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Catalytic Properties of Hierarchical Ti-MFI Zeolites in the Synthesis of Cyclic Carbonates from Styrene Under CO_2 Pressure

Abstract

Hierarchical titanosilicate zeolites of the MFI structural type (zeolites with the MFI framework topology according to the classification of the International Zeolite Association) showed a high catalytic activity in the synthesis of cyclic carbonate from styrene in the presence of *tert*-butyl hydroperoxide as an oxidant. It has been shown that the combination of the redox properties of titanium centers with the acid-base characteristics of the zeolite matrix ensures the efficient implementation of the tandem process within a single reaction medium. The results obtained confirm the potential of titanium-containing zeolites as an effective platform for creating heterogeneous catalysts for the direct conversion of olefins into cyclic carbonates.

Keywords: styrene; cyclic carbonate; styrene oxide; hierarchical zeolites; titanosilicates

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Каталітичні властивості ієрархічних Ti-MFI цеолітів у процесі одержання циклічних карбонатів зі стиролу під тиском CO_2

Анотація

Ієрархічні титаносилікатні цеоліти структурного типу MFI (цеоліти з топологією MFI згідно з класифікацією Міжнародної цеолітної асоціації) показали високу каталітичну активність у реакціях одержання циклічного карбонату зі стиролу в присутності *трет*-бутилгідропероксиду як окисника. Доведено, що поєднання окисно-відновних властивостей титанових центрів із кислотно-основними характеристиками цеолітної матриці забезпечує ефективну реалізацію тандемного процесу в одному реакційному середовищі. Отримані результати підтверджують перспективність титановмісних цеолітів як ефективної платформи для створення гетерогенних каталізаторів прямого перетворення олефінів на циклічні карбонати.

Ключові слова: стирол; циклічний карбонат; епоксид стиролу; ієрархічні цеоліти; титаносилікати

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Introduction

Carbon dioxide (CO_2) is one of the main greenhouse gases produced by human activity, and is a factor in global warming and climate change [1]. The atmospheric concentration of carbon dioxide has been steadily increasing over the past two centuries [1]. Carbon dioxide is also an abundant carbon resource that can be utilized in the chemical industry. It is an inexpensive, non-flammable, and non-toxic carbon source. Therefore, the synthesis of chemical compounds using CO_2 has attracted significant scientific interest [2–4]. However, the chemical inertness and high activation energy of reactions involving CO_2 limit the range of possible applications [3]. The use of materials capable of catalyzing reactions involving CO_2 can reduce the activation energy and enable its efficient utilization. Therefore, the development of effective catalytic systems that enhance the interaction of CO_2 with organic molecules is of great importance.

Cyclic carbonates are compounds that can be synthesized from carbon dioxide and readily available organic substrates. They are components of biodegradable polymers [5, 6], precursors for polyurethanes [7], electrolytes in lithium-ion batteries [8], solvents [9, 10], and valuable intermediates in the synthesis of functional compounds [11].

Cyclic carbonates can be obtained from epoxides [3, 12, 13], olefins in the presence of an oxidant [14–16], and diols [17, 18].

The production of cyclic carbonates directly from olefins in the presence of an oxidant and CO_2 is one of the key directions for the CO_2 chemical utilization. In contrast to the traditional approach involving the separate synthesis of epoxides, this method combines the epoxidation and CO_2 cycloaddition in a single reaction medium, reducing the number of steps and material losses [15, 16]. The process proceeds *via* a tandem mechanism, in which the epoxide is formed *in situ* and subsequently converted into a five-membered cyclic carbonate.

The oxidative carboxylation is a complex catalytic process as it involves two stages with distinct requirements: the epoxidation requires an oxidant and a metal center for the C=C bond activation, whereas the cycloaddition step involves the nucleophilic or Lewis acidic activation of the epoxide and CO_2 [16]. Therefore, effective systems are typically multicomponent, comprising metal-based catalysts and cocatalysts (e.g., halide salts and ionic liquids).

The combination of multiple catalysts in a single reactor, in particular epoxidation catalysts and catalysts for the cycloaddition of CO_2 to the resulting epoxide, is a promising approach. At the same time, it is of interest to investigate the use of various titanosilicate zeolites in the synthesis of cyclic carbonates from olefins and to compare the performance of hierarchical and non-hierarchical zeolite systems in this two-step process.

The aim of this study was to determine the effect of the reaction mixture composition, particularly the titanium content, on the adsorption, acidic, and catalytic properties of hierarchical zeolites in the oxidation of styrene with *tert*-butyl hydroperoxide under CO_2 pressure and in its absence.

Materials and methods

MFI-type zeolites were synthesized according to the procedure described in [19, 20], using the structure-directing agent $C_{16\text{-}6\text{-}6}(OH)_2$, proposed by *Ryoo et al.* in 2009 [21]. The numbers in the sample names correspond to the Si/Ti ratio in the reaction mixture used for the synthesis, while 'am' denotes the partially amorphous sample. A detailed description of the synthesis of the structure-directing agent is provided in [21]. The synthesis of this template was carried out in two stages. At the first stage, 1 mol of hexadecyl bromide was added dropwise to 3 mol of *N*,*N*,*N'*,*N'*-tetramethylhexane-1,6-diamine. The reaction was performed in the acetonitrile/1,4-dioxane (1:1) solution. The reaction mixture was maintained at 80 °C for 72 h, after which the solution was concentrated using a rotary vacuum evaporator. The resulting gel-like mixture was washed with methyl *tert*-butyl ether and dried on a rotary evaporator. The yield of $C_{16\text{-}6}$ varied from 50 to 75%. At the second stage, 0.5 mol of $C_{16\text{-}6}$ and 0.5 mol of hexyl bromide were mixed, heated in acetonitrile (150 mL), and maintained at 80 °C for 16–18 h. The solution was evaporated, and the resulting precipitate was washed with methyl *tert*-butyl ether. The synthesis of hierarchical zeolites was carried out according to the following procedure [20]: tetraethyl orthosilicate (TEOS) was added to an aqueous solution containing the structure-directing agent (SDA, **Figure 1**), and the mixture was stirred for 20 min. The reaction mixture was cooled to 6–8 °C, and the calculated amount of titanium butoxide was added. The mixture was then stirred at 60 °C for 3 h in an open vessel until the complete removal

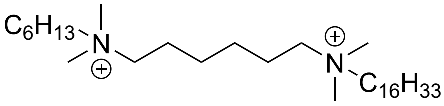

Figure 1. The SDA used for the synthesis of hierarchical MFI zeolites

of ethanol formed during the TEOS hydrolysis. The amount of water evaporated within 3 hours was added to the reaction gel. The composition of the reaction mixtures (RM) was as follows – $24SiO_2$: (0,24–0,48) TiO_2: 1,8–2,3 SDA: 1706 H_2O. The reaction mixtures (RM) were subjected to the hydrothermal treatment at 150 °C for 16–18 days in a stirred oven. After the hydrothermal treatment, the zeolite synthesized powder was filtered, washed with distilled water and ethanol, and dried at 100 °C for 1 h. The removal of the SDA was carried out by heating the zeolite samples to 550 °C at a rate of 5 °C min^{-1}, followed by keeping at this temperature for 5 h.

The results of the elemental analysis of the zeolites obtained are presented in **Table 1**.

The crystalline structure and the presence of impurity zeolite phases were determined by the X-ray diffraction using a Bruker D8 Advance diffractometer (Germany) with the Cu Kα radiation. The diffraction patterns were recorded in the 2θ range of 3–45° with a step size of 0.05°. The signal acquisition time was 3 s per step.

The adsorption properties of the materials were studied using low-temperature nitrogen adsorption-desorption at −196 °C. Based on the analysis of nitrogen adsorption isotherms, the following parameters were determined: the specific surface area ($S_{BET,}$ calculated by the Brunauer–Emmett–Teller method), the mesopore surface area (S_{MESO}), the micropore volume (V_{micro}, the t-plot method [22]), the mesopore volume (V_{meso}), and the mesopore diameter (d_{meso}, the Barrett–Joyner–Halenda method [23]).

The Si/Ti ratio was determined by the X-ray fluorescence (XRF) spectroscopy using an ElvaX Plus instrument and the Elva4 software.

The morphology of the sample was examined using a Mira 3 Tescan scanning electron microscope. Prior to measurements, the samples were coated with a 40 nm thick platinum-gold alloy layer.

The diffuse reflectance UV–Vis spectra were recorded using a Specord M40 spectrophotometer in the 200–700 nm range. The reflectance spectra were converted to absorption using the Kubelka–Munk theory [24].

The acidic properties of hierarchical zeolites were evaluated by the stepwise pyridine (Py) ad/desorption. Tablets (up to 0.01 g, surface area 0.0064 m^2) were placed in a spectral cell and evacuated at 450 °C for 1 h. The activated samples were heated to 150 °C and exposed to pyridine for 30 min, followed by the evacuation to remove physically adsorbed pyridine. IR spectra were recorded on a PerkinElmer Spectrum One spectrometer. The concentrations of acid sites were determined using extinction coefficients reported in [25].

Zeolite catalysts were evaluated in the TBHP-mediated oxidation of styrene under the excess CO_2 pressure using RVD-2-250 autoclaves (Riva Stal, Ukraine) equipped with Teflon liners. Prior to the reaction, the catalysts were activated at 150 °C for 16 h and introduced into the reaction mixture while hot. The autoclave was charged with 5 mL of CH_3CN, 0.1 mL of 1,3,5-trimethylbenzene (internal standard), 50 mg of the catalyst, 0.07 g of tetrabutylammonium iodide (TBAI, a cocatalyst for the second stage), 0.43 mL of styrene, and 0.67 mL of a 70% aqueous *tert*-butyl hydroperoxide (TBHP) solution. CO_2 was introduced to an initial pressure of 20 atm. The reaction mixture was heated to 100 °C for 1 h and then stirred at this temperature for 22 h, after which the autoclave was cooled and depressurized to atmospheric pressure. A 1 mL aliquot was withdrawn from the reaction mixture, and the catalyst was separated by the centrifugation. A 0.2 mL portion of the supernatant was diluted to 1 mL with methanol and analyzed by the gas

Table 1. The elemental composition and adsorption properties of the hierarchical zeolites studied in this work

Sample	Si/Ti ratio in		S_{BET}, $m^2 g^{-1}$	S_{meso}, $m^2 g^{-1}$	V_{Σ}, $cm^3 g^{-1}$	V_{meso}, $cm^3 g^{-1}$	V_{micro}, $cm^3 g^{-1}$
	RM[1]	Samp[2]					
TS-1	50	55	420	5	0.19	0.01	0.18
Ti-MFI_100	100	95	410	250	0.48	0.42	0.06
Ti-MFI_75	75	70	500	115	0.71	0.54	0.17
Ti-MFI_50	50	45	440	40	0.47	0.39	0.18
Ti-MFI_50_am	50	40	105	50	0.12	0.10	0.02

Note: [1]The Si/Ti ratio in the reaction mixture; [2]The Si/Ti ratio in the sample obtained was determined by the X-ray fluorescence spectroscopy chromatography–mass spectrometry (GC–MS; GC7820A, Agilent Technologies).

■ Results and discussion

Figure 2A shows the X-ray diffraction patterns of the zeolite samples obtained in this study. All samples exhibit reflections at 2θ = 7,8; 8,7°; 23,1°; 23,8°; 29,9°, characteristic of the MFI structure. Some reflections corresponding to the [0k0] planes are absent in the Ti-MFI_100–Ti-MFI_50 series, which may indicate the formation of thin nanosheet-like particles, similar to those previously reported [19–21]. The Ti-MFI_50_am sample exhibited a significantly lower degree of the crystallinity compared to the other samples. For the Ti-MFI_100–Ti-MFI_50 series, an increase in the titanium content led to an increase in the crystallinity; in particular, for Ti-MFI_50, a reflection at 2θ = 24.2° and several less intense reflections characteristic of the microporous TS-1 analog were observed. No impurity crystalline phases of other zeolites or bulk silicate phases were detected in the diffraction patterns of the samples studied. Crystalline phases corresponding to titanium oxide were also not observed.

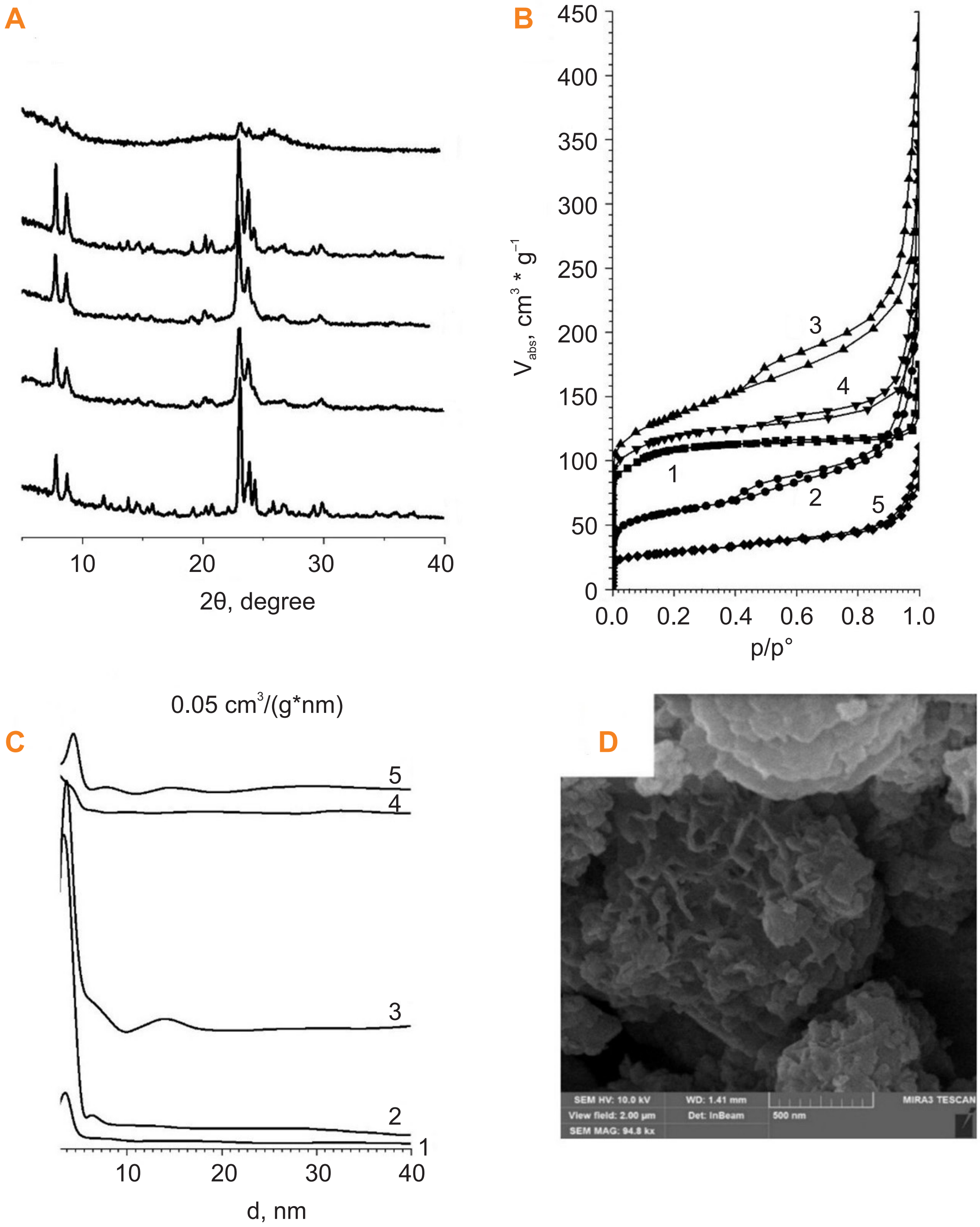

Figure 2. (A) X-ray diffraction patterns of Ti-MFI zeolites, (B) adsorption isotherms, (C) mesopore size distribution curves, and the (D) SEM image of a hierarchical MFI-type zeolite (1 – TS-1, 2 – Ti-MFI_100, 3 – Ti-MFI_75, 4 – Ti-MFI_50, 5 – Ti-MFI_50_am)

Figure 2B shows the nitrogen adsorption isotherms, and **Table 1** summarizes the corresponding adsorption characteristics of MFI-type zeolites. **Figure 2**C presents the mesopore size distribution curves. As shown, the samples are characterized by the presence of mesopores with diameters in the range of 2–10 nm, with relatively narrow size distributions.

Figure 2D shows a typical SEM image of a hierarchical MFI-type zeolite. As observed, the zeolites consist of sponge-like particles a few micrometers in size, composed of multilamellar layers. The typical number of layers is about 10–20.

Figure 3A shows the diffuse reflectance UV–Vis spectra of the series of samples studied. According to the literature, this method allows the coordination state of heteroatoms (Ti, Sn, Zr, etc.) in the zeolite framework to be evaluated [26]. The deconvolution of the spectra obtained reveals several absorption bands, including maxima at 220 and 251 nm. According to the literature, the band at 220 nm is attributed to the charge transfer from O^{2-} to tetrahedrally coordinated Ti^{4+} cations [20], whereas the band at 251 nm corresponds to the extra-framework isolated and non-isolated Ti^{4+} species in the octahedral coordination.

For all samples except Ti-MFI_50_am, no band at ~255 nm is observed, indicating that titanium is present predominantly in the tetrahedral coordination. In the case of Ti-MFI_50_am, the significant amount of the extra-framework titanium may be attributed to the low crystallinity of the sample and, consequently, the high content of the amorphous phase.

Figure 3B shows the Fourier-transform IR spectra of the adsorbed pyridine for Ti-MFI zeolites; the concentrations of different types of acid sites were calculated according to the method described in [25]. The spectra of the adsorbed pyridine for all samples exhibit bands at 1446 cm^{-1}, which can be attributed to C–N stretching vibrations of the pyridine coordinated to Lewis acid sites of various types. According to the literature, the band at 1446 cm^{-1} can be attributed both to the Lewis acid sites formed by Ti^{4+} cations incorporated into the zeolite framework [27] and to the pyridine adsorbed on weakly acidic hydroxyl groups that are inactive in the process studied. As shown in **Table 2**, the maximum concentration of Lewis acid sites associated with titanium was observed for Ti-MFI-75. Further increases in the titanium content in the reaction mixture and in the samples do not result in a significant increase in their concentration. The Ti-MFI_50_am sample is characterized by a relatively low concentration of Lewis acid sites, which is attributed to the high content of extra-framework titanium species.

As shown in **Table 1**, Ti-MFI_100 possesses a lower specific surface area and a lower concentration of Lewis acid sites, which may reduce the

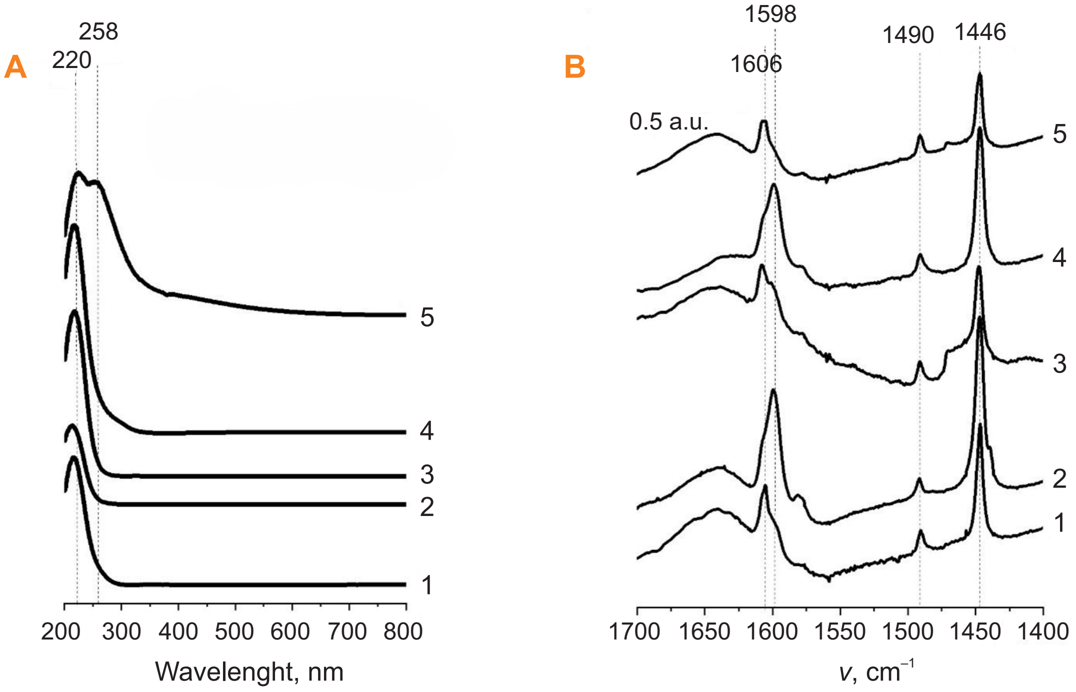

Figure 3. (A) Diffuse reflectance UV–Vis spectra; (B) IR spectra of the adsorbed pyridine for Ti-MFI zeolites. (1 – TS-1, 2 – Ti-MFI_100, 3 – Ti-MFI_75, 4 – Ti-MFI_50, 5 – Ti-MFI_50_am)

Table 2. Acidic properties of hierarchical BEA-type zeolites and their catalytic activity in the conversion of styrene to cyclic carbonate

Samples	Center concentration, μmole g^{-1}		Styrene conversion, %	Cyclic carbonate selectivity, %	Cyclic carbonate yields, %
	Ti^{4+} Lewis	OH groups			
TS-1	91	22	85.7	97.4	83.2
Ti-MFI_100	66	180	72.1	100	72.1
Ti-MFI_75	143	25	90.7	100	90.7
Ti-MFI_50	61	20	98	67.5	66.1
Ti-MFI_50_am	35	139	100	10.4	10.4

accessibility and the number of catalytically active titanium centers. Hierarchical zeolites outperform conventional TS-1 in the catalytic performance, which may be attributed to their larger surface area. The low catalytic activity of the Ti-MFI_50_am sample can also be associated with the reduced surface area of the material and the lower concentration of Lewis acid sites.

Conclusions

Hierarchical Ti-MFI zeolites have been shown to be effective catalysts for the one-pot synthesis of cyclic carbonates from styrene under CO_2 pressure. Their catalytic performance is governed by the balance between framework tetrahedral Ti^{4+} species and accessible Lewis acid sites, as well as the presence of the hierarchical porosity. The Ti-MFI_75 sample has exhibited optimal properties, providing the highest conversion and near-quantitative selectivity to cyclic carbonate. The results demonstrate that combining redox and acid functionalities within a single zeolite enables the efficient tandem catalysis and represents a promising strategy for the sustainable CO_2 utilization.

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