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RESEARCH ARTICLE

Hydroisomerization of *n*-hexadecane on Pt-containing silicone aluminum phosphate molecular sieves SAPO-11 with different silicon content

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Abstract

Objectives. To obtain silicone aluminum phosphate molecular sieves SAPO-11 with different silicon content, to determine their pore space and acidic properties, to apply 0.5 wt % to their surface Pt, and to evaluate the effectiveness of the use of *n*-hexadecane in the hydroisomerization reaction.

Methods. The chemical composition of the SAPO-11 molecular sieves obtained was determined by means of X-ray fluorescence spectroscopy using a Shimadzu EDX-7000P device. The radiographs of non-calcined SAPO-11 were recorded on a Shimadzu XRD-7000 diffractometer in CuKα radiation.

Results. Silicone aluminum phosphate molecular sieves SAPO-11 containing 0.5 wt % Pt on the surface were obtained from gels. As the SiO_2/Al_2O_3 ratio increases, the number of acid centers increases. This then leads to an increase in the conversion of *n*-hexadecane. The selectivity of the formation of hydrocarbons of the structure decreases at the same time.

Conclusions. Silicone aluminum phosphate molecular sieves Pt-SAPO-11 were obtained. It was also found that the samples undergo *n*-hexadecane conversion.

Keywords
molecular sieves, silicone aluminum phosphate SAPO-11, catalysts for hydroisomerization of *n*-hexadecane

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НАУЧНАЯ СТАТЬЯ

Гидроизомеризация *н*-гексадекана на Pt-содержащих силикоалюмофосфатных молекулярных ситах SAPO-11 с различным содержанием кремния

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Аннотация

Цель. Получить силикоалюмофосфатные молекулярные сита SAPO-11 с различным содержанием кремния, изучить свойства их пористой структуры и кислотные свойства; приготовить на их основе бифункциональные Pt-содержащие катализаторы (0.5 мас. % Pt) и оценить их каталитические свойства в реакции гидроизомеризации *н*-гексадекана.

Методы. Анализ химического состава синтезированных силикоалюмофосфатов SAPO-11 проводили на приборе Shimadzu EDX-7000P методом рентгенофлуоресцентной спектроскопии. Фазовый состав образцов SAPO-11 анализировали методом рентгеновской порошковой дифракции на дифрактометре Shimadzu XRD-7000 в Cu-Kα излучении.

Результаты. Синтезированы из реакционных гелей силикоалюмофосфатные молекулярные сита SAPO-11 с различным содержанием Si и приготовлены на их основе бифункциональные Pt-содержащие катализаторы гидроизомеризации высших *н*-парафинов. С ростом соотношения SiO_2/Al_2O_3 увеличивается число кислотных центров, что приводит к возрастанию конверсии *н*-гексадекана. Селективность образования углеводородов *изо*-строения при этом снижается.

Выводы. Установлено, что соотношение SiO_2/Al_2O_3 в исходных реакционных гелях оказывает влияние как на концентрацию кислотных центров, так и на свойства пористой структуры силикоалюмофосфатов SAPO-11. Показано, что соотношение SiO_2/Al_2O_3 оказывает существенное влияние на каталитические свойства SAPO-11 в гидроизомеризации *н*-гексадекана.

Ключевые слова

молекулярные сита, силикоалюмофосфат SAPO-11, катализаторы гидроизомеризации *н*-гексадекана

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INTRODUCTION

Zeolite catalysts are widely used in modern oil refining and petrochemistry [1–2]. Silicoaluminophosphate molecular sieves (SAPO-*n*) are used [3–10] in particular, for the hydroisomerization of *n*-paraffins C_{14}–C_{18}. Their efficiency is determined by acidity, pore size and the nature of adsorbed metal. The catalytic properties of SAPO-*n* zeolites are largely determined by the silicon content and the preparation technology, on which the pore size and acidity depend [3, 11–19].

We have previously described the preparation of SAPO-11 molecular sieves (structured type AEL) with a one-dimensional channel system and a pore size of 4.0–6.5 Å and SiO_2/Al_2O_3 ratio of 0.1 and 0.5 (SAPO-11-0.1 and SAPO-11-0.5), used in the oligomerization process of α-methylstyrene [20]. Continuing this research, we deposited 0.5 wt % metallic platinum on these zeolites. After extruding and grinding, particles of 20–40 μm size were used as catalysts for the hydroisomerization of *n*-hexadecane.

MATERIALS AND METHODS

SAPO-11 Synthesis

SAPO-11 silicoaluminophosphate molecular sieves were synthesized according to the method described in [21] from reaction gels with the following compositions: $1.0Al_2O_3 : 1.0P_2O_5 : (0;\ 0.2;\ 0.5)\ SiO_2 : 1.0(\text{Templat}) : 45H_2O$. Phosphoric acid ($H_3PO_4$, 85%, *Reakhim*, Russia), pseudobemite (AlO(OH), 72% Al_2O_3, *Ishimbay Specialized Chemical Catalyst Plant*, Russia), white soot (*SIGMA*, USA), di-*n*-propylamine (99%,

Table 1. Chemical and phase composition of reaction gels and their crystallization products (SAPO-11 and AlPO-11 molecular sieves)

Sample	Chemical composition of the initial reaction mass	Chemical composition of SAPO-11	Crystallinity, %
AlPO-11-0	$Al_{1.00}P_{0.98}Si_{0.00}$	$Al_{1.00}P_{0.98}Si_{0.00}$ (AlPO-11)	~93
SAPO-11-0.1	$Al_{1.00}P_{0.98}Si_{0.05}$	$Al_{1.00}P_{0.90}Si_{0.03}$ (SAPO-11)	~92
SAPO-11-0.5	$Al_{1.00}P_{0.98}Si_{0.25}$	$Al_{1.00}P_{0.91}Si_{0.18}$ (SAPO-11)	~95

Acros Organics, Belgium) and distilled water were used in the preparation of reaction gels. After the addition of all components, the reaction mixture was stirred at room temperature (25°C) for 1 h. The resulting thick gel was then incubated in an air thermostat for 24 h at 90°C. It had previously been found that preliminary aging of the gel at 90°C allows silicoaluminophosphate SAPO-11 of high phase purity and degree of crystallinity to be selectively obtained during its further crystallization [21]. After the aging step, the gels were loaded into Teflon-coated autoclaves (*TEFIC*, China) and crystallized at 200°C for 24 h. After crystallization, the reaction mass was centrifuged, washed with distilled water and dried at 100°C for 24 h. The SAPO-11 samples obtained from gels with SiO_2/Al_2O_3 ratios of 0.1 and 0.5 are designated as SAPO-11-0.1 and SAPO-11-0.5. Furthermore, a sample from a silicon-free gel was synthesized and designated as AlPO-11.

Preparation of catalytic systems

Bifunctional catalysts containing platinum (Pt/SAPO-11) were prepared in the following way: SAPO-11 samples with different silicon contents calcined at 600°C for 6 h in air were impregnated with an aqueous solution of $H_2PtCl_6{\cdot}6H_2O$ at the rate of 0.5 wt % Pt per weight of catalyst, then dried at room temperature at 25°C for 48 h and at 100°C for 24 h. The samples containing platinum compounds were then calcined in a muffle furnace at 600°C for 6 h in air. Next, the samples containing platinum compounds were subjected to calcination in a muffle furnace (*LIOP*, China) at 550°C for 5 h. After calcination, the samples were extruded, pulverized and sieved, in order to obtain a particle size fraction of 20–40 µm. The samples containing Pt SAPO-11-0.1 and SAPO-11-0.5 are hereinafter designated as Pt/SAPO-11-0.1 and Pt/SAPO-11-0.5, respectively. The silicon-free catalyst sample is designated as Pt/AlPO-11.

Methods of material analysis

The chemical composition of the synthesized SAPO-11 silicoaluminophosphates was analyzed using an EDX-7000P (*Shimadzu*, Japan) instrument by means of X-ray fluorescence spectroscopy.

The phase composition of SAPO-11 samples was analyzed by means of X-ray powder diffraction on an XRD-7000 diffractometer (*Shimadzu*, Japan) in Cu-Kα radiation. Scanning was carried out in the region of 2θ angles from 5° to 40° with a step of 1 deg/min. The processing of X-ray images and phase analysis were performed using the Shimadzu XRD program. The crystallinity was evaluated by the content of amorphous halo in the region from 20° to 30° of 2θ angles using the Shimadzu XRD program.

More detailed properties of the synthesized silicoaluminophosphate molecular sieves SAPO-11 are described in the dissertation of O.S. Travkina[1].

Hydroisomerization of *n*-hexadecane

The hydroisomerization reaction of *n*-hexadecane ($n\text{-}C_{16}H_{34}$, 99%, *ReaKhim*, Russia) was carried out in an integral flow reactor under a pressure of 3.0 MPa at 280–350°C with a molar ratio of $H_2/n\text{-}C_{16}H_{34}$ = 12 mol/mol and a mass feed rate of 2 h^{-1}. The reaction products were analyzed by means of gas–liquid chromatography on HRGC 5300 Mega Series Carlo Erba chromatograph (*Carlo Erba*, Italy) using a flame ionization detector (glass capillary column 50 m, SE-30). The products were identified by means of chromatography-mass spectrometry on a chromatography-mass spectrometer GCMS-TQ8050 (*Shimadzu*, Japan) using the WILEY mass-spectrum library. The physicochemical characteristics of the products obtained corresponded to the data available in literature [9].

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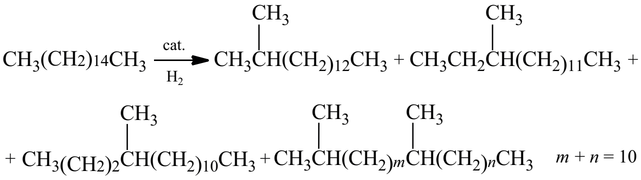
[1] Travkina O.S. *Granular zeolites A, X, Y, mordenite and ZSM-5 of a high degree of crystallinity with a hierarchical porous structure: synthesis, properties and application in adsorption and catalysis*. Diss. Dr. Sci. (Chem.). Ufa; 2023. 332 p. (in Russ.).

RESULTS AND DISCUSSION

The hydroisomerization of *n*-hexadecane *n*-$C_{16}H_{34}$ upon the Pt/SAPO-11-0.1, Pt/SAPO-11-0.5, and Pt/AlPO-11 samples in the temperature range 280–340°C was studied (Fig. 1). It was found that the reaction products mainly contain methyl pentadecanes. Among them the corresponding 2-, 3-, and 4-monomethyl derivatives are the main isomers.



$$CH_3(CH_2)_{14}CH_3 \xrightarrow[H_2]{cat.} CH_3\overset{CH_3}{\overset{|}{C}}H(CH_2)_{12}CH_3 + CH_3CH_2\overset{CH_3}{\overset{|}{C}}H(CH_2)_{11}CH_3 +$$
$$+ CH_3(CH_2)_2\overset{CH_3}{\overset{|}{C}}H(CH_2)_{10}CH_3 + CH_3\overset{CH_3}{\overset{|}{C}}H(CH_2)_m\overset{CH_3}{\overset{|}{C}}H(CH_2)_nCH_3 \quad m + n = 10$$

Fig. 1. Hydroisomerization of *n*-hexadecane *n*-$C_{16}H_{34}$

It follows from [22] that the predominant formation of such products (Table 2) is associated with the structure of the microporous structure of silicoaluminophosphates Pt/SAPO-11. In parallel, low hydrocarbons C_6–C_8 (cracking products) are formed in small amounts.

When the loading ratio of SiO_2/Al_2O_3 is increased to 0.5, the selectivity of their formation increases up to 30% (310°C). As temperature increases from 310 to 340°C, the selectivity of isomerization sharply decreases, and the main priority is cracking (Fig. 2).

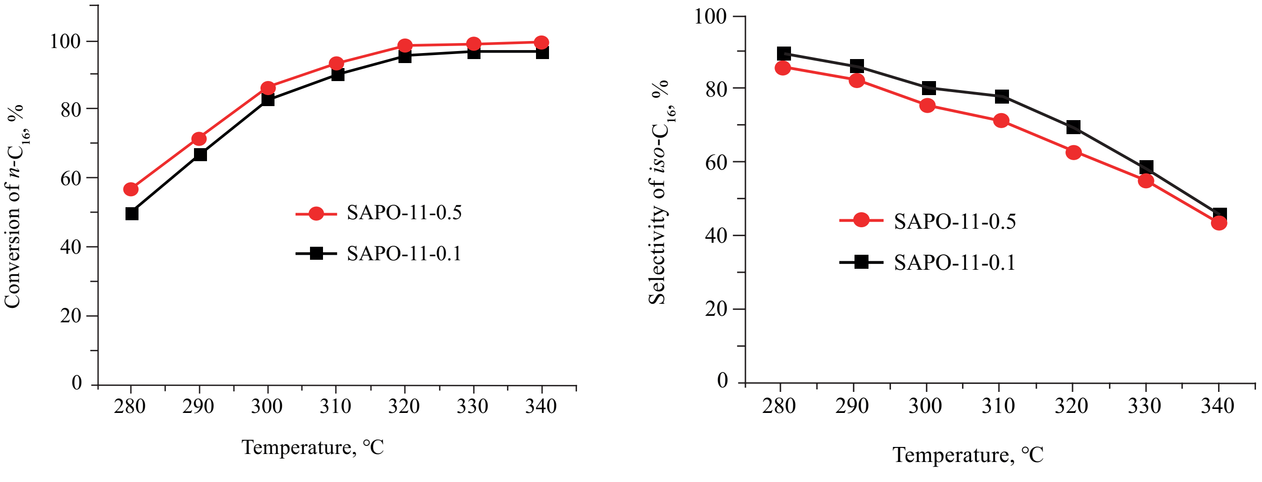
It follows from the data obtained (Table 2) that the catalyst Pt/SAPO-11-0.1 is more effective for isomerization processes, providing a higher total yield of isomers. We assume that this is due to the number of acid centers in the sample Pt/SAPO-11-0.5, which are 5 times greater, including on the outer surface of the crystals, relative to the sample Pt/SAPO-11-0.1.

Table 2. Results of hydroisomerization of *n*-hexadecane at 310°C on Pt-containing samples of silicoaluminophosphate molecular sieves

Name	Pt/SAPO-11-0.1	Pt/SAPO-11-0.5
Conversion of *n*-C_{16}, %	90	94
Selectivity of Σi-C_{16}, %	78	71
Composition of reaction products, %		
2-MeC_{15}, %	21	20
3-MeC_{15}, %	18	16
4-MeC_{15}, %	13	11
5-MeC_{15}, %	5	3
6-MeC_{15}, %	9	6
$(CH_3)_2$–C_{14}, %	12	15
ΣC_6–C_8, %	22	29

Symbols: selectivity of Σi-C_{16} is the total selectivity for C_{16} isomers; $(CH_3)_2$–C_{14} is di- and trimethylisomers of C_{16}; ΣC_6–C_8 are cracking products.

It should be noted that in the silicon-free aluminophosphate Pt/AlPO-11, the hydroisomerization of *n*-$C_{16}H_{34}$ under these conditions is not observed. We attribute this to the fact that the acid centers in silicoaluminophosphates, responsible for the course of skeletal isomerization, are formed only when silicon oxide is introduced into the material. This was shown in a previous study [23].



(a)

(b)

Fig. 2. Hydroisomerization of *n*-hexadecane over Pt/SAPO-11 catalysts: (a) conversion of *n*-C_{16}; (b) selectivity of *iso*-C_{16} formation

CONCLUSIONS

This study examined the effect of different silicon content on the degree of crystallinity, morphology and size of SAPO-11 silicoaluminophosphates. Samples SAPO-11-0.1 and SAPO-11-0.5 with 0.5 wt % Pt deposited were investigated as catalysts for the hydroisomerization of *n*-hexadecane. It was shown that the samples of bifunctional catalysts at 300°C, 3 MPa, 2.0 h^{-1} and H_2/C_{16} = 12 mol/mol enable *iso*-hexadecane yields of at least 70% to be obtained, with *n*-hexadecane conversion of more than 90%.

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Authors' contributions

M.R. Agliullin—conducting research, collecting and processing material, and writing the text of the article.

A.N. Khazipova—review of publications on the topic of the article.

A.F. Akhmetov—planning consultations.

O.A. Baulin—conceptualization of the research paper, critical revision with the introduction of valuable intellectual content.

The authors declare no conflicts of interest.

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