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Development of a pyrolysis catalyst of low molecular weight hydrocarbon raw materials

Tatyana A. Lavrentieva*✉*, Olga N. Karatun,
Alina A. Pustakhailova, Aylar S. Amansaryeva

Astrakhan State Technical University,
Astrakhan, Russia, lavrentievata@rambler.ru✉

Abstract. The article provides an analysis of the current state and prospects for the development of pyrolysis complexes in Russia and analyzes the main directions of development of the pyrolysis process of hydrocarbon raw materials. The characteristics of the feedstock and high-silica zeolites of the pentasil family of the CVN and HCVM types are given. The choice of a flow-through research method to identify the patterns of transformation of low-molecular-weight hydrocarbon fractions is justified, which is characterized by simplicity, accessibility and provides the ability to determine the catalytic activity at a steady state of the catalyst at any stage of the reaction. The method of conducting research on a laboratory flow-through unit of the thermal and catalytic pyrolysis process of the propane fraction and the method of preparing samples of catalysts for research are described. The effect of the concentration of pentasil family zeolites of the CVN and HCVM types on the catalytic properties of samples during heterogeneous pyrolysis of the propane fraction is considered. A comparative analysis of the results of thermal pyrolysis of the propane fraction and in the presence of prepared pentasyl-containing catalysts with the same technological parameters was carried out. The effect of the zeolite concentration in a heterogeneous catalyst on the activity and selectivity of the obtained catalytic systems during pyrolysis of the propane fraction is shown. A comparative analysis of coke formation in the presence of prepared catalyst samples as a result of high-temperature transformations of the propane fraction is carried out. It has been established that catalytic pyrolysis has an advantage over thermal pyrolysis, and the high catalytic activity and selectivity of the studied high-silica pentasyl-containing catalysts in the process of pyrolysis of low-molecular-weight hydrocarbon fractions makes it possible to develop active and effective catalysts based on them for the production of low-molecular-weight olefins. The optimal technological parameters of the pyrolysis process of low-molecular-weight hydrocarbon fractions are analyzed. The possibilities of implementing catalytic pyrolysis of the propane fraction within the framework of investment projects aimed at the development of the Astrakhan gas condensate field are considered.

Keywords: heterogeneous pyrolysis, propane, ethylene, propylene, high-silica zeolites, pentasyl-containing catalysts

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Научная статья

Разработка катализатора процесса пиролиза низкомолекулярного углеводородного сырья

Татьяна Алексеевна Лаврентьева*✉*, Ольга Николаевна Каратун,
Алина Александровна Пустахайлова, Айлар Сердаровна Амансарыева

Астраханский государственный технический университет,
Астрахань, Россия, lavrentievata@rambler.ru✉

Аннотация. В статье приведен анализ современного состояния и перспективы развития пиролизных комплексов в России и проанализированы основные направления развития процесса пиролиза углеводородного сырья. Приведены характеристики исходного сырья и высококремнеземных цеолитов семейства пентасила типа ЦВН и НЦВМ. Обоснован выбор проточного метода исследований для выявления закономерностей превращения низкомолекулярных углеводородных фракций, отличающийся простотой, доступностью и обеспечивающий возможность определения каталитической активности при стационарном состоянии катализатора на любой стадии реакции. Описана методика проведения исследований на лабораторной проточной установке термиче-

ского и каталитического процесса пиролиза пропановой фракции и методика приготовления образцов катализаторов для исследований. Рассмотрено влияние концентрации цеолитов семейства пентасила типа ЦВН и НЦВМ на каталитические свойства образцов в процессе гетерогенного пиролиза пропановой фракции. Проведен сравнительный анализ результатов термического пиролиза пропановой фракции и в присутствии приготовленных пентасилсодержащих катализаторов при тех же технологических параметрах. Показано влияние концентрации цеолита в составе гетерогенного катализатора на активность и селективность полученных каталитических систем в процессе пиролиза пропановой фракции. Проведен сравнительный анализ коксообразования в присутствии приготовленных образцов катализаторов в результате высокотемпературных превращений пропановой фракции. Установлено, что каталитический пиролиз имеет преимущество по сравнению с термическим, и высокая каталитическая активность и селективность исследуемых высококремнеземных пентасилсодержащих катализаторов в процессе пиролиза низкомолекулярных углеводородных фракций позволяет разработать на их основе активные и эффективные катализаторы для получения низкомолекулярных олефинов. Проанализированы оптимальные технологические параметры процесса пиролиза низкомолекулярных углеводородных фракций. Рассмотрены возможности реализации каталитического пиролиза пропановой фракции в рамках реализации инвестиционных проектов, направленных на развитие Астраханского газоконденсатного месторождения.

Ключевые слова: гетерогенный пиролиз, пропан, этилен, пропилен, высококремнеземные цеолиты, пентасилсодержание катализаторы

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Introduction

The petrochemical industry plays a key role in the modern economy, providing the production of materials without which the development of related industries is impossible. Petrochemical products – polymers, rubbers, synthetic fibers, and a wide range of organic compounds – have become the basis for progress in mechanical engineering, construction, transportation, and the household sector. For Russia, the development of the petrochemical complex is of strategic importance, as it allows to increase the depth of processing of hydrocarbon raw materials, reduce dependence on imports and increase the internal added value of the resources produced.

Pyrolysis of hydrocarbon raw materials is one of the most important processes underlying petrochemical production. This method provides the production of low molecular weight unsaturated and aromatic hydrocarbons, which serve as raw materials for the synthesis of plastics, rubbers and other valuable products. The pyrolysis process is flexible, as it allows the use of various raw materials, from light hydrocarbon gases and straight-run gasoline fractions to heavier residual raw materials. However, in industry, thermal pyrolysis requires high temperatures (770-900 °C) and is accompanied by the formation of coke.

The current trend in the development of pyrolysis process technology is associated with the introduction of catalysts capable of controlling the direction of decomposition reactions, increasing the yield of target products and reducing the formation of by-products [1, 2]. Catalytic pyrolysis is necessary to reduce energy consumption and improve production performance. The use of catalysts makes it possible to intensify the cracking of hydrocarbons, change the mechanism of bond breaking and increase the selectivity for light C_2–C_4 olefins.

Russian patents and publications describe specific examples of studies of catalytic pyrolysis of hydrocarbon raw materials. Catalysts are most often used in the processing of light fractions – ethane, propane, propane-butane, a wide fraction of light hydrocarbons, straight-run gasoline, where targeted production of target products is required. On an industrial scale, pyrolysis is still mainly carried out without a catalyst, however, catalytic systems are actively considered as a way to modernize and increase the selectivity of the process [3–15].

With the development of technology, approaches to the creation of catalysts are becoming more complex.

The use of catalysts in pyrolysis processes opens up opportunities for optimizing parameters, increasing the yield of target products and reducing energy costs. The Russian Scientific School is actively developing this area, adapting acid, oxide, metal composite and other systems to various types of raw materials. Catalytic pyrolysis is one of the most promising ways of technological modernization of the petrochemical industry and forms the basis for the transition to a new generation of energy-efficient and environmentally friendly industries.

An important step in the development of pyrolysis technology will be the transition from thermal to catalytic pyrolysis. Catalytic pyrolysis will improve the technical, economic and operational costs for the production of low molecular weight olefin hydrocarbons. This, in turn, will affect not only the economic performance of these installations, but will also lead to a reduction in the cost of high-margin products from petrochemical plants, the raw materials of which are low molecular weight olefins.

The experimental part

During the study, the propane fraction was used as the starting material, the characteristics of which are given in Table 1.

Table 1

Characteristics of the propane fraction

Indicator	CH_4	CO_2	C_3H_8	*i*-C_4H_{10}	н-C_4H_{10}
Component content, mass. %	0.01	0.01	99.97	0	0.01

Currently, the catalytic properties of zeolites of the pentasyl family are of great interest. They meet all the basic requirements for industrial catalysts: they have high mechanical strength, are resistant to coking, the action of sulfur, water and other oxygen-containing compounds [5, 8–10, 16].

During the experimental studies, CVM and CVN type zeolites belonging to the zeolites of the pentasyl family were used as initial zeolites. They have some differences in crystallographic structure, adsorption physico-chemical and catalytic properties. CVN type zeolites are obtained by direct synthesis and contain insignificant amounts (less than 0.1 mass. %) of sodium oxide. High-silica zeolites of the pentasil family of the CVN and CVM types were synthesized at the Nizhny Novgorod Sorbents CJSC in Nizhny Novgorod. The characteristics of the initial zeolites are given in Table 2.

Table 2

Characteristics of zeolites of the pentasyl family

№	Naming of the indicator	Powdered zeolite CVM		Powdered zeolite CVN	
		the norm for TS 38.102168-85	actual value	the norm for TS 38.102168-85	actual value
1	Appearance	Powder	Accordance	Powder	Accordance
2	Silica module	Nevertheless 30	31.2	Nevertheless 50	69.0
3	Content Al_2O_3, %	–	0.1	–	0.1
4	Static water vapor capacity at $P / P_c = 0.1$ cm^3/g	No more than 0,08	0.08	No more than 0.06	0.05
5	Static capacity of heptane vapor at $P / P_c = 0.4$ cm^3/g	No more than 0,14	0.17	No more than 0.16	0.16
6	X-ray phase analysis		Type CVM		Type CVN

Decationated zeolites were molded with a binder γ-Al_2O_3. Moreover, Al_2O_3 was previously peptized with concentrated nitric acid, then mixed with zeolite. The resulting pellet was granulated, the granules were dried at room temperature, and then dried at 120 °C for 2-3 hours.

The characteristics of the prepared pentasyl-containing catalysts are given in Table 3.

Table 3

Characteristics of pentasyl-containing catalysts

Designation	The amount of zeolitein the catalyst composition, mass. %
CVN-1	20
CVN-2	30
CVN-3	40
HCVM-1	20
HCVM-2	30
HCVM-3	40

The flow method was chosen to identify the patterns of transformation of low-molecular-weight hydrocarbon fractions on zeolite catalysts. This method is characterized by simplicity and accessibility, provides the ability to determine the catalytic activity at a steady state of the catalyst at any stage of the reaction and is used quite often in laboratory practice.

The studies were carried out in a laboratory flow-through unit at atmospheric pressure [16].

The feed rate of raw materials and water was calculated before the start of experimental studies, guided by the specified experimental conditions: temperature, contact time, water vapor: raw material ratio. The duration of the experiment was set taking into account the need to obtain pyrolysis products in sufficient quantities for their study.

The pyrolysis process was carried out in a quartz reactor, which made it possible to exclude the influence of the wall material on the experimental results. The reactor consisted of a quartz tube with an inner

diameter of $d_1 = 2.0 \times 10^{-2}$ m and a pocket for a thermocouple coaxially located in it with an outer diameter of $d_2 = 8.0 \times 10^{-3}$ m.

To carry out the catalytic pyrolysis process, the reactor was loaded $(0,5 \times 10^{-5})$–$(1,0 \times 10^{-5})$ m^3 of catalyst. During thermal pyrolysis, quartz glass of a similar volume was loaded into the reactor.

The coked catalyst was regenerated for 2 hours at a temperature of 500 °C in an air flow supplied through a gearbox and a flow regulator at a rate of 1 l/h.

Based on the literature data, temperature ranges of 600-800 °C were selected to identify the patterns of transformations of low-molecular-weight hydrocarbon fractions during thermal and catalytic pyrolysis. The contact time was 0.25 seconds, and the mass ratio of water vapor: raw materials was 0.4 : 1. The duration of the experiments ranged from 15 minutes to 30 hours.

Due to the formation of a wide range of hydrocarbons as a result of the conversion of the propane fraction, the composition of the resulting products was determined according to a multi-stage scheme using gas-liquid chromatography.

Results and discussions

For comparability of the data obtained, a series of experiments under thermal pyrolysis conditions was conducted at the initial stage of the study. The results of the data obtained confirm that an increase in temperature has a beneficial effect on the yield of the target pyrolysis products of the propane fraction. Thus, at a temperature of 600 °C, the conversion of raw materials practically did not occur, and the significant formation of ethylene begins at 700 °C. It should be noted that the yields of the target products, sufficient for industry, are achieved only at a temperature of 800 °C. Thus, at a temperature of 800 °C, the yield of ethylene during thermal pyrolysis of the propane fraction was 21.53 mass. %, and the yield of propylene was 9.58 mass. %. The yield of the sum of unsaturated hydrocarbons C_2–C_4 at a temperature of 800 °C was 31.12 mass. % with a propane conversion rate of 74.02 mass. %.

Thus, on an industrial scale, the pyrolysis process can only be carried out at temperatures above 800 °C. Carrying out the process in such conditions is associated with an increase in equipment costs due to the use of heat-resistant materials, as well as directly creating such a high temperature.

At the next stage of the research, the pyrolysis process of the propane fraction was carried out in the presence of prepared samples of pentasyl-containing catalysts under the same conditions.

The results of the experiments are presented in Tables 4 and 5.

Table 4

Results of catalytic pyrolysis of propane fraction on catalysts CVN-1, CVN-2 and CVN-3

Indicator	Process temperature, °C				
	600	650	700	750	800
CVN-1					
Output of C_2H_4, mass. %	3.04	18.20	21.27	26.79	23.35
The output of the sum of unsaturated hydrocarbons C_2–C_4, mass. %	3.04	18.20	21.27	30.84	31.38
Propane conversion, mass. %	3.71	39.05	49.10	66.58	49.97
CVN-2					
Output of C_2H_4, mass. %	2.74	5.85	13.79	21.19	30.44
The output of the sum of unsaturated hydrocarbons C_2–C_4, mass. %	2.74	5.85	14.42	24.41	40.33
Propane conversion, mass. %	3.47	7.44	24.34	41.70	68.09
CVN-3					
Output of C_2H_4, mass. %	5.09	9.12	12.06	35.62	37.73
The output of the sum of unsaturated hydrocarbons C_2–C_4, mass. %	5.09	14.79	20.33	45.65	47.94
Propane conversion, mass. %	6,31	34.45	31.51	76.39	83.53

Table 5

Results of catalytic pyrolysis of propane fraction on catalysts HCVM-1, HCVM-2 and HCVM-3

Indicator	Process temperature, °C				
	600	650	700	750	800
HCVM-1					
Output of C_2H_4, mass. %	2.83	10.33	16.10	20.55	23.95
The output of the sum of unsaturated hydrocarbons C_2–C_4, mass. %	2.83	10.33	20.10	27.72	30.68
Propane conversion, mass. %	5.51	20.65	33.55	42.18	49.19
HCVM-2					
Output of C_2H_4, mass. %	3.38	12.44	18.88	23.09	29.14
The output of the sum of unsaturated hydrocarbons C_2–C_4, mass. %	3.38	12.44	21.92	29.34	38.33
Propane conversion, mass. %	7.14	22.59	36.13	46.70	65.20

Ending of Table 5

Indicator	Process temperature, °C				
	600	650	700	750	800
HCVM-3					
Output of C_2H_4, mass. %	6.19	12.62	16.61	36.93	38.62
The output of the sum of unsaturated hydrocarbons C_2–C_4, mass. %	6.19	15.27	25.33	46.19	48.16
Propane conversion, mass. %	9.53	30.45	31.51	78.03	85.15

An analysis of the data obtained indicates that the pyrolysis process in the presence of pentasyl-containing catalysts leads to an increase in the yield of the target products and a decrease in the process temperature compared with the results of thermal pyrolysis.

High yields of ethylene were obtained already at 650 °C. In the presence of the CVN-1 catalyst at 650 °C, the yield of ethylene was 18.20 mass. %, and with thermal pyrolysis of the propane fraction, such a yield can be obtained at temperatures above 750 °C. In the presence of the HCVM-1 catalyst at 650 °C, the yield of ethylene was 10.33 mass. %. It should be noted that the yield of ethylene in the presence of pentasyl-containing catalysts over the entire temperature range studied was higher than the yield of ethylene during thermal pyrolysis with similar technological parameters.

An increase in the zeolite concentration in the catalyst led to an increase in the yields of unsaturated hydrocarbons at high temperatures (800 °C).

Thus, as a result of a study on the CVN-2 catalyst, an ethylene yield of 30.44 mass. % was obtained at 800 °C, and on the HCVM-2 catalyst in the amount of 29.14 mass. %, which is significantly more than in thermal pyrolysis with the same temperature (21.53 mass. %).

In the presence of the CVN-3 catalyst at 800 °C, the highest yield of unsaturated hydrocarbons was obtained, which amounted to 47.94 mass. % at the maximum yield of ethylene – 37.73 mass. %, and in the presence of the HCVM-3 catalyst at the same temperature, the maximum yield of unsaturated hydrocarbons was 48.16 mass. % with a maximum ethylene yield of 38.62 mass. %. The yield of ethylene in the presence of the CVN-3 catalyst was more than 75% higher than during thermal pyrolysis of the propane fraction. The yield of unsaturated C_2–C_4 hydrocarbons in the presence of the CVN-3 catalyst was more than 54% higher than during thermal pyrolysis of the propane fraction. A similar trend is observed as a result of the conversion of the propane fraction in the presence of the catalyst HCVM-3. The yield of ethylene and the sum of unsaturated C_2–C_4 hydrocarbons was higher by more than 57 and 79%, respectively, than during thermal pyrolysis of the propane fraction.

The data obtained indicate that in the presence of pentasyl-containing catalysts, the reaction rate of ethylene formation increases faster than the rate of formation of other unsaturated low-molecular-weight hydrocarbons. The conversion rate of the raw material (propane) in the presence of the CVN-3 catalyst at 750 and 800 °C was 76.39 and 83.53 mass. %, respectively, and in the presence of the HCVM-3 catalyst at the same process temperatures – 78.03 and 85.15 mass. %, respectively.

It should be noted that when using catalysts, the yield of coke and tarry substances increases compared to the results of the thermal pyrolysis process. Coke is deposited in the pores of the catalyst, blocks the active sites and reduces the activity of the catalyst. An increase in the amount of zeolite in the composition of the catalysts led to an increase in the yield of hydrogen, methane, tarry substances and coke. At the same time, the yield of resinous substances and coke was higher by 0.1-0.45 mass. % in the presence of pentasyl-containing catalysts of the HCVM type, than in the presence of pentasyl-containing catalysts of the CVN type.

The results obtained indicate that catalytic pyrolysis has an advantage over thermal pyrolysis. The yields of ethylene and the amounts of unsaturated C_2-C_4 hydrocarbons, which are the target products of the pyrolysis process, increase significantly, and the high catalytic activity and selectivity of the studied high-silica catalysts in the pyrolysis of low-molecular-weight hydrocarbon fractions makes it possible to develop active and effective catalysts based on them for the production of low olefins, which are valuable raw materials for petrochemical and organic synthesis.

The plans for the development of the Astrakhan gas condensate field [17] provide for the construction of a complex for the production of plastics. It is planned to use hydrocarbon fractions produced at the Astrakhan Gas Processing Plant, a branch of Gazprom Pererabotka LLC, as raw materials for their production. The conducted studies show the possibility of involving propane and propane fractions in the production of low-molecular-weight unsaturated hydrocarbons, as well as the prospects of using the catalytic pyrolysis process to obtain them [5]. Reducing the cost of heating hydrocarbon raw materials will improve the economics of the process and increase production efficiency.

Conclusion

A comparative analysis of the results of thermal and catalytic pyrolysis of the propane fraction in equal technological parameters showed that the yields of ethylene and unsaturated hydrocarbons C_2–C_4 were higher in the presence of pentasyl-containing catalysts

such as CVN and HCVM compared with the thermal process at optimal technological parameters, and the yield of the target products increased with an increase in the amount of zeolite in the catalyst. The largest number of target products as a result of the pyrolysis process of the propane fraction was obtained using the CVN-3 and HCVM-3 catalysts.

The results obtained allow us to assert the possibility of using pentasyl-containing catalysts in the process of propane pyrolysis, which will lead to a decrease in the process temperature and, as a result, to an improvement in the operational and economic performance of pyrolysis process plants. However, these results also make it possible to determine the direction of further research in this area, namely, to improve the efficiency of catalytic systems, it is necessary to work out the issue of finding modifiers that would increase the yield of target products and increase the stability of operation in order to increase the interregeneration period of using catalytic systems in the pyrolysis of low-molecular-weight hydrocarbons.

The search for effective modifiers for obtaining active and stable catalytic systems in the pyrolysis of low molecular weight fractions will continue in the future at the Department of Chemical Technology of Oil and Gas Refining of Astrakhan State Technical University.

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Information about the authors / Информация об авторах

Tatyana A. Lavrentieva – Candidate of Technical Sciences, Assistant Professor; Assistant Professor of the Department of Chemical Technology of Oil and Gas Refining; Astrakhan State Technical University; lavrentievata@rambler.ru

Татьяна Алексеевна Лаврентьева – кандидат технических наук, доцент; доцент кафедры химической технологии переработки нефти и газа; Астраханский государственный технический университет; lavrentievata@rambler.ru

Olga N. Karatun – Doctor of Technical Sciences, Professor; Professor of the Department of Chemical Technology of Oil and Gas Refining; Astrakhan State Technical University; karatunon@mail.ru

Alina A. Pustakhailova – Master's Course Student, training area "Chemical Technology"; Astrakhan State Technical University; lina.pustakhaylova@yandex.ru

Aylar S. Amansaryeva – Master's Course Student, training area "Chemical Technology"; Astrakhan State Technical University; aylar210203@mail.ru

Ольга Николаевна Каратун – доктор технических наук, профессор; профессор кафедры химической технологии переработки нефти и газа; Астраханский государственный технический университет; karatunon@mail.ru

Алина Александровна Пустахайлова – магистрант, направление «Химическая технология»; Астраханский государственный технический университет; lina.pustakhaylova@yandex.ru

Айлар Сердаровна Амансарыева – магистрант, направление «Химическая технология»; Астраханский государственный технический университет; aylar210203@mail.ru