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CATALYST SELECTION FOR THE CATALYTIC DEHYDROAROMATIZATION REACTION OF METHANE

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Abstract. This article investigates the technology for obtaining aromatic hydrocarbons—benzene, toluene, and xylenes—via the non-oxidative catalytic dehydroaromatization of methane. The reaction was carried out in a quartz tubular reactor, and molybdenum-containing nanocatalysts were synthesized. The catalysts were prepared using the sol–gel method, and their physicochemical properties were studied by X-ray diffraction, adsorption, and thermal desorption techniques. The results of the study showed that the conversion of methane into aromatic hydrocarbons proceeds efficiently under high-temperature conditions.

Keywords: methane, dehydroaromatization, benzene, toluene, xylenes, nanocatalyst, zeolite, molybdenum, gas chromatography.

Аннотация. В данной статье исследуется технология получения ароматических углеводородов — бензола, толуола и ксилолов — посредством неокислительной каталитической дегидроароматизации метана. Реакция проводилась в кварцевом трубчатом реакторе, при этом были синтезированы нанокатализаторы на основе молибдена. Катализаторы

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получали методом сол-гель, а их физико-химические свойства изучались с использованием методов рентгеновской дифракции, адсорбции и термической десорбции. Результаты исследования показали, что превращение метана в ароматические углеводороды эффективно протекает при высокотемпературных условиях.

Ключевые слова: метан, дегидроароматизация, бензол, толуол, ксилолы, нанокатализатор, цеолит, молибден, газовая хроматография.

Annotatsiya. Ushbu maqolada metanni oksidlovsiz katalitik degidroaromatizatsiya qilish orqali aromatik uglevodorodlar — benzol, toluol va ksilollarni olish texnologiyasi o‘rganilgan. Reaksiya kvartsli quvurli reaktorda olib borildi hamda molibden asosidagi nanokatalizatorlar sintez qilindi. Katalizatorlar sol-gel usuli yordamida tayyorlandi va ularning fizik-kimyoviy xossalari rentgen difraksiyasi, adsorbsiya hamda termik desorbsiya usullari orqali tahlil qilindi. Tadqiqot natijalari shuni ko‘rsatdiki, metanning aromatik uglevodorodlarga aylanish jarayoni yuqori harorat sharoitida samarali kechadi.

Kalit so‘zlar: metan, degidroaromatizatsiya, benzol, toluol, ksilollar, nanokatalizator, zeolit, molibden, gaz xromatografiyasi.

INTRODUCTION.

Methane, which is the main component of natural gas, is an important raw material in the chemical industry [1-3]. At present, methane is mainly used as a fuel, however, its level of chemical processing is very low. The conversion of methane into high-value products is one of the urgent directions of the gas chemical industry [4-5].

Aromatic hydrocarbons—benzene, toluene and xylenes—can be obtained through the catalytic dehydroaromatization (MDA) process of methane. These products are widely used in the production of plastics, synthetic fibers, pharmaceuticals and many other chemical products [6-8].

Benzene is the simplest aromatic hydrocarbon and is a colorless liquid with a characteristic odor. Its main physical properties are: boiling point – 80.1 °C, melting point – 5.5 °C, density – 0.879 g/cm³. Toluene and xylenes also belong to the class of aromatic hydrocarbons and are used as solvents as well as in many organic synthesis processes [9-10].

In the process of converting methane into aromatic hydrocarbons, Mo-containing zeolite catalysts are considered the most effective [11].

RESEARCH METHODOLOGY.

The non-oxidative dehydroaromatization reaction of methane in a catalytic dehydroaromatization unit was carried out in a quartz tubular reactor. The reactor

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had a diameter of 12 mm and a length of 250 mm, and it was filled with catalyst particles of 0.5–1.0 mm in size (Figure 1).

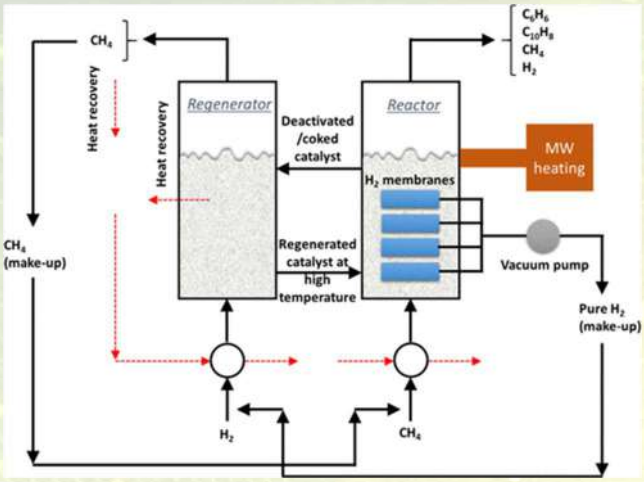


Figure 1. Schematic diagram of the quartz tubular reactor used for the non-oxidative dehydroaromatization of methane.

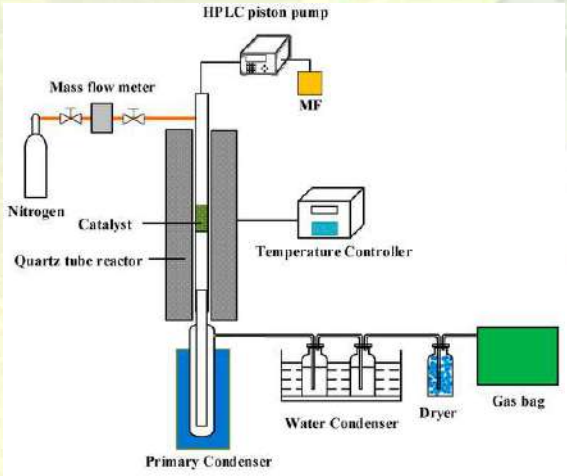


Figure 2. Catalytic dehydroaromatization unit for methane

The process was carried out under the following conditions: temperature: 750 °C, pressure: 0.1 MPa, gas mixture: CH_4: N_2 = 1: 1, space velocity: 500–1500 h^{-1}.

Methane was supplied from a cylinder to the reactor through a pressure regulator [12-15]. The reactor temperature was controlled using an electric furnace and measured by a thermocouple. The reaction products were collected through a condenser and subsequently analyzed by gas chromatography (Figure 2).

CATALYST SYNTHESIS.

Molybdenum-containing zeolite catalysts were prepared for the dehydroaromatization of methane. The catalysts were synthesized using the sol–gel method.

Catalyst composition: MoO_3, ZrO_2, ZnO, high-silica zeolite (HSZ).

Ammonium heptamolybdate was used as the molybdenum source [16-20].

The stages of catalyst preparation:

1. Modification of the zeolite with acidic and alkaline solutions
2. Impregnation with the molybdenum salt
3. Drying (80 °C)
4. Calcination (500 °C, 4 hours)

As a result, a Mo/HSZ nanocatalyst was obtained.

FORMATION OF AROMATIC PRODUCTS.

As a result of the catalytic dehydroaromatization of methane, the following main products are formed: benzene (C_6H_6), toluene (C_7H_8), and xylenes (C_8H_{10}).

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Gas chromatography was used to analyze the reaction products in order to determine the composition of the gaseous and liquid products formed during the catalytic dehydroaromatization of methane. The analyses were carried out using a "Kristallyuks-4000M" gas chromatograph. This instrument allows the separation and quantitative determination of components in gas mixtures.

The following main components were identified during chromatographic analysis: hydrogen (H_2), methane (CH_4), ethane (C_2H_6), ethylene (C_2H_4), propylene (C_3H_6), benzene (C_6H_6), toluene (C_7H_8), and xylenes (C_8H_{10}).

The analysis results were calculated using the external standard method. In this method, a calibration curve is constructed using standard gas mixtures of known concentrations, and the quantities of components in an unknown mixture are determined. Gas chromatographic analysis results are shown in Figure 3.

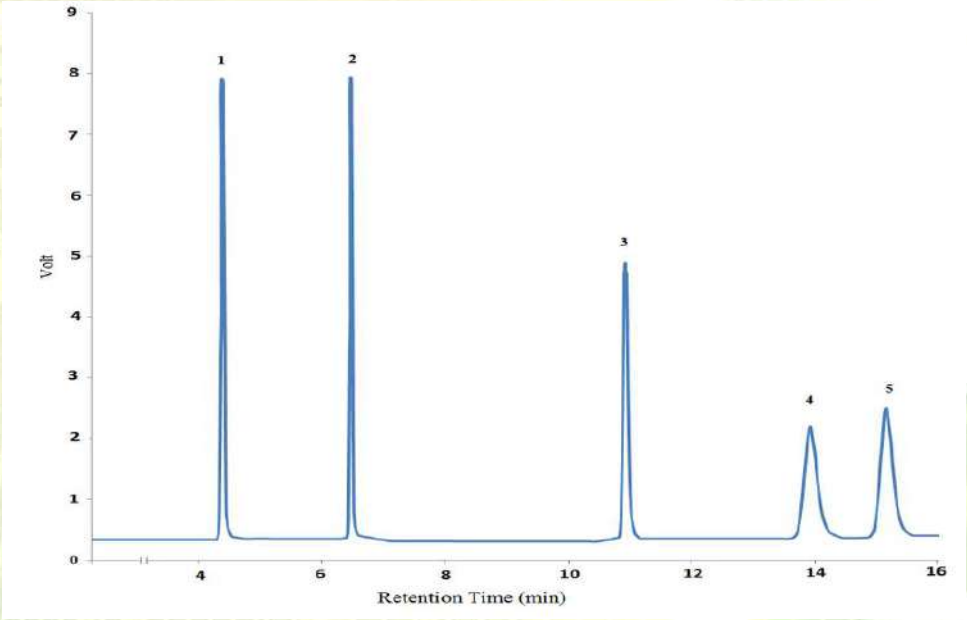


Figure 3. The chromatogram typically shows the following sequence: 1-H_2, 2-CH_4, 3-benzene, 4-toluene, 5-xylenes

As a result of gas chromatographic analysis, peaks corresponding to different components were observed on the chromatogram. The area of each peak is proportional to the amount of the component in the mixture.

Aromatic hydrocarbons usually appear in the later part of the chromatogram due to their higher boiling points. These conditions ensure good separation of the components in the gas mixture. The composition of the reaction products is presented in Table 1.

Table 1.

The following table shows the representative composition of products obtained from the catalytic reaction.

№	Component	Chemical Formula	Volume Fraction (%)

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1	Hydrogen	H_2	45.2
2	Methane	CH_4	28.6
3	Benzene	C_6H_6	7.5
4	Toluene	C_7H_8	4.2
5	Xylenes	C_8H_{10}	2.2

The results indicate that benzene is formed as the main aromatic product in the catalytic dehydroaromatization of methane.

PHYSICOCHEMICAL PROPERTIES OF THE CATALYSTS.

In the catalytic dehydroaromatization of methane, the activity, selectivity, and stability of the catalysts are directly related to their physicochemical properties. Therefore, the synthesized catalysts were studied using a set of comprehensive physicochemical characterization methods.

The following methods were applied in the study: BET method based on nitrogen adsorption, temperature-programmed desorption of ammonia (NH_3-TPD), determination of pore structure and distribution (BJH method), and surface area measurement (BET method).

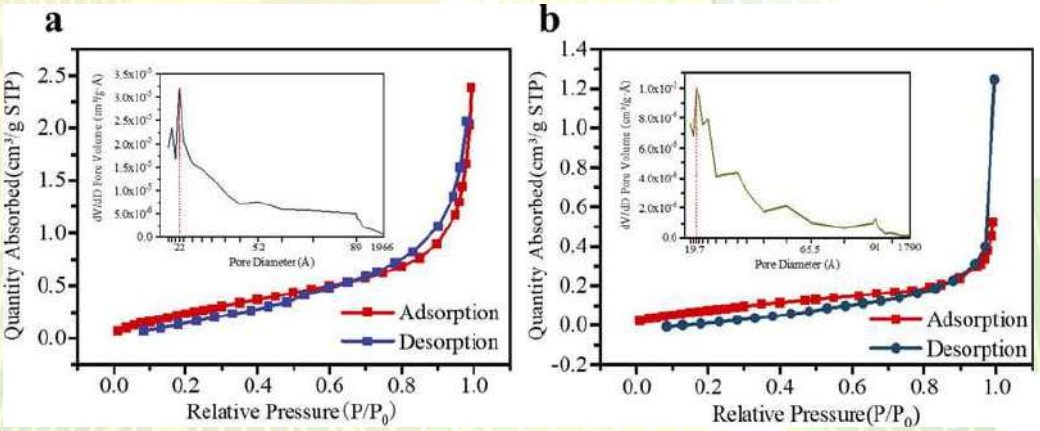


Figure 4. The specific surface area of the catalysts was determined using the Brunauer–Emmett–Teller (BET) method. The measurements were carried out at 77 K using nitrogen adsorption.

The experimental conditions were as follows: relative pressure: 0.05–0.99, adsorbate gas: N_2, instrument: ASAP-2400.

The BET method allowed the determination of the following parameters: specific surface area (m^2/g), pore volume, and pore diameter. The results showed that a larger catalyst surface area increases the number of active sites, which leads to higher methane conversion.

Ammonia Temperature-Programmed Desorption (NH_3-TPD). The NH_3-TPD method was used to identify the acidic sites of the catalysts. The experiment was conducted in the following stages: the catalyst sample was activated at 500 °C

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under an inert gas flow, saturated with NH_3, and then the NH_3 desorption was measured as the temperature was gradually increased.

The peaks observed in the NH_3-TPD spectrum indicate the strength of the catalyst acidic sites:

- 100–250 °C — weak acidic sites
- 250–450 °C — medium acidic sites
- above 450 °C — strong acidic sites

The results demonstrated that zeolite-based catalysts possess a high number of acidic sites.

Pore Structure (BJH Method)

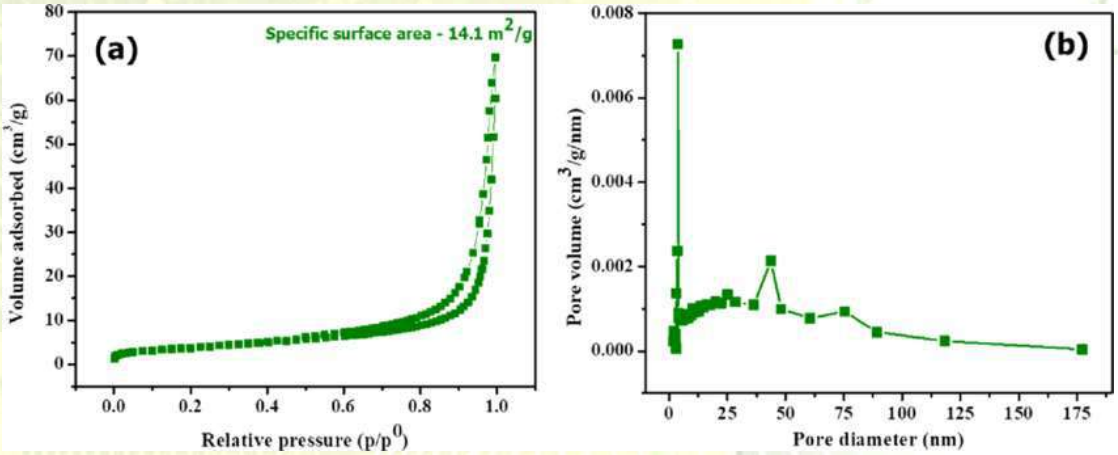


Figure 5. The pore structure and pore sizes of the catalysts were determined using the Barrett–Joyner–Halenda (BJH) method.

The BJH method, based on adsorption–desorption isotherms, allows the determination of the following parameters: pore diameter, pore volume, and pore size distribution. According to the study results, the catalysts possess a mesoporous structure (2–50 nm), which facilitates the diffusion of reactants and products into and out of the pores during the reaction.

CONCLUSION.

Aromatic hydrocarbons can be obtained through the catalytic dehydroaromatization of methane. Mo-containing zeolite catalysts exhibit high catalytic activity. Nanocatalysts prepared using the sol–gel method enhance the efficiency of the process. The reaction products were accurately analyzed using gas chromatography.

The conversion of methane into aromatic hydrocarbons represents a promising direction in the gas chemical industry. Gas chromatography is a highly precise and sensitive method for determining the products of the catalytic dehydroaromatization of methane. Analysis showed that the main products of the reaction are benzene, toluene, and xylenes, and the methane conversion and selectivity toward aromatic hydrocarbons were determined.

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Experimental results indicated that methane conversion increases with temperature, the molybdenum content affects the catalyst activity, and the zeolite structure enhances the selectivity toward aromatic products.

The best results were obtained using the $Mo/ZrO_2/ZnO/HSZ$ catalyst. Physicochemical analysis of the catalysts revealed the following: the BET method showed that the catalysts have a large specific surface area; NH_3-TPD confirmed the presence of acidic sites; and the BJH method identified a mesoporous structure in the catalysts. These properties ensure high catalytic activity in the methane catalytic dehydroaromatization reaction.

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