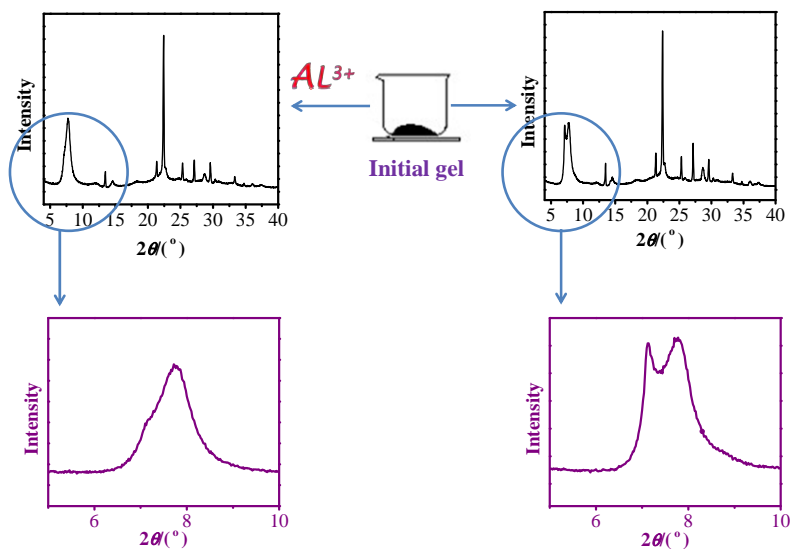


An efficient Zn-containing HZSM-5 catalyst for ethylene aromatization was prepared by physically mixing. Both Zn(OH)^+ and ZnO species facilitated the dehydrogenation of light alkanes, and promoted the formation of aromatics.

Chin. J. Catal., 2015, 36: 889–896 doi: 10.1016/S1872-2067(14)60300-4

Influence of Al³⁺ on polymorph A enrichment in the crystallization of beta zeolite

Tingting Lu, Pan Gao, Jun Xu, Yongrui Wang, Wenfu Yan*, Jihong Yu, Feng Deng, Xuhong Mu, Ruren Xu
*Jilin University; Wuhan Institute of Physics and Mathematics, Chinese Academy of Sciences;
 Research Institute of Petroleum Processing, SINOPEC*



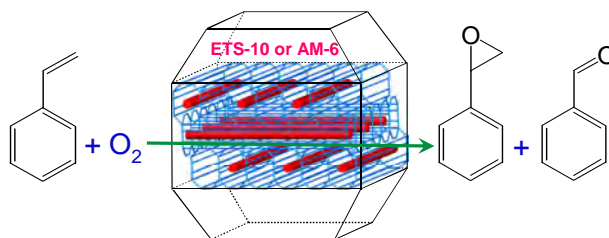
Polymorph A-enriched silica beta zeolite was synthesized. The degree of enrichment of chiral polymorph A was decreased owing to five coordinated Al species.

Chin. J. Catal., 2015, 36: 897–905 doi: 10.1016/S1872-2067(15)60864-6

Co-ETS-10 and Co-AM-6 as active catalysts for the oxidation of styrene to styrene oxide and benzaldehyde using molecular oxygen

Shuvo Jit Datta, Kyung Byung Yoon*
Sogang University, Korea

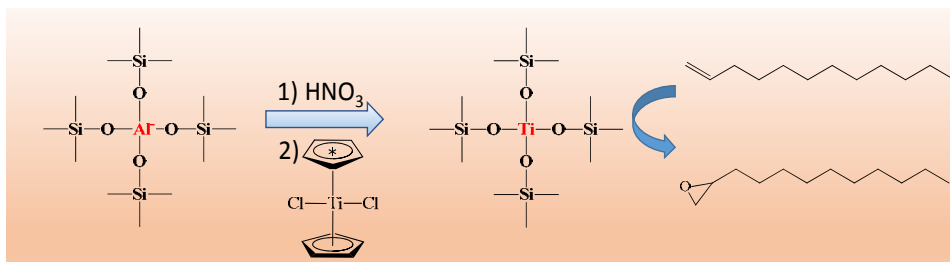
ETS-10, AM-6, and their corresponding Co²⁺-exchanged forms were examined as catalysts toward the oxidation of styrene in oxygen atmosphere. The conversion increased, whereas the styrene epoxide/benzaldehyde product ratio decreased with increasing degrees of Co²⁺ exchange. In the absence of Co²⁺, pristine ETS-10 and AM-6 were catalytically active toward the formation of styrene epoxide.



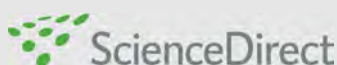
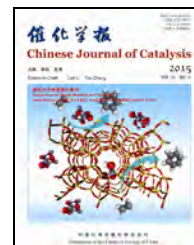
Chin. J. Catal., 2015, 36: 906–912 doi: 10.1016/S1872-2067(14)60267-9

Post-synthesis of hierarchically structured Ti-β zeolites and their epoxidation catalytic performance

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Hierarchical Ti-β zeolites were prepared using a two-step post-synthesis procedure. This solid-state procedure is simple and Ti-content tunable. The synthesized hierarchical Ti-β zeolites had higher catalytic activities than microporous Ti-β in bulky molecule epoxidation.

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Editorial (Special Issue on Zeolite Materials and Catalysis)

Preface to Special Issue on Zeolite Materials and Catalysis

Zeolites are crystalline microporous materials with regular channels and cages of molecular size. Due to their unique structures and acidic properties, they have been widely used in the petrochemical and chemical industries since the late 1950s. Zeolites have been used as catalysts, for instance, in fluid catalytic cracking and hydrocracking.

Faujasites were the first molecular sieves applied for catalytic purposes. Today, FAU is still the most important zeolite structure type used in catalytic transformations. Industrially important zeolites are produced synthetically. Although most industrial catalysts are drawn from the FAU, MFI, MOR, FER and BEA structure types, there are also worldwide attempts to synthesize new structure types to improve the understanding of how the zeolite structure, composition, and physical properties affect the catalytic transformation. As of now, 225 types of zeolite topology have been identified by the Structure Commission of the International Zeolite Association (IZA-SC), including over 40 naturally occurring zeolite frameworks.

This special issue is an update of the state-of-art of "Zeolite Materials and Catalysis" which covers the synthesis and characterization of zeolites and their catalytic reactions. One review, 2 communications, and 13 research articles are included in this special issue. Review paper is focused on recent advance in solid-state NMR characterization of zeolites (Feng Deng).

Communications cover the solvent-free synthesis of SAPO-5 zeolite with plate-like morphology in the presence of surfactants (Feng-Shou Xiao, Xiangju Meng) and the incorporation of cerium atoms into Al-free beta zeolite framework for catalytic application (Naijia Guan, Weili Dai). Research articles are focused on utilization of ZSM-5 zeolites with different silica/alumina ratios as FCC catalyst additives for residue cracking (Jihong Yu), synthesis and characterization of Eni carbon silicates prepared in the Si/Ga system (Giuseppe Bellussi, Michela Bellettato), One-pot synthesis of 5-Hydroxymethyl furfural from glucose using bifunctional [Sn,Al]-Beta catalysts (Peng Wu), activation of Mo/HZSM-5 for methane aromatization (Emiel J. M. Hensen), ordered mesoporous silicalite-1 zeolite assembled from colloidal nanocrystalline precursors (Weiguo Song), heterogeneous oxidation of cyclohexanone catalyzed by titanium silicate zeolite TS-1: combined experimental and DFT studies (Xiaodong Zou), ionothermal synthesis of zeolitic imidazolate frameworks and the synthesis dissolution-crystallization mechanism (Zhijian Tian), one-pot synthesis of hierarchically structured ZSM-5 zeolites using single micropore-template (Jianlin Shi, Zile Hua), location and orientation of the *N,N*-diisopropyl ethylamine template in the AEI framework by X-ray synchrotron diffraction and molecular modelling (Sankar Gopinathan), influence of zinc species on the reaction process



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of ethylene aromatization (Jianguo Wang, Weibin Fan), the influence of Al^{3+} on the enrichment of polymorph A in the crystallization of zeolite beta (Wenfu Yan), Co-ETS-10 and Co-AM-6 as active catalysts for facile oxidation of styrene into styrene oxide and benzaldehyde with molecular oxygen (Kyung Byung Yoon), and post-synthesis of hierarchically structured Ti-BEA

zeolites and their epoxidation catalytic performance (Jianlin Shi, Zile Hua). The high quality of the papers written by international well-known research groups in this highly exciting research area will render this special issue a good reference for future research activity.

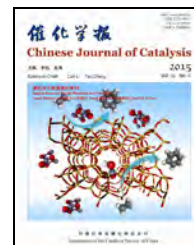
We express our sincere thanks to all the authors.



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Communication (Special Issue on Zeolite Materials and Catalysis)

Incorporation of cerium atoms into Al-free Beta zeolite framework for catalytic application

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ABSTRACT

Ce(IV)-containing zeolite Beta was successfully prepared by a reproducible and scalable two-step post-synthesis strategy. This consists of creating vacant T sites that are associated with silanol groups by the dealumination of the H-Beta zeolite and a subsequent dry impregnation of the resultant Si-Beta zeolite with cerium (IV) isopropoxide. XRD, FT-IR, UV-Vis and ¹H MAS NMR confirmed that Ce(IV) was successfully incorporated into the Beta zeolite framework by the post-synthesis procedures and that they exist as isolated atoms with tetrahedral coordination. The mechanism of Ce incorporation into the framework of Beta zeolite was confirmed by DRIFT spectroscopy. The ring-opening hydration of epoxides to their corresponding 1,2-diols was selected as a model reaction for an evaluation of the catalytic performance of the as-synthesized Ce-Beta zeolite.

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Zeolites are widely used as catalysts in the refining, petrochemical, and fine chemical industries because of their high specific surface area, large pore volume, uniform microporous channels and excellent thermal and hydrothermal stability [1,2]. The incorporation of transition metals into the zeolite framework can deliver heteroatomic zeolite materials with new remarkable catalytic activity. Transition metals such as Ti, Sn, Zr, V, and Nb, have been introduced into the framework of different zeolitic structure allowing their application in new fine chemical processes as highly active and selective heterogeneous catalysts [3]. One of the most interesting classes of materials is highly hydrophobic BEA-type metallosilicate zeolites. They exhibit superior catalytic activity in novel biomass-related transformations in water. Nevertheless, few

studies have been reported on isomorphous substitution with lanthanides (Ln) in BEA zeolites and the resultant catalytic properties of these microporous crystalline materials [4,5]. A feasible method for the preparation of framework-substituted lanthanide zeolites still remains a significant challenge because of size incompatibility between lanthanides and the Si⁴⁺ ion. The reason for this is the longer ≡Si–O–Ln≡ bonds with large bond-angle strain in the Ln-substituted rigid silica networks.

For the preparation of heteroatomic zeolites, different methods such as conventional hydrothermal crystallization [6], dry-gel conversion [7], fluoride modification [8] and microwave-assisted methods [9] have been adopted. Although highly active metallosilicates can be obtained using direct synthesis strategies, most of these routes are complicated and time-con-

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suming resulting in both a lack of reproducibility and feasibility for large-scale synthesis. Therefore, alternative post-synthesis strategies have been developed for the preparation of these materials [10–14]. Recently, we reported an improved post-synthesis approach for the preparation of Ti(IV)- and Sn(IV)-containing Beta zeolites [15,16]. The dry impregnation (DI) of a dealuminated Beta zeolite with an organometallic precursor followed by simple thermal treatment has been shown to be an attractive route for the incorporation of metal ions into a zeolite framework. A significant amount of active centers can be created.

In this study, we adopted the following strategy to incorporate Ce(IV) into the framework of Beta zeolite. Vacant T sites with associated silanol groups were first created by treating commercial H-Beta zeolite with a concentrated nitric acid solution and the resulting Si-Beta zeolite was subsequently ground with the organic Ce precursor, Ce(IV) isopropoxide, to give a Si-Beta/Ce(*iso*-OC₃H₇)₄ mixture, which was further calcined to give Ce-Beta zeolite [15,16]. The as-synthesized sample was characterized by X-ray diffraction (XRD), FT-IR, DRIFT, diffuse reflectance ultraviolet-visible (UV-Vis) spectroscopy and multinuclear solid-state nuclear magnetic resonance (NMR) spectroscopy. The catalytic performance was evaluated by the hydration of epoxides.

To determine if structural changes occurred during the post-synthesis procedures, the XRD patterns of the parent H-Beta together with those of the post-treated samples were collected and are shown in Fig. 1. All the samples are characteristic of typical BEA topology with comparable diffraction intensity apart from the zeolite framework collapse during the dealumination and Ce incorporation processes. The dealumination and incorporation of Ce species into the framework of the Beta zeolite were evident by the contraction/expansion of the framework as shown by the detectable changes in the position of the diffraction peak (302) at $2\theta = 22^\circ$. This is similar to that previously reported by Dzwigaj's group [17,18]. The d_{302} spacing, obtained from the corresponding 2θ value decreased from 0.3921 nm (H-Beta, $2\theta = 22.64^\circ$) to 0.3871 nm (Si-Beta, $2\theta = 22.95^\circ$). This suggests the contraction of the Beta matrix after the dealumination procedure. The d_{302} spacing of the Ce-Beta zeolite after dry impregnation increased to 0.3946 nm ($2\theta =$

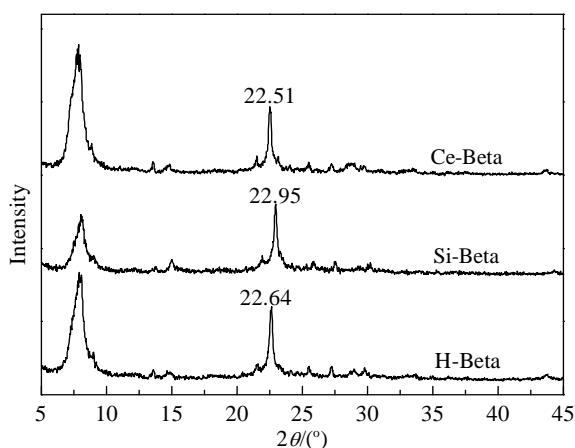


Fig. 1. XRD patterns of the H-Beta, Si-Beta, and Ce-Beta samples.

Table 1
Physicochemical properties of the H-Beta, Si-Beta, and Ce-Beta samples.

Catalyst	Si/Al ^a	Ce loading ^a (wt%)	Surface area ^b (m ² /g)	Micropore volume ^c (cm ³ /g)
H-Beta	13.5	—	590	0.204
Si-Beta	> 1800	—	620	0.220
Ce-Beta	> 1800	3.4	600	0.195

^a Determined by ICP. ^b Obtained by the BET method. ^c Calculated from the *t*-plot.

22.51°). This phenomenon is clear evidence of BEA framework expansion indicating the successful incorporation of Ce into the Beta zeolite.

The physicochemical properties of H-Beta, Si-Beta, and Ce-Beta are summarized in Table 1. All the samples have a similar BET surface area (590–620 m²/g) and micropore volume (0.195–0.220 cm³/g). This confirms that the textural properties of the Beta zeolite were well preserved after dealumination and Ce incorporation. ICP analysis results reveal that the $n_{\text{Si}}/n_{\text{Al}}$ ratio of the parent H-Beta increases significantly from 13.5 to more than 1800 for the dealuminated Si-Beta and this indicates that the latter is essentially free of Al. After the incorporation of Ce the actual Ce loading determined by ICP is close to the design value indicating the efficiency of the dry impregnation method.

The FT-IR spectra of H-Beta, Si-Beta and Ce-Beta samples are given in Fig. 2. No obvious IR bands are present in 900–1000 cm⁻¹ for the parent H-Beta sample while a band at ~948 cm⁻¹ is present for the Si-Beta sample. This newly-formed band can be assigned to the presence of a large amount of defect sites as a result of the extraction of Al atoms from the Beta framework [19]. After the incorporation of Ce the 948 cm⁻¹ band was absent indicating an interaction between Ce(IV) isopropoxide and the defect sites. Furthermore, a new band centered at 967 cm⁻¹ is present in the Ce-Beta sample and this is indicative of the successful incorporation of Ce into the framework of the Beta zeolite.

To investigate the coordination behavior of the incorporated Ce species, a diffuse reflectance UV-Vis spectrum was obtained for the as-synthesized Ce-Beta sample. As shown in

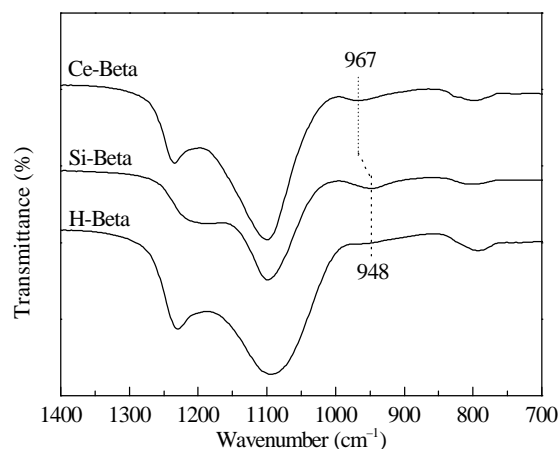


Fig. 2. FT-IR spectra showing the skeletal vibration region for H-Beta, Si-Beta, and Ce-Beta.

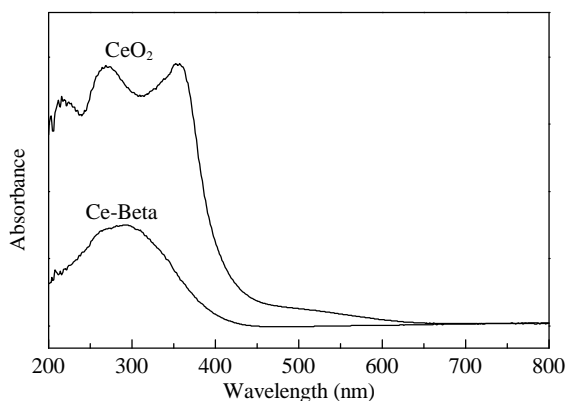


Fig. 3. UV-Vis spectra of the Ce-Beta and CeO₂ samples.

Fig. 3, the Ce-Beta zeolite sample has a main strong absorbance maximum at ~ 290 nm in strong contrast to the CeO₂ reference sample. According to the literature [5,20], this band may be attributed to a ligand-to-metal charge transfer from O²⁻ to Ce⁴⁺. Therefore, incorporated Ce is present in a highly isolated tetrahedrally coordinated form and it is generally accepted to be the catalytically active Ce species during liquid-phase oxidation reactions.

The dealumination of H-Beta and the incorporation of Ce are associated with the evolution of the silanols related to the vacant sites that formed during the post-synthesis procedures. This is further supported by the DRIFT spectra (Fig. 4). Several characteristic bands are present for the dehydrated H-Beta zeolite sample in the hydroxyl stretching region of 3800–3400 cm⁻¹. The bands at 3740 and 3605 cm⁻¹ come from isolated external Si-OH groups and the bridging hydroxyls in Si-OH-Al, respectively. Additional bands are present at 3660 and 3525 cm⁻¹, and these are associated with the Al-OH of the extra-framework aluminum species and the hydrogen-bonded Si-OH groups, respectively [15,16]. As shown in Fig. 2, the treatment of H-Beta with a concentrated HNO₃ solution results in the absence of the bands at 3660 and 3605 cm⁻¹, which are representative of Al species. This suggests the complete elimi-

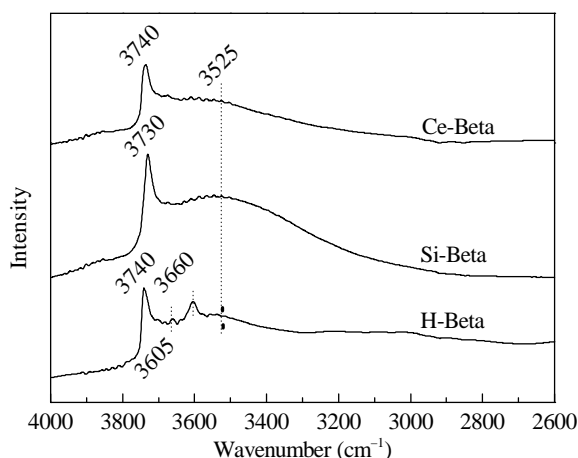


Fig. 4. DRIFT spectra in the hydroxyl stretching vibration region of H-Beta, Si-Beta, and Ce-Beta.

nation of Al atoms from the Beta framework. Simultaneously, an increase in the intensity of the band at 3730 cm⁻¹ because of the isolated internal Si-OH groups and at 3525 cm⁻¹ because of the H-bonded silanols occurs indicating the formation of vacant T sites during acid treatment. This is in accordance with earlier studies [10–12,17,18]. Subsequent dry impregnation of the Si-Beta zeolite sample with Ce(IV) isopropoxide results in an obvious decrease in the intensity of the bands at 3730 and 3525 cm⁻¹ because of the hydroxyls located at the vacant T sites. This observation indicates a reaction between the Ce species and the silanols proving their incorporation into the Beta zeolite framework.

In the ¹H MAS NMR spectra of H-Beta, a strong signal is present at 1.5 ppm and two weak signals are present at 2.5 and 3.9 ppm (Fig. 5). These signals come from silanol groups in the framework defects (1.5 ppm), hydroxyl groups from the extra-framework Al species (2.5 ppm), and bridging hydroxyl (Si(OH)Al) groups (3.9 ppm), respectively [21]. The dealumination process is characterized by the absence of hydroxyl groups associated with the framework as well as the extra-framework Al species, which are responsible for the signals at 2.5 and 3.9 ppm, respectively [21]. The intensity of the signal from the Si-OH groups in the framework defects (1.5 ppm) increases significantly and a new signal is present at 2.7 ppm. This is assigned to the Si-OH groups that are involved in hydrogen bonding to neighboring framework oxygen species within the silanol nests [15]. The incorporation of Ce into Si-Beta zeolite results in the absence of silanol groups in the framework defects and the hydroxyl nests. The remaining unreacted silanols have a chemical shift of 1.7 ppm.

Based on IR and ¹H MAS NMR spectroscopy results, the Ce-Beta zeolite can be prepared in a manner similar to the synthesis of Ti- and Sn-Beta zeolite as reported in our previous work [15,16]. The strategy employed in this study consists of creating vacant T sites with associated silanols by the dealumination of H-Beta zeolite with HNO₃ and the subsequent dry impregnation of the resulting Si-Beta zeolite with organic ce-

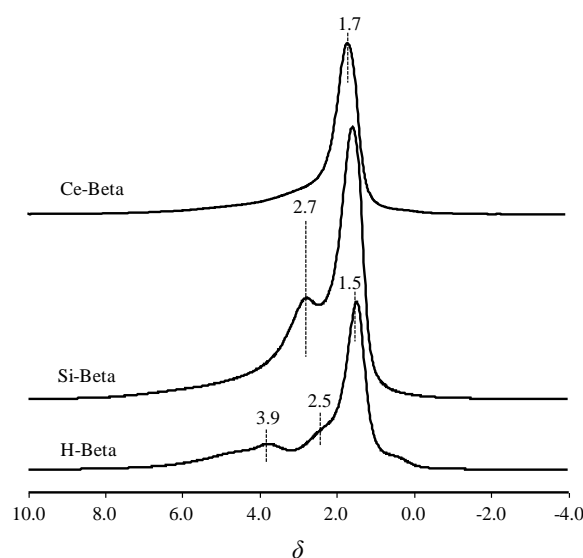
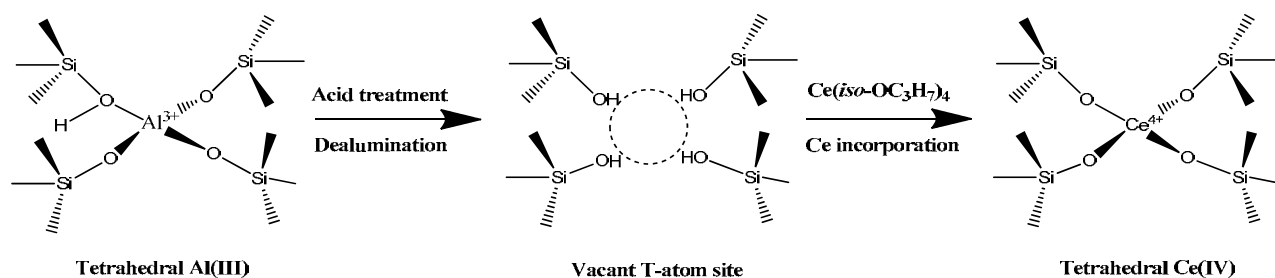


Fig. 5. ¹H MAS NMR spectra of the dehydrated H-Beta, Si-Beta, and Ce-Beta samples.

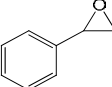
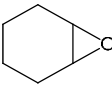



Scheme 1. Schematic representation of the incorporation of tetrahedrally coordinated Ce(IV) species into Beta zeolite.

rium(IV) isopropoxide, as illustrated in Scheme 1.

The ring-opening hydration of epoxides was selected as a model reaction to evaluate the catalytic performance of the post-synthesized Ce-Beta zeolite. Target 1,2-diols are widely used as important intermediates for the production of polyester resins, antifreeze, cosmetics, and other products. Typically, epoxide hydration is performed in a 25 mL round-bottom glass vessel with a cryogenic-liquid condenser under atmospheric pressure. In our test the vessel was charged with a mixture of 10 mmol epoxide, 20 mmol H₂O and 0.1 g catalyst, which was mixed vigorously by a magnetic stirrer. Samples from the reaction were qualitatively analyzed using a Shimadzu 2010 GC (Japan, Agilent HP-5MS column, 30 m × 0.25 mm × 0.25 μm; FID detector) with octanol as the internal standard. The GC peaks were identified by comparison with the retention time of known standard samples and also by a Shimadzu GC-MS QP2010 SE equipped with an Agilent HP-5MS column. As shown in Table 2, considerable epoxide conversion as well as high selectivity toward 1,2-diols was obtained (ethers from the dimerization/trimerization were the main by-products) accompanied by the stoichiometric incorporation of water in the ring-opening reaction. However, under identical reaction conditions very little product (1%) was obtained for the cyclohexene oxide hydration reaction in the absence of the catalyst. The use of Si-Beta zeolite as the catalyst also resulted in low catalytic activity (< 3%). The influence of preparation method, i.e. DI (dry impregnation), WI (wet impregnation) or SSIE (solid-state ion-exchange) on the activity of Ce-Beta zeolite during the ring-opening hydration of epoxides was also investigated.

Table 2
Hydration of different epoxide substrates catalyzed by Ce-Beta zeolite.

Substrate	Temperature (K)	Time (h)	Epoxide conversion (%)	1,2-Diol selectivity (%)
	333	12	89.5	> 99
	353	12	45.8	90.5
	333	6	51.2	72.9

Reaction conditions: 10 mmol epoxide, 20 mmol H₂O, 0.1 g catalyst.

*Reaction was performed in a 10 mL autoclave under a N₂ pressure of 1.0 MPa.

Outstanding catalytic performance for Ce-Beta zeolite was only obtained when using the DI route. This shows the superiority of the two-step post-synthesis strategy that was developed for the preparation of Ce-Beta in this study. The hydration of epoxides is generally carried out with a large excess of water ($n_{\text{H}_2\text{O}}/n_{\text{epoxide}} = 20\text{--}25$) at elevated temperatures (> 413 K) and/or in the presence of solvents to obtain high substrate conversion as well as the desired product selectivity. The catalytic results obtained with a lower $n_{\text{H}_2\text{O}}/n_{\text{epoxide}}$ of 2 confirm that the Ce(IV)-containing Beta zeolite can be regarded as a promising catalyst for the sustainable production of 1,2-diols.

In conclusion, a simple and scalable two-step post-synthesis procedure was developed to prepare Ce-Beta zeolite. This includes the dealumination of H-Beta zeolite and dry impregnation of the resultant Si-Beta zeolite with the organic precursor, Ce(IV) isopropoxide. We thus present a novel and simple preparation method for the synthesis of lanthanide-containing zeolites and we also expand its catalytic application.

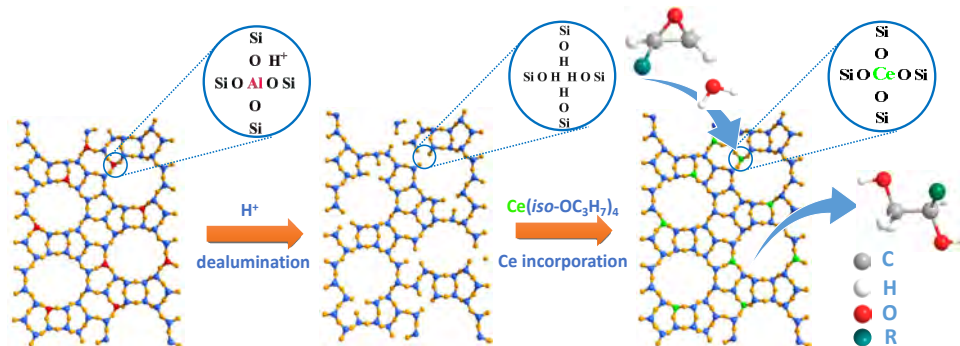
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Graphical Abstract

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Incorporation of cerium atoms into Al-free Beta zeolite framework for catalytic application

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Preparation of Al-free Ce-Beta zeolite was realized for the first time by a facile and scalable two-step post-synthesis procedure and the material was used as a catalyst in a ring-opening hydration reaction.

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Ce-Beta分子筛的制备及其催化性能

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摘要: 采用简单、易重现的两步后合成法成功制备了Ce-Beta分子筛。制备过程中, H-Beta分子筛首先经过脱铝处理得到Si-Beta分子筛, 然后再以异丙醇铈为前驱体, 通过干法浸渍的途径向Si-Beta分子筛引入Ce(IV)。利用XRD, FT-IR, UV-Vis和¹H MAS NMR等对其结构进行了表征, 结果表明, Ce(IV)物种以四配位的形式成功引入Beta分子筛的骨架, Ce原子进入分子筛骨架的机理通过DRIFT光谱得以证实。通过环氧化物水合制备1,2-二醇反应对所制的Ce-Beta催化剂进行了催化性能评价。

关键词: Ce-Beta分子筛; 后合成; 脱铝; 开环水合; 环氧

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