

High-efficient hierarchical [B]-ZSM-5 catalyst by simultaneously using of CTAB surfactant and boron promoter for methanol to olefins reaction

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Abstract

In this work, a one-pot hydrothermal route was applied to prepare the hierarchical high-silica ZSM-5 catalyst (Si/Al=200), including boron promoter in the structure and CTAB surfactant. XRD, FE-SEM, BET, NH₃-TPD, and FT-IR techniques were applied to evaluate the physical and chemical properties. The effect of different amounts of secondary template (CTAB) and different operating conditions was studied on the ZSM-5 catalyst preparation and performance in methanol to olefins reaction. The results showed the high surface area (391.8 m²g⁻¹), mesoporous structure, high crystallinity, and well-adjusted acidity for the modified catalyst. The prepared catalyst with CTAB/TPABr molar ratio of 1 led to the high surface area, short diffusion path length by mesopore structure, and optimum acidity. It was found that the optimum temperature and weight hourly space velocity were 480 °C and 7.2 h⁻¹, respectively.

Keywords Methanol to olefins · Catalyst · Secondary template · Promoter · ZSM-5

Introduction

The high oil price and environmental issues have ignited interest in developing methanol to olefins (MTO) process, which can be a dominant process for the olefins industry [1, 2]. The development of a catalyst with high performance is a crucial point for enhancing the efficiency and selectivity of the MTO reaction. The

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performance of a variety of catalysts like ZSM-5 [3, 4], SAPO-34 [5], MOR [6], and HZSM-22 [7] has been evaluated. Among them, ZSM-5 with microsize pores showed excellent performance due to its high thermal stability, resistance against coke formation, and surface area [8]. In addition, acidity and specific shape selectivity are the other important parameters, which can be controlled easily for ZSM-5 [9]. Despite ZSM-5 promising features, its microporous structure accelerates catalyst deactivation due to blocking microspore channels by large hydrocarbon molecules. One strategy to overcome this drawback of ZSM-5 is modification of the microporous structure of ZSM-5 by adding mesopores into the porous structure of the catalyst [10]. The mesopore ZSM-5 can be synthesized by either chemical leaching or templating routes. Unfortunately, the chemical leaching method suffers from reducing the high amount of acid sites [11]. On the other hand, the amount of acid sites can be controlled by synthesis conditions (e.g., template type) in a templating synthesis route (TSR) [12]. In this regard, a secondary template such as CTAB [13] has been developed to prepare mesoporous catalysts. It is added as a structuredirecting agent (SDA) and its hydrophobic tail is responsible for mesopores [14]. The formed mesopores increase the diffusion rate of reactants, surface area, and the number of accessible acidic sites compared with conventional ZSM-5 [15, 16]. The acidity of catalyst is the other parameter that plays a vital role in MTO reaction. The high density and strong acid sites accelerate coke formation and so catalyst deactivation [17]. In addition, the selectivity of products can be controlled by tuning acidity of catalyst. Therefore, promoter incorporation and metal doping routs have been used to change the selectivity of products by controlling acidity of the catalyst [18]. Incorporation of boron promoter in the structure of the ZSM-5 catalyst can adjust the acidity, leading to the better performance of the ZSM-5 catalyst in the MTO reaction. It is reported that strong acid sites are responsible for the side reactions and coke formation [14, 19, 20]. It is accepted that the high adsorption of hydrocarbons on the MTO catalyst surface results in the high catalyst activity and more cracking reactions. Sacchetto et al. [21] studied the adsorption of gaseous hydrocarbons on high-silica ZSM-5. They found that high Si/Al ratio favored hydrocarbon adsorption on ZSM-5 surface. Furthermore, it is reported that chemical and thermal stability, as well as the yield of synthesis of the high-silica ZSM-5, is more than the low-silica ZSM-5 [22, 23], favoring the economy of the catalyst preparation for the methanol conversion. On the other hand, promoter is necessary to achieve appropriate acidity properties as well as the high catalytic performance. It is accepted that acid sites are responsible for the main and side reactions in the MTO process. Therefore, the well-adjusted acidity and structural properties of the catalyst play a crucial role for developing of the improved catalyst for the MTO reaction. The minimizing of the synthesize steps, including post-treatment steps such as addition of promoter and modification of catalyst structure, leads to the one-pot synthesize of the optimum catalyst as a cost-effective and fast route. To the best of our knowledge, among all researches in this field, there is no report about the modifying chemistry and structure of high-silica ZSM-5 by simultaneously using CTAB and B promoter. Therefore, we focused on the effect of CTAB as a SDA agent and B promoter on the physic-chemical properties as well as the performance of the catalyst in the MTO reaction.

Materials and method

Materials

Silicic acid (SiO₂.xH₂O, 99 wt.%), sodium aluminate (NaAlO₂, Al₂O₃ 55 wt.%). sulfuric acid (H₂SO₄, 98 wt.%), ammonium nitrate (NH₄NO₃, 99 wt.%), sodium hydroxide (NaOH, 99.6 wt.%), boric acid (H₃BO₃, 99.8 wt.%), tetrapropyl ammonium bromide (TPABr), and cetyl-trimethylammonium bromide (CTAB) were applied for the catalyst synthesis. All the chemicals were used as received from Merck Company (Germany).

Catalyst synthesis

Firstly, NaAlO₂, H₃BO₃, and TPABr were dissolved in NaOH solution for 30 min (solution A). Solution B was prepared by adding silicic acid and CTAB to NaOH solution. The solution A was gradually added to the solution B under stirring. After adjusting pH at 10.5 by concentrated sulfuric acid, the solution was stirred for 2 h. Table 1 shows the molar composition of the final solutions. The crystallization was carried out at 180 °C for 48 h in an autoclave. The sample was dried at 110 °C for 12 h, followed by calcination at 540 °C for 24 h with 3°Cmin⁻¹ heating rate. In the final step, the catalyst was ion-exchanged with 1 M NH₄NO₃ solution for 10 h at 90 °C, dried at 110 °C for 12 h, and calcined at 550 °C for 12 h. The catalysts were denoted by CTZM-y where y was the CTAB/TPABr molar ratio.

The details of the characterization methods were provided in the supplementary information.

Experiments

The MTO reaction was carried out in a fixed bed reactor with a 450 mm length and 11 mm inner diameter. The reactor was connected to a gas chromatograph (GC), which contained a flame ionization detector (FID) and HP-plot Q column with 30 m length and 0.53 mm inner diameter. The applied catalyst had 16–25 mesh particle size. The reaction was carried out at different temperatures (450, 480, and 500 °C) at atmospheric pressure. The feed was a 50/50 wt.% methanol/water solution, which was charged into the reactor with determined weight hourly space velocity (WHSV).

Molar composition of synthesize solutions	Catalysts	Compositions						
		Al ₂ O ₃	SiO_2	TPABr	Na ₂ O	B_2O_3	CTAB	H ₂ O
	CTZM-0	0.05	20	1	1.511	0	0	209
	CTZM-0.75	0.05	20	1	1.511	0.05	0.75	209
	CTZM-1	0.05	20	1	1.511	0.05	1	209
	CTZM-1.5	0.05	20	1	1.511	0.05	1.5	209

Table 1 the to s The methanol conversion and product selectivity were defined by the following equations:

$$x_{\text{MeOH}} = \frac{N_{\text{MeOH}}^i - N_{\text{MeOH}}^o}{N_{\text{MeOH}}^i} * 100 \tag{1}$$

$$S_{C_x H_y} = \frac{x * N_{C_x H_y}^o}{N_{MeOH}^i - N_{MeOH}^o} * 100$$
 (2)

where N is the mole number of components. The superscript i and o stand for components at the inlet and outlet of the reactor, respectively.

Results and discussion

Physic-chemical properties

The X-ray diffraction (XRD) pattern for the synthesized CTZM-y samples is shown in Fig. 1. The results confirm an MFI-structure (JCPDS NO. 00-042-0023) [24] for the prepared samples. The relative crystallinity of the modified catalysts is determined by the height of the peak at $2\theta = 23^{\circ}$ based on the CTZM-0 catalyst, and the crystallinity change is determined by Eq. (3):

$$\Delta C\% = \left(\frac{I}{I_0} * 100\right) - 100\tag{3}$$

where I and I_0 are the height of the peak at $2\theta = 23^{\circ}$ for the modified and parent catalysts, respectively [25]. As shown in Table 2, the CTZM-0.75 and CTZM-1 catalysts show positive crystallinity change while the CTZM-1.5 catalyst shows a negative crystallinity change. The crystallinity is improved in the limited range of the added CTAB because structure-directing characteristic of CTAB molecules assists the growth of crystal. On the other hand, if the amount of the added CTAB exceeds from the certain value, the overloaded CTAB forms some amorphous phases and



Table 2 Crystallinity and crystallite size change for the modified catalysts	Catalysts	Crystallinity change (%)	Crystallite size change (%)
	CTZM-0.75	+8.7	+ 30.0
	CTZM-1	+3.3	+22.1
	CTZM-1.5	-6.5	- 10.9

decreases the crystallinity [26]. The CTZM-1.5 has the highest amount of CTAB and the lowest crystallinity. The crystallite size of the catalysts is calculated using Debby–Scherrer equation. Table 2 presents the estimated crystallite size change for the catalysts compared with the CTZM-0 catalyst (33.0 nm). The crystallite size of both CTZM-0.75 and CTZM-1 catalysts are smaller than parent catalyst, showing positive crystallite size change (+30.0 and+22.1%, respectively). Therefore, the low CTAB/TPABr ratio (<1.5) favors the high crystallinity and the small crystallite size, which can be resulted in better catalytic performance. Since CTZM-1.5 catalyst has the negative crystallinity and crystallite size change, this catalyst is ruled out for the rest of the characterizations.

The BET surface area, pore volume, and pore diameter of the catalysts (Table 3) are determined using N₂ absorption–desorption isotherm (Fig. 2). The BET surface area for the CTZM-0.75 and CTZM-1 catalysts (377.5 and 391.8 m^2g^{-1} , receptivity) is higher than the CTZM-0 catalyst (321.1 m^2g^{-1}) due to the formation of mesoporous structure by CTAB incorporation. It is reported that hydrophobic interaction between long-chain alkyl groups of CTAB formed meso-scale micellar structure [27]. As can be seen in Fig. 2, the samples have type-I and type-IV isotherms, which are assigned to the micro- and mesopores,

Catalysts	$S_{BET} (m^2 g^{-1})$	$S_{BET} (m^2 g^{-1})$	V _{micro} (cm ³ g ⁻¹)	$V_{meso} (cm^3g^{-1})$	Mean pore diameter(nm)
CTZM-0	321.10	0.15	0.13	0.02	1.00
CTZM-0.75	377.51	0.21	0.11	0.10	2.08
CTZM-1	391.85	0.25	0.14	0.11	2.12

 Table 3
 Textural data of the catalysts





respectively. The nitrogen sorption and desorption in the initial pressures belong to the microporous structure, and the loop in the $P/P_0 > 0.4$ can be attributed to the presence of mesoporosity [28]. A low slope for the CTZM-1 catalyst at P/ $P_0 > 0.4$ can explain the excellent appropriate coverage [29]. The results show that the mesopore volume is increased significantly after adding CTAB, while the micropore volume remained constant. Interestingly, the structure directing effect of CTAB increases the pore size of the modified catalysts (up to 112%), favoring the low mass transfer resistance and high catalytic activity. Since the surface area, pore volume, and pore size of the CTZM-1 catalyst is higher than the CTZM-0.75 catalyst, the CTZM-1 catalyst is selected for the rest of characterizations and experiments. Figure 3 represents the FE-SEM images of the CTZM-0 and CTZM-1 catalysts, including the semi-spherical particles. The FT-IR spectra of the catalysts are presented in Fig. 4. The band at 450 cm⁻¹ is associated with TO_4 (T=Al, Si) units as internal tetrahedrons. The band at 550 cm^{-1} is attributed to the formation of double five rings in ZSM-5. The bands near 800 and 1100 cm⁻¹ are assigned to external symmetric bonds and internal asymmetric stretching of T–O (T=Al, Si) linkages. The bands in the range of



Fig. 3 FE-SEM images of the catalysts a CTZM-1 and b CTZM-0



Fig. 4 FT-IR spectra of the catalysts

 $3500-3800 \text{ cm}^{-1}$ are related to the hydroxyl groups [30, 31]. The FT-IR crystallinity change is calculated using the following equation:

$$\Delta C\% = \left(\frac{I}{I_0} * 100\right) - 100\tag{4}$$

where *I* and I_0 are the intensity of the 550 and 450 cm⁻¹ bands, respectively. The IR crystallinity of the CTZM-1 catalyst has slightly a positive change (+1.0%) compared with the CTZM-0 catalyst, supporting the XRD results. According to the literature [32], the IR crystallinity is related to the crystallinity of the aluminosilicate phase in the zeolite while the XRD crystallinity is attributed to the structure. The band at ~1370 cm⁻¹ for the CTZM-1 catalyst confirms that the boron is coordinated in a trigonal form [27, 33].

NH₃-TPD measurement compares the strength and amount of the acid sites (Fig. 5). The first and second peaks are attributed to the ammonia desorption from the weak and strong acid sites, respectively. The peak temperature shows the strength, and the peak area is associated with density of the acid sites. Table 4 presents the calculated density and peak temperature for the catalysts. The density of both strong and weak acid sites for the CTZM-1 catalyst is lower than the CTZM-0 catalyst due to the presence of boron in the structure, which is inconsistent with the literature [19, 34]. Briefly, the observed diminishing for the acid sites results from substitution of Al atoms in Si–OH–Al bonds by B, and subsequently formation of Si–OH–B bonds. Stave et al. [35] found that the large bond length between B and OH in a trigonal structure decreased the interaction between boron and hydroxyl group as well as the acidity of the catalyst.





 Table 4
 Acidity of the catalysts

Catalyst	Concentration of acid sites (NH3 mmol. g-1)						
	Strong		Strong/weak	Total weak			
CTZM-0	0.63	0.32	0.51	0.95			
CTZM-1	0.20	0.08	0.40	0.28			

Catalytic performance

Effect of CTAB/TPABr ratio

The performance of the parent and CTZM-x catalysts was evaluated in the fixed bed reactor in specific operating conditions: T = 480 °C, WHSV = 7.2 h⁻¹, and P = 1 atm (Fig. 6). The MTO reaction follows two steps: (1) dimethyl ether (DME) formation by dehydration of methanol (2) DME conversion to a wide range of hydrocarbons [36]. Since the molecular size of methanol is smaller than the pore size of the catalysts [37], the appropriate physic-chemical properties of the catalysts lead to the full methanol conversion thought the whole time on stream. The CTZM-1 catalyst has higher $C_2^{=}$ selectivity (12.7%) than the CTZM-0 catalyst (8.5%). The moderate acidity of the CTZM-1 catalyst controls hydrogen transfer reactions, which is a critical factor for the formation of the by-products like aromatics. The CTZM-1 catalyst shows better performance for the propylene selectivity (48.56%) due to the high BET surface area (391.85 m^2g^{-1}), high mesoporous volume (0.11 cm^3g^{-1}), and the small crystallite size, reducing diffusion resistance as well as the secondary reactions in the catalyst. Svelle et al. [35] explained propylene and ethylene production with two different cycles: (1) propylene is produced by methylation of alkenes and cracking cycle; (2) ethylene is formed through (poly)methylbenzene methylation and alkylation cycle. Zhu et al. [38] studied the application of H-B-ZSM-5 borosilicate (Si/B=41) in the MTO reaction. They found that B incorporation in the ZSM-5 structure improved the propylene selectivity owing to the methylation of ethylene with methanol. This phenomenon can be explained by the weak acid sites of the modified catalyst, leading to the low hydrogen-transfer reactions. Wang et al. [39]



Fig. 6 The variation of **a** ethylene selectivity, **b** propylene selectivity, and **c** light olefins selectivity over the catalyst with time on stream at T=480 °C and WHSV=7.2 h⁻¹

reported that bulkier methyl benzenes (tetraMB and pentaMB) and light methyl benzenes (p/m-diMB) intermediate compounds improved the propylene and ethylene formation, respectively. The low density of strong acid sites and the less adsorption of light MBs (owing to mesoporous structure) increase the propylene formation because the light MBs can diffuse out of zeolite channel quickly and bulkier MBs remain longer in the catalyst. It is worth noting that the change in the propylene selectivity is more than the ethylene selectivity over the catalysts. Since the molecular size of propylene is larger than ethylene, so diffusion limitation about this molecule significantly decreases with an increase at the pore size of the catalyst [40]. In this regard, the P/E (propylene to ethylene) molar ratio for the CTZM-1 catalyst (6.3) is higher than the CTZM-0 catalyst (4.2), which is a crucial and economical factor for the MTO process. Furthermore, the CTZM-1 catalyst provides the highest light olefins $(C_2^{-}-C_4^{-})$ selectivity (71%) that represents 18% improvement in comparison with the parent catalyst. Therefore, the results show that CTAB/TPABr ratio of 1 leads to the best textural and acidity properties as well as the catalytic activity in the MTO reaction. The CTZM-1 catalyst is selected as optimum catalyst for further investigation.

Effect of reaction temperature

The performance of the CTZM-1 catalyst at different temperatures (450, 480, and 500 °C) is shown in Fig. 7. The highest propylene selectivity is obtained (48.56%) at 480 °C. It is accepted that the high temperature favors the high propylene selectivity [41]. Based on thermodynamic, the increasing temperature at the constant range of the olefins partial pressure leads to more production of the light olefins [41]. In this regard, the ethylene is the main product at the low temperature (450 °C) while propylene is the dominant at the high temperatures. When the temperature is 450 °C, the rate of the reaction is low. Indeed, the acid sites do not have enough energy for producing olefins. On the other side, when the temperature is elevated from 480 to 500 °C, the cracking reactions are accelerated and propylene is converted to smaller molecules. Specifically, (poly)methylbenzene methylation and alkylation cycle are more favorable at 500 °C. It is worth noting that more increasing temperature (500 °C) enhances the coke formation and the side reactions, resulting in conversion of the high olefins to by-products. The coke formation blocks the catalyst pores and hinders the fast transfer of the light olefins out of the pores. Cyclization, aromatization, methylation, and hydrogen transfer reactions convert the light olefins to higher hydrocarbons, which are the main source of coke [42-44]. Therefore, the highest propylene and light olefins selectivity are obtained at 480 °C (48.56 and 71%, respectively).

Effect of WHSV

The effect of WHSV on the product distribution over the CTZM-1 catalyst is shown in Fig. 8. It is clear that the high WHSV favors more light olefins production. The highest propylene (48.56%) and light olefins selectivity (71%) are obtained at WHSV of 7.2 h⁻¹. It is worth noting that increasing WHSV could have dual effect on the MTO reaction. The high WHSV results in more methanol feeding and the low conversion. On the other hand, it leads to short residence time in the catalyst bed, reducing the possibility of the side reactions to convert the light olefins. Therefore, the acidity and structural properties of the catalyst significantly influence WHSV effects and the resulted product distribution. The preferred properties, including the high specific surface area and uniform pore size distribution, can compensate negative effects of the reduced feed residence time and increase production rate without methanol conversion drop. The high WHSV over the CTZM-1 catalyst bed results in more products without methanol conversion reduction, which can be attributed to











surface area, the high pore volume, and mesopore size that lead to the low diffusion resistance and hinder coke formation. The ethylene selectivity decreases by increasing WHSV due to the cracking of oligomers [45, 46]. Zhu et al. [38] reported the methylation of ethylene with methanol to production of propylene over ZSM-5 catalyst. Their results showed that the weak acidity hindered the formation of ethylene oligomers and the methylation of ethylene with methanol resulted in the high propylene selectivity. The hydrogen transfer plays a key role in the product distribution of the MTO reaction. At the low WHSV, the hydrogen transfer reactions are accelerated owing to the long contact time, leading to more conversion of the light olefins.

Table 5Comparison of the propylene selectivity in the literature	No	Catalyst	Si/Al	Propylene selec- tivity (%)	Reference
	1	ZSM-5	200	37.35	[12]
	2	ZSM-5/SiC	44	44.87	[47]
	3	ZSM-5	78	42.03	[48]
	4	ZSM-5	175	41.96	[49]
	5	ZSM-5	30	44.04	[50]
	6	ZSM-5	200	43.56	[51]
	7	CTZM-1	200	48.56	This study

Therefore, there is no time for ethylene and propylene conversion to paraffins and aromatics through the secondary reaction at the high WHSV [45, 46].

Table 5 compares the propylene selectivity of the CTZM-1 catalyst and the reported results in the literature. As can be seen, the developed catalyst has the high performance, including the fast and one-pot synthesis process. The simultaneous incorporation of CTAB secondary template and B promoter improves the textural and acidity properties of the catalyst. The low acidity suppresses the side reactions and coke formation. The low acidity hinders hydrogen transfer, cyclization, alkylation, and poly-condensation reactions. These reactions convert the light olefins to paraffins, aromatics, naphthalenes, and higher olefins. Furthermore, CTAB template results in the mesopore structure, which reduces the mass transfer resistance and coke formation possibility. In fact, hierarchical ZSM-5 catalyst shifts coke formation from inside of the micropores to the external surface and/or mesopores, increasing the acid sites accessibility as well as the catalyst life time.

Conclusion

In summary, the hierarchical high-silica ZSM-5 catalysts were synthesized hydrothermally in one-pot, including different amounts of CTAB surfactant as the second template and boron as a promoter. It was obtained that the CTAB/TPABr molar ratio of 1 was the best, resulting in the optimum textural and acidity properties as well as the catalytic activity in the MTO reaction. The results showed the high surface area, mesoporous structure, high crystallinity, and well-adjusted acidity for the modified catalyst. The optimum operating conditions were 480 °C and WHSV of 7.2 h⁻¹, which led to the highest propylene selectivity (48.56%), the highest light olefins selectivity (71%), and the highest P/E ratio (6.3) in the MTO reaction. The results confirmed the significant role of CTAB and boron introduction in the MTO catalyst development.

Supplementary information The online version contains supplementary material available at https://doi.org/10.1007/s11164-021-04465-z.

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