Dimethyl Ether Conversion to Gasoline Hydrocarbons over Nanosized Zeolite Catalysts: Effect of Modifier Nature

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Abstract—The effect of modifier nature on the catalytic properties of nanosized zeolite catalysts used for the synthesis of liquid hydrocarbons from dimethyl ether is studied. It is shown that selectivity of the catalysts for liquid hydrocarbons depends on both the modifier nature and the initial feedstock composition. The best activity and selectivity for liquid hydrocarbons are exhibited by the samples modified with zinc and magnesium. The content of aromatic hydrocarbons in the products decreases appreciably when the catalysts are additionally modified with noble metals and the reaction is carried out in the medium of hydrogen.

Keywords: liquid hydrocarbons, selectivity, dimethyl ether, nanosized zeolite ZSM-5 **DOI:** 10.1134/S0965544119120119

At the beginning of the 21st century, views about the future of world energetics and the role of various sources of the hydrocarbon feedstock changed considerably. The discovery of zeolite catalysts based on ZSM-5 [1], which efficiently converted light oxygenates, in particular methanol, to hydrocarbons C_{5+} , invoked development of a new method for producing liquid hydrocarbons from alternative carbonaceous raw materials, for example, natural gas [2]. The most large-scale methane consumer is the process of syngas production. This process is in most common use in the chemical industry, for example, for the production of methanol, which in turn can be converted to valuable chemical products, such as liquid hydrocarbons. The processes of synthesizing liquid hydrocarbons from methanol have been developed over the past three decades by a number of companies and are currently being actively implemented in the industry.

At present, the study of processes for producing liquid hydrocarbons from dimethyl ether (DME) is in progress. Compared with methanol methods the pathway of methane conversion via dimethyl ether to these products offers a number of advantages. For example, a more favorable thermodynamics makes it possible to synthesize dimethyl ether at a lower pressure than the methanol synthesis and to reach a higher syngas conversion per pass. Energy and capital expenditure are reduced, the degree of beneficial use of natural gas is increased, and the production price is decreased. Other advantages of this method are reduction in heat stress at the stage of hydrocarbon synthesis from DME (because of removal of methanol dehydration exothermic reaction from this stage) and higher activity and selectivity of DME in the synthesis of hydrocarbons compared with methanol [3, 4]. The most efficient catalysts for this process are catalysts based on zeolite ZSM-5 [5] modified with such metals as Zn [6], Ga [7], Ni [8], and La [9], which favors ethylene oligomerization to hydrocarbons C_{5+} and ensures the occurrence of dehydrogenation reactions of paraffins to olefins and naphthenes to aromatic hydrocarbons.

Zeolite ZSM-5 is a microporous crystalline aluminosilicate known for its catalytic activity and selectivity in the processes of MeOH and DME conversion to gasoline hydrocarbons owing to the developed surface and high thermal stability and chemical resistance [10]. The activity and selectivity of zeolite ZSM-5 depend on its acidity (the strength and ratio of Brønsted and Lewis acid sites), which can be varied by either introducing active elements [6–9] or postsynthesis treatment [11, 12]. However, in microporous structures, such as zeolite ZSM-5, the internal diffusion of bulky molecules is hindered; only active sites close to the entrance of zeolite pores on their external surfaces are accessible to them. In recent years, the interest of many researchers has been focused on the use of nanosized zeolites with the developed surface in various processes; owing to a small crystal size mass transfer and heat transfer processes are improved and the activity and lifetime of the catalyst grow [13]. One of the most available methods for synthesizing nanosized zeolites ZSM-5 is the ultrasonic treatment of microporous zeolite ZSM-5 [14].

Despite a wide variety of papers addressing the use of catalysts based on nanosized zeolite ZSM-5 in methanol conversion to liquid hydrocarbons [15–20],

Catalyst	[Me], wt %	Crystallite size (SEM data), nm	BET total specific surface area, S_{BET} , m ² /g	Total pore volume at $P/P_0 = 0.99$, V_{total} , cm ³ /g	Micropore volume, t-plot, $V_{\rm micro}$, cm ³ /g	Mesopore volume $V_{\text{meso}} = V_{\text{total}} - V_{\text{micro}},$ cm^3/g
MFI	_	600	451.9	0.249	0.120	0.129
MgMFI	1.0	600	378	0.241	0.115	0.126
ZnMFI	2.0	600	426.3	0.236	0.117	0.119
LaMFI	1.0	600	425.0	0.239	0.114	0.125
ZrMFI	1.0	700	424.7	0.238	0.116	0.112
CuMFI	2.0	800	390.7	0.220	0.108	0.112

 Table 1. Physicochemical properties of the synthesized nanosized catalysts

almost no data are available on the study of DME conversion over analogous catalysts.

This study concerns the effect of active component nature on the catalytic behavior of catalysts based on nanosized zeolite ZSM-5 in the process of DME conversion to gasoline hydrocarbons.

EXPERIMENTAL

Zeolite CBV (MFI) with a molar ratio of $SiO_2/Al_2O_3 = 80$ (Zeolyst Int.) was used in this study.

Nanosized zeolites were synthesized by the sonication-driven dispersion of the original zeolite in distilled water at a temperature of 25°C for 1 h in an Elmasonic P30H ultrasonic bath (an ultrasound frequency of 80 kHz and a power of 130 W).

Catalysts containing magnesium, zirconium, or copper were prepared by the incipient wetness impregnation of the nanosized zeolite with the aqueous solutions of respective nitrate salts, dried for a day in an oven, and calcined in air at a temperature of 500° C for 6 h. The nanosized zeolite was modified with zinc by treating the zeolite with a zinc nitrate aqueous solution at 92–95°C under stirring for 2 h. Afterwards the solution was evaporated and the sample was dried at 100°C, and calcined at 500°C for 6 h. Modification of the zeolite with lanthanum was carried out in a similar manner using a lanthanum nitrate solution. The data on the modifier content are listed in Table 1.

The introduction of rhodium in an amount of 0.05 wt % into the catalyst composition was conducted as follows: the preformed suspension composed of chitosan, which was preliminarily dissolved in water, and a RhCl₃ aqueous solution was added to the modified zeolite, and the resulting mixture was held for a day; after that the sample was dried and calcined at 500°C for 4 h. The suspension composed of chitosan dissolved in water and RhCl₃ aqueous solution was solution was solution was solution on the zeolite [21].

The palladium sample (palladium content, 0.5 wt %) was prepared by impregnating the nanosized zeolite MFI, which was modified with zinc in an amount of 2 wt %, with the aqueous solution of PdCl₂.

Upon solution evaporation the sample was dried at 100° C and calcined at 500° C for 6 h.

Powdered catalyst samples were preliminarily pressed and calcined at 500°C. Before loading (3 g) a fraction with a grain size of 2–3 mm was prepared. After loading in a reactor the catalyst used for the synthesis of hydrocarbons was subjected to reduction for 2 h in a hydrogen stream at a pressure of 0.1 MPa and a temperature of 380°C; then, the temperature was reduced to a running temperature of 340°C and DME was fed at a weight hourly space velocity of 2.6 h⁻¹.

The DME conversion was run on a micropilot flowcirculation unit at a temperature of 340° C and a pressure of 10 MPa. The catalytic properties of the zeolite catalysts were tested in the medium of hydrogen (100 vol %) or syngas (30 vol % CO and 70 vol % H₂).

The experimental technique and the analysis of reaction products were described in detail in [19]. According to the analysis of the feed gas containing CO, CH₄, CO₂, DME, H₂O, and N₂ and the end gas as well as the reaction water for the DME content the conversion of DME X_{DME} was calculated as

$$X_{DME} = (V_{inlet} \times DME_{inlet} - V_{outlet};$$

$$\times DME_{outlet};)/V_{inlet} \times DME_{RX},$$

where V_{inlet} and V_{outlet} are the amounts of gas at the inlet and outlet of the unit, L/h; and DME_{inset} and DME_{outlet} are the DME concentrations at the inlet and outlet of the unit, vol %. Gasoline selectivity $S_{C_{5+}}$ was calculated according to the formula:

$$S_{\rm C5+} = \rm{DME}_{\rm{benzene}} / (\rm{DME}_{\rm{benzene}} + \rm{DME}_{\rm{gas C1-C4}}) \times 100\%,$$

where $DME_{gasoline}$ is the amount of gasoline in terms of converted DME and $DME_{gas C1-C4}$ is the amount of gas C_1-C_4 in terms of converted DME.

The total amount of hydrocarbons was calculated from the results of analysis of gaseous and liquid hydrocarbons for the whole testing time.

The content of metals in zeolite samples was determined by X-ray fluorescence analysis (XRF) on an ARL Perform'x sequential XRF spectrometer (Thermo Fisher Scientific, Atkinson, NH, United

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States) equipped with a rhodium tube (3.5 kW). The metal content in the samples was 1-2 wt %.

Scanning electron microscopic (SEM) images of the samples were taken on a Hitachi TM3030 scanning electron microscope. Before imaging the samples were decorated with gold by vacuum spraying.

Specific surface area was determined by low-temperature nitrogen adsorption on a Micromeritics ASAP-2000 porosimeter. Specific surface area was calculated using the Brunauer–Emmett– Teller (BET) method at a relative partial pressure of $P/P_0 = 0.2$. All samples were preliminarily evacuated to 4×10^{-1} Pa at a temperature of 350°C for 6 h. The adsorption of N₂ was conducted at a temperature of 77 K.

The acidic properties of the zeolites were tested by the temperature-programmed desorption of ammonia (TPD-NH₃) on an USGA-101 universal sorption gas analyzer (Unisit). A weighed portion (100 mg), which was preliminarily calcined in air, was placed in a quartz reactor, heated in a helium stream at temperature of 500°C for 1 h, cooled to a temperature of 60°C, and saturated with ammonia in an ammonia–nitrogen stream for 15 min. To remove weakly bound ammonia the sample was held at 70°C in a dry helium stream for 1 h. The sample was then rapidly cooled to room temperature in a helium stream (30 mL/min), and the TPD-NH₃ curves were registered in the mode of linear temperature rise to 800°C at a rate of 8°C/min using a thermal conductivity detector.

RESULTS AND DISCUSSION

The catalytic properties of the modified zeolite catalysts are influenced by many factors. The modifier nature, its concentration, introduction technique, and catalyst pretreatment conditions exert a strong effect on the properties of zeolite catalysts [15, 19].

Nitrogen adsorption isotherms obtained for MeMFI catalysts (Fig. 1) are typical type I isotherms with micropores [22]. A well-defined hysteresis observed at a relative pressure of $P/P_0 = 0.4-0.9$ suggests the presence of mesopores connected to the external surface through micropores [23]. Modification of the zeolites with metals leads to a slight reduction in the specific surface areas of the zeolite and the volumes of micropores and mesopores (Table 1). Reduction in the micropore volume in the catalysts compared with the original zeolite indicates a partial arrangement of the modifier in zeolite channels. Among the modified samples the highest specific surface area (426.3 m^2/g) is exhibited by ZnMFI; the lowest specific surface area, by MgMFI and CuMFI. Sample MgMFI is characterized by a considerable mesopore volume (0.126 cm^3/g), while the sample promoted by copper has the lowest micropore volume $(0.112 \text{ cm}^3/\text{g}).$



Fig. 1. Isotherms of nitrogen adsorption on catalysts: (1) nanosized MFI, (2) ZnMFI, (3) LaMFI, (4) CuMFI, (5) ZrMFI, and (6) MgMFI.

Figure 2 shows the TPD-NH₃ profiles of the original MFI and modified samples. All tested samples have two desorption peaks. The first peak at 150– 300°C, which is classified as low-temperature related to the desorption of a weakly bound NH₃, corresponds to the content of weak and medium-strength acid sites. In a similar manner the high-temperature peak at 320-550°C corresponds to the desorption of strongly bound NH₃ and defines the content of strong acid sites.

As follows from Table 2, the total acidity decreases in the sequence MFI > CuMFI > ZrMFI > LaMFI > MgMFI > ZnCMFI (from 631 to 565 μ mol/g). Note that modification of the nanosized zeolite with active metals causes a reduction in the proportion of strong acid sites (ratio II/I decreases from 1.1 for MFI to 0.46 for CuMFI).

The reaction was run out in the medium of syngas (30 vol % CO and 70 vol % H₂) and hydrogen (100 vol %). Hydrogen was selected since the two-step technology developed for the production of hydrocarbons C₅₊ from CO and H₂ through DME [3, 4] involves the use of a unit composed of two reactors: a reactor for DME synthesis from CO and H₂ and a reactor for the synthesis of hydrocarbons from DME; these reactors are united by a common circulation contour. When the syngas of composition: N₂, 1; CO, 17; CO₂, 7; and H₂, 75 (vol %) enters the reactor of DME synthesis, then the end gas fed to the reactor of hydrocarbon synthesis consists almost exclusively of hydrogen (CO, 1–2 vol %; H₂, 90–95 vol % (Table 3).

In the presence of all catalysts the reaction occurs with a fairly high conversion in the medium of both syngas and hydrogen (Table 3) except ZrMFI, and selectivity for liquid hydrocarbons considerably





depends on the nature of the active element. For example, in the syngas medium, the sample MgMFI exhibits the highest selectivity for liquid hydrocarbons (86.9 wt %) and is characterized by the highest amount of aromatic hydrocarbons (33.5 wt %) and the lowest amount of isoparaffins (54.5 wt %). The ZnMFI sample also shows high selectivity for liquid hydrocarbons (85.0 wt %) with a high content of isoparaffins (68.2 wt %) and the lowest content of aromatic hydrocarbons (17.8 wt %). Note that for the zinc-containing sample the aqueous phase of the liquid product contains a much higher amount of methanol (12.5 wt %).

These results showed that the composition of the concomitant gas exerts a strong effect on the distribution of liquid hydrocarbons. For example, in the presence of ZnMFI a high selectivity for isoparaffins (78.3 wt %) and the lowest amount of aromatic hydrocarbons (10.9 wt %) are observed. This is explained by the fact that in the hydrogen medium hydrogenation reactions are accelerated; as a result, a smaller amount of aromatic hydrocarbons is formed [24].

Table 2. Acidic properties of the catalysts according to TPD-NH $_3$

Catalyst	Numb	11/1			
Catalyst	type I type II		total	11/1	
MFI	309	322	631	1.04	
CuMFI	419	192	615	0.46	
ZrMFI	348	252	600	0.72	
LaMFI	359	230	595	0.64	
MgMFI	344	241	585	0.70	
ZnMFI	310	255	565	0.82	



Fig. 3. Hydrocarbon group composition of the mixture of liquid hydrocarbons obtained in the presence of Rh-ZnMFI in the medium of (1) syngas and (2) hydrogen.

It is known that in the presence of group VIII metals (often more accessible Pd in an amount of up to 1 wt %) the isomerization and hydrogenation reactions are accelerated over zeolite catalysts. In this regard, Rh is a poorly studied modifier. Therefore, the synthesized catalysts were additionally modified with rhodium and tested in DME conversion to liquid hydrocarbons (Table 4).

As is clear from Table 4, all samples are characterized by a high conversion in both hydrogen and syngas media. However, selectivity for liquid hydrocarbons decreases upon the introduction of Rh into the catalyst composition. In the presence of rhodium catalysts the liquid product contains a somewhat higher amount of isoparaffins and a lower amount of aromatic hydrocarbons; this effect is more pronounced in the hydrogen medium. The hydrocarbon group composition of the mixture of liquid hydrocarbons synthesized in the presence of sample Rh-ZnMFI is shown in Fig. 3.

When Rh-MgMFI and Rh-CuMFI catalysts are used, the highest amount of methanol is detected in the aqueous phase of the liquid product (Table 4). In the case of ZSM-5 methanol is formed via the interaction of methoxy groups with H_2O . Most probably, in the presence of these catalysts the highest amount of methoxy groups is formed; as a consequence, the amount of the produced methanol is increased [25].

Table 5 presents the comparative data on the effect of two hydrogenating metals Pd (0.5 wt %) and Rh (0.05 wt %) on the catalytic behavior of nanozeolite zinc-containing (2 wt %) catalysts. The nature of the additional modifier considerably affects the behavior of the catalyst. At an almost similar DME conversion, the rhodium catalyst is characterized by a much higher selectivity than the palladium catalyst. However, the resulting mixture contains a higher amount of isoparaffins, while the amount of aromatic hydrocarbons is small and the concentration of methanol in the aqueous phase of the liquid product is low (6.8 wt %).

	DME conversion, $C_{\rm DME}, \%$	Selectivity for liquid hydrocarbons, $S_{C_{5+}}$, wt %	Gasoline composition, wt %					
Catalyst			isoparaffins	aromatic hydrocarbons	MeOH, wt %			
Syngas medium								
MFI	99.8	81.4	56.7	26.7	3.9			
ZrMFI	99.9	78.7	55.1	28.9	2.5			
ZnMFI	98.9	85.0	68.2	17.8	12.8			
LaMFI	99.8	80.0	56.2	28.3	4.0			
MgMFI	99.2	86.9	54.5	33.5	2.7			
CuMFI	99.8	79.8	58.7	24.5	3.5			
Hydrogen medium								
MFI	99.9	79.8	64.9	17.3	2.5			
ZrMFI	99.9	76.2	65.1	16.5	1.3			
ZnMFI	98.3	84.2	78.3	10.9	20.4			
LaMFI	99.7	77.8	68.9	13.5	5.9			
MgMFI	99.5	83.8	61.1	20.9	3.8			
CuMFI	99.5	75.3	63.2	17.8	5.7			

Table 3. Effect of modifiers on catalyst activity and selectivity in DME conversion to liquid hydrocarbons

Table 4. Influence of Rh on the activity and selectivity of catalysts of DME conversion to liquid hydrocarbons

		Selectivity	Gasoline composition, wt %				
Catalyst	DME conversion, $C_{\rm DME}, \%$	for liquid hydrocarbons, $S_{C_{5+}}$, wt %	isoparaffins	aromatic hydrocarbons	MeOH, wt %		
Syngas medium							
Rh-ZrMFI	99.9	78.4	57.4	25.1	2.8		
Rh-ZnMFI	98.8	85.6	69.7	16.7	12.8		
Rh-LaMFI	99.8	79.8	64.2	20.3	5.0		
Rh-MgMFI	99.2	84.9	60.5	27.5	12.7		
Rh-CuMFI	99.8	76.8	63.7	19.5	3.5		
Hydrogen medium							
Rh-ZrMFI	99.9	71.2	68.8	14.2	2.3		
Rh-ZnMFI	99.6	77.8	86.8	3.7	6.9		
Rh-LaMFI	99.4	60.1	70.2	12.5	7.8		
Rh-MgMFI	96.9	68.6	68.7	18.4	27.1		
Rh-CuMFI	98.8	47.7	70.3	11.4	14.8		

Thus, the effect of modifier nature on the activity and selectivity of nanosized zeolite catalysts of dimethyl ether conversion to liquid hydrocarbons is studied. The nature of the active element considerably influences both the physicochemical and catalytic properties of the catalyst. In the presence of all of the catalysts a fairly high DME conversion is reached in both syngas and hydrogen media. Introduction of the additional modifier (Rh) entails an increase in the amount of isoparaffins and a decrease in the content of aromatic hydrocarbons and methanol in the aqueous phase of the liquid product.

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	DME conversion, $C_{\rm DME}, \%$	Selectivity for liquid hydrocarbons, S _{C₅₊, wt %}	Gasoline composition, wt %		
Catalyst			isoparaffins	aromatic hydrocarbons	MeOH, wt %
Rh-ZnMFI	99.6	77.8	86.8	3.7	6.9
Pd-ZnMFI	97.4	83.9	83.8	6.8	28.9

Table 5. Effect of Pd and Rh on the nanosized zinc catalyst for synthesizing liquid hydrocarbons from DME. Tests were run in the hydrogen medium

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CONFLICT OF INTEREST

A.L. Maximov is the editor-in-chief of the journal *Petroleum Chemistry*; other authors declare that there is no conflict of interest.

ADDITIONAL INFORMATION

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